Site Investigation at the G-8 Burning Ground Former Plum Brook Ordnance Works Sandusky, Ohio

Revision 1

Includes:

Quality Assurance Project Plan
Sampling and Analysis Plan
Safety and Health Plan

IT Project Number 764678

May 1996

Site Safety and Health Plan
Site Investigation at the G-8 Burning Ground
at the Former Plum Brook Ordnance Works
Sandusky, Ohio

Revision 1

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IT Project Number 764678

May 1996

Site Investigation at the G-8 Burning Ground
Former Plum Brook Ordnance Works
Sandusky, Ohio

I have read and approve this site safety and health plan with respect to project hazards, regulatory requirements, and IT procedures.

Don C Burton

Don Burton, PE
Project Manager

5/10/96
Date

Don L Smith, CIH for M. Smith

Melissa G. Smith, CIH
Health and Safety Manager

5/10/96
Date

Site Safety and Health Plan
Acknowledgement Form

I have been informed of, and will abide by the procedures set forth, in this safety and health plan and its amendments for the activities at the former Plum Brook Ordnance Works, Sandusky, Ohio.

Printed Name	***Signature***	***Representing***	***Date***

Table of Contents

Page

List of Appendices vi
List of Tables vii
List of Figures vii
List of Acronyms viii
1.0 Introduction 1-1
1.1 Objectives 1-1
1.2 Facility and Location Description 1-1
1.3 Policy Statement 1-2
1.4 References 1-2
2.0 Responsibilities 2-1
2.1 All Personnel 2-1
2.2 Project Manager: Don Burton 2-1
2.3 Health and Safety Manager: Melissa G. Smith 2-1
2.4 Site Safety and Health Officer: George H. Yu 2-2
2.5 Heavy Equipment Operators 2-3
2.6 Power Auger Operators 2-3
2.7 Subcontractors 2-3
2.8 On-Site Personnel and Visitors 2-3
2.9 Occupational Physician 2-4
3.0 Job Hazard Analysis 3-1
3.1 Scope of Work 3-1
3.2 Job Hazard Analysis by Task 3-1
3.3 Physical Hazards 3-1
3.3.1 Heavy Equipment Hazards 3-1
3.3.2 Sampling Hazards 3-2
3.4 Chemical Hazards 3-2
3.5 Noise 3-2
3.6 Anticipated Biological Hazards 3-3
3.6.1 Snakes 3-3
3.6.2 Insects 3-3
3.6.3 Poisonous Plants 3-3

Table of Contents (Continued)

Page

3.7 Heat Stress 3-4
4.0 Safety Program and Procedures 4-1
4.1 General Practices 4-1
4.2 Operating Procedures 4-2
4.2.1 Power Auger Operators 4-2
4.2.2 Drum Handling Safety 4-2
4.2.3 Heavy Equipment Operating Safety 4-3
4.2.4 Lifting Safety 4-4
4.2.5 Confined-Space Entry 4-5
4.3 Heat Stress Prevention 4-5
4.4 Hearing Conservation 4-6
4.5 Sanitation 4-7
4.5.1 Potable Water 4-7
4.5.2 Nonpotable Water 4-7
4.5.3 Toilet Facilities 4-7
4.5.4 Trash Collection 4-7
5.0 Personal Protective Equipment 5-1
5.1 Respiratory Program 5-1
5.2 Levels of Protection 5-3
5.2.1 Level A Protection 5-3
5.2.2 Level B Protection 5-3
5.2.3 Level C Protection 5-4
5.2.4 Level D-Modified Protection 5-4
5.2.5 Level D Protection 5-4
5.2.6 Selection of PPE 5-5
5.3 Using PPE 5-5
5.3.1 Donning Procedures for Level C PPE 5-5
5.3.2 Doffing Procedures for Level C PPE 5-5
6.0 Site Control 6-1
6.1 Authorization to Enter 6-1
6.2 Site-Specific Orientation 6-1
6.3 Documentation of Certificates 6-1

Table of Contents *(Continued)*

		Page
	6.4 Entry Log	6-1
7.0	Decontamination	7-1
	7.1 Personnel Decontamination	7-1
	7.2 Equipment Decontamination	7-2
	7.3 PPE Decontamination	7-2
8.0	Site Monitoring	8-1
	8.1 Air Monitoring	8-1
	8.1.1 Air Monitoring Frequency	8-1
	8.1.2 Air Monitoring Equipment	8-1
	8.1.3 Monitoring Equipment Maintenance and Calibration	8-1
	8.2 Other Hazardous Conditions	8-2
	8.3 Noise Monitoring	8-2
	8.4 Monitoring Records	8-2
	8.5 Notification	8-3
9.0	Employee Training	9-1
	9.1 General	9-1
	9.1.1 Site-Specific Orientation	9-1
	9.1.2 Tailgate Safety Meetings	9-1
	9.2 Site Workers' Basic Course	9-1
	9.3 Supervisors' Course	9-2
	9.4 First Aid and Cardiopulmonary Resuscitation	9-2
	9.5 Instructors	9-3
10.0	Medical Surveillance Program	10-1
	10.1 Physical Examinations	10-1
	10.1.1 Preplacement Examination	10-2
	10.1.2 Annual Examination	10-2
	10.1.3 Exit Examination	10-2
	10.2 First-Aid and Medical Treatment	10-2
	10.3 Medical Restriction	10-3
	10.4 Medical Records	10-3
11.0	Emergency Procedures	11-1
	11.1 General	11-1

Table of Contents *(Continued)*

Page

11.2 First Aid 11-1
11.3 Emergency Response 11-1
11.3.1 Pre-Emergency Planning 11-1
11.3.2 Project Manager's Responsibilities 11-1
11.3.3 Evacuation Signal 11-2
11.3.4 Emergency Equipment/Facilities 11-2
11.3.5 Medical Emergencies 11-2
11.3.6 Emergency Contact/Notification Listing 11-2
11.3.7 Key Project and IT Personnel 11-3
11.3.8 Directions to the Nearest Hospital 11-3
11.4 General Emergency Procedures 11-3
11.4.1 Personal Injury 11-4
11.4.2 Fire or Explosion 11-4
11.5 Evacuation Routes and Procedures 11-4
12.0 Bloodborne Pathogen Exposure Control Plan 12-1
12.1 Definition 12-1
12.1.1 Hepatitis B Virus 12-1
12.1.2 Human Immunodeficiency Virus 12-1
12.2 Exposure Determination 12-2
12.3 Measures for Prevention 12-3
12.3.1 Universal Precautions 12-3
12.3.2 Engineering Controls 12-3
12.3.3 Work Practice Controls 12-3
12.3.4 Personal Protective Equipment 12-4
12.3.5 Waste Handling 12-4
12.3.6 Waste Disposal 12-5
12.4 Medical Requirements 12-5
12.4.1 Hepatitis B Vaccination 12-5
12.4.2 Postexposure Procedures and Evaluation 12-5
12.4.3 Postexposure Medical Evaluations 12-6
12.5 Hazard Communication 12-6
12.5.1 Warning Labels 12-6

Page

12.5.2 Warning Signs 12-6
12.5.3 Employee Training Program 12-6
12.6 Record Keeping 12-7
12.6.1 Training Records 12-7
12.6.2 Medical Records 12-7
12.6.3 Incident Recording 12-7

List of Appendices

Appendix A - Subcontractor Certification
Appendix B - Material Safety Data Sheets
Appendix C - IT Procedure HS603
Appendix D - Supervisor's Employee Accident Report
Appendix E - Site and Hospital Location Map

List of Tables

Table	Title	Follows Page
3-1	Activity Hazard Analysis	3-1
8-1	Action Levels	8-1
8-2	Air Monitoring Frequency and Location	8-1

List of Figures

Figure	Title	Follows Page
1-1	Site Location Map	1-1

List of Acronyms

AIDS	Acquired Immune Deficiency Syndrome
ANSI	American National Standards Institute
CFR	Code of Federal Regulations
CPR	cardiopulmonary resuscitation
CRZ	contamination reduction zone
dBA	decibels
°F	degrees Fahrenheit
DNT	dinitrotoluene
DOL	U.S. Department of Labor
EPA	U.S. Environmental Protection Agency
EZ	exclusion zone
FR	Federal Register
H&S	health and safety
HBV	hepatitis B virus
HEPA	high-efficiency particulate air
HIV	human immunodeficiency virus
IT	IT Corporation
NASA	National Aeronautics and Space Administration
NIOSH	National Institute of Occupational Safety and Health
NPDES	National Pollutant Discharge Elimination System
NRR	Noise Reduction Rating
OSHA	Occupational Safety and Health Administration
PBS	Plum Brook Station
PBOW	Plum Brook Ordnance Works
PE	professional engineer
PEL	permissible exposure limit
PID	photoionization detector
PM	project manager
PPE	personal protective equipment
QAPP	quality assurance project plan
SAP	sampling and analysis plan
SEIR	Supervisor's Employee Injury Report
SSHO	site safety and health officer

SSHP	site safety and health plan
TNT	trinitrotoluene
USACE	U.S. Army Corps of Engineers
VOC	volatile organic compound

1.0 Introduction

1.1 Objectives

The Nashville District, U.S. Army Corps of Engineers (USACE-Nashville) has retained IT Corporation (IT) to conduct a site investigation at G-8 Burning Ground at the former Plum Brook Ordnance Works (PBOW) in Sandusky, Ohio. This site safety and health plan (SSHP) is being prepared in support of the activities and will be used in conjunction with the sampling and analysis plan (SAP) and quality assurance project plan (QAPP).

This SSHP establishes the work practices necessary to ensure the protection of IT personnel during field activities.

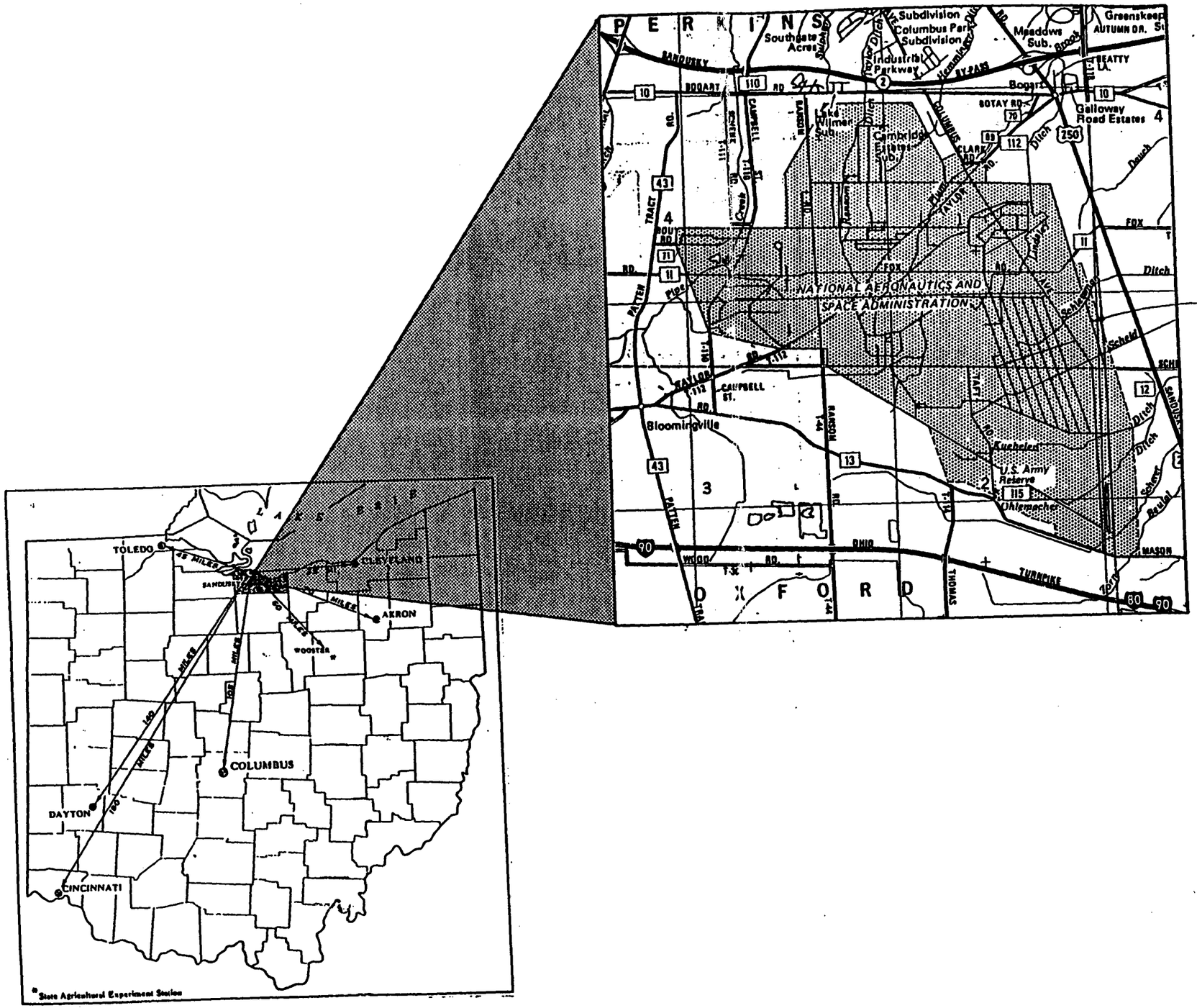
The objective of this plan is to provide a mechanism for the establishment of safe working conditions at the site. The safety organization and health and safety (H&S) procedures have been established following an analysis of potential hazards at the site. Specific hazard control methodologies have been evaluated and selected in an effort to minimize the potential of accident or injury.

All site operations will be performed in accordance with applicable state, local, and IT corporate regulations and procedures, Occupational Safety and Health Administration (OSHA) requirements, and USACE guidelines contained within EM385-1-1. All IT employees and IT subcontractors must comply with the requirements of this plan.

1.2 Facility and Location Description

The former PBOW is located approximately 4 miles south of Sandusky, Ohio and is specifically located in the Perkins, Milan, Huron, and Oxford Townships. The site is bounded on the north by Bogart Road, on the south by Mason Road, on the east by U.S. Highway 250, and on the west by County Road 43 (Figure 1-1). The former PBOW site is consisted of approximately 9,000 acres and lies in an area that is primarily rural and agricultural with low population density. National Aeronautics and Space Administration (NASA) currently owns 6,400 acres of the original site.

Previous activities conducted at the site in the early 1940s included manufacturing trinitrotoluene (TNT), dinitrotoluene (DNT), pentolite, nitric acid, and sulfuric acid. In 1945, manufacturing operations ceased and decontamination of TNT, acid, pentolite, and DNT



FORMER PLUM BROOK ORDNANCE WORKS BOUNDARY

FIGURE 1-1
VICINITY MAP

FORMER PLUM BROOK ORDNANCE WORKS
SANDUSKY, OHIO

INTERNATIONAL TECHNOLOGY CORPORATION

STARTING DATE: 4-26-89	DATE LAST REV.: 2-27-90	INITIATOR: D. BURTON	DRAWING NO.: 409658 - B - WP08
DRAWN BY: S. MOORE	DRAWN BY: P.L. SUAREZ	PROJ. MGR.: D. BURTON	PROJECT NO.: 409658

BRUNING 72425

KD00834

manufacturing lines were completed. NASA acquired PBOW in 1963 and is presently using the site to conduct space research. Over the years, several thousand acres of the original PBOW have been sold off by NASA. The details of these sales are listed in the site management plan and can be found at the NASA Plum Brook Station (PBS).

The G-8 Burning Ground is located in the southwestern corner of the former PBOW site, east of Guard House G-8. Guard House G-8 was formerly located at the corner of Campbell Street and Patrol Road. The area of the burning pit is estimated to be approximately 2 acres and consists of an open area surrounded by hardwood forest. During previous investigations, soil samples indicated the presence of polynuclear aromatic hydrocarbons at 61 to 140 micrograms per kilogram. 2-Butanone was detected in the surface water sample at 7 micrograms per liter. A geophysical survey was conducted over a half-acre area and no apparent anomalies of burn debris/residue or buried metallic objects were found.

1.3 Policy Statement

It is IT's policy to provide a safe and healthful work environment for all its employees. IT considers no phase of operations or administration to be of greater importance than injury and illness prevention. Safety takes precedence over expediency or shortcuts. IT believes every accident and every injury is preventable, and will take every reasonable step to reduce the possibility of injury, illness, or accident.

This SSHP prescribes the procedures that must be followed during the activities at PBOW. Operational changes that could affect the H&S of personnel, the community, or the environment will not be made without the prior approval of the USACE, the IT project manager (PM), and the IT H&S manager.

The provisions of this plan are mandatory for all IT personnel and subcontractors assigned to the project. IT requires all visitors to the work site to abide by the requirements of the plan.

1.4 References

This SSHP complies with applicable OSHA and U.S. Environmental Protection Agency (EPA) regulations. This plan follows the guidelines established in the following:

- *Standard Operating Safety Guidelines* (EPA, November 1984)
- *Occupational Safety and Health Guidance Manual for Hazardous Waste Site Activities* (National Institute of Occupational Safety and Health [NIOSH] 86-116)

- Title 29 of the Code of Federal Regulations (CFR), Part 1910.120, U.S. Department of Labor (DOL)/OSHA

- Title 29 of the CFR, Part 1926.65, DOL/OSHA.

The contents of this plan are consistent with the IT H&S policies and procedures.

2.0 Responsibilities

2.1 All Personnel

Each person is responsible for his safety and that of his coworkers, for completing tasks in a safe manner, and reporting any unsafe acts or conditions to his supervisor. All personnel are responsible for continuous adherence to these H&S procedures during the performance of their work. No person may work in a manner that conflicts with the safety and environmental precautions expressed in this SSHP. After due warnings, IT will dismiss from the site any person who violates safety procedures. IT's employees are subject to progressive discipline and may be terminated for blatant or continued violations. All on-site personnel will be trained in accordance with 29 CFR 1910.120, 29 CFR 1926.65, and this document.

2.2 Project Manager: Don Burton

The PM, Don Burton, is ultimately responsible for ensuring that all project activities are completed in accordance with requirements set forth in this plan. The PM must coordinate at least one on-site safety review during the project. He is responsible for ensuring all accidents and incidents on the project are reported and thoroughly investigated. The PM must approve in writing any addenda or modifications of the SSHP.

2.3 Health and Safety Manager: Melissa G. Smith

The H&S manager for this project, Melissa G. Smith, is responsible for developing and coordinating the SSHP and addenda as required. This plan complies with 29 CFR 1910.120 and 29 CFR 1926.65 in all respects and includes medical surveillance and training requirements, hazard assessment, personal protective equipment (PPE) specifications, field implementation procedures, and audits. The H&S manager is the contact for regulatory agencies on matters of safety and health. Other H&S manager responsibilities include:

- Administering general H&S program
- Determining the level of personnel protection required
- Updating equipment or procedures based on information obtained during site operations
- Establishing air monitoring parameters based on expected contaminants
- Establishing employee exposure monitoring notification programs

- Investigating significant accidents and illnesses and implementing corrective action plans

- Performing regular site inspections

- Developing site-specific employee/community emergency response plans as required based on expected hazards.

2.4 Site Safety and Health Officer: George H. Yu

The site safety and health officer (SSHO) has the ultimate responsibility to stop any operation that threatens the H&S safety of the team or surrounding populace, or that causes significant adverse impact to the environment. Other responsibilities include, but are not limited to:

- Implementing all safety procedures and operations on site

- Observing work crew members for symptoms of on-site exposure or stress

- Upgrading or downgrading, in coordination with the H&S manager and the PM, the levels of personal protection based upon site observations and monitoring results

- Informing the project H&S manager of significant changes in the site environment that require equipment or procedure changes

- Arranging for the availability of first aid and on-site emergency medical care, as necessary

- Determining evacuation routes, establishing and posting local emergency telephone numbers, and arranging emergency transportation

- Ensuring that all site personnel and visitors have received the proper training and medical clearance prior to entering the site (refer to Chapter 6.0 of this plan)

- Establishing exclusion, contamination reduction, and support zones (refer to Chapter 7.0 of this plan)

- Presenting tailgate safety meetings and maintaining attendance logs and records

- Ensuring that the respiratory protection program is implemented (refer to Chapter 5.0 of this plan)

- Ensuring that decontamination procedures meet established criteria
- Ensuring that there are qualified first-aid persons on site who are trained in universal precautions and the use of PPE (refer to Chapter 12.0 of this plan).

2.5 Heavy Equipment Operators

Heavy equipment operators shall be responsible for the safe operation and maintenance of the equipment they operate and other equipment and supplies associated with heavy equipment operations. Operators shall possess appropriate and current licenses/certifications/permits required by local, state, or federal authorities or required by their contract for each project. Heavy equipment operators are responsible for complying with the controls requirements of the Chapter 4.0 subsection entitled "Heavy Equipment Operating Safety."

2.6 Power Auger Operators

Operators of power auger equipment shall be responsible for the safe operation and maintenance of the equipment they operate. Operators shall possess appropriate and current licenses/certifications or permits required by local, state, or federal authorities or required by their contract for each project. Power auger operators are responsible for complying with the controls requirements of the Chapter 4.0 subsection entitled "Power Auger Equipment Operations."

2.7 Subcontractors

On-site subcontractors and their personnel are responsible for understanding and complying with all site requirements. Subcontractors are required to read and acknowledge the subcontractor certification (Appendix A) and follow all of the provisions set forth in this SSHP.

2.8 On-Site Personnel and Visitors

All on-site personnel and visitors are required to comply with the provisions of this SSHP and all applicable federal, state, local, and USACE regulations. Each person is responsible for his/her own safety and health and for completing tasks in a safe manner. All on-site personnel will report any unsafe acts or conditions to his/her supervisor or the IT representative. Personnel will monitor themselves and their fellow employees for signs and symptoms of heat/cold stress and chemical exposure.

2.9 Occupational Physician

The occupational physician for this project, Dr. David Barnes, is board-certified in occupational medicine with more than 20 years of experience. Dr. Barnes is the Medical Director of Environmental Medical Resources, a consulting service for IT.

3.0 Job Hazard Analysis

3.1 Scope of Work

IT will conduct the site investigation, which will include the following activities:

- Surveying
- Soil sampling
- Sediment/seep water sampling
- Excavating/trenching.

Soil sampling will be done by hand augering; however, power augering may be needed at some locations. Decontamination of sampling equipment will be conducted using Alconox.

3.2 Job Hazard Analysis by Task

The job hazard analysis identifies potential safety, health, and environmental hazards and provides for the protection of personnel, the community, and the environment. Because of the complexity and constant change of remediation projects, supervisors must continually inspect the work site to identify hazards that may harm site personnel, the community, or the environment. The PM, site superintendent, and SSHO must be aware of these changing conditions and discuss them with the H&S manager. The H&S manager will write addenda to modify job hazard analyses as necessary. Detailed activity hazard analyses by task are provided in Table 3-1.

3.3 Physical Hazards

The primary physical hazards for this project are associated with the use of power auger, heavy equipment, and supporting vehicles; and the collection of soil, water, and sediment samples.

The potential physical hazards of the work site are as follows:

- Power augering
- Heavy equipment hazards
- Sampling hazards.

3.3.1 Heavy Equipment Hazards

Physical hazards associated with operation of heavy equipment are listed in Table 3-1.

Table 3-1

Activity Hazard Analysis
Plum Brook Ordnance Works, Sandusky, Ohio

Activity	Potential Hazards	Recommended Controls
Staging equipment	Slip, trip, and fall hazards	• Determine best access route before transporting equipment. • Practice good housekeeping; keep work area picked up and clean as feasible. • Continually inspect the work area for slip, trip, and fall hazards. • Look before you step; ensure safe and secure footing.
	Heavy lifting	• Use proper lifting techniques. Lifts greater than 60 pounds require assistance or mechanical equipment.
	Falling objects	• Stay alert and clear of materials suspended overhead; wear hard hat and steel-toed boots.
	Flying debris, dirt, dust, etc.	• Wear safety glasses/goggles; ensure that eye wash is in proper working condition
	Pinch points	• Keep hands, fingers, and feet clear of moving/suspended materials and equipment. • Beware of contact points. • Stay alert at all times!
	Bees, spiders, and snakes	• Inspect work area carefully and avoid placing hands and feet into concealed areas.
	Fire	• Fire extinguishers shall be suitably placed, distinctly marked, readily accessible, and maintained in a fully charged and operable condition.
	Contact with moving equipment/vehicles	• Work area will be barricaded/demarcated. • Backup alarms on vehicles. • Equipment will be laid out in an area free of traffic flow.
	Hazard communication	• Label all containers as to contents and dispose of properly. • Ensure MSDSs are available for hazardous chemicals used on site.
	Noise	• Sound levels above 85 dBA mandates hearing protection.

Table 3-1

Activity Hazard Analysis
Plum Brook Ordnance Works, Sandusky, Ohio

Activity	Potential Hazards	Recommended Controls
Power Augering	Faulty or damaged equipment being utilized to perform work	• All machinery or mechanized equipment will be inspected by a competent mechanic and be certified to be in safe operating condition. • Equipment will be inspected before being put to use and at the beginning of each shift. • Faulty/unsafe equipment will be tagged and if possible locked out.
	Uneven terrain, poor ground support, inadequate clearances, contact with utilities	• Inspections or determinations of road conditions and structures shall be made in advance to ensure that clearances and load capacities are safe for the passage or placing of any machinery or equipment. • All mobile equipment and areas in which they are operated shall be adequately illuminated. • Aboveground and belowground utilities will be located prior to staging equipment. • Whenever the equipment is parked, the parking brake shall be set. • Equipment parked on inclines will have the wheels chocked. • Inspect brakes and tire pressure on drill rig before staging for work. • Obtain trenching/drilling permit prior to operation.
	Inexperienced operator	• Machinery and mechanized equipment shall be operated only be designated personnel. • Heavy equipment operators shall inform their supervisor(s) of any prescribed medication that they are taking that would impair their judgment.
	Falling objects	• Remove unsecured tools and materials before raising or lowering the derrick. • Stay alert and clear of materials suspended overhead.
	Pinch points	• Keep feet and hands clear of moving/suspended materials and equipment. • Stay alert at all times!
	Fire	• Mechanized equipment shall be shut down prior to and during fueling operations. • Have fire extinguishers inspected and readily available.
	Fall hazards	• Personnel are not allowed to work off of machinery or use them as ladders. • Use safety belts and lifeline when working above 6 feet
	Noise	• Hearing protection is mandatory above 85 dBA.
	Contact with rotating or reciprocating machine parts	• Machine guards, use long-handled shovels to remove auger cuttings. • Safe lockout procedures for maintenance work.

Table 3-1

Activity Hazard Analysis
Plum Brook Ordnance Works, Sandusky, Ohio

Activity	Potential Hazards	Recommended Controls
Power Augering (cont'd)	Heavy lifting	• Use proper lifting techniques. Lifts greater than 60 pounds require assistance or mechanical equipment; size-up the lift.
	Slip, trip, and fall hazards	• Good housekeeping, keep work area picked up and clean as feasible. • Continually inspect the work area for slip, trip, and fall hazards.
	Contact with potentially contaminated materials	• Real time air monitoring will take place. If necessary, proper personal protective clothing and equipment will be utilized.
Heavy equipment operation	Faulty or damaged equipment being utilized to perform work	• All machinery or mechanized equipment will be inspected by a competent mechanic and be certified to be in safe operating condition. • Equipment will be inspected before being put to use and at the beginning of each shift. • Faulty/unsafe equipment will be tagged and if possible locked out.
	Uneven terrain, poor ground support, inadequate clearances, contact with utilities	• Inspections or determinations of road conditions and structures shall be made in advance to assure that clearances and load capacities are safe for the passage or placing of any machinery or equipment. • All mobile equipment and areas in which they are operated shall be adequately illuminated. • Aboveground and belowground utilities will be located prior to staging equipment. • Whenever the equipment is parked, the parking brake shall be set. • Equipment parked on inclines will have the wheels chocked. • Inspect brakes and tire pressure on drill rig before staging for work. • Obtain trenching/drilling permit prior to operation.
	Inexperienced operator	• Machinery and mechanized equipment shall be operated only by designated personnel. • Heavy equipment operators shall inform their supervisor(s) of any prescribed medication that they are taking that would impair their judgment.
	Fire	• Mechanized equipment shall be shut down prior to and during fueling operations. • Have fire extinguishers inspected and readily available.
	Fall hazards	• Personnel are not allowed to work off of machinery or use them as ladders. • Use safety belts and lifeline when working above 6 feet
	Noise	• Hearing protection is mandatory above 85 dBA.

Table 3-1

Activity Hazard Analysis
Plum Brook Ordnance Works, Sandusky, Ohio

Activity	Potential Hazards	Recommended Controls
Heavy equipment operation (cont'd)	Vehicle traffic	• Site workers shall establish hand signals when verbal communication becomes difficult. • Seats should be provided for each occupant of the equipment. • Equipment operated on the highway shall be equipped with headlights, taillights, brake lights, back-up lights, and turn signals visible from the front and rear. • Never walk or work directly in back of or to the side of heavy equipment without the operator's knowledge. • Assign flag person(s) if necessary to direct traffic.
	Fall hazards	• Personnel are not allowed to work off of machinery or use them as ladders. • Use safety belts and lifeline when working above 6 feet
	Noise	• Hearing protection is mandatory above 85 dBA.
	Heat Rash	• Keep the skin clean and dry. • Change perspiration-soaked clothing, as necessary. • Bathe at end of work shift or day. • Apply powder to affected areas.
	Heat Cramps	• Drink plenty of cool fluids when not thirsty. • Provide cool fluid for work crews. • Move victim to shaded, cool area.
	Heat Exhaustion	• Conduct physiological worker monitoring as needed (i.e., heart rate, oral temperature). • Set up work/rest periods. • Use the buddy system. • Allow workers time to accumulate. • Have ice packs available for use. • Take frequent breaks.
	Heat Stroke	• Evaluate possibility of night work. • Perform physiological monitoring on workers during breaks. • Wear body cooling devices.

Table 3-1

Activity Hazard Analysis
Plum Brook Ordnance Works, Sandusky, Ohio

Activity	Potential Hazards	Recommended Controls
Soil sampling	Cross-contamination and contact with potentially contaminated materials	• Sampling technicians will wear proper protective clothing and equipment to safeguard against potential contamination. • Only essential personnel will be in the work area. • Initial real-time air monitoring will take place before and during sampling activities. • All personnel will follow good hygiene practices. • Proper decontamination procedures will be followed. • All liquids and materials used for decontamination will be contained and disposed of in accordance with federal, state, and local regulations.
	Cut hazards	• Use care when handling glassware. • Wear adequate hand protection.
	Slip, trip, fall	• Site workers will be required to wear hard hat, safety glasses with side shields, work gloves, and steel-toe/shank boots when working in the field. • Whenever possible, avoid routing cords and hoses across walking pathways. • Flag or cover inconspicuous holes to protect against falls.
	Bees, spiders, and snakes	• Workers shall inspect the work area carefully and avoid placing hands and feet into concealed areas. • Evaluate need for sensitive workers to have prescribed antibiotic or medicine to combat onset of symptoms.
	Poison ivy/oak/sumac	• Avoid plant areas if possible. • Wear long sleeves and long pants. • Promptly wash clothing that has contacted poisonous plants. • Wash affected areas immediately with soap and water.
	Heat Rash	• Keep the skin clean and dry. • Change perspiration-soaked clothing, as necessary. • Bathe at end of work shift or day. • Apply powder to affected areas.
	Heat Cramps	• Drink plenty of cool fluids when not thirsty. • Provide cool fluid for work crews. • Move victim to shaded, cool area.

Table 3-1

Activity Hazard Analysis
Plum Brook Ordnance Works, Sandusky, Ohio

Activity	Potential Hazards	Recommended Controls
Soil sampling (cont')	Heat Exhaustion	• Conduct physiological worker monitoring as needed (i.e., heart rate, oral temperature). • Set up work/rest periods. • Use the buddy system. • Allow workers time to accumulate. • Have ice packs available for use. • Take frequent breaks.
	Heat Stroke	• Evaluate possibility of night work. • Perform physiological monitoring on workers during breaks. • Wear body cooling devices.
	Lightning Strikes	• Whenever possible, halt activities and take cover. • If outdoors, stay low to the ground. • Limit the body surface area that is in contact with the ground, (i.e., kneeling on one knee s better than laying on the ground). • Seek shelter in a building if possible. • Stay away from windows. • If available, crouch under a group of trees instead of one single tree. • Keep all body parts in contact with the ground as close as possible. • Remain 6 feet away from tree trunk if seeking shelter beneath tree(s). • If in a group, keep 6 feet of distance between people.
	Thunderstorms, tornados	• Listen to radio or TV announcements for pending weather information. • Cease field activities during thunderstorms or tornado warnings. • Seek shelter. Do not try to outrun a tornado.

Table 3-1

Activity Hazard Analysis
Plum Brook Ordnance Works, Sandusky, Ohio

Activity	Potential Hazards	Recommended Controls
Moving and shipping collected samples	Heavy lifting	• Use proper lifting techniques. Lifts greater than 60 pounds require assistance or mechanical equipment; size-up the lift.
	Pinch points	• Keep hands, fingers, and feet clear of moving/suspended materials and equipment. • Beware of contact points. • Stay alert at all times!
	Cut hazards	• Wear adequate hand protection. Use care when handling glassware.
	Hazard communication	• Label all containers as to contents and associated
Material storage	Flammable and combustible liquids	• Store in NO SMOKING AREA. • Fire extinguisher readily available. • Transfer only when properly grounded and bonded.

3.3.2 Sampling Hazards

Field sampling operations consist of the collection of subsurface soil, surface water, and sediment samples for subsequent analysis. The physical hazards of this operation are primarily associated with the sample collection methods and procedures utilized.

The primary hazards associated with these specific sampling procedures are not potentially serious; however, other operations in the area, or the conditions under which samples must be collected, may present certain chemical and physical hazards. The hazards of these types of sampling procedures are generally limited to strains/sprains and potential eye hazards resulting from sampling activities.

3.4 Chemical Hazards

The analytical results from previous investigations indicate the following contaminants may be present at the site:

- Nitroaromatics and Nitramines
  - Trinitrotoluene
  - Nitrobenzene
  - Dinitrotoluene
  - Nitrotoluene
  - Dinitrobenzene

- Priority Pollutant Metals
  - Antimony
  - Arsenic
  - Beryllium
  - Cadmium
  - Chromium
  - Copper
  - Lead
  - Manganese
  - Mercury
  - Nickel
  - Selenium
  - Silver
  - Thallium
  - Zinc

- Polychlorinated biphenyls
  - Aroclor 1242
  - Aroclor 1250.

3.5 Noise

Noise exposure at or above the OSHA action level (85 decibels [dBA]) is likely during some activities. Exposure to noise levels in excess of 90 dBA, the OSHA permissible exposure limit (PEL) for noise, is likely during heavy equipment operation. Sound level monitoring will not be conducted during these activities because previous noise monitoring during similar

operations revealed noise levels at or above 85 dBA near the motor side of the operating equipment. A hearing conservation program shall be required for heavy equipment operators.

Exposure to noise over the OSHA action level can cause temporary impairment of hearing; prolonged and repeated exposure can cause permanent damage to hearing. The risk and severity of hearing loss increases with the intensity and duration of exposure to noise. In addition to damaging hearing, noise can impair voice communication, thereby increasing the risk of accidents on site.

3.6 Anticipated Biological Hazards

3.6.1 Snakes

Workers at the site have potential for encountering poisonous snakes such as water moccasins, rattlesnakes, and copperheads. All personnel shall be cautioned to be alert to the presence of snakes. If a snake bite occurs, attempts should be made to kill the snake for positive identification. The victim should be transported to the nearest hospital within 30 minutes. First aid consists of applying a pressure bandage; omit cutting and sucking (unless medical care cannot be obtained within 30 minutes). Snake venoms are complex and include proteins, some of which have enzymatic activity. The effects produced by venoms include neurotoxic effects with sensory, motor, cardiac, and respiratory difficulties; cytotoxic effects on red blood cells, blood vessels, heart muscle, kidneys, and lungs; defects in coagulation; and effects from local release of substances by enzymatic actions. Other noticeable effects of snake bites include swelling, edema and pain around the bite, and the development of ecchymosis (the escape of blood into tissues from ruptured blood vessels).

3.6.2 Insects

All IT personnel and subcontractors should be aware of the potential presence of deer ticks in wooded areas during the months of April through October. Precautionary methods include familiarity with the signs/symptoms and potential health hazards of Lyme disease, and the proper implementation of appropriate work practices.

During the months of April through October, particular caution must be exercised to prevent being bitten by ticks and potentially contracting Lyme disease, Rocky Mountain Fever, and other tick-borne diseases. Specific precautions include:

- Wear hooded coveralls to cover your body as much as possible. Light color clothing makes spotting of ticks easier.

- Try to eliminate possible paths by which the deer tick may reach unprotected skin. For example, tuck bottoms of pants into socks or boots and sleeves into gloves. (Duct tape may be utilized to help seal cuffs and ankles.) If heavy concentrations of ticks or insects are anticipated or encountered, Tyvek coveralls may be utilized.

- Conduct periodic and frequent (e.g., hourly) surveys of your clothing for the presence of ticks. Remove any ticks/insects that become attached to clothing.

- Use insect/tick repellents. Apply repellents in accordance with manufacturers' recommendations. These repellents are readily available and include such brands as Deep Woods OFF and Maximum Strength OFF. Check the ingredients of the repellent. IT field personnel should check with the PM to ensure use of insect repellents will not interfere with sample collection procedures.

3.6.3 Poisonous Plants

If work sites are located in areas where poisonous plants such as poison ivy, poison sumac, and poison oak may be encountered, personnel should wear long pants, long sleeves, and gloves to minimize the possibility of exposure. In some areas, the use of a Tyvek or other protective suit may be advisable.

3.7 Heat Stress

Wearing PPE puts site personnel at considerable risk of heat stress. Heat stress effects range from transient heat fatigue to serious illness and death. Heat stress is caused by a number of interacting factors, including environmental conditions, clothing, workload, and the individual chracteristics of the worker. Because heat stress is one of the most common and potentially serious illnesses during drilling operations, preventive measures and alertness to the symptom are vital.

Heat stress monitoring shall commence when personnel are wearing PPE, including Tyvek coveralls, and the ambient temperature exceeds 70 degrees Fahrenheit (°F). If impermeable garmets are not worn, heat stress monitoring shall commence at 85°F. The protocols of heat stress monitoring and symptoms of heat stress are discussed in Section 4.3.

4.0 Safety Program and Procedures

The following work practices will be observed during all site activities.

4.1 General Practices

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

- Ensure that no one is required to lift more than 60 pounds.

- Legible and understandable precautionary labels that comply with the hazard communication standard shall be affixed prominently to all containers of contaminated scrap, waste, debris, and clothing.

- No food or beverages shall be present or consumed in the work areas.

- No tobacco products shall be present or used in the work areas.

- Cosmetics shall not be applied within the work areas.

- All crew personnel on site shall use the buddy system (working in pairs or teams). If protective equipment or noise levels impair communications, then prearranged hand signals shall be used for communication. Visual contact shall be maintained between crew members at all times, and crew members must observe each other for signs of toxic exposure. Indication of adverse effects include, but are not limited to:

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

- Employees shall inform their partners or fellow team members of nonvisible effects of overexposure to toxic materials. The symptoms of such overexposure may include:

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

- Visitors to the site shall abide by the following:

  - "Visitor" means persons not involved in routine site work activities.

  - All visitors shall be instructed to stay outside the work area and remain within the support area during the extent of their stay. Visitors shall be cautioned to avoid skin contact with contaminated or potentially contaminated surfaces.

4.2 Operating Procedures

4.2.1 Power Auger Operations

Specific elements of this section may not be applicable, depending on the type of power augering equipment used and/or for the anticipated scope of this project. Prior to the start of site work, the subcontractor will inspect all equipment in the presence of the site supervisor.

General Power Augering Practices

- The departing operator shall inform the oncoming operator of any special hazards or ongoing work that may affect the safety of the crew.

- Fire-fighting equipment shall not be tampered with and shall not be removed for other than the intended fire-fighting purposes or for servicing.

- If lubrication fittings are not accessible with guards in place, machinery shall be stopped for oil and greasing.

- Rigging material equipment for material handling shall be checked prior to use on each shift and as often as necessary to ensure it is safe. Defective rigging shall be removed from service.

- Work areas and walkways shall not be obstructed.

4.2.2 Drum Handling Safety

Operations shall be organized to minimize the amount of drum or container movement. Employers shall be warned of the potential hazards associated with the contents of the drums or containers during tailgate safety meetings prior to the beginning of disposition operations. Ignition sources during transfer operations shall be minimized by using proper equipment and procedures. Standing upon or working from drums or containers is prohibited.

4.2.3 Heavy Equipment Operating Safety

The physical hazards and control methods shown in Table 3-1 shall be reviewed for each task and communicated to all affected personnel. As new hazards are discovered, the hazards will be promptly communicated to site personnel. Prior to the start of site work, the heavy equipment operators will inspect all equipment in the presence of the site supervisor.

All heavy equipment will be used in the manner it was intended. Drivers will operate all equipment in accordance with the manufacturers instructions and within the safe operating parameters as defined by the manufacturer.

Ground personnel should always make eye contact and wait for a signal to proceed before passing close to or in front of operating equipment or moving vehicles. Where possible, heavy equipment in stationary operation should be barricaded (with hazard tape) at a sufficient distance for ground personnel to avoid swinging cabs, counter weights, and brooms. When ground personnel are working in the vicinity of heavy equipment, they should inform the flagman of their presence.

All drivers and operators will be expected to adhere to the speed limits, signs, and road markings.

All heavy equipment will carry a multipurpose dry chemical fire extinguisher (at least 5 pounds).

Backhoes and other noncrane heavy equipment will be used to "pull" (lift) equipment only when this is within the normal design parameter of the equipment. If a piece of equipment such as a trackhoe is used to make a lift, it will have a lifting eye mounted on or near the bucket. This lifting eye must be standard equipment for the make and the model of equipment.

During trenching, IT Procedure HS307 on excavation and trenching must be followed. This procedure complies with OSHA regulations 1926.650 through 1926.652. Any excavation greater than 5 feet deep into which persons must enter must be shored, sloped, or otherwise made safe for entry. The shoring or sloping system must be designed by a registered professional engineer (PE) licensed in the state in which the excavation will take place. The design must be present at the site, and the system must be installed as designed.

All excavation shall be performed from a stable ground position. Daily inspections of the excavation shall be made by a competent person who has received training in excavation safety. The inspector shall determine the likelihood of a cave-in, and remedial action such as sloping or shoring shall be taken if the walls appear to be unstable.

All spoil shall be located at least 2 feet from the edge of the excavation to prevent it from falling back into the excavation. The excavation shall be guarded on all sides by barricades or caution tape at least 2 feet from the edge.

All project personnel shall participate in the daily tailgate safety meetings and be instructed on the following requirements:

- Operations must be suspended and the area vented if the airborne volatile organic compound (VOC) concentration reaches 25 parts per million continuously using the HNu photoionization detector (PID).
- VOC reading of the general work area will be made regularly.

4.2.4 Lifting Safety

Back strain or injury may be prevented by using proper lifting techniques. The fundamentals of proper lifting include:

- Consider the size, shape, and weight of the object to be lifted. Two persons must lift an object if it cannot be lifted safely alone (e.g., greater than 60 pounds).
- The hands and the object should be free of dirt or grease that could prevent a firm grip.
- Gloves must be used, and the object inspected for metal slivers, jagged edges, burrs, rough or slippery surfaces.
- Fingers must be kept away from points that could crush or pinch them, especially when putting an object down.
- Feet must be placed far enough apart for balance. The footing should be solid and the intended pathway should be clear.
- The load should be kept as low as possible, close to the body, with the knees bent.

- To lift the load, grip firmly and lift with the legs, keeping the back as straight as possible.

4.2.5 Confined-Space Entry

Confined-space entry will not be performed during activities at the sites. If confined-space entry must be performed, an SSHP addendum shall be prepared and approved prior to work start.

4.3 Heat Stress Prevention

One or more of the following control measures can be used to help control heat stress and are mandatory if any site worker has a heart rate in excess of 110 beats per minute (measured immediately prior to rest period):

- Site workers will be encouraged to drink plenty of water throughout the day. They will be advised to slightly increase their salt intake by lightly salting their food.

- On-site drinking water will be kept cool (50 to 60°F) to encourage personnel to drink frequently.

- A work regimen that will provide adequate rest periods for cooling down will be established as required using environmental temperatures.

- All personnel will be advised of the dangers and symptoms of heat stroke, heat exhaustion, and heat cramps.

- Cooling devices such as vortex tubes or cooling vests should be used when personnel must wear impermeable clothing in conditions of extreme heat.

- Employees should be instructed to monitor themselves and coworkers for signs of heat stress and to take additional breaks as necessary.

- A shaded rest area must be provided. All breaks should take place in the shaded rest area.

- Employees shall not be assigned to other tasks during breaks.

- Employees shall remove impermeable garments during rest periods. This includes white Tyvek-type garments.

- All employees shall be informed of the importance of adequate rest, acclimation, and proper diet in the prevention of heat stress disorders.

The signs of heat stress disorders follow.

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

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

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

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

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

4.4 Hearing Conservation

All on-site IT and subcontractor personnel shall wear hearing protection with a Noise Reduction Rating (NRR) of at least 20 when noise levels exceed 85 dBA. All site personnel who may be exposed to noise shall also receive baseline and annual audiograms and training as to the causes and prevention of hearing loss. Noise monitoring is discussed in Section 8.3.

Whenever possible, equipment that does not generate excessive noise levels will be selected for this project. If the use of noisy equipment is unavoidable, wherever possible, barriers or increased distance will be used to minimize worker exposure to noise.

4.5 Sanitation

4.5.1 Potable Water

The following rules apply for all project field operations:

- An adequate supply of potable water will be provided at each work site.
- Portable containers used to dispense drinking water shall be capable of being tightly closed, and shall be equipped with a tap dispenser. Water shall not be drunk directly from the container.
- Containers used for drinking water shall be clearly marked and not used for any other purpose.
- Disposable cups will be supplied; both a sanitary container for unused cups and a receptacle for disposing of used cups shall be provided.

4.5.2 Nonpotable Water

Outlets for nonpotable water shall be identified to clearly indicate that the water is unsafe and is not to be used for drinking, washing, or cooking purposes. There shall be no cross connection (open or potential) between potable and nonpotable water systems. Nonpotable and potable water systems shall be separated so as to minimize confusion and possible cross contamination.

4.5.3 Toilet Facilities

Toilet facilities shall be available for employees. If permanent toilet facilities are not available (i.e., the work site is more than 500 feet from a building with an accessible toilet), then a portable chemical toilet(s) will be provided.

4.5.4 Trash Collection

Trash collected from the contaminated reduction zone (CRZ) will be separated as routine hazardous waste. Trash collected in the support and break areas will be disposed of as nonhazardous waste. Labeled trash receptacles will be set up in the CRZ and in the support zone.

5.0 Personal Protective Equipment

The PPE outlined in this chapter has been selected according to the site characterization and analysis, job tasks, site hazards, intended use, and duration of potential employee exposures. Maintenance and storage of PPE, decontamination, donning and doffing procedures, inspection and monitoring of effectiveness, and PPE limitations are outlined in this chapter.

5.1 Respiratory Program

A comprehensive respiratory protection program has been established by IT and is required in all locations where use of such equipment is intended to lessen the potential for adverse health affects to any employee.

As part of the respiratory training program, each employee is instructed in the following elements:

- Nature of the respiratory hazard on the work site and the appraisal of potential consequences if the respiratory protection is not utilized
- Use and proper fitting of the respirator
- Cleaning, disinfecting, inspection, maintenance, and storage of the respirator
- Proper selection, capabilities, and limitations.

Routinely used respiratory equipment will be inspected, cleaned, and disinfected daily to help assure proper hygienic practices. An inspection of these breathing devices will include the following:

- Examine the head straps for breaks, loss of elasticity, broken or malfunctioning buckles and other attachments.
- Examine the face piece for excessive dirt, cracks, tears, distortion, holes, or inflexibility.
- Examine the exhalation and inhalation valves for any foreign material, cracks, tears, or distortion in the valve. Additional checks will be made to inspect for proper insertion, defective valve covers, or improper installation.
- Examine the air purifying elements for incorrect cartridge, expired shelf life of the cartridge, cracks or dents in the cartridge or cartridge-holder.

- Examine for proper insertion of the cartridges into the face-piece and a check of the gaskets inside the cartridge holder.

- Examine air cylinders for adequate air volume. Only Grade D air will be utilized for breathing air.

When Level C protection is required, respirator cartridges will be changed daily or if increased breathing resistance is detected. All respirators will be inspected prior to each day's use. If broken or malfunctioning parts are found during the cleaning process, these parts will be replaced or new respiratory equipment will be issued to the user.

The respiratory protective equipment will be stored in an area protected from any mechanical damage. These devices will also be stored in a location that provides protection against dust, heat, excessive moisture, or damage by chemical contact. The storage area for the respirators should be in a readily accessible location.

- Only employees who have been trained to wear and maintain respirators properly will be allowed to use respiratory protection.

- Selection of respirators, as well as any decisions regarding upgrading or downgrading of respiratory protection, will be made by the H&S manager or designee.

- Positive and negative pressure checks will be performed each time the respirator is donned.

- Only employees who have been fit tested within the last 12 months will be allowed to work in atmospheres where respirators are required.

- Respirator users will be instructed in the proper use and limitations of respirators.

- If an employee has difficulty in breathing during the fit test or during use, he will be evaluated medically to determine if he can wear a respirator safely while performing assigned tasks.

- No employee will be assigned to tasks requiring the use of respirators if, based upon the most recent examination, a physician determines that the health or safety of the employee will be impaired by respirator use.

- Contact lenses will not be worn while using any type of respiratory protection.

- Air-supplied respirators will be assembled according to manufacturer's specifications. Hose length, couplings, valves, regulators, manifolds, and all accessories

will meet American National Standards Institute (ANSI) and the manufacturer's requirements.

- Respirators will be cleaned and sanitized daily after use.
- Respirators will be stored in a convenient, clean, and sanitary location on site.
- Respirators will be inspected during cleaning. Worn or deteriorated parts will be replaced.
- Facial hair that might interfere with a good face-piece seal or proper operation of the respirator is prohibited.
- The IT PM will review the respiratory protection program daily to ensure employees are properly wearing and maintaining their respirators and that the respiratory protection is adequately protecting the employees.
- The H&S manager and the PM will evaluate the respiratory protection program monthly to ensure the continuing effectiveness.
- Respirators used for emergency response will be inspected weekly by the SSHO.

5.2 Levels of Protection

The level of protection used in the exclusion zone (EZ) is based on site-specific information. Specific levels of protection will be changed whenever site conditions change. Levels of protection can either be increased to the next higher level or decreased to the next lower level. If the site superintendent requests a change in levels of protection, he must contact the IT H&S manager and PM. If the need arises to protect safety and health, the site superintendent can upgrade protection levels without input from the H&S manager or PM. He will then review his decision with the H&S manager, SSHO, and PM. Levels of protection will not be downgraded without prior approval from the H&S manager.

5.2.1 Level A Protection

Level A protection is not required.

5.2.2 Level B Protection

Level B protection is not required.

5.2.3 Level C Protection

Level C protection will not be used unless air monitoring data indicates the need for upgrade; however, the equipment shall be readily available on site. The following equipment will be used for Level C protection:

- NIOSH/Mine Safety and Health Agency-approved full face, air purifying respirators equipped with organic vapor cartridge in combination with high-efficiency particulate air (HEPA) filter
- Hooded, polyethylene-coated Tyvek, taped at gloves, boots, and respirator
- Nitrile gloves (outer)
- Latex gloves (inner)
- Neoprene steel-toed boots or polyvinyl chloride overbooties/steel toed safety boots
- Hard hat
- Hearing protection (when working near/adjacent to operating equipment).

5.2.4 Level D-Modified Protection

The following equipment will be used for Level D-Modified protection:

- Permeable Tyvek, Kleenguard, or its equivalent
- Latex boot covers
- Nitrile, heavy work, or latex gloves
- Steel-toed safety boots
- Safety glasses
- Hard hat
- Hearing protection (when working near/adjacent to operating equipment).

5.2.5 Level D Protection

The minimal level of protection that will be required of IT personnel at the site will be Level D. The following equipment will be used for Level D protection:

- Coveralls or work clothing
- Steel-toed safety boots
- Safety glasses
- Hard hat
- Hearing protection (when working near/adjacent to operating equipment).

5.2.6 Selection of PPE

The selection of the PPE has been done after a thorough evaluation of the hazards involved during each sampling task. The initial levels of PPE are as follows:

Task	Initial Level of PPE
Staging equipment	Level D
Power Augering	Modified Level D*
Soil boring and sampling	Modified Level D*
Excavation/trenching	Modified Level D*

*Initial level will be raised to Level C or higher if air monitoring results in worker's breathing zone are above action levels stated in Chapter 8.0.

5.3 Using PPE

All persons entering the EZ will don the required PPE according to established procedures in this plan to minimize exposure potential. When leaving the EZ, PPE will be removed according to the established procedures to minimize the spread of contamination.

5.3.1 Donning Procedures for Level C PPE

- Put on boots and boot covers and tape the coveralls.
- Put on gloves.
- Tape the coveralls over the gloves at the wrist.
- Don respirator and check for secure fit.
- Put hood or head covering over the respirator.
- Put on remaining protective equipment, i.e., hard hat, safety glasses, etc.

One person will remain outside the work area to check that each person entering has the proper protective equipment. No persons will be allowed to enter an EZ improperly attired.

5.3.2 Doffing Procedures for Level C PPE

Whenever a person leaves the work site, the following proper decontamination sequence will be followed:

- Upon entering the CRZ, rinse contaminated mud and debris from boots or remove boot covers.

- Clean reusable protective equipment.

- Remove protective garments and equipment. All disposable clothing should placed in plastic bags and labeled as contaminated waste.
- Remove respirator.
- Proceed to the clean area and dress.
- Clean respirator and prepare for next use.

All disposable equipment, garments, and PPE will be bagged in two 6-mil plastic bags and properly labeled for disposal.

6.0 Site Control

6.1 Authorization to Enter

Access to work areas where intrusive activities are conducted is regulated and limited to authorized personnel. Only persons who have completed the following training and medical requirements will be allowed to enter in this work zone:

- Completion of 40 hours of hazardous waste operations training as defined under 29 CFR 1910.120 or 29 CFR 1926.65
- Completion of the 40-hour hazardous waste operations training within the past 12 months or completion of an 8-hour hazardous waste operations refresher
- Certification by a physician of fitness for hazardous waste operations within the last 12 months.

Personnel not meeting these requirements may enter the designated support zone only. The SSHO will maintain a list of persons authorized to enter contaminated work areas, and only personnel on the authorized persons list will be allowed within the work zone.

6.2 Site-Specific Orientation

No person shall be allowed on any field site without first being given a site-specific H&S orientation. This orientation will provide training on the potential H&S hazards and procedures specific to the site, and will discuss the provisions of this SSHP. All personnel will acknowledge their attendance by signing the SSHP Acknowledgement Form. IT subcontractors must also acknowledge and sign the form in Appendix A, Subcontractor Certification.

6.3 Documentation of Certificates

Personnel entering the site to work will have satisfied the medical and training requirements of 29 CFR 1910.120. The project file will contain copies of certificates documenting status for all on-site personnel. Personnel not entering the work zone need not meet the requirements in Section 6.1. The PM will accommodate requests from representatives of regulatory agencies to review documentation. All visitors must present documentation of current training and medical status before being granted authorization to enter the work zone.

6.4 Entry Log

The PM shall keep a daily roster of all on-site personnel and record the time of entry into and exit from the work zone for each person.

7.0 Decontamination

The project areas will be divided into three work zones: the EZ, a CRZ, and a support zone. The PM will be responsible for designation of the work zones. The EZ will be located in an approximately 20-foot radius around the power auger or heavy equipment operation and will be marked with barrier tape or other means to warn personnel of the hazards.

Only IT personnel and authorized visitors who have met the requirements of Section 6.1 and who are wearing the required PPE will be allowed within this zone.

Immediately adjacent to the EZ, a decontamination area for equipment and personnel will be established. This area will also be delineated with traffic cones and/or barrier tape. Each side of the decontamination zone will be posted.

The remainder of the IT project area will be designated as the support zone. No special markings or warning labels are required for this area.

7.1 Personnel Decontamination

All personnel working in the EZ must undergo personnel decontamination prior to entering the support zone. The personnel decontamination area shall consist of the following stations.

Station 1. Personnel leaving the EZ will remove the gross contamination from their outer clothing and boots.

Station 2. Equipment for this station may include plastic-lined waste receptacle, chair, clean damp cloths or paper towels, and plastic bags. At Station 2, personnel will remove their Tyvek coveralls and gloves and deposit them in the lined waste receptacles. Personnel will wipe their respirators (if used), hard hats, and boots with clean, damp cloths and then remove those items. Those items are then hand carried to the next station.

Station 3. Equipment for this station may include a wash basin with soap and water and respirator sanitation station. At this station, personnel will thoroughly wash their hands and face before leaving the decontamination zone. Respirators will be sanitized and then placed in a clean plastic ziplock bag.

7.2 Equipment Decontamination

Any heavy equipment that has entered the EZ shall be decontaminated prior to leaving the decontamination zone using methods specified in design package drawings.

7.3 PPE Decontamination

Where and whenever possible, single-use, external protective clothing shall be used for work within the EZ or CRZ. This protective clothing shall be disposed of in marked containers. Depending upon subsequent analysis, this protective clothing may require disposal as hazardous waste.

Reusable protective clothing will be rinsed at the site with detergent and water. The rinsate will be collected for possible disposal as hazardous waste. A Material Safety Data Sheet must be present on site for any hazardous chemicals used during decontamination procedures (see Appendix B).

Respiratory protective equipment will be wiped with a damp cloth while in the CRZ and bagged. After the respirator has been removed from the CRZ, it will be thoroughly cleaned with soap and water. The respirator face piece will be cleaned at the end of each work shift.

8.0 Site Monitoring

8.1 Air Monitoring

The primary contaminants of concern at G-8 Burning Ground are constituents of TNT manufacturing process: toluene, TNT, and DNT.

Measurements of airborne VOCs will be conducted in the breathing zone of employees in each work area and their respective support zones by using an HNu PID with an 10.2 electron volt lamp. All air monitoring activities shall be documented on the IT Real Time Air Monitoring Log or Field Activity Daily Log form.

The action level for airborne contaminants is provided in Table 8-1.

All real-time air monitoring results will be provided to the USACE as these results become available.

8.1.1 Air Monitoring Frequency

Air monitoring frequency and location is provided in Table 8-2.

8.1.2 Air Monitoring Equipment

An HNu Model PI-101 PID or equivalent will be available for on-site utilization. All equipment shall be maintained in such quantity and condition to adequately monitor and assess all site operations.

8.1.3 Monitoring Equipment Maintenance and Calibration

All IT HNu PIDs will be calibrated in accordance with IT Procedure HS603 in Appendix C. Preventive maintenance and repairs will be conducted in accordance with the respective manufacturer's procedures.

All direct reading instrumentation calibrations should be conducted under the approximate environmental conditions the instrument will be used. All air monitoring equipment calibrations and maintenance activities shall be documented on the IT Real Time Instrument Calibration Log form or Field Activity Daily Log form. All completed H&S documentation/forms shall be maintained by the SSHO for review by the H&S manager.

Table 8-1

Action Levels
Plum Brook Ordnance Works, Sandusky, Ohio

When in Level C PPE

Analyte	Action Level	Required Action[a]
Volatile organic hydrocarbons	≥ 25 ppm above background in breathing zone (BZ)	Stop work

When in Level D Modified/D PPE

Analyte	Action Level	Required Action[b]
Volatile organic hydrocarbons	≥ 5 ppm above background in BZ	Upgrade to Level C PPE.

When in Support Zone

Analyte	Action Level	Required Action
Volatile organic hydrocarbons	≥ 1 ppm above background in BZ	Evacuate support zone and re-establish perimeter of EZ.

[a]Four instantaneous peaks in any 15-minute period or a sustained reading for 5 minutes in excess of the action level will trigger a response.
[b]Contact with the H&S manager must be made prior to continuance of work. The H&S manager may then initiate perimeter/integrated air sampling along with additional engineering controls.

No one is permitted to downgrade levels of PPE without authorization from the H&S manager.

Table 8-2

Air Monitoring Frequency and Location
Plum Brook Ordnance Works, Sandusky, Ohio

Work Activity	Instrument	Frequency	Location
Staging equipment	HNu	Initially for area	Breathing zone (BZ) of employees
Sampling	HNu	Initially, then periodically	BZ of employees Support zone
Excavation/trenching	HNu	Initially, then periodically	BZ of employees Support zone

If an instrument is found to be inoperative or suspected of giving erroneous readings, the SSHO shall be responsible for immediately removing the instrument from service and obtaining a replacement unit. The specific IT or subcontractor operation for which this equipment is essential shall cease until an appropriate replacement unit is obtained. The SSHO shall be responsible for ensuring a replacement unit is obtained and/or repairs are initiated on the defective equipment.

When applicable, only manufacturer-trained and/or authorized IT personnel will be allowed to perform instrument repairs or preventive maintenance.

8.2 Other Hazardous Conditions

The SSHO will take affirmative action to limit exposures. If unknown chemicals or contamination are encountered, operations will cease until the situation is evaluated. The SSHO will contact the H&S manager to evaluate any potentially hazardous situations, or any situation with elevated contamination levels. Operations will only be resumed if they can be accomplished in a safe manner.

8.3 Noise Monitoring

Noise monitoring will not be conducted because previous noise monitoring during similar drilling and heavy equipment operations has characterized noise levels greater than 85 dBA within a 5-foot radius around the rear end (motor side) of heavy equipment. Hearing protection is mandatory for all employees in or when working near/adjacent to operating heavy equipment. Noise monitoring will occur when new heavy equipment is brought on site and when new tasks are started that have not previously had their noise levels characterized.

8.4 Monitoring Records

The PM shall ensure that site monitoring records are complete and incorporated into the project file. Any personnel or area air monitoring results will be incorporated into the IT H&S files. The host H&S manager will be responsible for establishing, maintaining, and forwarding to other IT offices (as necessary) all required monitoring information as follows for placement in individual employee files:

- Employee name, social security number, and payroll number
- The date, time, pertinent task information, and exposure information
- Weather conditions (wind direction, precipitation, temperature, etc.)

- Description of the analytical methods, equipment used, equipment identification number, and calibration data
- Type of PPE worn
- Engineering controls used to reduce exposure.

8.5 Notification

Within 5 working days after receipt of monitoring results, the project H&S staff and the host office H&S staff will ensure that each employee is informed in writing of the results which represent that employee's exposure. Monitoring results representative of an employee's exposure shall be reported to the affected employee on the IT Employee Notification of Industrial Hygiene Monitoring Results Form.

Whenever the results indicate that the representative employee exposure exceeds the PEL, the Employee Notification of Industrial Hygiene Monitoring Results Form shall state that the PEL was exceeded, and shall provide a description of the corrective action taken to reduce exposure to a level below the PEL.

IT will provide industrial hygiene monitoring results to subcontractor companies, if the exposure of subcontractor employees to airborne contaminants is elevated. Notification of subcontractor personnel of industrial hygiene monitoring results is the responsibility of the subcontractor.

9.0 Employee Training

9.1 General

All on-site project personnel shall have completed at least 40 hours of hazardous waste operations training, as required by 29 CFR 1910.120 and 29 CFR 1926.65. In addition, all field employees shall have received a minimum of 3 days of actual field experience under the direct supervision of a trained, experienced supervisor. Those personnel who completed the 40-hour training more than 12 months prior to the start of the project shall have completed an 8-hour refresher course within the past 12 months. Supervisors shall have completed an additional 8 hours of relevant H&S training. Subcontractor personnel must meet these training requirements. Documentation of IT and subcontractor training shall be maintained with the project files.

9.1.1 Site-Specific Orientation

All personnel, before performing any work at the site, are required by the NASA PBS to go through a safety orientation, which will include a short briefing at the guard house, and a 10-minute orientation video. In addition, all on-site project personnel will be given a site-specific H&S orientation. This orientation will provide training on the potential H&S hazards and procedures specific to the site, and will discuss the provisions of this SSHP. All personnel will acknowledge their attendance by signing the SSHP Acknowledgement Form. Additionally, subcontractors must acknowledge the Subcontractor Certification Form in Appendix A.

9.1.2 Tailgate Safety Meetings

The SSHO shall conduct a tailgate safety meeting at the beginning of each shift or whenever new employees arrive at the job site once the job commences. The topics discussed at the tailgate safety meeting will include H&S considerations for the day's activities, necessary protective equipment, potential for encounters with biological hazards, problems encountered, and new operations. Attendance records and meeting notes will be maintained with the project files.

9.2 Site Workers' Basic Course

The following is a list of the topics covered in IT's 40-hour training course:

- General site safety

- Physical hazards (fall protection, noise, heat stress, cold stress)
- Names and titles of key personnel responsible for site H&S
- Safety, health, and other hazards typically present at hazardous waste sites
- Use of PPE
- Work practices by which employees can minimize risks from hazards
- Safe use of engineering controls and equipment on site
- Medical surveillance requirements including recognition of symptoms and signs that might indicate overexposure to hazards
- Worker right-to-know (Hazard Communication OSHA 1910.1200)
- Routes of exposure to contaminants
- Engineering controls and safe work practices
- Components of the site H&S program
- Decontamination practices for personnel and equipment
- Confined-space entry procedures
- Emergency response plan.

9.3 Supervisors' Course

Management and supervisors must receive an additional 8 hours of training presented by the IT Training Department that includes:

- General site safety and health programs
- PPE programs
- Air monitoring techniques.

9.4 First Aid and Cardiopulmonary Resuscitation

At least two employees current in first aid/cardiopulmonary resuscitation (CPR) will be assigned to the work crew and will be on the site whenever operations are ongoing. First aid and CPR training courses are offered to all IT employees. Annual refresher training in CPR and triannual refresher training in first aid is required to maintain the currency of the certificate.

9.5 Instructors

The IT Training Department, headquartered in Torrance, California, teaches the 40-hour hazardous waste operations classes using certified environmental trainers. When training needs exceed the capacity of the training division, IT uses outside institutions. IT is recognized by EPA and listed in the Federal Register (53 FR 3982). Only similarly recognized outside training institutions may be used with prior approval of the IT Training Department.

10.0 Medical Surveillance Program

10.1 Physical Examinations

All on-site project personnel shall within the past 12 months have completed a comprehensive medical examination that meets the requirements of OSHA regulations 29 CFR 1910.120 and 29 CFR 1926.65. The annual medical includes the following elements:

- Medical and occupational history questionnaire
- Physical examination
- Complete blood count, with differential
- Liver enzyme profile
- Chest X-ray, once every 3 years, for nonasbestos workers
- Pulmonary function test
- Audiogram
- Electrocardiogram for persons older than 35 years of age, or if indicated during the physical examination
- Drug screening
- Visual acuity
- Follow-up examinations, at the discretion of the examining physician or the corporate medical director.

All employee medical clearance forms are maintained by the H&S group within the worker's home profit center, or for subcontractors at the subcontractor's office. The examining physician provides the employee with a letter summarizing his findings and recommendations. Each employee also has the right to inspect and copy medical records.

The examining physician provides the employer with a letter confirming the worker's fitness for work and ability to wear a respirator. A copy of this letter for all project workers will be kept on site during all project site work.

Subcontractors will verify on the certification form that all their employees have successfully completed a physical examination by a qualified physician. The physical examinations shall meet the requirements of 29 CFR 1910.120, 29 CFR 1910.134, HS100, and HS101. Subcontractors must submit a completed Subcontractor Certification Form (Appendix A) and supply copies of the medical examination certificate for each on-site employee.

10.1.1 Preplacement Examination

All employees will receive a preplacement medical examination prior to assignment to field operations.

10.1.2 Annual Examination

Each year subsequent to the placement examination, all employees and subcontractors must undergo an annual examination similar in scope to the placement examination. IT employees hired prior to 1985 are not required to submit to drug screening. Chest x-rays are taken every third year. The medical and occupational history is updated with each examination.

10.1.3 Exit Examination

IT employees receive an exit examination upon leaving the company if they have not been examined within the previous 6 months. The exit examination consists of the annual examination without drug screen. The employee's immediate supervisor is to notify the home office H&S assistant within a reasonable time before the termination to allow for the necessary arrangements.

10.2 First-Aid and Medical Treatment

All persons on site must report any near-miss incident, accident, injury, or illness to their immediate supervisor. First aid will be provided by the designated site first aider. Injuries and illnesses requiring medical treatment will be accompanied by an Authorization for Treatment form. The employee's supervisor will complete the Supervisor's Employee Injury Report (SEIR) (Appendix D) and conduct an accident investigation as soon as emergency conditions no longer exist and first aid and/or medical treatment has been rendered. The results of the investigation should be reported on the accident/injury investigation report. The SEIR must be completed and submitted to the H&S manager within 24 hours of the incident. The accident/injury investigation report must be submitted within 72 hours of the incident.

If first-aid treatment is required, first-aid kits are available at the CRZ and in all IT vehicles. If treatment beyond first aid is required, the injured should be transported to the medical

facility shown in Appendix E. If the injured is not ambulatory or shows any sign of not being in a comfortable and stable condition for transport, then an ambulance/paramedics should be summoned. If there is any doubt as to the injured worker's condition, it is best to let the local paramedic or ambulance service examine and transport the worker. In case of any emergency, the IT field supervisor needs to contact the PBS Communications Center located in the front gate for assistance using the portable station radio provided by the PBS.

10.3 Medical Restriction

When a medical care provider identifies a need to restrict work activity, the employee's home office H&S assistant will communicate the restriction to the employee, his supervisor, and the H&S manager. The terms of the restriction will be discussed with the employee and his supervisor. Every attempt will be made to keep the employee working, while not violating the terms of the medical restriction.

10.4 Medical Records

Medical and personal exposure monitoring records will be maintained according to the requirements of 29 CFR 1910.20 and HS103, and shall be kept for 30 years after employment. Employee confidentiality shall be maintained. Employees and their authorized representatives have access to these records through the H&S assistant.

11.0 Emergency Procedures

11.1 General

This SSHP has been developed to allow site activities to be conducted without adversely impacting the safety of the workers, the community, and the environment. Procedures included in this section address the action required in the event extraordinary conditions occur at the site.

11.2 First Aid

If first aid and/or CPR is administered by site personnel, the SSHO must ensure full compliance with the requirements of Chapter 12.0, Bloodborne Pathogen Exposure Control Plan. First-aid kits shall be maintained at the control access point between the decontamination and support zones and in all support vehicles. If the victim cannot be safely moved from the contaminated area, first aid necessary to stabilize the victim for safe transport shall be administered at the accident location. Appropriate decontamination of all clothing and equipment shall be followed upon leaving the contaminated area. The SSHO is responsible for administering first aid.

11.3 Emergency Response

11.3.1 Pre-Emergency Planning

During the site-specific orientation and at the daily tailgate safety meetings, all employees will be trained in and reminded of the provisions of this emergency response plan, the communication systems and evacuation routes. This plan will be reviewed and revised, if necessary, on a regular basis. This will ensure that the plan is adequate and consistent with prevailing site conditions.

11.3.2 Project Manager's Responsibilities

The PM has primary responsibility for responding to and correcting emergency situations. This includes taking appropriate measures to ensure the safety of site personnel and the public. Possible actions may involve evacuation of personnel from the site area. The PM is additionally responsible for ensuring that corrective measures have been implemented, appropriate authorities notified, and follow-up reports completed. In the absence of the PM, the site superintendent shall assume the responsibilities of the PM. All project personnel are responsible for assisting the PM in responding to emergency situations within the capabilities of their skills, equipment, and training.

11.3.3 Evacuation Signal

Vehicle or portable air horns will be used for evacuation signals as follows:

- One long, continuous blast: Emergency evacuation of the site.

11.3.4 Emergency Equipment/Facilities

All individuals shall be familiar with the site and be able to identify the locations of the following emergency equipment:

- "IT-Approved" first-aid kits
- Fire extinguishers (minimum of 20-pound A; B; C)
- Emergency eye wash/portable shower that complies with ANSI Z-358.1
- Cellular telephone
- PPE.

11.3.5 Medical Emergencies

First aid should be administered by the SSHO while awaiting an ambulance or paramedics. In the event of serious injury on site, all activities will be immediately suspended and resources will be diverted until it can be ensured that response procedures are adequate and that proper assistance has been provided to ambulance or paramedics responsible for treating the injury.

All injuries and illnesses must be immediately reported to the PM or the SSHO, who will then notify appropriate off-site personnel and organizations as necessary.

For ambulance, fire, or police contacts, give the name of the road and the nearest intersection to the Communication Center (Front Gate). Notify the IT PM, the USACE representative, and the IT H&S manager after primary emergency contacts have been made.

Attending emergency physicians should be given the telephone number of the IT medical director to obtain immediate access to an employee's medical records and for consultation purposes.

11.3.6 Emergency Contact/Notification Listing

In case of any emergency, the PBS Communication Center will be contacted for appropriate services. The PBS Communication Center will coordinate Station access and response.

Local, State, and National emergency numbers as follows:

Police Department	(419) 627-5863
Fire Department	(419) 627-5837
Ambulance	911
Hospital	(419) 621-7000
National Response Center	(800) 424-8802
Poison Control Center	(800) 542-4225
Ohio EPA Emergency Spill Number	(800) 282-9378

11.3.7 Key Project and IT Personnel

Project Manager	Don Burton, PE	(423) 690-3211
H&S Manager	Melissa Smith, CIH	(423) 690-3211
SSHO	George Yu	(423) 690-3211

11.3.8 Directions to the Nearest Hospital

The nearest hospital is located at:

Providence Hospital
1912 Hayes Avenue
Sandusky, Ohio 44870

Directions to Providence Hospital:

Exit from the front gate of Plum Brook Station; turn right to Taylor Road; go approximately 1/4 mile; turn left to Botay Road; at the stop sign, make a left turn to Bogart Road (County Road No. 10); take Bogart Road all the way to State Road No. 4; (Wright Brothers Memorial Highway); make a right turn at State Road No. 4; Providence Hospital will be on your right-hand side.

Estimated travel time to the hospital from PBS is between 15 and 30 minutes, depending on traffic condition.

See Appendix E, Site and Hospital Location Map.

11.4 General Emergency Procedures

- If a member of the field crew experiences any adverse effects or symptoms of exposure while on the scene, the entire field crew shall immediately halt work and act according to the instructions provided by the SSHO.

- The discovery of any condition that would suggest the existence of a situation more hazardous than anticipated shall result in the evacuation of the field team and re-evaluation of the hazard and the level of protection required.

- If an accident occurs, the site supervisor must immediately complete the SEIR (Appendix B). If necessary, follow-up action shall be taken to correct the situation that caused the accident.

11.4.1 Personal Injury

The SSHO is trained in American Red Cross first-aid procedures and shall administer appropriate first-aid treatment, including CPR, in emergency situations. The following general emergency procedures shall be carried out in the event of injury:

- Notify the SSHO of the incident.

- Administer first aid.

- Call for ambulance transport, or transport victim to nearest hospital or emergency medical center, as appropriate.

- Notify the IT PM, the USACE representative, and the IT H&S manager of the incident and describe the emergency response actions taken.

11.4.2 Fire or Explosion

In the event of a fire of explosion:

1. Immediately evacuate injured personnel and leave the area.

2. Administer first aid, as appropriate.

3. Notify emergency services through the PBS Communication Center.

4. Notify the IT PM, the USACE representative, and the IT H&S manager.

11.5 Evacuation Routes and Procedures

Evacuation routes and procedures will be developed by IT upon arrival at the site in coordination with the NASA PBS personnel.

12.0 Bloodborne Pathogen Exposure Control Plan

This exposure control plan presents H&S guidelines for voluntary first aid and CPR care providers. To meet the requirements of OSHA 29 CFR 1910.151 and EM 385-1-1, during day shift operations, at least one person on site will adequately trained in first aid and CPR, and in the requirements of the Bloodborne Pathogens Standard as listed in 29 CFR 1910.1030, IT Procedure HS512, and the contents of this plan.

12.1 Definition

Bloodborne pathogens are those agents (i.e., bacteria, virus, fungi) found in blood components, certain body fluids, and other materials, objects or surfaces that have had contact with blood that are capable of causing human disease or death to unprotected people who came into contact with blood or blood-affected items. Diseases caused by bloodborne pathogens include, but are not limited to, hepatitis B virus (HBV), human immunodeficiency virus (HIV), hepatitis C, malaria, and syphilis. The most significant and of greatest concern are HBV and HIV.

12.1.1 Hepatitis B Virus

HBV is the major bloodborne pathogen hazard that first aid/CPR care providers are most likely to encounter. The HBV can remain infectious for up to 10 days even in dried blood. The virus adversely affects 8,000 to 10,000 workers annually, resulting in approximately 200 deaths each year.

Hepatitis Exposure Symptoms. Hepatitis means "inflammation of the liver" causing severe liver damage or cirrhosis. Exposure symptoms include fever, fatigue, nausea, vomiting, muscle aches, loss of appetite, and jaundice (yellowing of the eyes or skin). Hepatitis diagnosis is difficult because some symptoms are similar to the flu and may remain mild for an extended period of time.

Presently, no cure exists for hepatitis, but it can be prevented with a vaccination.

12.1.2 Human Immunodeficiency Virus

HIV attacks and deteriorates the body's immune system and eventually weakens it to the point that infection sets in causing the disease Acquired Immune Deficiency Syndrome (AIDS). HIV is primarily transmitted through sexual contact, but may also be transmitted

through contact with blood and body fluids. HIV is not transmitted by touching or working with people who are HIV-positive.

Human Immunodeficiency Virus Exposure Symptoms. HIV leads to AIDS-related illnesses that eventually cause neurological problems, cancer, pneumonia, and death. People can carry the virus for many years without experiencing any symptoms. Upon development, symptoms may include weight loss, skin lesions, dry cough, fever, fatigue, diarrhea, or swelling of the lymph glands.

Presently, no cure exists for HIV or AIDS, and no vaccination is currently available.

12.2 Exposure Determination

The purpose of the guidelines in this plan are designed to limit occupational exposure of site workers to infectious blood materials that could result in disease or possibly death. The contents of this plan are intended to protect the IT employees trained in first aid and CPR who are responsible for administering medical assistance to site workers.

Means of Transmission. The major activity that may expose any of these IT employees to bloodborne pathogens is their response and care to on-site personal injuries or decontamination of equipment/surfaces contaminated by blood or other potentially infectious materials during the incident.

These IT employees could be subject to bloodborne pathogens during rendering of first aid or CPR by accidental exposure due to:

- Punctures through the skin with a contaminated sharp object (i.e., scissors)
- Contact or absorption of blood or blood-contaminated objects through open or broken skin (i.e., cuts, scratches, rashes)
- Blood splashes to their eyes, nose, or mouth, or other mucous membranes.

Workers can reduce their risk of contacting HBV or HIV by implementing the recommended work practices (outlined in this plan) before, during, and after responding to emergency medical incidents involving personal injuries.

12.3 Measures for Prevention

The establishment of work practice controls is an integral part of an effective exposure control plan in preventing accidental infection of employees. These work practices are designed to protect employees from reasonably foreseeable occupational exposures to bloodborne pathogens from blood and other potentially infectious material. The work practice controls outlined in this section are applicable to the administration of first aid in emergency situations and subsequent cleanup only.

12.3.1 Universal Precautions

Universal precautions is an approach to infection control that operates on the assumption that all human blood and bodily fluids are to be treated as if they are known to be contaminated with HIV, HBV, or other infectious diseases. Universal precautions shall be implemented whenever there exists a foreseeable potential for contact with blood or bodily fluids.

12.3.2 Engineering Controls

Due to the remote location of the work site, the nature of work in outdoor locations with potential exposure to airborne chemical contaminants, and the potential for exposure being limited to emergency situations, the implementation of engineering controls is not feasible. Exposure control shall be accomplished through implementation of work practice controls and use of PPE.

12.3.3 Work Practice Controls

Work practice controls shall be instituted whenever foreseeable potential contact with, or exposure to, blood and bodily fluid exists. Examples of situations in which these controls are to be implemented include, but are not limited to, accidents or injuries in which administration of first aid is required, application of bandages to minor cuts and abrasions of another person, and contact with sores, wounds, or broken skin.

Following are specific work practice controls that shall be implemented:

- Open wounds or cuts will be promptly bandaged.
- Hands and face will be washed as soon as possible after administering first aid or CPR. If wash facilities are not readily available, stock disposable one-time use towelettes.
- No eating, drinking, or smoking is allowed in any work area where a potential exists for occupational exposure to bloodborne pathogens.

- Nondisposable equipment or materials that have or may have blood or infectious fluid contact must be washed immediately after their use. (A 1-to-10 solution of bleach and water is recommended for proper decontamination.)

- Any clothing that comes into contact with blood or infectious fluids shall be removed as soon as possible after administering first aid or CPR.

- No personal clothing that comes into contact with blood or infectious fluids shall be laundered off site.

- First-aid kits on site will be equipped with a pair of surgical gloves and CPR mouth pieces.

Minimization of Contact. Direct contact with blood and bodily fluids should be kept to an absolute minimum, as required in a particular situation. In situations where direct contact is likely, PPE shall be worn to help prevent infection.

Based upon professional judgment, an employee may choose to temporarily forego the use of PPE if he determines that the use of the PPE will further jeopardize his well-being or that of the injured worker. This limited application must be carefully evaluated by the employee.

If this situation does occur, IT is obligated to investigate and document the circumstances in an effort to provide alternative means to avoid further occurrence.

12.3.4 Personal Protective Equipment

The following are specific PPE items that shall be implemented:

- Always use CPR mouthpieces or ventilation devices.

- Inspect PPE prior to use to ensure it is in good working order and without flaws.

- Do not reuse gloves once removed.

- After use, remove gloves from top to bottom inside-out, not allowing unprotected skin to contact the exterior of the gloves.

12.3.5 Waste Handling

Disposable items that have or may have blood contact must be bagged separately from other trash. These wastes must be placed in leak-proof containers or bags and labeled.

A collection container for contaminated articles will be available on site. Wastes used in medical emergency treatment (i.e., gloves, towels, gauze) shall be disposed in the infectious waste container(s). The container will be replaced as needed and will not be overfilled.

12.3.6 Waste Disposal

The waste will remain on site in approved container(s) until an approved disposal facility capable of receiving medical wastes is identified. Disposal of the infectious waste container(s) shall be in accordance with applicable local, state, and federal regulations.

12.4 Medical Requirements

The medical requirements of the exposure control plan include provision of a hepatitis B vaccination to all exposed employees, and postexposure procedures and evaluation.

12.4.1 Hepatitis B Vaccination

All potentially exposed employees will have made available to them at no cost a hepatitis B vaccination. The employee will also receive training as to the vaccine's efficacy, safety, benefits, and consequences prior to administration. The vaccination series shall be initiated within 24 hours of providing first aid/CPR in an incident and shall be administered under the supervision of a licensed physician. Employees may at their own discretion decline the vaccination, in which case documentation of declination will be completed and employees may be assigned immediately. If an employee covered by this exposure plan decides to accept the vaccination at a later date, the vaccination will be offered at that time at no cost to the employee.

12.4.2 Postexposure Procedures and Evaluation

Subsequent to all reported exposure incidents, a confidential medical evaluation and follow-up shall be made available to each employee exposed in the incidents.

Documentation Procedures. Documentation of the exposure incident shall be recorded as soon as possible, and shall include the route(s) of exposure, the circumstances surrounding the incident, and the identification of the source individual. Additionally, each incident shall be placed on the "first aid incident list" attached to the location OSHA Log of Occupational Injuries and Illnesses.

Blood Testing

Source Individuals. As soon as feasible, the source individual in an exposure incident will be asked to consent to a blood test to determine HBV and HIV infectivity. Where applicable laws require employee consent, documented consent shall be obtained prior to testing. If an employee refuses the blood test, documentation of the refusal will be made. Documentation of the test results shall be made available to the exposed employee(s). All results should be kept confidential; criminal and civil penalties may be charged against persons negligently or willfully releasing such information, depending on local laws.

Exposed Employees. Exposed employees will be asked to consent to a blood test for HBV and HIV serological status. If consent to HIV testing is denied, the blood sample will be preserved for 90 days; within this time, the employee may elect to consent to the HIV test.

12.4.3 Postexposure Medical Evaluations

Exposed employees shall receive a health care professional's written opinion for postexposure evaluations. The written opinion shall include the results of the evaluation and any medical conditions resulting from the exposure incident that require further medical treatment.

12.5 Hazard Communication

12.5.1 Warning Labels

Containers used for disposal of blood contaminated supplies and waste will be labeled in accordance with the word "biohazard."

12.5.2 Warning Signs

There are no designated areas for medical treatment on site, because first aid will be provided on an emergency basis only; therefore, warning signs are not applicable. In cases of potential exposure, observers and nonessential personnel should be verbally warned to keep a safe distance from injured personnel.

12.5.3 Employee Training Program

All associates who are first aid/CPR trained and may provide assistance shall be trained in the requirements for voluntary providers as described in HS512, this SSHP and its addenda, and the general provisions of HS512.

12.6 Record Keeping

12.6.1 Training Records

All employees selected to attend the training program that covers the contents of this plan shall sign the Acknowledgment Form and the Training Attendance Form.

The training record will contain the date, training outline, name and qualifications of the trainer, and names and job titles of attendees.

At the completion of the training program, all participants must take and pass the training quiz.

The training records will be maintained by the IT Training Department for at least 3 years from the training date.

12.6.2 Medical Records

Medical records necessary for IT employees must include documentation of HBV vaccination status, medical follow-up, postexposure testing, and a medical professional's written evaluation.

Confidentiality. The employee medical records will be forwarded to Washington Occupational Health Associates Consulting Services for inclusions in the employee's medical file.

Maintenance and Transfer of Records. IT shall maintain the employee medical records for the duration of the employee's employment plus 30 years thereafter.

If, for whatever reason, IT no longer does business and no successor exists, IT will notify the director of NIOSH in writing 3 months prior to the disposal of records. If so directed, the records shall be transferred to the director of NIOSH.

12.6.3 Incident Recording

An incident that occurs as a result of rendering emergency medical care will be recorded on the OSHA 200 log as OSHA defines work-related injuries and illnesses. All injuries involving the release of blood or bodily fluids must be immediately reported to the H&S department to ensure proper reporting and followup.

APPENDIX A

SUBCONTRACTOR CERTIFICATION

SUBCONTRACTOR CERTIFICATION

I, _____ as an agent of _____, do hereby certify that the following employees have successfully completed a 40-hour training course that complies with the provisions of 29 CFR 1910.120/29 CFR 1926.65, and respiratory protection training that complies with 29 CFR 1910.134. Each employee has successfully completed a medical examination that complies with these regulations.

Individual copies of certification of successful completion of the required training and medical examinations are attached for each employee.

_____ _____

Signature Date

APPENDIX B

MATERIAL SAFETY DATA SHEETS

lallinckrodt
laterial Safety Data

Mallinckrodt provides the information contained herein in good faith but makes no representation as to its comprehensiveness or accuracy. Individuals receiving this information must exercise their independent judgment in determining its appropriateness for a particular purpose.

Mallinckrodt makes no representation, or warranties, either express or implied, of merchantability, fitness for a particular purpose with respect to the information set forth herein or to the product to which the information refers. Accordingly, Mallinckrodt will not be responsible for damages resulting from use of or reliance upon this information.

ergency Phone Number: 314-539-1600

Mallinckrodt, Inc., Science Products Division, P.O. Box 800, Paris, KY 40361.

XANES

DUCT IDENTIFICATION:

m: n-Hexane

CAS No.: 110-54-3

r Weight: 86.18

Formula: $CH_3(CH_2)_4CH_3$

c Ingredients: n-Hexane
3-Methylpentane

AUTIONARY MEASURES

EXTREMELY FLAMMABLE. HARMFUL IF
RD OR INHALED.
ENTRAL NERVOUS SYSTEM. MAY CAUSE
N.

from heat, sparks and flame.
iner closed.
lequate ventilation.
thing vapor.
act with eyes, skin and clothing.
oughly after handling.

GENCY/FIRST AID

hazard.
!, DO NOT INDUCE VOMITING!
uantities of water or milk if available. Never give
mouth to an unconscious person. If inhaled, remove to
not breathing, give artificial respiration. If
difficult, give oxygen. In case of contact,
flush skin or eyes with plenty of water for at least 15
all cases call a physician.
ON 5.

d Class: Flammable Liquid

SECTION 1 Physical Data

Appearance: Clear, colorless solution.

Odor: Odorless.

Solubility: Insoluble in water.

Boiling Point: ca. 68°C (154°F).

Melting Point: ca. -95°C (-139°F)

Specific Gravity: ca. 0.7

Vapor Density (Air = 1): 3.0

Vapor Pressure (mm Hg): 124 @ 20°C (68°F)

Evaporation Rate: No information found.

SECTION 2 Fire and Explosion Information

Fire:
Extremely Flammable. Dangerous fire hazard when exposed to heat or flame.
Flashpoint: -22 to -26°C (-7 to -15°F).
Autoignition temperature: 240-260°C (464-500°F).
Flammable limits in air, % by volume:
lel: 1.1; uel: 7.5.

Explosion:
Above flash point, vapor-air mixtures are explosive within flammable limits noted above. Contact with oxidizing materials may cause extremely violent combustion.

Fire Extinguishing Media:
Dry chemical, foam or carbon dioxide. Water spray may be used to keep fire exposed containers cool.

Special Information:
In the event of a fire, wear full protective clothing and NIOSH-approved self-contained breathing apparatus with full faceplece operated in the pressure demand or other positive pressure mode. Water spray may be used to keep fire exposed containers cool. Use chemical safety goggles. Contact lenses should not be worn when working with this material. Vapors can flow along surfaces to distant ignition source and flash back.

SECTION 3 Reactivity Data

Stability:
Stable under ordinary conditions of use and storage. Heat will contribute to instability.

Hazardous Decomposition Products:
Toxic gases and vapors may be released if involved in a fire.

Hazardous Polymerization:
This substance does not polymerize.

Incompatibilities:
Strong oxidizers.

SECTION 4 Leak/Spill Disposal Information

Ventilate area of leak or spill. Remove all sources of ignition. Clean-up personnel require protective clothing and respiratory protection from vapors. Contain and recover liquid when possible. Use non-sparking tools and equipment. Collect as hazardous waste and atomize in a suitable RCRA approved combustion chamber, or absorb with vermiculite, dry sand, earth or similar material for disposal as hazardous waste in a RCRA approved facility. Do not flush to sewer!

Ensure compliance with local, state and federal regulations.

NFPA Ratings: Health: 1 Flammability: 3 Reactivity: 0

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ITION 5 Health Hazard Information

EXPOSURE / HEALTH EFFECTS

lation:
'rritant to the respiratory tract. Overexposure may cause cadedness, nausea, and blurred vision. Greater exposure may unconsciousness and death.

tion:
use abdominal pain, nausea. Other symptoms parallel tion. Aspiration into the lungs can produce severe lung c.

Contact:
use redness, irritation.

'ontact:
may cause irritation. Splashes may cause redness and pain.

-lc Exposure:
c inhalation may cause peripheral nerve disorders.

ration of Pre-existing Conditions:
with pre-existing skin disorders or eye problems or d respiratory function may be more susceptible to the of the substance.

B. FIRST AID

Inhalation:
Remove to fresh air. If not breathing, give artificial respiration. If breathing is difficult, give oxygen. Call a physician.

Ingestion:
Aspiration hazard.
If swallowed, DO NOT induce vomiting. Give large quantities of water or milk if available. Call a physician immediately. Never give anything by mouth to an unconscious person.

Skin Exposure:
Remove any contaminated clothing. Wipe off excess from skin. Wash skin with soap and water for at least 15 minutes. Get medical attention if irritation develops or persists

Eye Exposure:
Wash eyes with plenty of water for at least 15 minutes. If irritation develops, get medical attention.

C. TOXICITY DATA (RTECS, 1991)

Hexane: Oral rat LD50: 28710 mg/kg. Irritation eye rabbit 10 mg mild Investigated as a tuporigen, mutagen and reproductive effector.

SECTION 6 Occupational Control Measures

Airborne Exposure Limits:
n-Hexane [110-54-3]:
-OSHA Permissible Exposure Limit (PEL): 50 ppm (TWA)

-ACGIH Threshold Limit Value (TLV): 50 ppm (TWA)

Ventilation System:
A system of local and/or general exhaust is recommended to keep employee exposures below the Airborne Exposure Limits. Local exhaust ventilation is generally preferred because it can control the emissions of the contaminant at its source, preventing dispersion of it into the general work area. Please refer to the ACGIH document, "Industrial Ventilation, A Manual of Recommended Practices", most recent edition, for details.

Personal Respirators: (NIOSH Approved)
If the TLV is exceeded a full facepiece chemical cartridge respirator may be worn, in general, up to 100 times the TLV or the maximum use concentration specified by the respirator supplier, whichever is less. Alternatively, a supplied air full facepiece respirator or airlined hood may be worn.

Skin Protection:
Gloves and lab coat, apron or coveralls.

Eye Protection:
Use chemical safety goggles and/or a full face shield where splashing is possible. Contact lenses should not be worn when working with this material. Maintain eye wash fountain and quick-drench facilities in work area.

SECTION 7 Storage and Special Information

Protect against physical damage. Store in a cool, dry well-ventilated location, away from direct sunlight and any area where the fire hazard may be acute. Store in tightly closed containers (preferably under nitrogen atmosphere).Outside or detached storage is preferred. Inside storage should be in a standard flammable liquids storage room or cabinet. Separate from oxidizing materials. Containers should be bonded and grounded for transfers to avoid static sparks. Storage and use areas should be No Smoking areas. Use non-sparking type tools and equipment.

I-CHEM

MATERIAL SAFETY DATA SHEET

CORPORATE OFFICE
23787-F Eichler Street
Hayward, CA 94545-2744
(510) 782-3905
Fax: (510) 782-3219

2 Boulden Circle, Suite 8
New Castle, DE 19720-2064
(302) 322-3808

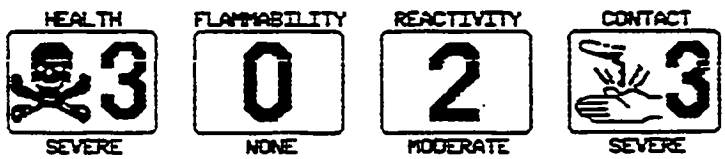
J.T.BAKER INC., 222 Red School Lane, Phillipsburg, NJ 08865

SECTION I - PRODUCT IDENTIFICATION

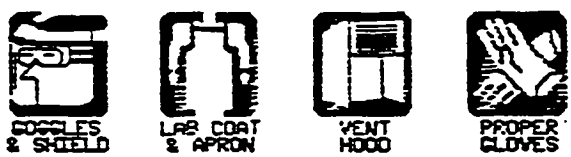
Product Name: Hydrochloric Acid
Common Synonyms: Muriatic Acid; Chlorohydric Acid; Hydrogen Chloride, Aqueous
Chemical Family: Inorganic Acids
Formula: HCl
Formula Wt.: 36.46
CAS No.: 7647-01-0
NIOSH/RTECS No.: MW4025000
Product Use: Laboratory Reagent
Product Codes: 9538,9535,4800,9542,9534,9549,9529,9547,9546,6900,9536,9540 9539,9548,5367,9544,5800,5214,5575,9543,9530,9537,5537

PRECAUTIONARY LABELING

BAKER SAF-T-DATA* System



Laboratory Protective Equipment



U.S. Precautionary Labeling

POISON! DANGER!
CAUSES SEVERE BURNS. MAY BE FATAL IF SWALLOWED OR INHALED.
Do not get in eyes, on skin, on clothing. Do not breathe vapor. Causes damag to Respiratory system (lungs), eyes and skin. Keep in tightly closed container. Loosen closure cautiously. Use with adequate ventilation. Wash thoroughly after handling. In case of spill neutralize with soda ash or lime and place in dry container.

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Continued on Page: 2

SECTION IV - FIRE AND EXPLOSION HAZARD DATA

Flash Point (Closed Cup): N/A NFPA 704M Rating: 3-0-0

Autoignition Temperature: N/A

Flammable Limits: Upper - N/A Lower - N/A

Fire Extinguishing Media
Use extinguishing media appropriate for surrounding fire.

Special Fire-Fighting Procedures
Firefighters should wear proper protective equipment and self-contained breathing apparatus with full facepiece operated in positive pressure mode. Move containers from fire area if it can be done without risk. Us water to keep fire-exposed containers cool. Do not get water inside containers.

Unusual Fire & Explosion Hazards
May emit hydrogen gas upon contact with metal.

Toxic Gases Produced
hydrogen chloride, hydrogen

Explosion Data-Sensitivity to Mechanical Impact
None identified.

Explosion Data-Sensitivity to Static Discharge
None identified.

SECTION V - HEALTH HAZARD DATA

Threshold Limit Value (TLV/TWA): 7 mg/m³ (5 ppm)

TLV (Ceiling) is for Hydrogen chloride.

Short-Term Exposure Limit (STEL): Not Established

Permissible Exposure Limit (PEL): 7 mg/m³ (5 ppm)

PEL (Ceiling) is for Hydrogen chloride.

Toxicity of components

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SECTION V - HEALTH HAZARD DATA (CONTINUED)

Emergency and First Aid Procedures

INGESTION: CALL A PHYSICIAN. If swallowed, do NOT induce vomiting. I conscious, give water, milk, or milk of magnesia.

INHALATION: If inhaled, remove to fresh air. If not breathing, give artificial respiration. If breathing is difficult, give oxygen.

SKIN CONTACT: In case of contact, immediately flush skin with plenty of water for at least 15 minutes while removing contaminated clothing and shoes. Wash clothing before re-use.

EYE CONTACT: In case of eye contact, immediately flush with plenty of water for at least 15 minutes.

SARA/TITLE III HAZARD CATEGORIES and LISTS

Acute: Yes Chronic: Yes Flammability: No Pressure: No Reactivity: No

Extremely Hazardous Substance:	Yes	Contains Hydrogen Chloride (RQ = 1 LB, T = 500 LBS)
CERCLA Hazardous Substance:	Yes	Contains Hydrochloric Acid (RQ = 5000 LB
SARA 313 Toxic Chemicals:	Yes	Contains Hydrochloric Acid
Generic Class:		C16
TSCA Inventory:	Yes	

SECTION VI - REACTIVITY DATA

Stability: Stable Hazardous Polymerization: Will not occur

Conditions to Avoid: heat, moisture

Incompatibles: most common metals, water, amines, metal oxides, acetic anhydride, propiolactone, vinyl acetate, mercuric sulfate, calcium phosphide, formaldehyde, alkalies, carbonates, strong bases, sulfuric acid, chlorosulfonic acid

Decomposition Products: hydrogen chloride, hydrogen, chlorine

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SECTION X - TRANSPORTATION DATA AND ADDITIONAL INFORMATION

Domestic (D.O.T.)

Proper Shipping Name: Hydrochloric acid, solution
Hazard Class: 8
UN/NA: UN1789 Reportable Quantity: 5000 LBS. Packaging Group: II
Labels: CORROSIVE
Regulatory References: 49CFR 172.101

International (I.M.O.)

Proper Shipping Name: Hydrochloric acid, solution
Hazard Class: 8 I.M.O. Page: 8183
UN: UN1789 Marine Pollutants: No Packaging Group: II
Labels: CORROSIVE
Regulatory References: 49CFR 172.102; Part 176; IMO

AIR (I.C.A.O.)

Proper Shipping Name: Hydrochloric acid, solution
Hazard Class: 8
UN: UN1789 Packaging Group: II
Labels: CORROSIVE
Regulatory References: 49CFR 172.101; 173.6; Part 175; ICAO/IATA=== We belie the transportation data and references contained here to be factual and the opinion of qualified experts. T data is meant as a guide to the overall classificatio of the product and is not package size specific, nor should it be taken as a warranty or representation fo which the company assumes legal responsibility.=== Th information is offered solely for your consideration, investigation, and verification. Any use of the information must be determined by the user to be in accordance with applicable Federal, State, and Local laws and regulations. See shipper requirements 49CFR 172.3 and employee training 49CFR 173.1.

U.S. Customs Harmonization Number: 28061000000

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J.T.BAKER INC. 222 RED SCHOOL LANE, PHILLIPSBURG, NJ 08865
M A T E R I A L S A F E T Y D A T A S H E E T
24-HOUR EMERGENCY TELEPHONE -- (908) 859-2151
CHEMTREC # (800) 424-9300 -- NATIONAL RESPONSE CENTER # (800) 424-8802

J.T.BAKER INC., 222 RED SCHOOL LANE, PHILLIPSBURG, NJ 08865

SECTION I - PRODUCT IDENTIFICATION

PRODUCT NAME: METHANOL
COMMON SYNONYMS: METHYL ALCOHOL; WOOD ALCOHOL; CARBINOL; METHYLOL; WOOD SPIRIT
CHEMICAL FAMILY: ALCOHOLS
FORMULA: CH3OH
FORMULA WT.: 32.04
CAS NO.: 67-56-1
NIOSH/RTECS NO.: PC1400000
PRODUCT USE: LABORATORY REAGENT
PRODUCT CODES: 9049,9063,9076,9091,5370,9074,6808,9127,9098,5807,9073,9071 5811,5217,9075,9090,9093,P704,9069,5536,9263,9070,9077,9072 9068

PRECAUTIONARY LABELING

BAKER SAF-T-DATA* SYSTEM

HEALTH	-	3	SEVERE (POISON)
FLAMMABILITY	-	3	SEVERE (FLAMMABLE)
REACTIVITY	-	1	SLIGHT
CONTACT	-	1	SLIGHT

LABORATORY PROTECTIVE EQUIPMENT

GOGGLES & SHIELD; LAB COAT & APRON; VENT HOOD; PROPER GLOVES; CLASS B EXTINGUISHER

U.S. PRECAUTIONARY LABELING

POISON DANGER
FLAMMABLE. HARMFUL IF INHALED. CANNOT BE MADE NON-POISONOUS. MAY BE FATAL OR CAUSE BLINDNESS IF SWALLOWED.
KEEP AWAY FROM HEAT, SPARKS, FLAME. DO NOT GET IN EYES, ON SKIN, ON CLOTHING. AVOID BREATHING VAPOR. KEEP IN TIGHTLY CLOSED CONTAINER. USE WITH ADEQUATE VENTILATION. WASH THOROUGHLY AFTER HANDLING. IN CASE OF FIRE, USE ALCOHOL FOAM, DRY CHEMICAL, CARBON DIOXIDE - WATER MAY BE INEFFECTIVE. FLUSH SPILL AREA WITH WATER SPRAY.

CONTINUED ON PAGE: 2

SECTION IV - FIRE AND EXPLOSION HAZARD DATA

FLASH POINT (CLOSED CUP): 12 C (54 F) NFPA 704M RATING: 1-3-0

AUTOIGNITION TEMPERATURE: 463 C (867 F)

FLAMMABLE LIMITS: UPPER - 36.0 % LOWER - 6.0 %

FIRE EXTINGUISHING MEDIA
USE ALCOHOL FOAM, DRY CHEMICAL OR CARBON DIOXIDE. (WATER MAY BE INEFFECTIVE.)

SPECIAL FIRE-FIGHTING PROCEDURES
FIREFIGHTERS SHOULD WEAR PROPER PROTECTIVE EQUIPMENT AND SELF-CONTAINED BREATHING APPARATUS WITH FULL FACEPIECE OPERATED IN POSITIVE PRESSURE MODE. MOVE CONTAINERS FROM FIRE AREA IF IT CAN BE DONE WITHOUT RISK. USE WATER TO KEEP FIRE-EXPOSED CONTAINERS COOL.

UNUSUAL FIRE & EXPLOSION HAZARDS
VAPORS MAY FLOW ALONG SURFACES TO DISTANT IGNITION SOURCES AND FLASH BACK. CLOSED CONTAINERS EXPOSED TO HEAT MAY EXPLODE. CONTACT WITH STRONG OXIDIZERS MAY CAUSE FIRE. BURNS WITH A CLEAR, ALMOST INVISIBLE FLAME.

TOXIC GASES PRODUCED
CARBON MONOXIDE, CARBON DIOXIDE, FORMALDEHYDE

EXPLOSION DATA-SENSITIVITY TO MECHANICAL IMPACT
NONE IDENTIFIED.

EXPLOSION DATA-SENSITIVITY TO STATIC DISCHARGE
NONE IDENTIFIED.

SECTION V - HEALTH HAZARD DATA

THRESHOLD LIMIT VALUE (TLV/TWA): 260 MG/M3 (200 PPM)

THE TLV LISTED DENOTES TLV (SKIN).

SHORT-TERM EXPOSURE LIMIT (STEL): 310 MG/M3 (250 PPM)

PERMISSIBLE EXPOSURE LIMIT (PEL): 260 MG/M3 (200 PPM)

CONTINUED ON PAGE: 4

SECTION V - HEALTH HAZARD DATA (CONTINUED)

EMERGENCY AND FIRST AID PROCEDURES

INGESTION: CALL A PHYSICIAN. IF SWALLOWED, IF CONSCIOUS, GIVE LARGE AMOUNTS OF WATER. INDUCE VOMITING.

INHALATION: IF INHALED, REMOVE TO FRESH AIR. IF NOT BREATHING, GIVE ARTIFICIAL RESPIRATION. IF BREATHING IS DIFFICULT, GIVE OXYGEN.

SKIN CONTACT: IN CASE OF CONTACT, IMMEDIATELY FLUSH SKIN WITH PLENTY OF WATER FOR AT LEAST 15 MINUTES WHILE REMOVING CONTAMINATED CLOTHING AND SHOES. WASH CLOTHING BEFORE RE-USE.

EYE CONTACT: IN CASE OF EYE CONTACT, IMMEDIATELY FLUSH WITH PLENTY OF WATER FOR AT LEAST 15 MINUTES.

SARA/TITLE III HAZARD CATEGORIES AND LISTS

ACUTE: YES CHRONIC: YES FLAMMABILITY: YES PRESSURE: NO REACTIVITY: NO

EXTREMELY HAZARDOUS SUBSTANCE:	NO	
CERCLA HAZARDOUS SUBSTANCE:	YES	CONTAINS METHANOL (RQ = 5000 LBS)
SARA 313 TOXIC CHEMICALS:	YES	CONTAINS METHANOL
GENERIC CLASS:		C05
TSCA INVENTORY:	YES	

SECTION VI - REACTIVITY DATA

STABILITY: STABLE HAZARDOUS POLYMERIZATION: WILL NOT OCCUR

CONDITIONS TO AVOID: HEAT, FLAME, OTHER SOURCES OF IGNITION

INCOMPATIBLES: STRONG OXIDIZING AGENTS, STRONG ACIDS, ZINC, ALUMINUM, MAGNESIUM

DECOMPOSITION PRODUCTS: CARBON MONOXIDE, CARBON DIOXIDE, FORMALDEHYDE

CONTINUED ON PAGE: 6

SECTION X - TRANSPORTATION DATA AND ADDITIONAL INFORMATION

DOMESTIC (D.O.T.)

PROPER SHIPPING NAME: METHYL ALCOHOL
HAZARD CLASS: FLAMMABLE LIQUID
UN/NA: UN1230 REPORTABLE QUANTITY: 5000 LBS.
LABELS: FLAMMABLE LIQUID
REGULATORY REFERENCES: 49CFR 172.101; 173.119

INTERNATIONAL (I.M.O.)

PROPER SHIPPING NAME: METHANOL
HAZARD CLASS: 3.2, 6.1 I.M.O. PAGE: 3251
UN: UN1230 MARINE POLLUTANTS: NO PACKAGING GROUP: II
LABELS: FLAMMABLE LIQUID, POISON
REGULATORY REFERENCES: 49CFR 172.102; PART 176; IMO

AIR (I.C.A.O.)

PROPER SHIPPING NAME: METHANOL
HAZARD CLASS: 3.2, 6.1
UN: UN1230 PACKAGING GROUP: II
LABELS: FLAMMABLE LIQUID, POISON
REGULATORY REFERENCES: 49CFR 172.101; 173.6; PART 175; ICAO/IATA=== WE BELIEVE THE TRANSPORTATION DATA AND REFERENCES CONTAINED HEREIN TO BE FACTUAL AND THE OPINION OF QUALIFIED EXPERTS. THE DATA IS MEANT AS A GUIDE TO THE OVERALL CLASSIFICATION OF THE PRODUCT AND IS NOT PACKAGE SIZE SPECIFIC, NOR SHOULD IT BE TAKEN AS A WARRANTY OR REPRESENTATION FOR WHICH THE COMPANY ASSUMES LEGAL RESPONSIBILITY.=== THE INFORMATION IS OFFERED SOLELY FOR YOUR CONSIDERATION, INVESTIGATION, AND VERIFICATION. ANY USE OF THE INFORMATION MUST BE DETERMINED BY THE USER TO BE IN ACCORDANCE WITH APPLICABLE FEDERAL, STATE, AND LOCAL LAWS AND REGULATIONS. SEE SHIPPER REQUIREMENTS 49CFR 172.3 AND EMPLOYEE TRAINING 49CFR 173.1.

U.S. CUSTOMS HARMONIZATION NUMBER: 29051100009

CONTINUED ON PAGE: 8

MALLINCKRODT

Material Safety Data Sheet

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Mallinckrodt, Inc. Science Products Division, P.O. Box M Paris, KY 40361

Emergency Telephone Number: 314-982-5001

NITRIC ACID, 70%

PRODUCT IDENTIFICATION:

Synonym: Aqua Fortis; Azotic Acid; Nitric Acid 70%

Formula CAS No.: 7697-37-2

Molecular Weight: 63.00

Chemical Formula: HNO_3

Hazardous Ingredients: Nitric acid

PRECAUTIONARY MEASURES

DANGER! STRONG OXIDIZER. CONTACT WITH OTHER MATERIAL MAY CAUSE FIRE.
CORROSIVE. LIQUID AND MIST CAUSE SEVERE BURNS TO ALL BODY TISSUE. MAY BE FATAL IF SWALLOWED. HARMFUL IF INHALED. INHALATION MAY CAUSE LUNG DAMAGE.

Do not get in eyes, on skin, or on clothing.
Avoid breathing mist.
Use only with adequate ventilation.
Wash thoroughly after handling.
Keep from contact with clothing and other combustible materials.
Do not store near combustible materials.
Store in a tightly closed container.
Remove and wash contaminated clothing promptly.
This substance is classified as a POISON under the Federal Caustic Poison Act.

EMERGENCY/FIRST AID

In case of contact, immediately flush skin or eyes with plenty of water for at least 15 minutes. If swallowed, DO NOT INDUCE VOMITING!
Give large quantities of water or milk if available. Never give anything by mouth to an unconscious person. If inhaled, remove to fresh air. If not breathing, give artificial respiration. If breathing is difficult, give oxygen. In all cases call a physician.
SEE SECTION 5.

DOT Hazard Class: Oxidizer

SECTION 1 Physical Data

Appearance: Clear, colorless to slightly yellow liquid.

Odor: Suffocating acrid.

Solubility: Infinite in water.

Boiling Point: 122°C (252°F)

Melting Point: -34°C (-29°F)

Specific Gravity: 1.41

Vapor Density (Air=1): 2-3 approximately

Vapor Pressure (mm Hg): 62 @ 20°C (68°F)

Evaporation Rate: No information found.

SECTION 2 Fire and Explosion Information

Fire:
Not combustible, but substance is a strong oxidizer and its heat of reaction with reducing agents or combustibles may cause ignition. Can react with metals to release flammable hydrogen gas.

Explosion:
Reacts explosively with combustible organic or readily oxidizable materials such as: alcohols, turpentine, charcoal, organic refuse, metal powder, hydrogen sulfide, etc.

Fire Extinguishing Media:
If involved in a fire, use water spray.

Special Information:
Increases the flammability of combustible, organic and readily oxidizable materials. In the event of a fire, wear full protective clothing and NIOSH-approved self-contained breathing apparatus with full facepiece operated in the pressure demand or other positive pressure mode.

SECTION 3 Reactivity Data

Stability:
Stable under ordinary conditions of use and storage. Container may burst when heated.

Hazardous Decomposition Products:
When heated to decomposition, emits toxic nitrogen oxides fumes and hydrogen nitrate. Will react with water or steam to produce heat and toxic and corrosive fumes.

Hazardous Polymerization:
Will not occur.

Incompatibilities:
A dangerously powerful oxidizing agent, concentrated nitric acid is incompatible with most substances, especially strong bases, metallic powders, carbides, hydrogen sulfide, turpentine, and combustible organics.

SECTION 4 Leak/Spill Disposal Information

Isolate or enclose the area of the leak or spill. Clean-up personnel should wear protective clothing and respiratory equipment suitable for toxic or corrosive fluids or vapors.
Small Spills: Flush with water and neutralize with alkaline material (soda ash, lime, etc.). Sewer with excess water
Larger spills and lot sizes: Neutralize with alkaline material, pick up with absorbent material (sand, earth, vermiculite) and dispose in a RCRA-approved waste facility or sewer the neutralized slurry with excess water if local ordinances allow. Provide forced ventilation to dissipate fumes.

Reportable Quantity (RQ)(CWA/CERCLA): 1000 lbs.

Ensure compliance with local, state and federal regulations.

NFPA Ratings: Health: 3 Flammability: 0 Reactivity: 0 Other: Oxidizer

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Mallinckrodt, Inc. Science Products Division, P.O. Box M Paris, KY 40361

Emergency Telephone Number: 314-982-5000

Addendum to Material Safety Data Sheet

REGULATORY STATUS

This Addendum Must Not Be Detached from the MSDS

Identifies SARA 313 substance(s)

Any copying or redistribution of the MSDS must include a copy of this addendum

(Chem.Key: NITRA)

Hazard Categories for SARA Section 311/312 Reporting

Acute	Chronic	Fire	Pressure	Reactive
X	X			X

Product or Components of Product:	SARA EHS Sect. 302 RQ (lbs.)	SARA EHS Sect. 302 TPQ (lbs.)	SARA Section 313 Chemicals Name List	SARA Section 313 Chemicals Chemical Category	CERCLA Sec.103 RQ (lbs.)	RCRA Sec. 261.33
NITRIC ACID, 70% (7697-37-2)	1000	1,000	Yes	No	1000	No

SARA Section 302 EHS RQ: Reportable Quantity of Extremely Hazardous Substance, listed at 40 CFR 355.
SARA Section 302 EHS TPQ: Threshold Planning Quantity of Extremely Hazardous Substance. An asterisk (*) following a Threshold Planning Quantity signifies that if the material is a solid and has a particle size equal to or larger than 100 micrometers, the Threshold Planning Quantity = 10,000 LBS.
SARA Section 313 Chemicals: Toxic Substances subject to annual release reporting requirements listed at 40 CFR 372.65.
CERCLA Sec. 103: Comprehensive Enviromental Response, Compensation and Liability Act (Superfund). Releases to air, land or water of these hazardous substances which exceed the Reportable Quantity (RQ) must be reported to the National Response Center, (800-424-8802); Listed at 40 CFR 302.4
RCRA: Resource Conservation and Reclamation Act. Commercial chemical product wastes designated as acute hazards and toxic under 40 CFR 261.33

APPENDIX C

IT PROCEDURE HS603

IT CORPORATION

STANDARD PROCEDURE

PROCEDURE NO. 9521.1

DATE October 29, 1979

SUPERSEDES ----

APPROVED David R. Smith

David R. Smith

SUBJECT:

GasTech GX-3 Maintenance

1. Purpose: To prescribe routine inspection and preventive maintenance for the GX-3 combustible gas/oxygen indicators.

2. Reference: Instruction Manual, 0102-072977-1-10, Model GX-3 Combination Combustible Gas/Oxygen Indicator and Alarm.

3. Attachments:

   A. Inventory list, GX-3 Combustible gas/oxygen indicator; instrument diagram, GX-3

   B. Operational tests.

4. Office of Primary Responsibility:

   Corporate Director of Occupational Safety and Health

5. Discussion:

   Reliable measurements of potentially hazardous atmospheres encountered by field personnel demand optimum performance from operations test instruments. These instruments are sensitive and can get out of calibration easily. In consequence, conscientious attention to proper accomplishment of necessary inspections and scheduled maintenance is mandatory. Periodic inspections and maintenance checks are prescribed in this directive.

6. Action: Before any instrument is issued to field personnel the following procedures will be accomplished:

   A. Daily Inspection

      1. The instrument shall be inspected to ensure that all items listed on the inventory list on Attachment A are with the instrument.

      2. The instrument shall be given the operations tests in Attachment B to verify proper operation.

B. Monthly Maintenance

1. The instrument shall be given the maintenance prescribed in Attachment B at no less a frequency than once a month.

2. An inspection report tag shall be attached to the instrument upon completion of monthly maintenance.

C. Special Precautions

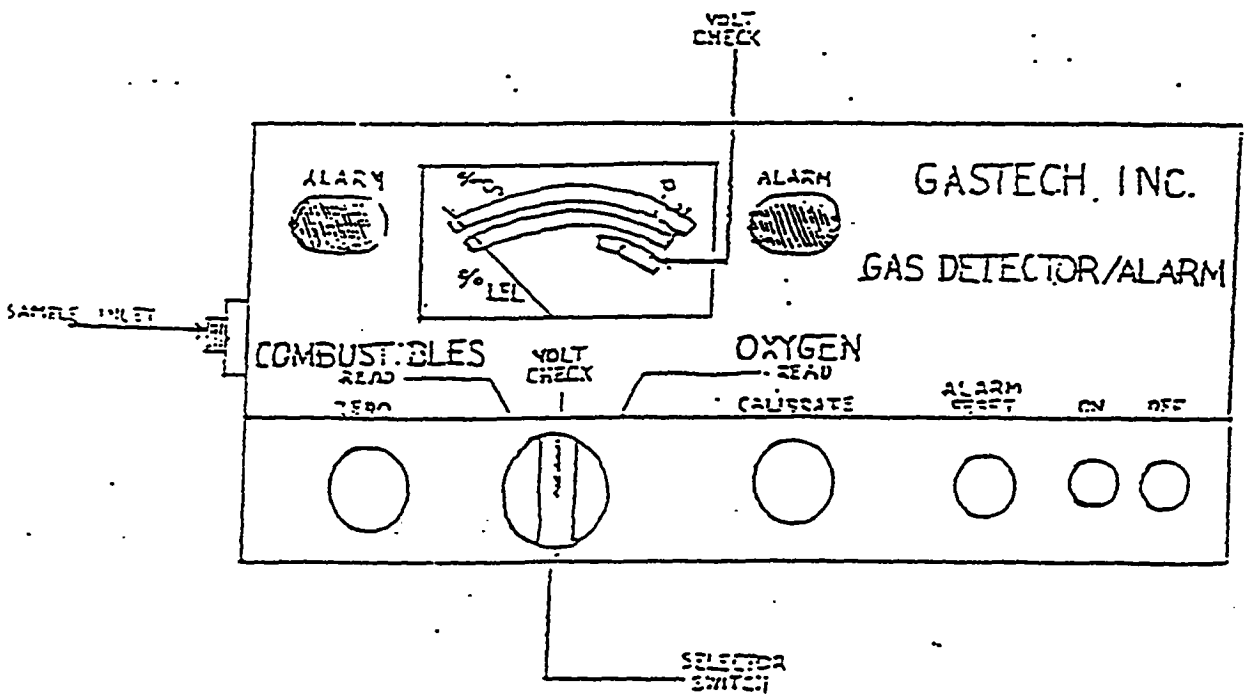
Instruments used in sampling heated spaces such as hot tanks or sampling spaces containing leaded products require additional precautionary checks. These are set forth in Attachment B. A most important consideration is the need for more frequent calibration of instruments using a known concentration of calibration gas to ensure reliability of measurements.

7. Exceptions: All instruments that are assigned to an individual Foreman or Supervisor shall be returned by them each week to the storeroom for inspection, maintenance, and calibration.

GasTech GX-3 Inventory List

1. GX-3 complete with carrying case.
2. Instruction Manual.
3. Probe with cotton filter, 10" fiberglass.
4. Hose, 3', complete with couplings.
5. Hose, 6', complete with couplings.

Instrument Diagram



1. Operational Tests - each instrument shall be inspected to verify normal operation by the following means:

   A. Check battery voltage. Turn instrument on and move selector switch to "volt check" position. Needle should rise to the black band near top of scale. Batteries should be replaced if the needle lies below the "V" on the volt check scale (see attachment A).

   B. Verify pump operation. With instrument turned on, verify pump operation by holding finger over sample inlet. Pump should slow down or stop.

   If pump does not slow appreciably, there is leakage at pump valves or elsewhere in system, and reliable gas indication may be impossible.

   C. Inspect Sampling System

      (1) Probe. Inspect 10" flexible plastic tube for general cleanliness, and remove all liquid and residue before issuing. Inspect filter unit and clean wire screen as necessary. Replace cotton plug if soiled.

      (2) Hoses. Inspect both 3' and 6' hose for damage to rubber surfaces, fittings, or teflon lining. Inspect "O" rings. Replace or repair as necessary.

      (3) Leak Test. Assemble sample system and attach to instrument. With instrument turned on, hold finger over hose inlet. Pump should stop or slow noticeably if there is no leak. Disassemble sample system and place it back in pouch in carrying case.

   D. Check Oxygen Alarm Setting. With instrument turned on, check oxygen alarm setting by moving selector switch to "Oxygen Read" position. Needle should rise to upper portion of scale. Lift and engage "calibrate" knob and rotate counterclockwise to move the needle to 20% oxygen. The oxygen alarm light will glow and an audible alarm will sound if the instrument is properly set at 20% oxygen. Do not issue the instrument unless the oxygen alarm is properly set at 20% oxygen. Return the needle position to 21% oxygen and press "Reset" button to reset alarm circuit.

   E. Check Combustibles Alarm Setting. Check combustibles alarm setting by turning selector switch to "combustibles" position. Meter should read close to zero. Lift and engage "zero" knob clockwise to move the needle to 10% LEL. The combustibles alarm light will glow and an audible alarm will sound if the instrument is properly set at 10% LEL. Return the needle to zero by rotating the zero knob counterclockwise.

2. Monthly Maintenance

A. Thoroughly clean external surfaces of instrument and carrying case using a mild detergent and water solution.

B. Calibrate instrument with a known concentration of methane gas using the GasTech Calibration Test Kit No. 81-0202, as per manufacturer's instructions.

C. Perform all operations checks described in 1. above.

D. Attach an inspection report tag indicating the date of inspection and the inspector's name.

3. Special Precautions. In addition to normal daily instrument inspection and monthly calibration checks, the following precautionary maintenance checks may be necessary for special operations:

A. Heated Samples. Sampling spaces such as hot tanks that are warmer than the instrument may result in condensation of water vapor as the sample passes through the cool sample line. This condensation can block the flow system, corrode the flame arrestor and cause pump valve sticking. Instruments that have been used for such sampling should be inspected for evidence of water vapor condensation.

B. Filament Poisoning. Sampling spaces that have contained leaded products such as leaded gasoline, or silicone vapors, may result in desensitization of the catalytic surface of the platinum filament.

Instruments that have been used for such sampling should be calibrated more frequently using a known concentration of methane gas to ensure reliability of measurements.

IT CORPORATION

STANDARD PROCEDURE

PROCEDURE NO. ITC PRO 9

DATE July 27, 1987

SUPERSEDES

APPROVED D.R. Smith
David R. Smith

SUBJECT:

Use, Calibration, and Maintenance of The HNU PI-101

I. PURPOSE

To provide direction for the use, calibration, limitations, and maintenance of the HNU.

II. REFERENCES

A. HNU Systems, Inc., Instruction Manual for Model PI 101 Photoionization Analyzer, 1975

B. E&E FIT Operation and Field Manual: HNU System PI 101 Photoionization Detector and Century Systems (Foxboro) Model OVA-128 Organic Vapor Analyzer

III. RESPONSIBILITIES

A. Corporate Director -- Health & Safety

1. Maintain this procedure, and

2. Specify training of IT employees as appropriate on the use, calibration, limitations, and maintenance of the HNU.

B. Each IT Location shall

1. Ensure that employees using the HNU are trained in use, limitations, calibration, maintenance, and interpretation of readings,

2. Implement the procedures outlined herein,

3. Maintain records of employee training and instrument calibration, and

4. Maintain HNU inventory control and accountability.

IV. DISCUSSION

A. Instrument Maintenance and Calibration

It is the responsibility of the instrument user to ensure that the instrument is calibrated and operating properly. When the instrument is scheduled for or requires maintenance, these functions should be conducted only by qualified individuals. If possible, maintenance responsibilities should be restricted to one or two individuals, who will also bear responsibilities for logging the equipment in and out.

Each regional office will be responsible for developing a documentation format which should include instrument user, dates of use, instrument identification number, and projects the instrument will be used on.

Documentation of maintenance and calibration functions will be discussed in the appropriate attachment.

B. Operator Qualifications

The HNU, although a relatively simple instrument to use, can be incorrectly operated if the user is not thoroughly familiar with the instrument's operation. An appropriate training and certification procedure should be developed and incorporated into each region's training procedures. All users should complete the training and be certified for HNU operation prior to using the instrument in the field.

V. PROCEDURES

A. Start-up/Shut Down Procedures

1. Start-up Procedures

a. Attach the probe to the read out unit. Match the alignment key, then twist the connector clockwise until a distinct locking is felt.

b. Turn the FUNCTION switch to the battery check position. Check to ensure that the indicator reads within or beyond the green battery arc on the scale plate. If the indicator is below the green arc, or if the red LED comes on the battery must be charged prior to using.

c. To zero the instrument, turn the FUNCTION switch to the STANDBY position and rotate the ZERO POTENTIOMETER until the meter reads zero. Wait 15-20 seconds to ensure that the zero adjustment is stable. If not, then readjust.

d. Check to see that the SPAN POTENTIOMETER is set at the appropriate setting for the probe being used.

e. Set the FUNCTION switch to the desired ppm range.

f. Listen for the fan operation to verify fan function.

g. Check instrument with an organic point source such as a magic marker prior to survey to verify instrument function.

2. Shut Down Procedures

a. Turn FUNCTION Switch to OFF.

b. Disconnect the probe connector.

c. Place the instrument on the charger.

B. Maintenance and Calibration Schedule

Function	Frequency
Routine Calibration	Prior to each use*
Factory Check-out & Calibration	Yearly or when malfunctioning
Wipe Down Read-Out Unit	After each use
Clean UV Light Source Window	Every month or as use and site conditions dictate
Clean the Ionization Chamber	Monthly
Recharge Battery	After each use

* It is recommended that for activities where the HNU-PI-101 is in the field for extended periods of time that calibration gas be brought into the field to check calibration. However, if necessary a single calibration conducted prior to an activity will be considered acceptable for periods up to one week.

C. Calibration Procedure 1

1. Run through start-up procedures as set forth in V.A.

2. Fill a sampling bag with HNU calibration gas (Analyzed).

3. Allow sample bag contents to be drawn into the probe and check response in ppm.

4. If the reading deviates ± 15% from the concentration of the calibration gas, the instrument requires maintenance.

5. Each region should develop a mechanism for the documentation of calibration results. This documentation should include:

   a. Date inspected

   b. Person who calibrated the instrument

   c. The instrument number (Serial # or EPA ID#)

   d. The results of the calibration (ppm, probe ev, span pot setting)

   e. Identification of the calibration gas (source, type, concentration, lot #).

D. Calibration Procedure 2 - (For HNU Calibration Canisters Equipped with a Regulator)

1. Run through start-up procedures as set forth in V.A.

2. Connect a sampling hose to the regulator outlet and the other end to the sampling probe of the HNU.

3. Crack the regulator valve.

4. Take reading after 5-10 seconds.

5. If the reading deviates ± 15% from the concentration of the calibration gas, the instrument requires maintenance.

6. Calibration documentation should be as above.

E. Cleaning the UV Light Source Window

1. Turn the FUNCTION switch to the OFF position and disconnect the sensor/probe from the Read Out/Control unit.

2. Remove the exhaust screw located near the base of the probe. Grasp the end cap in one hand and the probe shell in the other. Separate the end cap and lamp housing from the shell.

3. Loosen the screws on the top of the end cap and separate the end cap and ion chamber from the lamp and lamp housing, taking care that the lamp does not fall out of the lamp housing.

4. Tilt the lamp housing with one hand over the opening such that the lamp slides out of the housing into your hand.

5. The lamp window may now be cleaned with any of the following compounds using lense paper:

   a. HNU Cleaning Compound-All lamps except the 11.7 eV

b. Carbon tetrachloride-All lamps except the 11.7 eV

c. Methanol-All lamps

6. Following cleaning, reassemble by first sliding the lamp back into the lamp housing. Place the ion chamber on top of the housing making sure the contacts are properly aligned.

7. Place the end cap on top of the ion chamber and replace the two screws. Tighten the screws only enough to seal the O-ring. Do Not Overtighten.

8. Line up the pins on the base of the lamp housing with pins inside the probe shell, and slide the housing assembly into the shell. It will only fit one way.

F. Cleaning the Ionization Chamber

1. Turn the FUNCTION switch to the OFF position and disconnect the sensor/probe from the Read Out/Control unit.

2. Remove the exhaust screws located near the base of the probes. Grasp the end cap in one hand and the probe shell in the other. Separate the end cap and lamp housing from the shell.

3. Loosen the screws on the top of the end cap and separate the end cap and ion chamber from the lamp and lamp housing, taking care that the lamp does not fall out of the lamp housing.

4. The ion chamber may now be cleaned according to the following sequence:

   a. acetone rinse with agitation (10 min.)

   b. dry (preferably with oven at 100°C)

   c. methanol rinse with agitation (10 min.)

   d. dry (preferably with oven at 100°C)

5. Place the ion chamber on top of the housing making sure the contacts are properly aligned.

6. Place the end cap on top of the ion chamber and replace the two screws. Tighten the screws only enough to seal the O-ring. Do Not Overtighten.

7. Line up the pins on the base of the lamp housing with pins inside the probe shell, and slide the housing assembly into the shell. It will only fit one way.

G. Trouble Shooting

1. No meter response in any switch position (including BATT CHK)

   a. Broken meter movement

      Tip instrument rapidly from side to side. Meter needle should move freely, and return to zero.

   b. Electrical connection to meter is broken

      Check all wires leading to meter and clean the contacts of quick-disconnects.

   c. Battery is completely dead

      Disconnect battery and check voltage with volt-ohm meter.

   d. If none of the above solves the problem, consult the factory.

2. Meter responds in BATT CHK position, but reads zero or near zero for all others.

   a. Power supply defective

      Check power supply voltages per Figure 11 of the HNU owners manual. If any voltage is out of specification, consult the factory.

   b. Input transistor or amplifier has failed

      (1) Rotate zero control; meter should reflect up/down as control is turned.

      (2) Open probe. Both transistors should be fully seated in sockets.

   c. Input signal connection broken in probe or readout.

      (1) Check input connector on printed circuit board. The input connector should be firmly pressed down.

      (2) Check components on back side of printed circuit board. All connections should be solid, and no wires should touch any other object.

      (3) Check all wires in readout for solid connections.

3. Instrument responds correctly in BATT CHK, and STBY, but not in measuring mode.

Check to see that light source is on.

a. Check high voltage power supply.

b. Open end of probe, remove lamp and check high voltage on lamp contact ring.

c. If High voltage is present at all above points, light source has most likely failed. Consult the factory.

4. Instrument responds correctly in all positions, but signal is lower than expected.

a. Check span setting for correct value.

b. Clean window of light source.

c. Double check preparation of standards.

d. Check power supply 180 V output.

e. Check for proper fan operation. Check fan voltage.

5. Instrument responds in all switch positions, but is noisy (erratic meter movement)

a. Open circuit in feedback circuit. Consult the factory.

b. Open circuit in cable shield or probe shield. Consult the factory.

6. Instrument response is slow and/or irreproducible.

a. Fan operating improperly. Check fan voltage.

b. Check calibration and operation. See sections 2 and 3.

IT CORPORATION

STANDARD PROCEDURE

PROCEDURE NO. ITC PRO

DATE July 27, 1987

SUPERSEDES

APPROVED DRSmith

David R. Smi

SUBJECT:

Use, Calibration, and Maintenance of The OVA-128

I. PURPOSE

To provide direction for the use, calibration, limitations, and maintenance of the OVA.

II. REFERENCES

A. Region II FIT "Proposed Guidelines for the Organic Vapor Analyzer"

B. E&E FIT "FIT Operation and Field Manual: HNU Systems PI 101 Photoionization Detector and Century Systems (Foxboro) Model OVA-128 "Organic Vapor Analyzer"

C. Century Systems (Foxboro). "Service Procedures: Organic Vapor Analyzer; 128GC".

III. RESPONSIBILITIES

A. Corporate Director -- Health & Safety

1. Maintain this procedure, and

2. Specify training of IT employees as appropriate on the use, calibration, limitations, and maintenance of the OVA.

B. Each IT Location shall

1. Ensure that employees using the OVA are trained in use, limitations, calibration, maintenance, and interpretation of readings,

2. Implement the procedures outlined herein,

3. Maintain records of employee training and instrument calibration, and

4. Maintain OVA inventory control and accountability.

IV. DISCUSSION

A. Instrument Maintenance and Calibration

It is preferable to minimize the number of people responsible for maintenance and calibration of the OVA. If possible, one or two individuals should be given this responsibility. These individual(s) will also be responsible for logging the equipment in and out.

Each regional office will be responsible for developing a documentation format which should include instrument user, dates of use, instrument identification number, and projects the instrument will be used on.

Documentation of maintenance and calibration procedures will be discussed in the appropriate attachment.

B. Operator Qualifications

The OVA, although a relatively simple instrument to use, can be incorrectly operated if the user is not thoroughly familiar with the instrument's operation. An appropriate training and certification procedure should be developed and incorporated into each region's training procedures. A user should complete the training and be certified for OVA use prior to taking the instrument into the field.

V. PROCEDURES

A. Start-up/Shut Down Procedures

1. Start-up Procedures

a. Connect the probe/read out connectors to the side-pack assembly.

b. Check battery condition and hydrogen supply.

c. For measurements taken as methane equivalent, check that the GAS SELECT Dial is set at 300.

d. Turn the electronics on by moving the INSR switch to the ON position and allow five (5) minutes for warm-up.

e. Set CALIBRATE switch to x10, use CALIBRATE knob to set indicator at 0.

f. Open the H_2 tank valve all the way and the H_2 supply valve all the way. Check that the hydrogen supply gauge reads between 8.0 and 12.0 psig.

g. Turn the PUMP switch ON and check the flow system according to the procedures in Attachment 4.

h. Check that BACKFLUSH and INJECT valves are in the UP position.

i. To light the flame, depress the igniter switch until a meter deflection is observed. The igniter switch may be depressed for up to 5 seconds. Do not depress for longer than five (5) seconds, as it may burn out the igniter coil. If the instrument does not light, allow the instrument to run several minutes and repeat ignition attempt.

j. Confirm OVA operational state by sniffing an organic source, such as a magic marker.

k. Establish a background level in a clean area or by using the charcoal scrubber (depress the sample inject valve) and recording measurements referenced to background.

l. Set the alarm level, if desired.

2. Shut Down Procedures

a. Close H_2 supply valve and H_2 tank valve (Do Not Overtighten Valves).

b. Turn INSTR switch to OFF.

c. Wait until H_2 supply gauge indicates system is purged of H_2, then switch off pump.

d. Put instrument on electrical charger at completion of day's activities.

B. Maintenance and Calibration Schedule

Function	Frequency
Check Particle Filters	Weekly or as needed
Check Quad Rings	Monthly or as needed
Clean Burner Chamber	Monthly or as needed
Secondary Calibration Check	Prior to project start-up
Primary Calibration Check	Monthly or if secondary check is off by more than ± 10%
Check Pumping System	Prior to project start-up
Replace Charcoal	120 hours of use or when background readings are higher with the inject valve down than with the inject valve up in a clean environment.
Factory Service	At least annually

Instruments which are not in service for extended periods of time are not held to the above schedule. However, they must be given a complete check-out prior to their first use addressing the above maintenance items.

C. Calibration Procedure 1

1. Remove instrument guts from the instrument shell.

2. Turn on Electronics and Zero Instrument on x-10 scale. Gas select dial to 300.

3. Turn on Pump and Hydrogen. Ignite Flame. Go to Survey Mode.

4. Introduce a Methane Standard near 100 ppm.

5. Adjust R-32 Trimpot on Circuit Board to make meter read to standard.

6. Turn off hydrogen flame and adjust meter needle to read 40 ppm (calibrate @ x 10) using the calibration adjust knob.

7. Switch to X100 Scale. The meter should indicate 0.4 on the 1-10 meter markings. (0.4 x 100 = 40 ppm), if the reading is off, adjust with R33 Trimpot.

8. Return to X10 Scale and adjust meter needle to 40 ppm with calibration adjust knob if necessary.

9. At the X10 Scale adjust meter to read 0.4 on the 1-10 meter markings using the calibration adjust. Switch to X1 scale. The meter should read 4 ppm. If the reading is off, adjust using the R-31 Trimpot.

D. Calibration Procedure 2

1. Fill an air sampling bag with 100 ppm (certified) methane calibration gas.

2. Connect the outlet of the air sampling bag to the air sampling line of the OVA.

3. Record the reading obtained off the meter onto the calibration record.

E. Documentation

Each regional shall develop a system whereby the following calibration information is recorded.

1. Instrument calibrated (I.D. or Serial #)
2. Date of calibration
3. Method of calibration
4. Results of the calibration
5. Identification of person who calibrated the instrument
6. Identification of the calibration gas (source, type, concentration, lot #)

F. Pump System Check-Out

1. With Pump On, hold unit upright and observe flow gauge.

2. Ball level significantly below a reading of 2 is low flow.

3. Check connections at the sample hose.

4. Clean or replace particle filters if flow is impaired or it is time for scheduled service.

5. Reassemble and retest flow.

6. If flow still low, replace pump diaphragm and valves.

7. If flow normal, plug air intake. Pump should slow and stop.

8. If no noticeable change in pump, tighten fittings and retest.

9. If still no change, replace pump diaphragm and valves.

10. Document this function in the maintenance records developed for your region.

G. Burner Chamber Cleaning

1. Remove plastic exhaust port cover.

2. Unscrew exhaust port.

3. Use wire brush to clean burner tip and electrode. Use wood stick to clean Teflon.

4. Brush inside of exhaust port.

5. Blow out chamber with a gentle air flow.

6. Reassemble and test unit.

7. Document this function in the maintenance records developed for your region.

H. Quad Ring Service

1. Remove OVA guts from protective shell.

2. Remove clip ring from bottom of valve.

3. Unscrew nut from top of valve.

4. Gently pull valve shaft upward and free of housing.

5. Observe rings for signs of damage. Replace if necessary.

6. Lightly grease rings with silicone grease.

7. Reassemble valve. Do not pinch rings during shaft insertion.

8. Document this function in the maintenance records developed for your region.

I. Trouble Shooting

Indication	Possible Causes
High Background Reading (More Than 10 ppm)	Contaminated Hydrogen Contaminated Sample Line
Continual Flameout Dirty Burner Chamber Dirty Air Filters	Hydrogen Leak
Low Air Flow Pump Malfunction Line Obstruction	Dirty Air Filter
Flame Will Not Light Ignitor Broken Hydrogen Leak Dirty Burner Chamber Air Flow Restricted	Low Battery
No Power to Pump Short Circuit	Low Battery
Hydrogen Leak (Instrument Not in Use)	Leak in Regulator Leak in Valves

J. Shipping

Since the OVA-128 contains hydrogen, it is subject to shipping restrictions.

1. As Personal Luggage

   The OVA-128 can be taken on a plane as luggage as a permit has been issued from the Department of Transportation to the manufacturer (Foxboro).

2. Federal Express

   The following labels must be affixed to both sides of the OVA case when shipping OVA by Federal Express.

   Danger - Peligro
   Flammable Gas
   Inside Container Complies with DOT regulations
   Hydrogen UN #1049
   Name and Address of Recipient

A hazardous air bill must be filled out. The following information is requested.

Proper Shipping Name	Hydrogen
Classification	Flammable Gas
ID# UN 1049	
Net Quantity 75 Cubic Centimeters	

In addition, the shipping's certification must be signed and marked Cargo Aircraft Only.

K. Hydrogen Recharging

1. High-grade hydrogen (99.9996) is required.

2. Connect the fill hose to the REFILL FITTING on the side Pack Assembly, with the FILL/BLEED valve in the OFF position.

3. Open H_2 supply bottle valve.

4. Place FILL/BLEED Valve on fill hose in BLEED position momentarily to purge any air out of the system.

5. Crack the instrument TANK and SUPPLY VALVE.

6. Open REFILL Valve on instrument.

7. Place FILL/BLEED Valve in FILL position until the instrument PRESSURE GAUGE equalize with the H_2 SUPPLY BOTTLE PRESSURE GAUGE.

8. Shut REFILL Valve, FILL/BLEED Valve, and H_2 SUPPLY BOTTLE Valve, in quick succession.

9. Turn FILL/BLEED Valve to BLEED until hose pressure equalizes to atmospheric pressure.

10. Turn FILL/BLEED Valve to FILL position, then to the BLEED position, then to OFF.

11. Close TANK and SUPPLY VALVE on instrument.

12. Disconnect the FILL HOSE and replace protective nut on the REFILL FITTING.

L. Particle Filter Servicing

There are two points in the air sampling line of the OVA where filters have been placed to keep particulates from entering the

instrument. The location of these filters are indicated on the attached figure. The first filter is located in the probe assembly, and the second filter (primary filter) is located on the side pack assembly. Cleaning procedures are as follows:

1. Detach the probe assembly from the readout assembly.

2. Disassemble the probe (the components unscrew).

3. The particle filter located within the probe can be cleaned by blowing air through the filter.

4. Reassemble the probe.

5. The primary filter, located behind the sample inlet connector on the side pack assembly is accessed by removing the sample inlet connector with a thin-walled 7/16" socket wrench. Remove the filter and clean as above.

6. Reassemble the sample inlet fitting and filter to the side pack assembly.

7. Check sample flow rate.

APPENDIX D

SUPERVISOR'S EMPLOYEE ACCIDENT REPORT

INTERNATIONAL TECHNOLOGY CORPORATION

SUPERVISOR'S EMPLOYEE INJURY REPORT

This is an official document to be initiated by the employee's supervisor. Please answer all questions completely. This rept forwarded to the employee's Regional Health and Safety office within 24 hours of the injury.

Injured's Name_____ Sex____ S.S. No._____ Birthdate____

Home Address_____ City_____ State_____ Zip_____ Phone____

Job title_____ Employee P.C._____ Hire Date_____ Hourly wag

SUPERVISOR

Date of incident_____ Time_____ Time reported_____ To whom?_____

Client name_____ Client address_____ Time shift bega

Exact location of the incident_____ Did the employee leave work? ☐ No ☐ Yes When____

Has employee returned to work? ☐ No ☐ Yes When_____ Did employee miss a regularly scheduled shift? ☐ N

Doctor/Hospital name_____ Address_____

Witness name(s)_____ Statement attached? ☐ No

Nature of injury_____ Exact body part_____

Medical attention: ☐ None ☐ First aid on site ☐ Doctor's office ☐ Hospital ER ☐ Hospitalized

Job assignment at time of incident_____ Job:_____ Phase:_____ Task:_____ Subtask:

Describe incident_____

What unsafe physical condition or unsafe act caused the incident?_____

What corrective action has been taken to prevent recurrence?_____

Supervisor/Foreman _____ (Print) _____ Signature _____ Date

MANAGER

Comments on incident and corrective action_____

Manager's name _____ (Print) _____ Signature _____ Date

HEALTH AND SAFETY

Concur with action taken? ☐ No ☐ Yes Remarks_____

OSHA Classification:

☐ Incident only ☐ First aid ☐ Recordable, no lost work days ☐ Lost work days ☐ Restricted activity ☐ Fatal

Days away from work_____ Days restricted work_____ Total days charged_____

☐ State jurisdiction ☐ Federal L & H ☐ Date ER submitted _____ Which claims office_____

Coding: A. Injury type or illness____ B. Injured body parts____ C. Activity at time of accident____ D. Injury cause code_

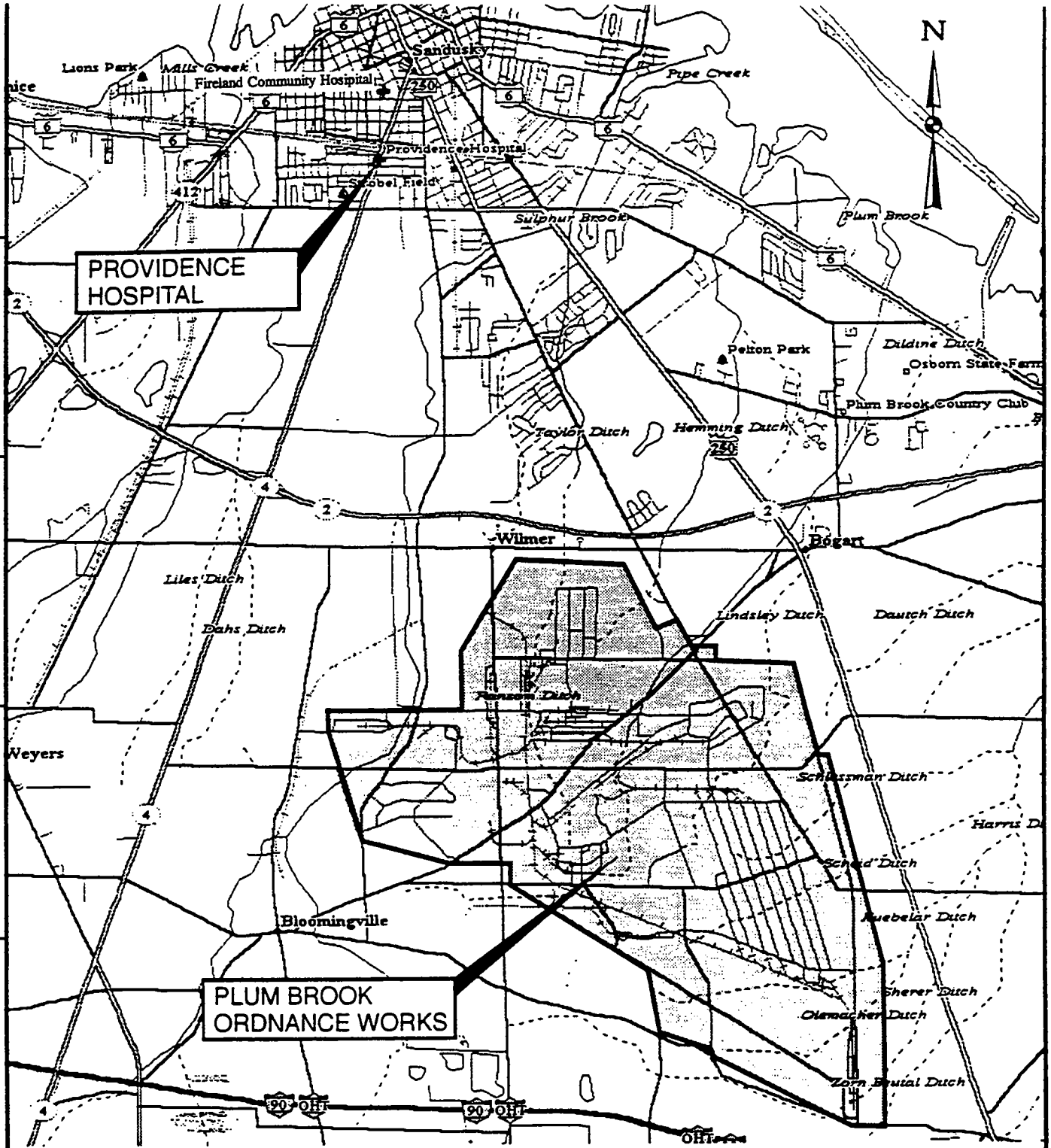
E. Agent code____ F. Safety rule violated code____ G. Accident prevention code

Name _____ (Print) _____ Signature _____ Date

Form HS0204 5/25/94 Submit copies to: Corporate HS, Corporate Risk Mgmt., Region HS, Profit/Cost Center Mgr. and EMR

APPENDIX E

SITE AND HOSPITAL LOCATION MAP



DWG. No.: 764676-A-
PROJ. NO.: 764676
INITIATOR: J. LI
PROJ. MGR.: D. BURTON
DRAFT. CHCK. BY: J. HUBBARD
ENGR. CHCK BY: G. YU
DATE LAST REV.:
DRAWN BY:
STARTING DATE: 10/26/95
DRAWN BY: D. HIGGS

EMAP5.CDR 10/26/95 DJH

APPENDIX C

SITE AND HOSPITAL LOCATION MAP

PROVIDENCE HOSPITAL
1912 HAYES AVE.
SANDUSKY, OHIO 44870

PH. (419) 621-7000

PLUM BROOK ORDNANCE WORKS
HURON COUNTY, OHIO

INTERNATIONAL TECHNOLOGY CORPORATION

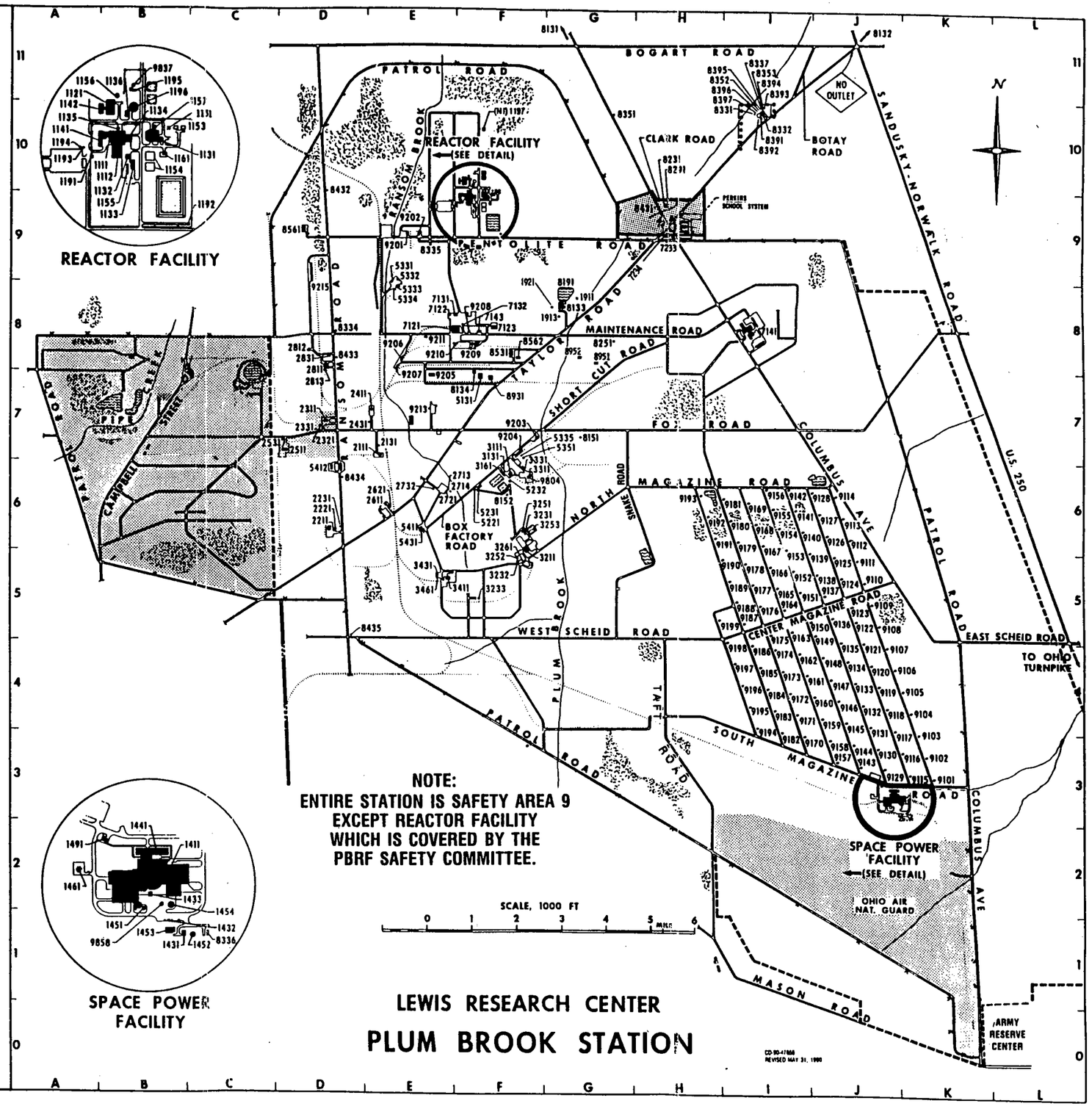
Direction to the Providence Hospital:

Exit from the front gate of Plum Brook Station; turn right to Taylor Road; go approximately 1/4 mile; turn left to Botay Road; at the stop, sign make a left turn to Bogart Road (County Road No. 10); take Bogart Road all the way to State Road No. 4; (Wright Brothers Memorial Highway); make a right turn at State Road No. 4; Providence Hospital will be on your right-hand side.

Estimated travel time to the hospital from PBS is between 15 and 30 minutes, depending on traffic condition.

1111 REACTOR BUILDING. F-9
1112 REACTOR HOT LABORATORY. F-9
1121 REACTOR ATS BUILDING. F-10
1131 REACTOR SERV. EQUIP. BLDG. F-9
1132 REACTOR FAN HOUSE. F-9
1133 REACTOR WASTE HANDLING BLDG. F-9
1134 REACTOR PRIMARY PUMP HOUSE. F-9
1135 REACTOR GAS SERVICES BLDG. F-10
1136 REACTOR COMPRESSOR BLDG. F-10
1141 REACTOR OFFICE & LABORATORY. F-9
1142 REACTOR OFFICE BUILDING. F-10
1151 REACTOR WATER TOWER. F-10
1153 REACTOR SLUDGE BASINS. F-10
1154 REACTOR COLD RETENTION BASINS. F-9
1155 REACTOR HOT RETENTION BASINS. F-9
1156 REACTOR ATS WATER STORAGE TANK (200K GAL). F-10
1157 REACTOR PRECIPITATOR. F-10
1161 REACTOR SUBSTATION (E). F-9
1191 REACTOR SECURITY BUILDING. F-9
1192 REACTOR EFFLUENT METERING STA. F-9
1193 REACTOR WEATHER TOWER HOUSE. E-9
1194 REACTOR WEATHER TOWER. E-9
1195 REACTOR CRYOGENIC AND GAS SUPPLY SYSTEM. F-10
1196 REACTOR GAS STOR. STRUC. F-10
1197 REACTOR MONITORING STATION (N1) F-10
1411 SPF TEST BUILDING. J-3
1431 SPF WATER TREATMENT BLDG. K-3
1432 SPF LN2 SERVICE BUILDING. J-3
1433 SPF BOTTLE STORAGE BUILDING. J-3
1441 SPF OFFICE BUILDING. J-3
1451 SPF STACK. J-3
1452 SPF WATER TOWER (G). K-3
1453 SPF COOLING TOWER. J-3
1454 SPF LN2 TANK (200K GAL.). J-3
1461 SPF SUBSTATION (H). J-3
1491 SPF SECURITY BUILDING. J-3
1911 100 KW WIND TURBINE. G-8
1913 WIND TURBINE WEATHER TOWER. G-8
1921 WTG SHOP G-8
2111 A SITE TEST BUILDING. E-7
2131 A SITE BOILER HOUSE. E-7
2211 C SITE TEST BUILDING. D-6
2221 C SITE SHOP BUILDING. D-6
2231 C SITE BOILER HOUSE. D-6
2311 D SITE TEST BUILDING. D-7
2321 D SITE SHOP BUILDING. D-7
2331 D SITE BOILER HOUSE. D-7
2411 E SITE TEST BUILDING. E-7
2431 E SITE BOILER HOUSE. E-7
2511 F SITE TEST BUILDING. D-7
2531 F SITE BOILER HOUSE. D-7
2611 I SITE TEST BUILDING. E-6
2621 I SITE SHOP BUILDING. E-6
2713 J SITE J5 TEST BUILDING. E-6
2714 J SITE J5 CONTROL TANK. E-6
2721 J SITE SHOP BUILDING. E-6
2732 J SITE BOILER HOUSE. E-6
2811 K SITE TEST BUILDING. D-8
2812 K SITE CONTROL BUILDING D-8
2813 K SITE VACUUM EQUIPMENT BUILDING D-8
2831 K SITE BOILER HOUSE D-8
3111 B1 TEST STAND. F-6
3131 B1 PUMP AND SHOP BUILDING. F-6
3161 B1 SUBSTATION (D). F-6
3211 B2 TEST BUILDING. F-5/6
3231 B2 REFRIGERATION BUILDING. F-6
3232 B2 UTILITY SERVICE BUILDING. F-5
3233 B2 LN2 TRANSFER BUILDING. F-5
3251 B2 COOLING TOWER. F-6
3252 B2 COOLING TOWER (TEST BLDG.). F-5
3253 B2 RETENTION POND (2.5M GAL). F-6
3261 B2 SUBSTATION (G). F-5
3311 B3 TEST STAND. F-6
3331 B3 BOILER HOUSE. F-6
3411 HTF TEST BUILDING. E-5
3431 HTF BOILER AND ELECTRICAL SWITCHGEAR HOUSE. E-5
3461 HTF SUBSTATION (F). E-5
5131 AIR COMPRESSOR BUILDING. F-7
5221 SHOP BUILDING. F-6
5231 BOILER BUILDING. F-6
5232 VALVE HOUSE. F-6
5331 GAS HANDLING HELIUM BUILDING. E-9
5332 GAS HANDLING N2 BUILDING. E-9
5333 GAS HANDLING H2 BUILDING. E-8
5334 GAS HANDLING STORAGE BLDG. E-8
5335 LH2 STOR. DEWAR CONT. BLDG. F-7
5351 LH2 STORAGE DEWAR (200K GAL). F-7
5411 B CONTROL AND DATA BUILDING. E-6
5412 H CONTROL AND DATA BUILDING. D-6
5431 GUARANTEE POWER BUILDING. E-6
7121 MAINTENANCE SHOP. E/F-8
7122 CARPENTER SHOP. E-8
7123 LOCOMOTIVE SHOP. F-8
7131 GARAGE. F-8
7132 VEHICLE SERVICE STATION. F-8
7141 ENGINEERING BUILDING. I-8
7143 CHEMICAL LABORATORY. F-8
7233 PLANT PROTECTION BUILDING. H-9
7234 PLANT PROTECTION BOILER HOUSE H-9
8132 RYE BEACH PUMPING STATION. J-11
8133 PUMP STATION NO. 1. G-8
8134 PUMP HOUSE. F-7
8151 RAW WATER TOWER (D). G-7
8152 RAW WATER TOWER (E). F-6
8191 RESERVOIR NO. 1. G-8
8231 DOMESTIC WATER PUMP HOUSE. H-9
8251 DOMESTIC WATER TOWER (A). G-8
8291 DOMESTIC WATER RESERVOIR. H-9
8331 SEWAGE PUMPING STATION. I-10
8332 SEWAGE PUMP & CHLORINE BLDG. I-10
8334 SEWAGE LIFT STATION. D-8
8335 SEWAGE LIFT STATION. E-9
8336 SEWAGE TREATMENT PLANT-SPF. K-3
8337 SEWAGE CHEMICAL BUILDING. I-10
8351 SEWAGE LIFT STATION. G-10
8352 SEWAGE CHLORINE CONTACT TANK. I-10
8353 SEWAGE MIXING CHAMBER. I-10
8391 SEWAGE SETTLING TANK. I-10
8392 SEWAGE DIGESTING TANK. I-10
8393 SEWAGE SLUDGE BEDS. I-10
8394 SEWAGE SLUDGE BEDS. I-10
8395 SEWAGE FLOCCULATOR AND FINAL SETTLING TANK. I-13
8396 SEWAGE DIVERSION CHAMBER. I-10
8397 SEWAGE TRICKLING FILTER. I-10
8431 GAS METER HOUSE. H-9
8432 GAS METER HOUSE. D-9/10
8433 GAS METER HOUSE. D-8
8434 GAS METER HOUSE. D-6
8435 GAS METER HOUSE. D-5
8531 POWER HOUSE NO. 1. F-8
8561 SUBSTATION A. D-9
8562 SUBSTATION B. F-8
8931 INCINERATOR BUILDING. F-7
8951 FUEL STORAGE TANK. E-8
8952 FUEL STORAGE TANK. E-8
9101-9199 MAGAZINES H/K-V6
9201 WAREHOUSE. E-9
9202 WAREHOUSE. E-9
9203 WAREHOUSE. F-7
9204 WAREHOUSE. F-7
9205 WAREHOUSE. E-7
9206 WAREHOUSE. E-8
9207 WAREHOUSE. E-8
9208 WAREHOUSE. F-8
9209 WAREHOUSE. F-8
9210 WAREHOUSE. F-8
9211 WAREHOUSE. E-8
9213 WAREHOUSE. E-7
9215 WAREHOUSE. D-9
9804 GN_2 FARM. F-6
9837 GHE FARM. F-10

(This Site Map is provided by NASA Plum Brook Station)



Quality Assurance Project Plan
Site Investigation at the
G-8 Burning Ground
Former Plum Brook Ordnance Works
Sandusky, Ohio

Revision 1

Prepared For:

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Quality Assurance Project Plan
Site Investigation at the
G-8 Burning Ground
Former Plum Brook Ordnance Works
Sandusky, Ohio

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Table of Contents

Page

List of Tables iv
List of Acronyms v
1.0 Project Description 1-1
1.1 Site Description 1-1
1.2 Project Objectives 1-2
1.3 Target Compounds 1-2
1.4 Sample Program and Rationale 1-2
1.5 Schedule 1-3
2.0 Project Organization 2-1
2.1 USACE Nashville District Project Manager 2-1
2.2 IT Project Manager 2-1
2.3 Quality Assurance Officer 2-2
2.4 Health and Safety Officer 2-3
2.5 Analytical Services QA/QC Director 2-3
2.6 Laboratory QC Coordinator 2-3
2.7 Data Validation Services 2-4
2.8 Subcontractor Activities 2-4
2.9 Qualifications and Training of Personnel 2-5
3.0 Quality Assurance Objectives for Measurement of Data 3-1
3.1 Data Quality Indicators 3-1
3.2 Project QA Objectives 3-2
3.3 Analytical Support Levels 3-3
3.4 Laboratory QC Criteria 3-4
4.0 Sampling Procedures 4-1
4.1 Prevention of Cross Contamination 4-2
4.2 Decontamination Requirements 4-2
4.2.1 Nonsampling Equipment 4-2
4.2.2 Sampling Equipment 4-2
4.3 Sample Identification 4-3
4.4 Sample Turnaround Time 4-4
4.5 Field Documentation 4-4

Table of Contents (Continued)

Page

4.6 Corrections to Documentation 4-5
4.7 Variance System 4-5
4.8 Nonconformances and Corrective Action Procedures 4-6
4.9 Field Quality Control Samples 4-7
4.9.1 Field Duplicates 4-7
4.9.2 Matrix Spike/Matrix Spike Duplicates 4-7
4.9.3 Split Samples 4-7
5.0 Sample Control Procedures 5-1
5.1 Field Custody Procedures 5-1
5.2 Sample Labeling 5-2
5.3 Transfer of Custody and Shipment 5-2
5.4 Preparation, Packaging, Handling, and Shipping 5-3
5.5 Laboratory Receipt and Entry of Samples 5-3
5.6 Preanalysis Storage 5-4
5.7 Postanalysis Storage 5-4
5.8 Final Evidence Files 5-4
5.8.1 Indexing and Filing of Project Records 5-5
5.8.2 Laboratory Analytical Project Files 5-5
6.0 Calibration Procedures 6-1
6.1 General Calibration Procedures and Frequency 6-1
6.2 Calibration Reference Standards 6-1
6.3 Calibration Failures 6-2
6.4 Calibration Records 6-2
6.5 Operational Calibration 6-3
6.6 Periodic Calibration 6-3
7.0 Analytical Procedures 7-1
7.1 Analytical Objective 7-1
7.2 Analytical Laboratories 7-1
7.3 Analytical Instrumentation 7-1
7.4 Analytical Methods and Procedures 7-2
8.0 Data Reduction, Validation, and Reporting 8-1
8.1 Data Reduction and Verification 8-1
8.2 Data Reports 8-3

Table of Contents *(Continued)*

Page

8.3 Deliverables 8-4
8.4 Data Validation 8-4
9.0 Internal Quality Control Samples 9-1
9.1 Surrogate Spikes 9-1
9.2 Duplicate Analyses 9-1
9.3 Matrix Spikes and Postdigestion Spikes 9-2
9.4 Internal Standards 9-2
9.5 Method Blanks 9-2
9.6 Reagent Blanks 9-2
9.7 Check Standard 9-3
9.8 Standard Reference Material 9-3
9.9 Blank Spike (Laboratory Control Samples) 9-3
9.10 Analytical Instrument Calibration 9-3
10.0 Performance and System Audits 10-1
10.1 Audit Schedule and Records 10-1
10.2 Performance and Systems Audits 10-2
10.2.1 Performance Audits 10-2
10.2.2 System Audits 10-3
10.2.3 Field Audits 10-3
11.0 Preventive Maintenance Procedures 11-1
11.1 Field Instruments and Equipment 11-1
11.2 Laboratory Instruments 11-1
12.0 Assessment Procedures for Accuracy, Precision, and Completeness 12-1
13.0 Corrective Action 13-1
14.0 QA Reports to Management 14-1
Appendix A - Quanterra Standard Operating Procedures

List of Tables

Number	Title	Follows Page
1-1	Summary of Chemical Analysis and Methodologies for Soil and Sediment Samples	1-2
4-1	Soil and Sediment Sample Designations and QA/QC Sample Quantities	4-7
5-1	Laboratory Sample Containers, Preservatives, and Holding Times	5-3
7-1	Target Compound List Volatile Organic Compounds and Laboratory Reporting Limits for Soil and Sediment Samples SW-846, 8260A	7-2
7-2	Target Compound List Semivolatile Organic Compounds and Laboratory Reporting Limits for Soil and Sediment Samples SW-846, 8270B	7-2
7-3	Target Compound List Pesticides/PCBs and Laboratory Reporting Limits for Soil and Sediment Samples SW-846, 8081	7-2
7-4	Nitroaromatic Compounds and Laboratory Reporting Limits for Soil and Sediment Samples SW-846, 8330	7-2
7-5	Target Analyte List Inorganic Compounds and Laboratory Reporting Limits for Soil and Sediment Samples SW-846, 6010A/7471A	7-2
9-1	Laboratory Quality Control Samples	9-1

List of Acronyms

AA	atomic absorption
ASTM	American Society for Testing and Materials
CLP	Contract Laboratory Program
COC/RFA	Chain of Custody/Request for Analysis
DNT	dinitrotoluene
DQO	data quality objective
EPA	U.S. Environmental Protection Agency
FADL	Field Activity Daily Log
GC/MS	gas chromatography/mass spectrometry
GSA	General Services Administration
HSO	health and safety officer
ICP	inductively coupled plasma spectroscopy
IT	IT Corporation
LCS	laboratory control sample
MS/MSD	matrix spike/matrix spike duplicate
NASA	National Aeronautics and Space Administrative
NIST	National Institute of Science and Technology
OEPA	Ohio Environmental Protection Agency
PBOW	Plum Brook Ordnance Works
PBS	Plum Brook Station
PCB	polychlorinated biphenyls
PE	performance evaluation
PM	project manager
POC	point-of-contact
QA	quality assurance
QAM	quality assurance manual
QAO	quality assurance officer
QAPP	quality assurance project plan
QC	quality control
RPD	relative percent difference
SAP	sampling and analysis plan
SOP	standard operating procedure
SOW	statement of work

SRM	standard reference material
SSHP	site safety and health plan
STEP	Solutions to Environmental Problems, Inc.
SVOC	semivolatile organic compound
TNT	trinitrotoluene
USACE	U.S. Army Corps of Engineers
VOC	volatile organic compound

1.0 Project Description

This quality assurance project plan (QAPP) describes the methods and procedures that will be used by IT Corporation (IT) at the former Plum Brook Ordnance Works (PBOW) (Sandusky, Ohio) site investigation at the G-8 Burning Ground to ensure quality, precision, accuracy, and completeness of the data collection during the investigation. This QAPP will be used in conjunction with the sampling and analysis plan (SAP) and the site safety and health plan (SSHP) during the site investigation. These activities will be conducted under Delivery Order 0012 of Contract DACA62-94-D-0030 with the Nashville District, U.S. Army Corps of Engineers (USACE) so that they satisfy the project objectives and comply with U.S. Environmental Protection Agency (EPA) and Ohio EPA (OEPA) requirements.

This QAPP has been prepared to provide assurance that the work to be performed will be of the quality required to satisfy the project objectives and is responsive to the applicable requirements of the EPA, the OEPA, and the Department of the Army.

1.1 Site Description

The 9,009-acre PBOW site was built in early 1941 as a manufacturing plant for 2,4,6-trinitrotoluene (TNT), dinitrotoluene (DNT), and pentolite. Production of explosives began in December 1941 and continued until 1945. It is estimated that more than one billion pounds of explosives were manufactured during the 4-year operating period.

After the plant was shut down, decontamination of TNT, acid, pentolite, and DNT processing lines began; decontamination was completed during the last quarter of 1945. The property was initially transferred to the Ordnance Department, then to the War Assets Administration after it was certified by the U.S. Army to be decontaminated. In 1949, PBOW was transferred to the General Services Administration (GSA).

National Aeronautics and Space Administration (NASA) acquired the PBOW in 1963 and is presently utilizing the site. On April 18, 1978, NASA declared approximately 2,152 acres of land as excess. The Perkins Township Board of Education acquired 46 acres of the excess and uses its area as a bus transportation center. GSA retains the remaining acreage and currently has a use agreement with the Ohio National Guard for 604 acres of the land. NASA presently controls about 6,400 acres and is using its site to conduct space research as a satellite operation of NASA's Lewis Research Center in Cleveland, Ohio. The details of

these land transactions are listed in the site management plan and can be found at the NASA Plum Brook Station (PBS).

The G-8 Burning Ground is located in the southwestern corner of the former PBOW site, east of Guard House G-8. Guard House G-8 was formerly located at the corner of Campbell Street and Patrol Road. The area of the burning pit was estimated to be approximately 2 acres. Environmental contaminants in this burning ground, according to previous investigations at the PBOW site and review of the site history, may include nitroaromatic compounds (explosive residual), volatile organic compounds (VOC), semivolatile organic compounds (SVOC), and metals.

1.2 Project Objectives

This investigation is intended to provide information concerning possible environmental contamination at the G-8 Burning Ground that can be used in making management decisions for the necessity of further work at the site. This investigation is preliminary in nature and information acquired will provide a foundation upon which other studies of the site, if deemed necessary and appropriate, should be based. Specifically, the objectives of this site investigation are:

- Define the location and the extent of the G-8 Burning Ground.
- Confirm or deny the presence of residual chemical contamination of sediment, surface, and subsurface soils.
- Determine whether or not soils are the sample media of importance and if other site media should be further evaluated.

1.3 Target Compounds

The targeted compounds for this site investigation are based on the possible contamination present in surface and subsurface soils and in sediment. The contaminants may include VOCs, SVOCs, pesticide/polychlorinated biphenyls (PCB), nitroaromatics, and metals.

Analytical parameters and methods to be used during this investigation are presented in Table 1-1.

1.4 Sample Program and Rationale

The sampling program and rationale for sample selection are presented in the SAP, Sections 4.4 and 4.5.

Table 1-1

Summary of Chemical Analysis and Methodologies for Soil and Sediment Samples Site Investigation at the G-8 Burning Ground Former Plum Brook Ordnance Works, Sandusky, Ohio

Analytical Parameters	Analytical Method
TCL Volatile Organic Compounds	EPA SW-846 8260A[a]
TCL Semivolatile Organic Compounds	EPA SW-846 3540/8270B[a]
TAL Metals	SW-846 3050A/6010A[b] for Ag, Al, As, Ba, Be, Ca, Cd, Co, Cr, Cu, Fe, K, Mg, Mn, Na, Ni, Pb, Sb, Se, Tl, V, Zn 7471A[a] for Hg
Nitroaromatic Compounds	EPA SW-846/8330[c]
TCL PCBs/Pesticides	EPA SW-846 8081[a]

[a]Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, Third Edition, Update II, September 1994.
[b]Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, Third Edition, Update I, July 1992.
[c]Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, Third Edition, Revision 1, December 1990.
TCL - Target compound list.
TAL - Target analyte list.
PCB - Polychlorinated biphenyl.

Data collected as outlined in the SAP and in this document, in conjunction with data from previous investigations, will be used in the decision-making process to determine the necessity of further action at the site. The investigative approach is discussed in detail in Chapter 4.0 of the SAP.

1.5 Schedule

Once approval for this work plan has been given, mobilization will take place in 2 weeks (depending on subcontractor availability). It is expected that field work will begin in the month of May 1996 and will take approximately 2 weeks to complete. Data analysis will then take place and a draft investigation report will be prepared, documenting the results of the site investigation work.

2.0 Project Organization

All project personnel, including field, laboratory, and subcontractor personnel are subject to the requirements of this QAPP. The key positions are described in the following sections. Figure 2-1 of the SAP shows the key project personnel and their reporting relationships.

2.1 USACE Nashville District Project Manager

The USACE Nashville District project manager (PM) will be an appointed representative of the USACE and will serve as the primary contact for the federal and state oversight agencies, as well as for the IT PM. The USACE Nashville District PM will facilitate the project from beginning to end, providing the necessary interface with the regulatory agencies and all contractors selected to support the project efforts. The USACE PM will also be responsible for the following:

- Scheduling of the site investigation activities
- Seeking the appropriate regulatory authorizations
- Seeking the required permits or certification necessary to complete site investigation
- Providing the necessary interface with the regulatory agencies as required
- Obtaining the authorization to proceed with the activities at the site
- Quality assurance review of IT's work plans
- Ultimately, for meeting the project's objectives.

2.2 IT Project Manager

The IT PM will serve as the central point of contact for government agencies, subcontractors, and other concerned parties. The IT PM will be responsible for management of the activities, ensuring compliance with the statement of work (SOW), QAPP, SSHP, and SAP. Specifically, the duties of the IT PM will be as follows:

- Assigning duties to the project staff and orienting the staff to the needs and requirements of the project
- Obtaining required approvals for proposed variances to the supplemental investigation SAP, QAPP, and SSHP

- Supervising the performance of the project staff
- Evaluating training needs of the project staff
- Providing budget and schedule control
- Reviewing subcontractor work and approving subcontractor invoices
- Establishing a project record system
- Ensuring that major project deliverables are reviewed for technical accuracy and completeness including data validity before their release
- Ensuring that the requirements of the QAPP are satisfied
- Regularly communicating the project status, progress, and any problems to the USACE project coordinator.

2.3 Quality Assurance Officer

The IT quality assurance officer (QAO) will be an independent representative reporting to the IT PM and/or upper management, responsible for ensuring compliance with the QAPP and other work-related documents. The specific duties of the QAO are as follows:

- Administering the overall QAPP
- Performing independent quality assurance (QA) audits
- Serving as the official contact for all project QA matters
- Identifying and responding to QA/quality control (QC) needs, resolving problems, and answering requests for assistance or guidance
- Reviewing, evaluating, and approving quality related changes to the QAPP, SAP, or SSHP
- Preparing and submitting QA/QC reports to the IT PM
- Verifying that appropriate corrective actions are taken for all nonconformances
- Actively tracking the progress of quality tasks in this QAPP and consulting periodically with the IT PM
- Other duties and responsibilities as assigned by the IT PM.

2.4 Health and Safety Officer

The health and safety officer (HSO) will ensure that site activities comply with the requirements of the SSHP. The HSO will have the authority to stop work, if necessary, and to implement corrective actions if SSHP requirements are not satisfied.

2.5 Analytical Services QA/QC Director

Laboratory services for this supplemental investigation will be provided by Quanterra Environmental Services, Knoxville, Tennessee.

The Quanterra QA/QC director will have the following responsibilities:

- Performing audits of the laboratory to verify compliance with the QAPP
- Verifying completion of corrective actions cited in previous audits
- Acting as the point-of-contact (POC) for proposed changes in the laboratory QAPP and initiating changes in the program
- Discussing with laboratory QC coordinators and managers any unresolved nonconformances.

2.6 Laboratory QC Coordinator

The Quanterra Laboratory QC coordinator will be responsible for implementing the requirements of this QAPP. The laboratory coordinator's responsibilities will include (as appropriate):

- Providing orientation and necessary training to laboratory personnel on the requirements of this QAPP
- Assisting the project staff in implementing sampling and testing programs
- Serving as liaison between the laboratory staff, the IT PM, task managers, and other project personnel
- Serving as the POC for laboratory staff reporting nonconformances and changes in laboratory activities
- Notifying the laboratory, QA personnel, the IT PM, and affected task managers of specific laboratory nonconformances and changes
- Maintaining laboratory data and checkprints while sampling is in progress

- Preparing and submitting QA/QC reports to the laboratory manager
- Releasing test data and results
- Calibrating laboratory equipment
- Storing and controlling samples submitted for analysis or archiving.

2.7 Data Validation Services

A Level IV data validation will be performed for 50 percent of the samples submitted to the laboratory for chemical analyses. Validation will be conducted by Solutions to Environmental Problems, Inc. (STEP), an independent subcontractor. Validation will involve data flagging, evaluation of blank data, evaluation of duplicates, and statistical evaluation of the data. The validation process references the EPA *Contract Laboratory Program National Functional Guidelines for Organic Data Review* (February 1993, EPA-540/R-94/012) and *Contract Laboratory Program (CLP) National Functional Guidelines for Inorganic Data Review* (February 1994, EPA-540/R-94/013). For parameters not specifically addressed by the EPA's CLP or validation guidelines, evaluation procedures similar to these references and the laboratory's submitted standard operating procedures (SOP) are followed.

2.8 Subcontractor Activities

The selection of qualified subcontractors will be in accordance with the IT procurement and QA procedures. Subcontractors such as drillers, geophysical specialists, surveyors, and environmental monitoring specialists must meet predetermined qualifications developed by the IT PM that are defined in the procurement bid packages. Each subcontractor bid submittal is reviewed by technical personnel, purchasing, and QA personnel to verify that the bidders are qualified and can satisfy bid requirements. Before starting work, IT will perform a quality check to determine if the subcontractor has fulfilled the procurement requirements necessary to begin activities. Subcontractors involved in environmental measurements will be monitored by the field coordinator to verify the use of calibrated equipment and qualified operators.

All subcontractors will be required to follow the procedures of the QAPP. Ongoing observation and monitoring of subcontractor performance during the field investigation will be the responsibility of the IT PM or the field coordinator. Periodic QC inspections of each subcontractor may be performed. Inspection will include (as appropriate):

- Type and conditions of equipment
- Calibration procedures
- Personnel qualifications
- Documentation.

2.9 Qualifications and Training of Personnel

Personnel assigned to the project, including field personnel and subcontractors, will be qualified to perform the tasks to which they are assigned. In addition to education and experience, specific training may be required to qualify individuals to perform certain activities. Training will be documented on the appropriate form and placed in the project file as a record. Project personnel will receive an orientation to the SAP, the SSHP, and the QAPP as appropriate to their responsibilities before participation in project activities. Training of field personnel will be provided by the field coordinator, the QA officer, or by a qualified designee. This orientation will be documented.

3.0 Quality Assurance Objectives for Measurement of Data

3.1 Data Quality Indicators

This QAPP facilitates implementation of the requirements of the SOW for the site investigation of the former Plum Brook G-8 Burning Grounds and applicable regulatory requirements principally to provide an internal means for control and review so that the environmentally related measurements and data collected by IT and its subcontractors are scientifically sound, defensible, and of known acceptable documented quality.

The procedures to be used for assessing the quality of the measurement data are as follows:

- ***Accuracy and Precision*** is the agreement between a measurement and the true value and the degree of variability in the agreement, respectively.

  To determine the precision of the method and/or laboratory analyst, a routine program of replicate analyses is performed. The results of the replicate analyses are used to calculate the relative percent difference (RPD), which is the governing QC parameter for precision.

  For replicate results $R_1 + R_2$

$$RPD = \frac{\frac{|R_1 - R_2|}{R_1 + R_2}}{2} \times 100\%$$

  To determine the accuracy, the evaluation is applied over the entire range of spiking concentrations. To determine the accuracy of an analytical method and/or the laboratory analyst, a periodic program of sample spiking is conducted (minimum 1 spike and 1 spike duplicate per 20 samples). The results of sample spiking are used to calculate the QC parameter for accuracy evaluation, the percent recovery (%R).

$$\%R = \frac{|S_1 - S_2|}{T_1} \times 100\%$$

  where:

  S_1 = Observed spiked sample concentration
  S_2 = Sample concentration
  T_1 = True concentration of the spike.

Accuracy and precision of data collected in the investigation will depend on the measurement standards used and their meticulous, competent use by qualified personnel.

- ***Completeness*** is the adequacy in quantity of valid measurements to prevent misinterpretation and to answer important questions. For this project the data completeness objective is 90 percent.

- ***Representativeness*** is the extent to which discrete measurements accurately describe the greater picture they are intended to represent.

  For this project, good representativeness will be achieved through careful, informed selection of sampling sites, drilling sites, drilling depths, and analytical parameters; and through the proper collection and handling of samples to avoid interferences and to minimize contamination and loss.

- ***Comparability*** is the extent to which comparisons among different measurements of the same quantity or quality will yield valid conclusions. For this project, comparability among measurements will be achieved through the use of standard procedures, standard field data sheets, and uniform concentration units.

- ***Quantitation Limits*** are the extent to which the equipment, laboratory or field, or analytical process can provide accurate, minimum data measurements of a reliable quality for specific constituents in replicate field samples. It is defined as the minimum concentration of a substance that can be measured and reported with 99 percent confidence that the value is above zero. The actual reporting limit for a given analysis will vary depending on instrument sensitivity, matrix effects, and dryness calculations.

- ***Traceability*** is the extent to which data can be substantiated by hard-copy documentation. Traceability documentation exists in two essential forms: one that links quantitation to authoritative standards and a second that explicitly describes the history of each sample from collection to analysis.

Typical quantitation limits applicable to this project are presented in each EPA-approved analytical method (see Section 7.0). The QA objectives for laboratory QC data are designed to screen out data of unacceptable precision or accuracy and to provide data that will meet the data quality goals for this project.

3.2 Project QA Objectives

Several procedures will be used for demonstrating the precision and accuracy objectives of this project. Sampling and analytical activities will be conducted following standard reference procedures whenever possible. All reference materials used as calibration standards or

surrogate compounds will be of the highest purity commercially available, typically greater than 99 percent. Each instrument used for the analysis will be calibrated daily before sample analyses to demonstrate accurate analytical performance levels. Absolute instrument responses will be within the ranges specified in the laboratory-specific SOP, typically within 10 percent of the most recent calibration of the instrument. Data precision and accuracy will be assessed by evaluating the results from the analysis of internal standards, laboratory blanks, calibration check standards, reagent blanks, method blanks, field and trip blanks, duplicate samples, and matrix or surrogate spiked samples. Sections 4.0 and 9.0 of this QAPP describe both the field and laboratory QC sample types which will be used to evaluate the precision and accuracy of the sampling and analytical activities associated with this project.

3.3 Analytical Support Levels

The analytical options available to support data collection activities are presented in two general levels: screening data, with definitive confirmation, and definitive data. These two data categories are associated with specific QA and QC elements, and may be generated using a wide range of analytical methods. The particular type of data to be generated depends on the qualitative and quantitative data quality objective (DQO) developed for the project. Definitive data will be required for this investigation.

Definition of Definitive Data. Definitive data are generated using rigorous analytical methods, such as approved EPA reference methods. Data are analyte-specific, with confirmation of analyte identity and concentration. Methods produce tangible raw data (e.g., chromatograms, spectra, digital values, etc.) in the form of paper printouts or computer-generated electronic files. Data may be generated at the site or at an off-site location, as long as the QA/QC requirements are satisfied. For the data to be definitive, either analytical or total measurement error must be determined.

Definitive Data QA/QC Elements

- Sample documentation (location, date and time collected, etc.)
- Chain of custody
- Sampling design approach (systematic, simple or stratified random, judgmental, etc.)
- Initial and continuing calibration

- Determination and documentation of detection limits
- Analyte(s) identification
- Analyte(s) quantification
- QC blanks (trip, method, rinsate)
- Matrix spike recoveries
- Performance evaluation (PE) samples (when specified)
- Analytical error determination (measures precision of analytical method): an appropriate number of replicate aliquots, as determined by the QAPP, are taken from at least one thoroughly homogenized sample and analyzed. From these samples, standard laboratory QC parameters such as variance, mean, and coefficient of variation should be calculated and compared to method-specific performance requirements defined in the QAPP.
- Total measurement error determination (measures overall precision of measurement system from sample acquisition through analysis): an appropriate number of colocated samples as determined by the QAPP are independently collected from the same location and analyzed following standard operating procedures. Based on these analytical results, standard laboratory QC parameters such as variance, mean, and coefficient of variation should be calculated and compared to establish measurement error goals. This procedure may be required for each matrix under investigation, and may be repeated for a given matrix at more than one location at the site.

Data Quality, Types, and Quantity. Soil and sediment will be sampled and analyzed to meet the objectives of the site investigation. QA/QC samples will be collected for all sample types according to the proportionality described in Section 4.0. All samples will be analyzed using EPA-approved methods and will comply with EPA definitive data criteria.

3.4 Laboratory QC Criteria

The primary analytical QA objective is to produce an analytical database during the data collection phase of the project that can be used to assess all site activities relative to the contracted DQOs, as well as other project-specific criteria established for the project. The specific project QC criteria for laboratory analyses are as stated in each of the individual methods.

4.0 Sampling Procedures

The site investigation SAP documents the scope and rationale of the sampling activities to be conducted at the former Plum Brook G-8 Burning Grounds. The on-site activities will include soil and sediment sampling.

The following considerations form the basis for the site-specific sampling program:

- Selection of sampling points
- Frequency of sampling
- Methods of sampling to be used
- Media to be sampled
- Number of samples to be collected
- Volume of samples to be collected
- Types and kinds of analyses to be performed in the field
- Types and kinds of analyses to be performed at the laboratories
- Sample turnaround time
- Procedures and precautions to be followed during sampling
- Methods of preservation and shipment.

Sampling will be conducted frequently enough to satisfy program objectives. The methods used during sampling will preserve the integrity of the material parameters. Field procedures for sample collection and analysis are presented in the SAP.

Any sample obtained during the field sampling should be representative of the sample location and free of contaminants from sources other than the immediate environment being sampled. The equipment and techniques that will be employed to obtain representative samples will be in accordance with procedures specified in this QAPP and the SAP.

The equipment and sample collection, preparation, and shipment techniques that will be used will be in accordance with EPA protocols. The rationale for the specific sampling program(s) are presented in the SAP.

The SAP presents sampling location design considerations; the numbers and types of samples to be collected; sampling equipment, procedures, and sample containers; methods of sample preservation; decontamination procedures; shipping and packaging methods; and a sampling schedule. Analytical tests that will be performed are also listed. Precleaned containers,

certified to meet all EPA protocols, will be purchased from a contractor for use on this project.

4.1 Prevention of Cross Contamination

All sampling equipment that will be in contact with the soil will be decontaminated before use, between each sampling point, and before leaving the site. Each decontamination activity will be recorded on the Field Activity Daily Log (FADL), as shown on Figure 5-1 of the SAP. Detailed procedures for decontamination of sampling equipment and disposal of decontamination byproducts are provided in the SAP.

4.2 Decontamination Requirements

The decontamination procedures to be used during the investigation and subsequent monitoring activities are described in the following sections. Documentation for all decontamination will be recorded in the FADLs.

4.2.1 Nonsampling Equipment

All nonsampling equipment will be decontaminated with high-pressure steam, scrubbed with a laboratory-grade nonphosphate detergent, rinsed with potable water from a source with known quality, and, if appropriate, covered with plastic sheeting to protect it from the elements.

4.2.2 Sampling Equipment

All equipment used in sample collection will be decontaminated before sampling begins and between samples. The decontamination procedure to be employed depends on the analyses to be performed and the media being collected.

The procedures to be used are as follows:

- ***Inorganic Sampling:***
  - Wash and scrub with detergent (laboratory grade).
  - Rinse with tap water.
  - Rinse with American Society for Testing and Materials (ASTM) Type II water.

- Air dry.
- Protect from fugitive dust or vapors by wrapping with plastic sheeting.

- ***Organic Sampling:***
  - Wash and scrub with detergent (laboratory grade).
  - Rinse with tap water.
  - Rinse with ASTM Type II water.
  - Rinse with methanol; follow with hexane rinse if testing for pesticides, PCBs, or fuels.
  - Air dry.
  - Wrap in aluminum foil.

Note: Detergents may be phosphatic only if phosphate is not a target analyte. ASTM Type II water as defined by Section D 1193-77 (reapproved 1983), must be used.

Solvents (methanol, hexane) used must be pesticide grade or better. Paint or coatings (such as rust) must be removed from any part of the equipment that may contact the sample.

4.3 Sample Identification

Samples will be put into sample containers that have been precleaned and treated with a preservative by the laboratory. The labels on the containers will state the type of preservative, if any, and the sample type and analysis for which the container is intended. As samples are collected and sealed in containers, the containers will be marked.

The sample numbering system to be used during this investigation will conform to the USACE Nashville District's numbering convention. Specifically, each sample will be assigned a unique sample identification number that describes where the sample was collected. Each number consists of a group of letters and numbers, separated by hyphens. The sample media and numbering system is described as follows.

Project Code	Year	Sample Type[a]	Site Number	Sample Number	Depth
PBOW	96	XX	BG8	XXXX	(XXXX)

[a]Sample type:

SD - sediment sample
SO - soil sample
TR - trench

The complete sample number will be recorded by the IT field geologist in the FADL and in the Boring Log or the Sediment Sample Collection Log.

4.4 Sample Turnaround Time

Sample analyses are based upon information received through previous investigations and will be consistent with the sample holding times required for the EPA analytical methods used for the generation of definitive data. The standard turnaround for sample analyses is 21 calendar days for hard copy and electronic delivery of the data.

4.5 Field Documentation

An integral part of the QAPP for the sampling activities will be maintaining a FADL (Figure 4-1 of the SAP). All information pertinent to field activities will be recorded on the FADLs. Entries on the logs will include, as a minimum:

- The names and affiliations of all personnel on site
- A general description of the day's field activities showing the sequence of events
- Documentation of weather conditions
- Readings from personnel safety instruments
- References to appropriate field data forms for details of each activity performed.

Appropriate field-generated data forms will be prepared based on the requirements presented in the SAP. Data to be recorded will include information such as the monitored location (e.g., boring, well, depth, sampling location, elevation, and field coordinates) and applicable sample analyses to be conducted. All handwritten documents must be recorded in such a

manner as to be completely legible. Errors are not to be obliterated, but crossed out with a single line, dated, and initialed.

Documents used to direct field activities will be controlled to ensure that they are correct and available to field personnel when the activities are conducted. Documents reporting data or results will be considered valid records only if stamped, initialed, or signed and dated by authorized personnel or otherwise authenticated.

4.6 Corrections to Documentation

All original data recorded in field log books, FADLs, sample labels, and Chain-of-Custody/Request-for-Analysis (COC/RFA) record will be written with permanent ink. None of the accountable serialized documents will be destroyed or thrown away, even if they are illegible or contain inaccuracies that require a replacement document.

If an error is made on an accountable document assigned to one individual, that individual will make all corrections simply by crossing a line through the error, entering the correct information, and initialing and dating the change. The erroneous information will not be obliterated. Any subsequent error discovered on an accountable document will be corrected by the person who made the entry. All subsequent corrections will be initialed and dated.

4.7 Variance System

Procedures that properly address all specific conditions encountered during a field program cannot be prepared. Variances from approved operating procedures presented herein will be documented on a Variance Log (Figure 4-2 of the SAP). Approval of the variance is required by the IT PM and QAO before work proceeds. The field coordinator will initiate and chronologically maintain the variances generated during the field work. Variances affecting project scope, costs, or schedule must be forwarded to the USACE PM within 48 hours of occurrence. Any variance from the SSHP must be signed off by the HSO. Approval by the IT PM can be initiated on a verbal basis via telephone with follow-up sign-off. Verbal approval will also be documented using a record-of-telephone-call memorandum. In no case will any subcontractor initiate a variance. If a variance is proposed by the client, it will be so recorded. Copies of the Variance Log will be kept on site until the field work is complete and originals will be sent to the project files.

4.8 Nonconformances and Corrective Action Procedures

Nonconforming equipment, items, activities, conditions, and unusual incidents that could affect compliance with project requirements will be identified, controlled, and reported in a timely manner. A nonconformance is defined as a malfunction, failure, deficiency, or deviation that renders the quality of an item unacceptable or indeterminate. The originator of a nonconformance report will describe the finding on the form provided for this purpose and will notify the IT PM and QAO. Each nonconformance will be reviewed and a disposition given for the item, activity, or condition. The disposition of a nonconformance will be documented and approved by the organization responsible for the issuance of the nonconformance. The QAO will concur with the disposition of the nonconformance.

The laboratory coordinator is responsible for the assessment of QC sample information. If data fall outside of the accepted limits, the QA/QC coordinator will immediately notify the laboratory PM and the responsible group leader. If the situation is not corrected, and an out-of-control situation occurs or is expected to occur, then the QAO will notify the IT PM. The IT PM and group leaders are responsible for identifying the source of the nonconformance and initiating corrective action. Completion of the corrective action should be evidenced by data returning to prescribed acceptable limits.

The modification, repair, rework, or replacement of nonconforming equipment, items, or activities will require the reverification of acceptability. In certain instances, as determined by the IT PM or QAO, these actions may require that corrective action be completed and verified before site work continues.

The equipment, item, or activity that has the deficiency may be temporarily stopped while the nonconformance is being investigated. If, in the opinion of the IT PM and the QAO, the nonconformance does not significantly affect the technical quality or use of the work, then the work may continue pending resolution of the nonconformance. The basis for such decisions will be documented on the nonconformance report and submitted to the QAO for review and approval. The documentation will include a statement that the decision was made prior to continuing the field work. The records of nonconformances and their dispositions will be kept in the project files.

In addition, the IT PM will notify the USACE Nashville District Project Coordinator within 48 hours of significant nonconformances that could impact the schedule or the SOW and will indicate the corrective action taken or planned.

4.9 Field Quality Control Samples

QC samples will be collected and analyzed by the laboratory for the purposes of assessing the quality of the project sampling and analytical efforts. The data obtained from the analysis of QC samples will be assessed with respect to all project DQOs before determining the usefulness of the project sample data.

QC samples will be collected during field sampling activities to provide a measure of QA for the project samples. The samples that will be collected include field duplicates, matrix spike/matrix spike duplicates (MS/MSD), and split samples. Table 4-1 includes a summary of the QC samples and frequency associated with each analytical parameter.

4.9.1 Field Duplicates

Field duplicate samples collected will be taken at a frequency of one per ten, or fewer, number of samples collected for laboratory analysis. All media samples will have at least one field duplicate collected. Field duplicate locations will be determined in the field by the supervising geologist or field coordinator. All field procedures for collection of samples will apply to the collection of the duplicate samples. Duplicate samples will be given a unique sample identification.

During the collection of soils for field duplicates it may be necessary to collect several samples from the sample depth to obtain the required volume of soils for duplicate samples. Soil samples to be split for VOC analysis are to be collected from the same sample run, splitting the material between the soil jars. Once the VOC sample has been collected, the remaining sample bottles shall be filled with soils that have been homogenized.

4.9.2 Matrix Spike/Matrix Spike Duplicates

To meet analytical QC requirements, MS/MSD samples will be collected for each medium sampled on a one-per-20 basis. At least one MS/MSD pair will be collected for each medium sampled. MS/MSD locations will be determined in the field by the supervising geologist or field coordinator. The MS/MSD location should be chosen so that high concentrations of contaminants are not expected.

4.9.3 Split Samples

Split samples will be collected on a one-per-ten basis collected in laboratory analysis. All media samples will have at least one split sample taken. The split sample locations will be the same as the field duplicate locations to be determined by the supervisory geologist or field

Table 4-1

Soil and Sediment Sample Designations and QA/QC Sample Quantities
Site Investigation at the G-8 Burning Ground
Former Plum Brook Ordnance Works, Sandusky, Ohio

Sample Designation	Sample Location	Sampling Depth[a] (ft)	QA/QC Sample Designation		
			Field Duplicates	Matrix Spike/ Matrix Spike Duplicate	Field Split Sample
Soil					
PBOW-96-SO01-BG8-xxxx-(0-0.5)	BG8-SB01	0-0.5'			
PBOW-96-SO01-BG8-xxxx-(2-3)		2-3'			
PBOW-96-SO02-BG8-xxxx-(0-0.5)	BG8-SB02	0-0.5'	PBOW-96-FD02-BG8-xxxx-(2-3)	PBOW-96-MS/MSD02-BG8-xxxx(2-3)	PBOW-96-FS02-BG8-xxxx-(2-3)
PBOW-96-SO02-BG8-xxxx-(2-3)		2-3'			
PBOW-96-SO03-BG8-xxxx-(0-0.5)	BG8-SB03	0-0.5'			
PBOW-96-S003-BG8-xxxx-(2-3)		2-3'			
PBOW-96-SO04-BG8-xxxx-(0-0.5)	BG8-SB04	0-0.5'			
PBOW-96-SO04-BG8-xxxx-(2-3)		2-3'			
PBOW-96-SO05-BG8-xxxx-(0-0.5)	BG8-SB05	0-0.5'			
PBOW-96-SO05-BG8-xxxx-(2-3)		2-3'			
PBOW-96-SO06-BG8-xxxx-(0-0.5)	BG8-SB06	0-0.5'			
PBOW-96-SO06-BG8-xxxx-(2-3)		2-3			
PBOW-96-SO07-BG8-xxxx-(0-0.5)	BG8-SB07	0-0.5'			
PBOW-96-SO07-BG8-xxxx-(2-3)		2-3'			

Table 4-1

Soil and Sediment Sample Designations and QA/QC Sample Quantities
Site Investigation at the G-8 Burning Ground
Former Plum Brook Ordnance Works, Sandusky, Ohio

Sample Designation	Sample Location	Sampling Depth* (ft)	QA/QC Sample Designation		
			Field Duplicates	Matrix Spike/ Matrix Spike Duplicate	Field Split Sample
Soil (continued)					
PBOW-96-SO08-BG8-xxxx-(0.0.5)	BG8-SB08	0-0.5'			
PBOW-96-SO08-BG8-xxxx-(2-3)		2-3'			
PBOW-96-SO09-BG8-xxxx-(0-0.5)	BG8-SB09	0-0.5'			
PBOW-96-SO09-BG8-xxxx-(2-3)		2-3'			
PBOW-96-SO10-BG8-xxxx-(0-0.5)	BG8-SB10	0-0.5'	PBOW-96-FD10-BG8-xxxx-(2-3)		PBOW-96-FS10-BG8-xxxx-(2-3)
PBOW-96-SO10-BG8-xxxx-(2-3)		2-3'			
PBOW-96-SO11-BG8-xxxx-(0-0.5)	BG8-SB11	0-0.5'			
PBOW-96-SO11-BG8-xxxx-(2-3)		2-3'			
PBOW-96-SO12-BG8-xxxx-(0-0.5)	BG8-SB12	0-0.5'			
PBOW-96-SO12-BG8-xxxx-(2-3)		2-3'			
Sediment					
PBOW-96-SD-BG8-xxxx	BG8-SD01	Water/sediment interface	PBOW-96-FD01-BG8-xxxx-(SD)		PBOW-96-FS01-BG8-xxxx-(SD)
PBOW-96-SD-BG8-xxxx	BG8-SD02	Water/sediment interface			

*Subsurface soil samples will be collected between depths 2 to 3 feet or at the soil/limestone interface, if encountered shallower.

coordinator. All field procedures for the collection of samples will apply to the collection of the split samples. Split samples will be given a unique sample identification.

The QA laboratory for this investigation is the USACE, Ohio River Division Laboratory (CEORD-PE-GL) located in Cincinnati, Ohio. All QA data sent to the QA laboratory will be bound and indexed. Split samples collected and submitted for QA analysis will be sent to:

U.S. Army Corps of Engineers
Ohio River Division Laboratory
Mr. John Adams
11275 Sebring Drive
Cincinnati, Ohio 45240-2714

5.0 Sample Control Procedures

Field personnel will be responsible for the identification, preservation, packaging, handling, shipping, and storage of samples obtained in the field so that all samples can be readily identified and will retain, to the extent possible, in situ characteristics to be determined through testing. All samples collected by field personnel will be documented through the use of COC/RFA forms (Figure 5-3 of the SAP).

5.1 Field Custody Procedures

Field custody procedural activity includes:

- Before sampling begins, the field coordinator will instruct site personnel in the COC procedures, as necessary.
- The quantity and types of samples and sample locations have been determined and are outlined in the SAP.
- The field coordinator determines whether proper custody procedures and report forms were used during the field work and documents results on the FADLs.
- The field coordinator has overall responsibility for the care and custody of the samples collected until they are transferred or properly dispatched to the laboratory. Each individual who collects a sample is responsible for sample custody until transferred to someone else via the COC/RFA.
- COC records initiated in the field shall be placed in a plastic bag and placed on top of the samples in the shipping container used for sample transport from the field to the laboratory.
- Additional samples that will be collected are classified as environmental or low-concentration samples.
- Shipping containers shall be secured using filament-reinforced strapping tape and custody seals to ensure that samples have not been disturbed during transport. The custody seals shall be placed on each container so that they cannot be opened without breaking the seal.
- Shipment information is recorded for shipment of samples at the end of the shift, day, or collection period on the FADL.

5.2 Sample Labeling

All physical samples for laboratory analysis obtained at the site will be placed in appropriate sample containers for shipment to the laboratory. Sample labels must contain sufficient information to uniquely identify the sample in the absence of other documentation. The following information will be included on the label:

- Project identification
- Sample identification
- Sample location
- Date of collection
- Time of collection
- Required analytical methods
- Sampler's initials.

An example sample label is shown in Figure 5-2 of the SAP.

5.3 Transfer of Custody and Shipment

Transfer of custody and shipping procedures include:

- A COC/RFA will be initiated in the field for each sample or group of samples up to ten. A copy of this record will accompany each sample shipment.

- The custody of individual sample containers will be documented by recording each container's identification on an appropriate COC/RFA.

- Each time responsibility for custody of the sample changes, the new custodian will sign the COC/RFA and note the date.

- The analyses to be performed for each sample will be recorded on a COC/RFA.

- The waybill number will be recorded on the COC/RFA when a commercial carrier is used.

- If the laboratory sample custodian judges sample custody to be invalid (e.g., samples arrive damaged or custody seals have been broken), a nonconformance report will be initiated. The IT PM will be advised immediately and the samples will not be analyzed unless the IT PM so authorizes. The IT PM, the laboratory coordinator, and QAO will be notified. The IT PM will make a decision as to the fate of the sample(s) in question on a case-by-case basis. The sample(s) will either be processed "as is" with custody failure noted along with the analytical data, or rejected with sampling rescheduled if necessary. The IT PM and QAO will sign-off the nonconformance report, noting the reason for disposition. Any problem with a sample will be noted in the appropriate data report.

- Upon sample destruction or disposal, the custodian responsible for the disposal will complete the COC/RFA, file a copy, and send a copy to the IT PM or to his designated representative for record keeping.

5.4 Preparation, Packaging, Handling, and Shipping

Samples will be placed in containers compatible with the intended analysis and properly preserved. Also, the controller of samples must consider the time interval between sample collection and sample analysis (holding time) so that the sample is representative. Table 5-1 presents the requirements for various analytical parameters with respect to the type of container, preservation method, and the maximum holding time.

Samples that are to be shipped off site for chemical analysis will be placed in ice chests containing "blue ice," or in double-bagged ice during shipment. The ice chest will be sealed, addressed, and placarded as appropriate.

As soon as field personnel have consigned samples to a commercial carrier, they shall notify the laboratory by telephone of the shipment. If the samples are transported by field personnel, then the estimated time of arrival at the laboratory should be given to the laboratory. Transportation will be scheduled to enable samples to arrive at the laboratory in time to permit testing within the established holding times.

5.5 Laboratory Receipt and Entry of Samples

Upon receipt at the laboratory, each sample is removed from the shipping container and the sample identification of the sample bottles is compared to the information contained in the COC documents. If discrepancies exist, appropriate notes (signed and dated) are made on the COC/RFA and the shipping and receiving supervisor is notified; this supervisor in turn determines if a nonconformance report needs to be initiated.

Upon receipt of samples, the following items are checked against the COC/RFA or the accompanying forms:

- The seals and tapes on the sample containers and cooler are unbroken and uncut.
- The sample containers in the cooler are intact and temperature of samples required to be preserved by cooling.
- The identification on the sample bottles corresponds to the entries on accompanying forms.

Table 5-1

Laboratory Sample Containers, Preservatives, and Holding Times
Site Investigation at the G-8 Burning Ground
Former Plum Brook Ordnance Works, Sandusky, Ohio

Matrix	Analytical Parameter	Analytical Method	Container Type & Quantity	Preservative	Holding Time
Soils and Sediments	Volatile Organics	8260A	2 x 125-mL Wide-Mouth Glass w/ Teflon-Lined Cap	Cool to 4°C	14 days from collection
	Semivolatile Organics	8270B	1 x 250-mL Wide-Mouth Amber Glass w/Teflon-Lined Cap	Cool to 4°C	Extract 14 days from collection, analyze 40 days from extraction
	Pesticides/PCB	8081			
	Metals	3050A/6010A 7471A	1 x 125-mL Wide-Mouth Glass w/Teflon-Lined Cap	Cool to 4°C	180 days from collection (Hg - 28 days) from collection
	Explosives	8330	1 x 125-mL Wide-Mouth Amber Glass w/Teflon-Lined Cap	Cool to 4°C	Extract 14 days from collection, analyze 40 days from extraction

- The number of sample containers received (i.e., bottles) is equal to the number of samples listed on the COC record or accompanying forms.

Laboratory identification numbers are stamped on stickers and securely wrapped about each sample. If samples are to be shipped from one laboratory to another, proper COC and packaging procedures will be maintained.

Individual SOPs addressing sample receiving, tracking, storage, and security have been provided by Quanterra.

5.6 Preanalysis Storage

Personnel from the appropriate laboratory group, receive, and log in the samples. These personnel have the responsibility of picking up samples that are specific to their group from shipping and receiving. The samples are then placed into temporary storage until analysis.

Samples are stored as prescribed in the laboratory-specific quality assurance manual (QAM). Methods of storage are generally intended to:

- Retard biological action.
- Retard hydrolysis of chemical compounds and complexes.
- Reduce volatility of constituents.
- Reduce adsorption effects.
- Reduce light exposure.

Preservation methods are generally limited to pH control, chemical addition, refrigeration, and freezing.

5.7 Postanalysis Storage

Anticipation of reanalysis prescribes proper environmental control. If reanalysis is not anticipated, environmental conditions are not observed, and the samples are stored at room temperature. Postanalysis environmental control of samples shall be specifically addressed in the laboratory-specific QAM. Disposal of samples will be in accordance with federal and state regulations.

5.8 Final Evidence Files

Records are prepared during the performance of the project to furnish documentary evidence of the conduct and quality of work. These records shall be controlled and retained.

5.8.1 Indexing and Filing of Project Records

Indexing and filing of records shall be performed only by designated filing personnel and maintained under the direction of the IT PM. The project record files will be organized by various project file categories.

Project files, including copies of all sampling and analytical data, are stored at the IT-Knoxville facility while the job is active. Once the job is completed and the files have been closed by the IT PM, records are transferred to off-site remote storage. Project files from the IT-Knoxville facility are managed by a local private storage company, Security Business Records Management.

5.8.2 Laboratory Analytical Project Files

Analytical data are stored by Quanterra for a period of 7 years unless specified differently by the client. Data are stored for 2 years at the laboratory facility; at the end of 2 years, records are transferred off site to a commercial records storage facility.

Evidentiary and document control/sample tracking SOPs for the Quanterra, Knoxville, Tennessee facility are provided in Appendix A.

6.0 Calibration Procedures

6.1 General Calibration Procedures and Frequency

Laboratory equipment will be identified and calibrated in accordance with the requirements of approved calibration procedures/instructions, such as those published by ASTM, EPA, or equipment manufacturers. Measuring equipment, test equipment, and reference standards will be calibrated at prescribed intervals and/or before use. Calibration frequency will be based on the analytical methods employed, the type of equipment, inherent stability, manufacturer's recommendations, values given in national standards, intended use, and experience as dictated by the laboratory-specific QAM.

In some cases, scheduled periodic calibration will not be performed because the equipment is not continuously in use. Such equipment will be calibrated before use and then at the required frequencies for as long as its use continues.

6.2 Calibration Reference Standards

Two types of reference standards may be used at the laboratories for calibration:

- Physical standards, such as weights for calibration balances and certified thermometers for calibrating working thermometers and ovens are generally used for periodic calibration.

- Chemical standards such as standard reference materials (SRM) provided by the National Institute of Science and Technology (NIST) or the EPA. These may include vendor-certified materials traceable to NIST or EPA SRMs. These are primarily used for operational calibration.

Whenever possible, physical reference standards shall have known relationships to nationally recognized standards (e.g., NIST) or accepted values of natural physical constants. If national standards do not exist, the basis for the reference standard shall be documented.

Physical reference standards shall be used only for calibration and shall be stored separately from equipment used in analyses. In general, physical reference standards shall be at least four to ten times as accurate as the requirements for the equipment that it is used to calibrate. In general, physical standards should be recalibrated every 3 years by a certified external agency.

Whenever possible, chemical reference standards shall be directly traceable to NIST SRMs. If SRMs are not available, compounds of vender-certified high purity will be used to prepare calibration standards.

6.3 Calibration Failures

Scheduled periodic calibration of testing equipment will not relieve personnel of the responsibility of employing properly functioning equipment. If an equipment malfunction is suspected, the device will be removed from service, tagged so it is not inadvertently used, and the laboratory coordinator notified, as appropriate, so that recalibration can be performed or substitute equipment can be obtained.

Equipment that can no longer be calibrated or becomes inoperable during use will be removed from service and either segregated to prevent inadvertent use or tagged to indicate that it is out of calibration. Such equipment will be repaired and recalibrated or replaced as appropriate. Any such action should be reported in the appropriate laboratory log.

6.4 Calibration Records

Records will be prepared and maintained for each piece of calibrated measuring and test equipment and each piece of reference equipment to indicate that established calibration procedures have been followed. Records demonstrating accuracy of preparation, stability, and proof of continuity of reference standards will also be maintained. Records for equipment used will be kept in the appropriate project file.

Records for periodically calibrated equipment will include, as a minimum:

- Identification number of equipment and type of equipment, or assigned unique equipment number
- Calibration frequency and acceptable tolerances
- Identification of calibration procedures used
- Date of calibration
- Identity of personnel performing the calibration
- Reference standards used for calibration
- Calibration data

- Certificates or statements of calibration provided by manufacturers and external agencies, and traceability to national standards

- Information regarding calibration acceptance or failure and any repair of failed equipment.

6.5 Operational Calibration

Operational calibration is generally performed as a part of the analytical procedure. Included may be the analysis of a method blank and the preparation of a standard response (standard calibration) curve. Operational calibration is dependent upon the instrumentation within the laboratory.

6.6 Periodic Calibration

Periodic calibration will be performed for equipment such as balances, thermometers, ovens, and furnaces that are required in analytical methods, but which are not routinely calibrated as part of the analytical procedure. Calibration requirements will be determined in the field and the laboratory depending upon the equipment used and its operating function.

7.0 Analytical Procedures

7.1 Analytical Objective

The analytical objective for this site remediation project is to provide data that most accurately reflect the constituents present at each sample location as indicated by the analytical results obtained from samples collected at each location. This objective will be met through the selection of the appropriate sample collection, sample preparation, and analytical methods. The process of selecting the analytical methods and procedures for this or any other project is based on the anticipated sample matrix, composition, required sample volume, and the analytes/compounds of interest.

7.2 Analytical Laboratories

The primary laboratory to be used for this investigation is Quanterra, Knoxville, Tennessee. Quanterra holds all necessary certifications for the execution of the analytical project.

The laboratories participating in the analytical program will be equipped with the proper analytical instrumentation necessary to complete the desired sample analysis while also meeting the project DQOs. The analysts performing the sample preparation and analysis shall be familiar with the preparation and analytical methodologies selected, proper instrument operation, instrument calibration, QC requirements, and instrument preventive maintenance. The laboratories utilized will provide a well documented analytical data package that meets or exceeds the project required deliverables. Chemical analysis for this investigation will be performed according to EPA SW-846 methodology as discussed in Section 7.4.

7.3 Analytical Instrumentation

Each instrument used to analyze samples in conjunction with this project will be set up, calibrated according to the procedures specified, and operated according to the selected analytical methodology. Instrument set-up, calibration, and operation will be documented in the run-log. Any deviations from these procedures also will be documented in the laboratory case narrative that precedes the analytical data packages of every sample delivery group. QC sample analyses will be performed according to the type and frequencies specified by each method in order to verify instrument performance periodically during routine analyses. All recommended calibration and calibration frequencies specified in the analytical methods will be met by the laboratory performing the analysis.

7.4 Analytical Methods and Procedures

The analytical methods that will be used for this project are listed in Table 1-1. Tables 7-1, 7-2, 7-3, 7-4, and 7-5 list laboratory reporting limits for all compounds and analytes of interest. Detection limits are matrix dependent and may vary from those listed in the tables.

Table 7-1

Target Compound List
Volatile Organic Compounds and Laboratory Reporting Limits for Soil and Sediment Samples SW-846, 8260A[a]
Site Investigation at the G-8 Burning Ground
Former Plum Brook Ordnance Works, Sandusky, Ohio

Compound	Reporting Limit[b] (µg/kg)
Chloromethane	10
Bromomethane	10
Vinyl Chloride	10
Chloroethane	10
Methylene Chloride	5
Acetone	20
Carbon Disulfide	10
1,1-Dichloroethene	5
1,1-Dichloroethane	5
1,2-Dichloroethene (total)	5
Chloroform	5
1,2-Dichloroethane	5
2-Butanone	20
1,1,1-Trichloroethane	5
Carbon Tetrachloride	5
Bromodichloromethane	5
1,2-Dichloropropane	5
cis-1,3-Dichloropropene	5
Trichloroethene	5
Chlorodibromomethane	5
1,1,2-Trichloroethane	5
Benzene	5
trans-1,3-Dichloropropene	5

Table 7-1

Target Compound List
Volatile Organic Compounds and Laboratory Reporting Limits for Soil and Sediment Samples SW-846, 8260A[a]
Site Investigation at the G-8 Burning Ground
Former Plum Brook Ordnance Works, Sandusky, Ohio

Compound	Reporting Limit[b] (µg/kg)
Bromoform	5
4-Methyl-2-pentanone	20
2-Hexanone	20
Tetrachloroethene	5
Toluene	5
1,1,2,2-Tetrachloroethane	5
Chlorobenzene	5
Ethyl Benzene	5
Styrene	5
Xylenes (total)	5

[a]*U.S. Environmental Protection Agency, Test Methods for Evaluating Solid Waste (SW-846), Physical/Chemical Methods,* Third Edition, Update II, September 1994.
[b]Specific reporting limits are highly matrix dependent. Reporting limits are adjusted based on dryness calculations, dilution factors, etc.

Table 7-2

Target Compound List
Semivolatile Organic Compounds and Laboratory Reporting Limits for Soil and Sediment Samples SW-846, 8270B[a]
Site Investigation at the G-8 Burning Ground
Former Plum Brook Ordnance Works, Sandusky, Ohio

Compound	Reporting Limits[b] (µg/kg)
Phenol	330
bis(2-Chloroethyl)ether	330
2-Chlorophenol	330
1,3-Dichlorobenzene	330
1,4-Dichlorobenzene	330
1,2-Dichlorobenzene	330
2-Methylphenol	330
2,2'-oxybis (1-Chloropropane)	330
4-Methylphenol	330
N-Nitroso-di-n-propylamine	330
Hexachloroethane	330
Nitrobenzene	330
Isophorone	330
2-Nitrophenol	330
2,4-Dimethylphenol	330
bis(2-Chloroethoxy) methane	330
2,4-Dichlorophenol	330
1,2,4-Trichlorobenzene	330
Naphthalene	330
4-Chloroaniline	330
Hexachlorobutadiene	330
4-Chloro-3-methylphenol	330
2-Methylnaphthalene	330
Hexachlorocyclopentadiene	1600

Table 7-2

Target Compound List
Semivolatile Organic Compounds and Laboratory Reporting Limits for Soil and Sediment Samples SW-846, 8270B[a]
Site Investigation at the G-8 Burning Ground
Former Plum Brook Ordnance Works, Sandusky, Ohio

Compound	Reporting Limits[b] (µg/kg)
2,4,6-Trichlorophenol	330
2,4,5-Trichlorophenol	330
2-Chloronaphthalene	330
2-Nitroaniline	1600
Dimethyl Phthalate	330
Acenaphthylene	330
2,6-Dinitrotoluene	330
3-Nitroaniline	1600
Acenaphthene	330
2,4-Dinitrophenol	1600
4-Nitrophenol	1600
Dibenzofuran	330
2,4-Dinitrotoluene	330
Diethyl Phthalate	330
4-Chlorophenyl-phenyl ether	330
Fluorene	330
4-Nitroaniline	1600
4,6-Dinitro-2-methylphenol	1600
N-nitrosodiphenylamine	330
4-Bromophenyl-phenyl ether	330
Hexachlorobenzene	330
Pentachlorophenol	1600
Phenanthrene	330
Anthracene	330

Table 7-2

Target Compound List
Semivolatile Organic Compounds and Laboratory Reporting Limits for Soil and Sediment Samples SW-846, 8270B[a]
Site Investigation at the G-8 Burning Ground
Former Plum Brook Ordnance Works, Sandusky, Ohio

Compound	Reporting Limits[b] (µg/kg)
Carbazole	330
Di-n-butyl Phthalate	330
Fluoranthene	330
Pyrene	330
Butyl Benzyl Phthalate	330
3,3'-Dichlorobenzidine	1600
Benzo(a)anthracene	330
Chrysene	330
bis(2-Ethylhexyl)phthalate	330
Di-n-octylphthalate	330
Benzo(b)fluoranthene	330
Benzo(k)fluoranthene	330
Benzo(a)pyrene	330
Indeno(1,2,3-cd)pyrene	330
Dibenzo(a,h)anthracene	330
Benzo(g,h,i)perylene	330

[a]*U.S. Environmental Protection Agency, Test Methods for Evaluating Solid Waste (SW-846), Physical/ Chemical Methods*, Third Edition, Update II, September 1994.
[b]Specific reporting limits are highly matrix dependent. Reporting limits are adjusted based on dryness calculations, dilution factor, etc.

Table 7-3

Target Compound List
Pesticides/PCBs and Laboratory Reporting Limits
for Soil and Sediment Samples SW-846, 8081[a]
Site Investigation at the G-8 Burning Ground
Former Plum Brook Ordnance Works, Sandusky, Ohio

Compound	Reporting Limit[b] (µg/kg)
alpha-BHC	1.7
beta-BHC	1.7
delta-BHC	1.7
gamma-BHC (Lindane)	1.7
Heptachlor	1.7
Aldrin	1.7
Heptachlor Epoxide	1.7
Endosulfan I	1.7
Dieldrin	1.7
4,4'-DDE	1.7
Endrin	1.7
Endosulfan II	1.7
4,4'-DDD	1.7
Endosulfan Sulfate	1.7
4,4'-DDT	1.7
Methoxychlor	3.3
Endrin Ketone	1.7
Endrin Aldehyde	1.7
alpha-Chlordane	1.7
gamma-Chlordane	1.7
Toxaphene	67
Aroclor-1016	33
Aroclor-1221	33
Aroclor-1232	33

Table 7-3

Target Compound List
Pesticides/PCBs and Laboratory Reporting Limits
for Soil and Sediment Samples SW-846, 8081[a]
Site Investigation at the G-8 Burning Ground
Former Plum Brook Ordnance Works, Sandusky, Ohio

(Page 2 of 2)

Compound	Reporting Limit[b] (µg/kg)
Aroclor-1242	33
Aroclor-1248	33
Aroclor-1254	33
Aroclor-1260	33

[a]*U.S. Environmental Protection Agency, Test Methods for Evaluating Solid Waste (SW-846), Physical/ Chemical Methods*, Third Edition, Update II, September 1994.
[b]Specific reporting limits are highly matrix dependent. Reporting limits are adjusted based on dryness calculations, dilution factor, etc.

Table 7-4

Nitroaromatic Compunds and Laboratory Reporting Limits for Soil and Sediment Samples SW-846, 8330[a] Site Investigation at the G-8 Burning Ground Former Plum Brook Ordnance Works, Sandusky, Ohio

Compound	Reporting Limit[b] (mg/kg)
4-Amino-2,6-dinitrotoluene	0.25
1,3-Dinitrobenzene	0.25
2,4-Dinitrotoluene	0.25
2,6-Dinitrotoluene)	0.25
HMX	0.50
Nitrobenzene	0.25
2-Nitrotoluene	0.25
3-Nitrotoluene	0.25
RDX	0.50
Tetryl	0.65
1,3,5-Trinitrobenzene	0.25
2,4,6-Trinitrotoluene	0.25

[a]*Test Methods for Evaluating Solid Waste; Physical/Chemical Methods*, Third Edition, Revision 1, December 1990.
[b]Specific reporting limits are highly matrix dependent. Reporting limits are adjusted based on dryness calculations, dilution factor, etc.

Table 7-5

Target Analyte List
Inorganic Compounds and Laboratory Reporting Limits for Soil and Sediment Samples SW-846, 6010A/7471A[a]
Site Investigation at the G-8 Burning Ground
Former Plum Brook Ordnance Works, Sandusky, Ohio

Analyte	Reporting Limit[b] (mg/kg)
Aluminum	20
Antimony	6.0
Arsenic	1.0
Barium	20
Beryllium	0.5
Cadmium	0.5
Calcium	500
Chromium	1.0
Cobalt	5.0
Copper	2.5
Iron	10
Lead	0.3
Magnesium	500
Manganese	1.5
Mercury	0.1
Nickel	4.0
Potassium	500
Selenium	0.5
Silver	1.0
Sodium	500
Thallium	1.0
Vanadium	5.0
Zinc	2.0

[a]*U.S. Environmental Protection Agency, Test Methods for Evaluating Solid Waste (SW-846), Physical/ Chemical Methods*, Third Edition, Update I, July 1992.
[b]Specific reporting limits are highly matrix dependent. Reporting limits are adjusted based on dryness calculations, dilution factor, etc.

8.0 Data Reduction, Validation, and Reporting

8.1 Data Reduction and Verification

Analytical data reduction, validation, and reporting will be performed as follows. Analytical data are generated by the gas chromatograph/mass spectrometer (GC/MS) computer software, the GC computer, the inductively coupled plasma spectroscopy (ICP) computer, atomic absorption (AA), ion chromatograph, and associated laboratory instrumentation. Examples of outputs include identifications of compounds, peak height or area, concentrations, retention times, and comparisons to standards. Outputs are in graphic form (chromatograms), bar graph (spectra), and printed tabular form in the standard formats specified for each analysis. These graphical or tabular outputs are used to calculate analyte concentrations using equations identified in the laboratory SOP for each method. Analyst will check computer output upon completion of sample analysis. If incomplete or incorrect outputs are received, corrective actions are taken according to procedures established for each type of analysis, consistent with manufacturer recommendations.

In the data review process, the data are compared to information such as the sample history, sample preparation, and QC sample data to evaluate the validity of the results. Corrective action is minimized through the development and implementation of routine internal system controls. Analysts are provided with specific criteria that must be met for each procedure, operation, or measurement system.

Laboratory data verification includes dated and signed entries by analysts and group leaders on the worksheets and logbooks used for samples, the use of sample tracking and numbering systems to track the progress of samples through the laboratory, and the use of QC criteria to reject or accept specific data.

Steps and checks used to validate precision and accuracy of the measured parameters and to support the representativeness, comparability, and completeness include:

- Description of the calibration performed
- Description of routine instrument checks (noise levels, drift, linearity, etc.)
- Documentation of the traceability of instrument standards, samples, and data
- Documentation of analytical methodology and QC methodology

- Description of the controls taken to determine and minimize interference contaminants in analytical methods (use of reference blanks and check standards for method accuracy and precision)

- MS recoveries, percent RPDs between the MS, and MSD and surrogate recoveries

- Description of routine maintenance performed

- Documentation of sample preservation and transport when shipped elsewhere.

Laboratory validation responsibilities are as follows:

- ***Level 1, Technical Data Review.*** Each laboratory analyst shall review the quality of his/her work based on an established set of guidelines. The review criteria as established in each method, in this instruction, and as stated within the laboratory QAM shall be used. The review shall at a minimum ensure that: (1) sample preparation information is correct and complete; (2) analysis information is correct and complete; (3) the appropriate SOPs have been followed; (4) analytical results are correct and complete; (5) QC samples are within established control appropriate QC limits; (6) special sample preparation and analytical requirements have been met; and (7) documentation is complete (any anomalies have been documented and forms complete, holding times documented, etc.). This data review shall be documented by using a checklist form and by signature and date of the reviewer.

- ***Level 2, Technical Review.*** The Level 2 review shall be performed by a supervisor or data review specialist whose function is to provide an independent review of the data package. This review shall also be conducted according to an established set of guidelines and is structured to ensure that: (1) all appropriate laboratory SOPs have been followed; (2) calibration data are scientifically sound, appropriate to the method, and completely documented; (3) QC samples are within established guidelines; (4) qualitative identification of sample components is correct; (5) quantitative results are correct; (6) documentation is complete and accurate (any anomalies have been documented and forms complete, etc.); (7) the data are ready for incorporation into the final report; and (8) the data package is complete and ready for data archive. Level 2 review shall be structured so that all calibration data and QC sample results are reviewed and all of the analytical results from at least 10 percent of the samples are checked back to the sample preparation and analytical bench sheets. If no problems are found with the data package, the review is complete. If any problems are found with the data package, an additional 10 percent of the sample results shall be checked back to the sample preparatory and analytical bench sheets. This cycle then repeats until either no errors are found in the data set checked or all data has been checked. All errors and corrections noted shall be documented. Level 2 data review shall also be documented on a checklist with the signature and date of the reviewer.

- ***Level 3, Administrative Data Review.*** Level 3 review is performed by the QA Officer or the program administrator at the laboratory. This review shall be similar to the review as provided in Level 2 except that it shall provide a total overview of the data package to ensure its consistency and compliance with this instruction. All errors noted shall be corrected and documented. Level 3 data review shall also be documented on a checklist with the signature and date of the reviewer.

8.2 Data Reports

The format and content of a data report depend on project needs such as client or contract requirements, government agency reporting formats, and if explanatory text is required. However, the following items are applicable to data presentation and will be included.

- The final data presentation shall be checked in accordance with data verification requirements and approved by the laboratory technical director.
- Data are presented in a tabular format whenever possible.
- Data will be formatted as a Certificate of Analysis.
- Each page of data is identified with the project number and name, date of issue, and, (if appropriate) client name.
- Data presentation include:
  - Sample identification number provided to the laboratory
  - Chemical parameters analyzed, reported values, and units of measurement
  - Detection limit of the analytical procedure if the reported value is less than the detection limit
  - Data for chemical parameters reported with consistent significant figures for samples
  - Results of QC sample analysis
  - Achieved accuracy, precision, and completeness of data if appropriate
  - Footnotes referenced to specific data if required to explain reported values
  - Analytical methods specifically referenced on all laboratory reports (any method modification will be included in the case narrative)

- Data for field QC samples reported in the same format as action samples (a modified data package consisting of QA/QC summary data sheets will be provided for all internal laboratory QC samples)

- Laboratory data stored so that complete data packages can be subsequently assembled for EPA-designated review.

The IT PM is responsible for preparing each technical report including the process of data validation.

8.3 Deliverables

The laboratory will provide the raw instrument data analytical results in a format based on the EPA CLP format or CLP-like format. These data packages will represent "stand alone" deliverables, which include sample preparation information, instrument data, parameter specific QC documentation and QC data such as method blanks, spikes, duplicates, surrogates, and internal standards, calibration and calibration check performance, and other instrument performance information. Deliverables will also include copies of the COC forms, along with validation narratives from the person(s) performing laboratory data validation.

8.4 Data Validation

A Level IV data validation will be performed on 50 percent of the samples analyzed by an independent third party. All data validations reference the EPA *Contract Laboratory Program National Functional Guidelines for Organic Data Review* (February 1993, EPA-540/R-94/012) and *Contract Laboratory Program National Functional Guidelines for Inorganic Data Review* (February 1994, EPA-540/R-94/013); for parameters that are not specifically addressed by the EPA's CLP or the validation guidelines, evaluation procedures similar to these references and the laboratory's submitted SOPs are followed.

Validation will involve data flagging, blank evaluation, evaluation of duplicates, and statistical evaluation of data.

9.0 Internal Quality Control Samples

This section addresses the QC samples that are routinely added to the normal laboratory sample stream to demonstrate that the laboratory is operating within prescribed requirements for accuracy and precision. The analytical laboratory will be required to include the following internal checks: instrument or method initial and continuing calibrations using traceable standards, duplicate analyses, surrogate spikes, MS/MSD samples, postdigestion spikes, internal standards (where appropriate), and method blanks. Table 1-1 summarizes the laboratory sample analyses and analytical method requirements for which the previously mentioned items must be maintained for this project. Table 9-1 summarizes laboratory QC samples and frequency of collection and analysis requirements. The analytical QC checks, frequencies, acceptance criteria, and corrective actions for each SW-846 analytical method or parameter are described in detail in the appropriate analytical method. SOPs for these particular methods are found in Appendix A.

9.1 Surrogate Spikes

The GC/MS and GC and high-performance liquid chromatography analytical procedures require that each sample be spiked with surrogates in order to evaluate the sample preparation method through the calculation of percent spike recoveries as a measure of accuracy. These standards are spiked onto the sample immediately before sample preparation. The surrogate spikes recommended in the SW-846 methods to be used for analysis will be spiked in the appropriate concentrations onto the project samples prior to sample preparation. Table 1-1 summarizes the analytical methods to be used in this project. The samples are prepared, analyzed, and the spike recoveries calculated. Following analyses, the surrogate recoveries will be calculated and checked against the acceptance criteria established in the analytical methods and in this QAPP. Surrogate spikes are used for volatile, semivolatile, and pesticide/PCB and explosives analyses only.

9.2 Duplicate Analyses

The intent of requesting a duplicate sample analysis will be to evaluate the variance in the applied analytical method. The collection of a composite sample will compensate for any variability in the sample components while providing adequate volume for the analysis. Duplicate samples may also be requested for samples collected during routine operations associated with project remediation. Duplicate analyses will be requested on the COC/RFA submitted to the laboratory with the samples.

Table 9-1

Laboratory Quality Control Samples
Site Investigation at the G-8 Burning Ground
Former Plum Brook Ordnance Works, Sandusky, Ohio

			Applicability			
Type	Purpose of Sample	Frequency	Inorganic	Organic	Accuracy and Precision Application	Introduced by Field Sampler/Analyst/QC Coordinator
Method Blank (Process Blank)	The analysis is performed using only the reagents and solvents used in the method. Determine cumulative interference. If interference cannot be eliminated, it must be considered when computations are performed.	With each group of samples, or beginning and end of each run. For GC/MS, method is spike with surrogates 1 out of 10.	X	X	Accuracy	Analyst
Reagent Blank	Determines the background of each reagent/solvent to be used in an analyses. Must use identical conditions to actual analyses including detection system. Background must not interfere with intended analyses.	Can be done as part of method blank; determine separately with each new batch of reagent/solvents.	X	X	Accuracy	Analyst
Duplicate	An aliquot of a sample known to analyst. Calculates relative percent difference.	1 out of 20 or at least 1/month/run.	X	X (MSD)	Precision	Analyst
Check Standard	Analysis of standard with concentrations at midpoint or low and of standard curve to verify standard calibration curve.	With each group of samples.	X		Accuracy	Analyst

Table 9-1

(Page 2 of 3)

Type	Purpose of Sample	Frequency	Applicability		Accuracy and Precision Application	Introduced by Field Sampler/Analyst/QC Coordinator
			Inorganic	Organic		
Surrogate Standards	For GC/MS and GC analysis, the addition of nonpriority pollutants as spikes in standards, method blanks, and samples. Three-point calibration curve. When used daily, determine Means and Warning and Control Limits.	All standards, method blanks, and samples.		X	Accuracy	Analyst
Standard Reference Materials	Standards prepared by recognized external agency and used to determine performance of entire system, instrumentation, and analysts.	Scheduled periodic basis such as quarter or semiannually	X	X	Accuracy	Analyst or QC Coordinator
Blank Spike (laboratory control sample)	Blank material spiked with parameters of interest. Recovery evaluated for proper execution of technique or analytical method.	1 out of 10 or at least 1/batch with MS/MSD pair.	X	X	Accuracy	Analyst
Analysis Matrix Spike	Sample or extract spiked just prior to analysis. Shows matrix effects during analysis.	As specified in methods, or as needed	X	X	Accuracy	Analyst
Internal Standards	Nonpriority pollutant spiked into sample and method blank after extraction to monitor instrument performance and sensitivity should surrogate standards indicate a problem.	Each sample and standard	GC/MS VOA and Base Neutral/Acid Analysis		Accuracy	Analyst
Initial Calibration	An analytical curve based on absorbance, emission intensity, or other measured characteristics of known standards. Data from subsequent analyses is considered valid as long as it falls within the linear range of the curve.	Instruments should be calibrated at the beginning of the project before any samples are analyzed, after each major disruption in analytical procedures, and whenever action limits are exceeded.	X	X	Accuracy	Analyst

Table 9-1

(Page 3 of 3)

			Applicability			
Type	Purpose of Sample	Frequency	Inorganic	Organic	Accuracy and Precision Application	Introduced by Field Sampler/Analyst/QC Coordinator
Continuing Calibration	Validity of the original calibration curve is confirmed.	GC/MS: Calibration should be checked at the beginning of each work shift, at least once every 12 hours (or every 10 to 12 analyses, whichever is more frequent) and after the last sampled analysis of each work shift. GC: Calibration should be checked at the beginning of each shift, every 6 hours (or every 6 samples, whichever is less frequent), and after the last sample analysis of each shift. ICP/AA: All work performed using continuing calibration. Ten percent of the samples or every 2 hours during analysis run, whichever is more frequent.	X	X	Accuracy	Analyst

9.3 Matrix Spikes and Postdigestion Spikes

Spiked samples will also include MS, MSD, and postdigestion spikes. MS will be conducted to evaluate accuracy and matrix recovery of selected compounds or analytes of interest. Post-digestion spikes will be applied to the samples receiving metals analysis. Postdigestion spikes are applied following sample preparation but prior to sample analysis. These spikes indicate any possible sample matrix effects. Specific spiking requirements are specified in the appropriate analytical methods.

9.4 Internal Standards

For volatiles and semivolatiles, internal standards are spiked after sample preparation and extraction. The data obtained from the analysis will not be used to evaluate accuracy, but rather to calculate response factors necessary for the quantitation of all compounds of interest. The recovery standards are used to calculate response factors for the internal standards, while the surrogate spikes are used to calculate response factors for the internal standards and to assess recovery of the internal standards for volatile and semivolatile compounds. The data will be reviewed for conformance with the acceptance criteria specified by the analytical method.

9.5 Method Blanks

A method blank sample will be prepared and analyzed by the laboratory in association with each batch of samples containing project samples. The method blanks will be prepared according to the same preparation methodology used for preparing the project samples. These blanks will consist of those required by the analytical method (method blanks prepared in the laboratory) to demonstrate the absence of fugitive contaminants in the laboratory reagents, materials, and glassware used during sample preparation. The resulting analytical data will be used to qualify the data obtained from project samples should the method blank indicate the presence of fugitive contaminants. Samples associated with this batch method blank will be qualified with a "B" for each analyte detected in the method blank. The method blank sample data will not be used to determine the usefulness or the acceptance of other project data. Use of this data will be restricted to notifying the analytical PM that an observed concentration detected in the project samples may be the result of fugitive contamination present in the laboratory during the preparation and analysis of samples.

9.6 Reagent Blanks

The reagent blank is an aqueous solution that is as free of analyte as possible and contains all the reagents in the same volume as used in the processing of the samples. The reagent blank

must be carried through the complete sample preparation procedure and contains the same reagent concentrations in the final solution as in the sample solution used for analysis. The reagent blank is used for determining possible contamination resulting from the preparation or processing of the sample.

9.7 Check Standard

A check standard is a material of known composition that is analyzed concurrently with test samples to evaluate a measurement process; it is an analytical standard that is analyzed to verify the calibration of the analytical system. One check standard should be analyzed with each analytical batch or every 20 samples, whichever is greater.

9.8 Standard Reference Material

SRM is a sample prepared from an independent standard at a concentration other than that used for calibration, but within the calibration range. An independent standard is defined as a standard composed of the analyte(s) of interest from a different source than that used in the preparation of standards for use in the standard curve. A QC reference sample is intended as an independent check of technique, methodology, and standards. This is applicable to all organic and inorganic analyses.

9.9 Blank Spike (Laboratory Control Samples)

A laboratory control sample (LCS) is a blank spiked with the parameters of interest or is a matrix of known parameter concentrations that is carried through the entire analytical procedure being used to analyze project samples. Analysis of this sample with acceptable recoveries demonstrates that the laboratory techniques for this method are in control. Where required, this sample will be analyzed in conjunction with MS/MSD samples. If the MS/MSD pair shows poor recoveries due to interferences of matrix effects, but the LCS is acceptable, strong evidence exists that the method has been performed correctly by the laboratory, but the sample matrix has affected the sample results.

9.10 Analytical Instrument Calibration

The analytical instrumentation used in the laboratory for analysis of project samples will undergo rigorous checks and re-checks of performance. Prior to sample analysis, initial and continuing calibrations will be performed to determine linearity of response versus concentration of known amounts of the analytes of interest. If acceptance criteria, as specified in the appropriate analytical methods for initial and/or continuing calibrations are not met, sample analysis will not proceed until the analytical problem has been corrected and the criteria met.

All reported sample results must be bracketed by two successful continuing calibration standards. Linearity checks will be used to verify that response has not shifted significantly from the most recent calibration. Additionally, internal standards will be analyzed to evaluate instrument and method performance.

10.0 Performance and System Audits

An individual audit plan will be developed to provide a basis for each audit. This plan will identify the audit scope, activities to be audited, audit personnel, any applicable documents, and the schedule. Checklists will be prepared by the auditors and used to conduct all audits. They will be developed to accomplish the necessary reviews and to document the results of the audit.

Audits may involve an on-site visit by the auditor. Items to be examined may, as appropriate, include the availability and implementation of approved work procedures, calibration and operation of equipment; packaging, storage, and shipping of samples obtained; performance documentation; and nonconformance documentation.

The records of operations will be reviewed to verify that laboratory and field-related activities were performed in accordance with appropriate approved procedures. Items reviewed will include, but will not be limited to, the calibration records of equipment, FADLs, COC documentation, and data resulting from field and laboratory operations.

10.1 Audit Schedule and Records

Audits will be conducted as the scheduled activities dictate. Audits will cover both field work as well as other project and laboratory actions. The QA officer is responsible for scheduling and performing audits. Within 20 working days of completion of an audit, the QA officer and/or his representative will prepare and submit an audit report. The report will be addressed to the IT PM and copies will be sent to the organization or group audited.

Within 30 working days after receipt of the audit report, the IT PM will prepare and submit to the QA officer, a reply to the audit. This reply will include, as a minimum, a plan for implementing the corrective action to be taken on nonconformances indicated in the audit report, the date by which such corrective action will be completed, and actions taken to prevent reoccurrence. If the corrective action has been completed, supportive documentation should be attached to the reply. The QA officer will ascertain (by reaudit or other means) whether appropriate and timely corrective action has been taken. Reaudits will be conducted and reported in the same manner as the original audit.

Records of audits will be maintained in the project files. Audit files will include, as a minimum, the audit report, the reply to audit, and any supporting documents. It is the responsibility of the IT PM to conform to the established procedures, particularly with timely replies to audit reports and implementation of such corrective action as may be indicated.

10.2 Performance and System Audits

Audits will be performed to review and evaluate the adequacy of laboratory performance, and to ascertain if the QAPP is being completely and uniformly implemented. The laboratory QC coordinator is responsible for such audits and will perform them according to a schedule planned to coincide with appropriate activities on the project schedule and sampling plans. Such scheduled audits may be supplemented by additional audits for one or more of the following reasons:

- When significant changes are made in the QAPP
- When it is necessary to verify that corrective action has been taken on a nonconformance reported in a previous audit
- When requested by the IT PM or laboratory manager.

The objectives of performance and systems audits are to verify that the QAPP developed for this project is being implemented according to the specified requirements, to assess the effectiveness of the QAPP, to identify nonconformances, and to verify that identified deficiencies are corrected. Upon discovery of any significant deviation from the QAPP, the IT PM shall be informed of the nature, extent, and corrective action taken to remedy the deviation.

In addition to these internal audits, surveillance of selected activities may be performed periodically.

10.2.1 Performance Audits

A performance audit can be defined as a review of the existing project and QC data to determine the accuracy of a total measurement system(s) or a component part of the system. The analysis of laboratory PE samples and the participation in scheduled interlaboratory studies may be included as part of the performance audit.

The laboratory will perform monthly internal audits covering all laboratory functions and activities. Performance audit reporting will be summarized in a monthly report to the technical director and the IT PM.

10.2.2 System Audits

A systems audit consists of an evaluation to determine if the components of a measurement system(s) were properly selected and are being used correctly. A system audit includes a careful evaluation of field and laboratory QC procedures.

System audits will be conducted on a semiannual basis by the QA/QC director. The system audit will be reported in formal audit reports.

10.2.3 Field Audits

Field audits can be performed either by internal personnel or by external personnel. An individual audit plan will provide a basis for each field audit. This plan will identify the audit scope, activities to be audited, audit personnel, any applicable documents, and the audit schedule. Checklists will be prepared by the auditors and used to conduct all audits. They will be developed to accomplish the review of necessary items and to document the results of the audit.

A field operations audit may involve an on-site visit by the auditor. Items to be examined may, as appropriate, include the availability and implementation of approved work procedures; calibration and operation of equipment; packaging, storage, and shipping of samples obtained; documentation procedures and instructions; and nonconformance documentation.

The records of field operations will be reviewed to verify that field-related activities were performed in accordance with appropriate procedures. Items reviewed may include, but are not limited to, the calibration records of field equipment, FADLs, COC/RFAs, and data resulting from field operations.

Field audits will be conducted during the field work. Field audit reports will be reported in formal audit reports distributed by the IT PM.

During an audit and upon its completion, the auditor(s) will discuss the findings with the individuals audited and discuss and agree on corrective actions to be initiated.

Minor administrative findings that can be resolved to the satisfaction of the auditors during an audit are not required to be cited as items requiring corrective action. Findings that are not resolved during the course of the audit and findings affecting the overall quality of the project will be noted on the audit checklists and included in the audit report.

11.0 Preventive Maintenance Procedures

11.1 Field Instruments and Equipment

Periodic preventive maintenance is required for all sensitive equipment. Instrument manuals will be kept on file for reference purposes should equipment need repair. The troubleshooting section of factory manuals may be used in assisting personnel in performing maintenance tasks. The frequency of preventive maintenance for field equipment is usually indicated in each operating instruction manual. Otherwise, the contractor will indicate when routine maintenance checks are necessary so that failures in the field can be minimized.

11.2 Laboratory Instruments

Preventive maintenance is an organized program of actions, such as equipment cleaning, lubricating, reconditioning, adjustment, and/or testing, taken to maintain proper instrument and equipment performance and to prevent instruments and equipment from failing during use. An adequate preventive maintenance program increases reliability of a measurement system.

The implementation of a preventive maintenance program is dependent upon the specific instruments and equipment used within the laboratories.

The laboratory manager is responsible for preparation and documentation of the program. All of the analysts will carry out the program, and the laboratory QC coordinator will review implementation to verify compliance. The laboratory preventive maintenance program will include the following:

- A listing of the instruments and equipment that are included in the program
- The frequency of maintenance considering the manufacturers' recommendations and/or previous experience with the equipment. The listing and maintenance frequency will be provided on a schedule, and the frequency will be stated in terms of monthly, quarterly, etc.
- A list of spare parts maintained by the laboratory for each instrument
- Documentation of external service contracts
- Items to be checked and/or serviced during maintenance and directions for performing maintenance (if external service is not provided, or if not stated in the manufacturers' instrument manuals).

Preventive maintenance records will be stored in the laboratory's central filing system. The master schedule will be kept at the beginning of the maintenance record. Behind the schedule, a separate file will be maintained for each instrument to include:

- Spare parts list
- External service contracts
- Checklist of items to be serviced and directions for maintenance or manufacturers' instrument manuals
- Record/results of periodic maintenance.

12.0 Assessment Procedures for Accuracy, Precision, and Completeness

The QA activities implemented in this study will provide a basis for assessing the accuracy and precision of the analytical measurements. Section 3.0 discusses the QA activities that will generate the accuracy and precision data for each sample type. The generalized form of the equations that will be used to calculate accuracy and precision are as follows:

Accuracy

$$\textit{Percent Recovery} = \left[\frac{(X-S)}{T}\right] \times 100$$

where:

X = experimentally determined concentration
T = true concentration of the spike
S = sample concentration before spiking

Precision

$$\textit{Relative Percent Difference (RPD)} = \left[\frac{|D_1 - D_2|}{\left(\frac{D_1 + D_2}{2}\right)}\right] \times 100$$

where:

D_1 and D_2 = results of duplicate measurements

or as the standard deviation relative to the average value expressed as:

$$\textit{Relative Standard Deviation (\% RSD)} = \left(\frac{\sigma_{(n-1)}}{\bar{X}\,(x_1..x_n)}\right) \times 100$$

where:

$\sigma_{(n-1)}$ = sample standard deviation of the sample data
n = number of duplicates
$\bar{X}\,(x_1..x_n)$ = arithmetic mean of the sample data

Completeness. Data completeness is a measure of the extent to which the database resulting from a measurement effort fulfills objectives for the amount of data required. For this remediation project, completeness will be defined as the percentage of valid data obtained from the total project. Completeness is assessed by the following equation:

$$\textit{Completeness, \%} = \left(\frac{D_r}{D_c} \right) \times 100$$

where:

D_r = number of samples for which valid results are reported
D_c = number of valid samples that are collected and reach the laboratory for analysis.

The completeness objective will help to evaluate the accuracy and precision of the analytical measurements.

13.0 Corrective Action

The need for corrective action occurs when a circumstance arises that threatens the quality of the data output. For corrective action to be initiated, awareness of a problem must exist. In most instances, the personnel conducting the field work and the laboratory analysis are in the best position to recognize problems that will affect data quality. Awareness on their part can frequently detect minor instrument changes, drifts, or malfunctions, which can then be corrected, thus preventing a major breakdown of the system. If major problems arise, personnel are in the best position to decide upon the proper corrective action and initiate it immediately, thus minimizing data loss. Therefore, the field sampling and laboratory analysis personnel will have a prime responsibility for recognizing the need for a nonconformance. Each nonconformance shall be documented by the personnel identifying or originating it. For this purpose, a variance log, testing procedure record, notice of equipment calibration failure, results of laboratory analysis QA tests, audit report, internal memorandum, or letter shall be used as appropriate. Documentation shall include:

- Identification of the individual(s) identifying or originating the nonconformance
- Description of the nonconformance
- Any required approval signatures
- Method(s) for correcting the nonconformance (corrective action) or description of the variance granted
- Schedule for completing corrective action.

Documentation in the form of a nonconformance report shall be made available to project and laboratory management and the QA/QC coordinator. It is the responsibility of the IT PM, laboratory analysis coordinator, and/or QA/QC coordinator to notify appropriate personnel of the nonconformance. Samples affected will be listed on the nonconformance report.

Decisions on whether to take corrective action and what action(s) to take will be made by the IT PM or the QA/QC coordinator. When a corrective action is taken by any of the operations or analytical laboratory personnel, they will be responsible for notifying the QA/QC coordinator so that, if deemed necessary, QA surveillance of the affected sampling or analysis system can be intensified. Nonconformance and corrective action reports will become part of the final report submittals or the supporting data files.

A second recognition level of the need for corrective action will be determined by the QA/QC coordinator, who will determine the need for corrective action from the results of the audits described in Section 10.0 and from review of the QA data generated during the study. The QA/QC coordinator will be responsible for initiating corrective action by immediately notifying the analytical PM during the sample analysis phase. The appropriate management will then be responsible for instituting corrective action and verifying that the corrective actions produce the desired results.

Ultimately, the personnel performing and checking the sampling and analysis procedures and results must participate in decisions to take corrective actions. To reach the proper decision, each individual must understand the program objectives and data quality required to meet the objectives established in the QAPP. DQOs for this program are presented in Section 3.0. Personnel involved in the project will receive or have available to them an approved copy of this QAPP and will be informed of these objectives. The individuals performing the analyses will have the responsibility to notify the analytical PM whenever a measurement system is not yielding data within these objectives. The analytical PM will then notify the IT PM of the problem.

If a situation arises requiring corrective action, the following closed-loop corrective action system will be used:

- Define the problem.
- Assign responsibility for investigating the problem.
- Investigate and determine the cause of the problem.
- Determine corrective action course to eliminate the problem.
- Assign responsibility for implementing the corrective action.
- Determine the effectiveness of the corrective action and implement the correction.
- Verify that the corrective action has eliminated the problem.
- If not completely successful, loop back to first step.

14.0 QA Reports to Management

Fundamental to the success of this QAPP is the active participation of management in the project. Management will be aware of project activities and will participate in development, review, and operation of the project. Management will be informed of QA activities through the receipt, review, and/or approval of:

- Project-specific QA project plans
- Corporate and project-specific QA/QC plans and procedures
- Post audit reports and audit closures
- Corrective action overdue notices
- Nonconformance reports.

Section 10.0 describes the performance and systems audits process to be used during the project. In addition, periodic assessment of QA/QC activities and data accuracy, precision, and completeness will be conducted and reported by the laboratory.

APPENDIX A

QUANTERRA STANDARD OPERATING PROCEDURES

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QUANTERRA STANDARD OPERATING PROCEDURE

TITLE: DETERMINATION OF VOLATILE ORGANICS BY GC/MS BASED ON METHODS 8240B AND 8260A

(SUPERSEDES: NONE)

Prepared by: Richard Burrows

Reviewed by: _____
Technology Standardization Committee

Approved by: _____
Director, Quality Assurance

Approved by: _____
Director, Environmental Health and Safety

Approved by: _____
Management

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TABLE OF CONTENTS

1. SCOPE AND APPLICATION 4

2. SUMMARY OF METHOD 4

3. DEFINITIONS 5

4. INTERFERENCES 7

5. SAFETY 7

6. EQUIPMENT AND SUPPLIES 8

7. REAGENTS AND STANDARDS 11

8. SAMPLE COLLECTION, PRESERVATION AND STORAGE 12

9. QUALITY CONTROL 13

10. CALIBRATION AND STANDARDIZATION 17

11. PROCEDURE 20

12. DATA ANALYSIS AND CALCULATIONS 25

13. METHOD PERFORMANCE 31

14. POLLUTION PREVENTION 31

15. WASTE MANAGEMENT 32

16. REFERENCES 32

17. MISCELLANEOUS 32

APPENDIX A

APPLICATION OF METHOD 8260A TO THE DRINKING WATER ANALYTE LIST 49

LIST OF TABLES

Table 1 Quanterra Primary Standard and Reporting Limits

Table 2 Quanterra Primary Standard Calibration Levels

Table 3 Quanterra Appendix IX Standard and Reporting Limits

Table 4 Quanterra Appendix IX Standard Calibration Levels

Table 5 Reportable Analytes for Quanterra Standard Tests, Primary Standard

Table 6 Reportable Analytes for Quanterra Standard Tests, Appendix IX Standard

Table 7 Internal Standards

Table 8 Surrogate Standards

Table 9 Matrix Spike / LCS Standards

Table 10 BFB Tune Criteria

Table 11 SPCC Compounds and Minimum Response Factors

Table 12 CCC Compounds

Table 13 Characteristic Ions

Table 14 Quanterra 8260A Drinking Water List Standard and Reporting Limits

Table 15 Internal Standards, Method 8260A Drinking Water List

Table 16 Surrogate Standards, Method 8260A Drinking Water List

Table 17 Calibration Levels, Method 8260A Drinking Water List

1. SCOPE AND APPLICATION

1.1. This method is applicable to the determination of Volatile Organic Compounds in waters, wastewaters, soils, sludges and other solid matrices. Standard analytes are listed in Tables 5 and 6.

1.2. This SOP is applicable to methods 8240B (capillary column) and 8260A.

1.3. This method can be used to quantify most volatile organic compounds that have boiling points below 200°C and are insoluble or slightly soluble in water. Volatile water soluble compounds can be included in this analytical technique; however, for more soluble compounds, quantitation limits are approximately ten times higher because of poor purging efficiency.

1.4. The method is based upon a purge and trap, gas chromatograph/mass spectrometric (GC/MS) procedure. Directions are provided for analysis based on methods 8240B and 8260A. The approximate working range is 5 to 200 µg/L for 5 mL waters, 1 to 60 µg/L for 25 mL purge waters, 5 to 200 µg/kg for low-level soils, and 630 to 25,000 µg/kg for medium-level soils. Reporting limits are listed in Tables 1, 3 and 14.

1.5. Method performance is monitored through the use of surrogate compounds, matrix spike/matrix spike duplicates, and laboratory control spike samples.

2. SUMMARY OF METHOD

2.1. The differences between method 8240B and 8260A as performed at Quanterra are summarized here.

2.1.1. Method 8240 was written as a packed column method; however, Quanterra has modified the method to use capillary columns for improved performance. Accordingly, when the surrogates and internal standards listed in this SOP are used, methods 8240B and 8260A are similar except for the minimum response factors listed in Table 11. ***Both*** methods may use 5 or 25 mL purge volumes, depending on the detection limits required, and both methods have the same calibration criteria.

2.1.2. When a method 8260A analysis is requested, a target compound list based on that contained in method 524.2 is frequently requested. One of the target compounds, bromochloromethane, is a method 8240B internal standard, so alternative surrogates and internal standards must be used. Quanterra's standard analyte list, surrogates and internal standards for method 8260A (drinking water analyte list) are listed in Appendix A, Tables 14, 15, and 16.

2.2. The volatile compounds are introduced into the gas chromatograph by the purge and trap method. The components are separated via the chromatograph and detected using a mass spectrometer which is used to provide both qualitative and quantitative information.

2.3. If the above sample introduction techniques are not applicable, a portion of the sample is dispersed in methanol to dissolve the volatile organic constituents. A portion of the methanolic solution is combined with water in a purging chamber. It is then analyzed by purge and trap.

2.4. In the purge and trap process, an inert gas is bubbled through the solution at ambient temperature (40°C for soils) and the volatile components are efficiently transferred from the aqueous phase to the vapor phase. The vapor is swept through a sorbant column where the volatile components are trapped. After purging is completed, the sorbant column (trap) is heated and backflushed with inert gas to desorb the components onto a gas chromatographic column. The gas chromatographic column is then heated to elute the components which are detected with a mass spectrometer.

2.5. Qualitative identifications are confirmed by analyzing standards under the same conditions used for samples and comparing the resultant mass spectra and GC retention times. Each identified component is quantified by relating the MS response for an appropriate selected ion produced by that compound to the MS response for another ion produced by an internal standard.

3. DEFINITIONS

3.1. Batch

The batch is a set of up to 20 samples of the same matrix processed using the same procedures and reagents within the same time period. Using this method, each BFB analysis will normally start a new batch. In the event that there is no delay (< 2 hours) between the last sample of one 12 hour analysis period and the BFB analysis that starts a new 12 hour analysis period, *and* no changes to instrumental parameters are made, a batch may extend for a maximum of 24 hours. Batches for medium level soils are defined at the sample preparation stage and may be analyzed on multiple instruments over multiple days, although reasonable effort should be made to keep the samples together.

3.1.1. The Quality Control batch must contain a matrix spike/spike duplicate (MS/MSD), a Laboratory Control Sample (LCS), and a method blank. In some cases, at client request, the MS/MSD may be replaced with a matrix spike and sample duplicate. Refer to the Quanterra QC Program document (QA-003) for further details of the batch definition.

3.2. Method Blank

A method blank consisting of all reagents added to the samples must be analyzed with each batch of samples. The method blank is used to identify any background interference or contamination of the analytical system which may lead to the reporting of elevated concentration levels or false positive data.

3.3. Laboratory Control Sample (LCS)

Laboratory Control Samples are well characterized, laboratory generated samples used to monitor the laboratory's day-to-day performance of routine analytical methods. The LCS, spiked with a group of target compounds representative of the method analytes, is used to monitor the accuracy of the analytical process, independent of matrix effects. Ongoing monitoring of the LCS results provides evidence that the laboratory is performing the method within accepted QC guidelines for accuracy and precision.

3.4. Surrogates

Surrogates are organic compounds which are similar to the target analyte(s) in chemical composition and behavior in the analytical process, but which are not normally found in environmental samples. Each sample, blank, LCS, and MS/MSD is spiked with surrogate standards. Surrogate spike recoveries must be evaluated by determining whether the concentration (measured as percent recovery) falls within the required recovery limits.

3.5. Matrix Spike/Matrix Spike Duplicate (MS/MSD)

A matrix spike is an environmental sample to which known concentrations of target analytes have been added. A matrix spike duplicate is a second aliquot of the same sample which is prepared and analyzed along with the sample and matrix spike. Matrix spikes and duplicates are used to evaluate accuracy and precision in the actual sample matrix.

3.6. Calibration Check Compound (CCC)

CCCs are a representative group of compounds which are used to evaluate initial calibrations and continuing calibrations. Relative percent difference for the initial calibration and % drift for the continuing calibration response factors are calculated and compared to the specified method criteria.

3.7. System Performance Check Compounds (SPCC)

SPCCs are compounds which are sensitive to system performance problems and are used to evaluate system performance and sensitivity. A response factor from the continuing calibration is calculated for the SPCC compounds and compared to the specified method criteria.

4. INTERFERENCES

4.1. Method interferences may be caused by contaminants in solvents, reagents, glassware, and other processing apparatus that lead to discrete artifacts. All of these materials must be routinely demonstrated to be free from interferences under conditions of the analysis by running laboratory method blanks as described in the Quality Control section. The use of ultra high purity gases, pre-purged purified reagent water, and approved lots of purge and trap grade methanol will greatly reduce introduction of contaminants. In extreme cases the purging vessels may be pre-purged to isolate the instrument from laboratory air contaminated by solvents used in other parts of the laboratory.

4.2. Samples can be contaminated by diffusion of volatile organics (particularly methylene chloride and fluorocarbons) into the sample through the septum seal during shipment and storage. A field blank prepared from reagent water and carried through the sampling and handling protocol can serve as a check on such contamination.

4.3. Matrix interferences may be caused by non-target contaminants that are coextracted from the sample. The extent of matrix interferences will vary considerably from source to source depending upon the nature and diversity of the site being sampled.

4.4. Cross-contamination can occur whenever high-level and low-level samples are analyzed sequentially or in the same purge position on an autosampler. Whenever an unusually concentrated sample is analyzed, it should be followed by one or more blanks to check for cross-contamination. The purge and trap system may require extensive bake-out and cleaning after a high-level sample.

4.5. Some samples may foam when purged due to surfactants present in the sample. When this kind of sample is encountered an antifoaming agent (e.g., J.T. Baker's Antifoam B silicone emulsion) can be used. A blank spiked with this agent must be analyzed with the sample because of the non-target interferences associated with the agent.

5. SAFETY

5.1. Procedures shall be carried out in a manner that protects the health and safety of all Quanterra associates.

5.2. The Chemical Hygiene Plan (CHP) gives details about the specific health and safety practices which are to be followed in the laboratory area. Personnel must receive training in the CHP, including the written Hazard Communication plan, prior to working in the laboratory. Consult the CHP, the Quanterra Health and Safety Policies and Procedures Manual, and available Material Safety Data Sheets (MSDS) prior to using the chemicals in the method.

5.3. Consult the Quanterra Health and Safety Policies and Procedures Manual for information on Personal Protective Equipment. Eye protection that satisfies ANSI Z87.1 (as per the Chemical Hygiene Plan) and a laboratory coat must be worn in the lab. Appropriate gloves must be worn while samples, standards, solvents, and reagents are being handled. Disposable gloves that have been contaminated will be removed and discarded; other gloves will be cleaned immediately. Disposable gloves shall not be reused.

5.4. The health and safety hazards of many of the chemicals used in this procedure have not been fully defined, therefore each chemical compound should be treated as a potential health hazard. Additional health and safety information can be obtained from the MSDS files maintained in the laboratory. The following specific hazards are known:

5.4.1. Chemicals that have been classified as carcinogens, or potential carcinogens, under OSHA include: Acrylonitrile, benzene, carbon tetrachloride, chloroform, 1,2-dibromo-3-chloropropane, 1,4-dichlorobenzene, and vinyl chloride.

5.4.2. Chemicals known to be flammable are: Methanol.

5.5. Exposure to chemicals must be maintained **as low as reasonably achievable**, therefore, unless they are known to be non-hazardous, all samples should be opened, transferred, and prepared in a fume hood, or under other means of mechanical ventilation. Solvent and waste containers will be kept closed unless transfers are being made.

5.6. The preparation of standards and reagents will be conducted in a fume hood with the sash closed as far as the operations will permit.

5.7. All work must be stopped in the event of a known or potential compromise to the health and safety of a Quanterra associate. The situation must be reported **immediately** to a laboratory supervisor.

5.8. Laboratory personnel assigned to perform hazardous waste disposal procedures must have a working knowledge of the established procedures and practices outlined in the Quanterra Health and Safety Manual. These employees must have training on the hazardous waste disposal practices initially upon assignment of these tasks, followed by an annual refresher training.

6. EQUIPMENT AND SUPPLIES

6.1. Microsyringes: 10 µL and larger, 0.006 inch ID needle.

6.2. Syringe: 5 mL glass with luerlok tip, if applicable to the purging device.

6.3. Balance: Analytical, capable of accurately weighing 0.0001 g, and a top-loading balance capable of weighing 0.1 g

6.4. Glassware:

6.4.1. Vials: 20 mL with screw caps and Teflon liners.

6.4.2. Volumetric flasks: 10 mL and 100 mL, class A with ground-glass stoppers.

6.5. Spatula: Stainless steel.

6.6. Disposable pipets: Pasteur.

6.7. pH paper: Wide range.

6.8. Gases:

6.8.1. Helium: Ultra high purity, gr. 5, 99.999%.

6.8.2. Compressed air: Used for instrument pneumatics.

6.8.3. Liquid nitrogen: Used for cryogenic cooling if necessary.

6.9. Purge and Trap Device: The purge and trap device consists of the sample purger, the trap, and the desorber.

6.9.1. Sample Purger: The recommended purging chamber is designed to accept 5 mL samples with a water column at least 3 cm deep. The purge gas must pass through the water column as finely divided bubbles, each with a diameter of less than 3 mm at the origin. The purge gas must be introduced no more than 5 mm from the base of the water column. Alternative sample purge devices may be used provided equivalent performance is demonstrated.

6.9.2. Trap: The trap should be at least 25 cm long and have an inside diameter of at least 0.105 inch. Starting from the inlet, the trap should contain the following amounts of absorbents: 1/3 of 2,6-diphenylene oxide polymer (Tenax-GC, 60/80 mesh or equivalent), 1/3 of silica gel (Davison Chemical, 35/60 mesh, grade 15, or equivalent), and 1/3 coconut charcoal. It is recommended to use a trap that also 1.0 cm methyl silicone packing at the inlet to extend its life. If it is not necessary to analyze for any fluorocarbons then the charcoal phase can be replaced with the polymer. Other traps, such as Supelco's Vocarb 3000 and 4000, may be used if the Quality Control criteria are met.

6.9.3. Desorber: The desorber should be capable of rapidly heating the trap to 180°C. Many such devices are commercially available.

6.9.4. Sample Heater: A heater or heated bath capable of maintaining the purge device at 40°C is used for low level soil analysis only.

6.10. Gas Chromatograph/Mass Spectrometer System:

6.10.1. Gas Chromatograph: The gas chromatograph (GC) system must be capable of temperature programming. The system must include or be interfaced to a purge and trap device. All GC carrier gas lines must be made from stainless steel or copper tubing.

6.10.2. Gas Chromatographic Columns: Capillary columns are used. Some typical columns are listed below:

6.10.2.1. Column 1: 105m x 0.53 ID Rtx-624 with 3 µm film thickness.

6.10.2.2. Column 2: 75 m x 0.53 ID DB-624 widebore with 3 µm film thickness.

6.10.3. Mass Spectrometer: The mass spectrometer must be capable of scanning 35-300 AMU every two seconds or less, using 70 volts electron energy in the electron impact mode and capable of producing a mass spectrum that meets the required criteria when 50 ng of 4-Bromofluorobenzene (BFB) are injected onto the gas chromatograph column inlet.

6.10.4. GC/MS interface: In general glass jet separators are used but any interface that achieves all acceptance criteria may be used.

6.10.5. Data System: A computer system that allows the continuous acquisition and storage on machine readable media of all mass spectra obtained throughout the duration of the chromatographic program. The computer must have software that allows searching any GC/MS data file for ions of a specified mass and plotting such ion abundances versus time or scan number. This type of plot is defined as an Extracted Ion Current Profile (EICP). Software must also be available that allows integrating the abundances in any EICP between the specified time or scan-number limits. Also, for the non-target compounds, software must be available that allows for the comparison of sample spectra against reference library spectra. The most recent release of the NIST/EPA mass spectral library should be used as the reference library. The computer system must also be capable of backing up data for long-term off-line storage.

6.10.6. Cryogenic Cooling: Some columns require the use of liquid nitrogen to achieve the subambient temperature required for the proper separation of the gases.

7. REAGENTS AND STANDARDS

7.1. Reagents

7.1.1. Methanol: Purge and Trap Grade, High Purity

7.1.2. Reagent Water: High purity water that meets the requirements for a method blank when analyzed. (See section 9.4) Reagent water may be purchased as commercial distilled water and prepared by purging with an inert gas overnight. Other methods of preparing reagent water are acceptable.

7.2. Standards

7.2.1. Calibration Standard

7.2.1.1. Stock Solutions: Stock solutions may be purchased as certified solutions from commercial sources or prepared from pure standard materials as appropriate. These standards are prepared in methanol and stored in Teflon-sealed screw-cap bottles with minimal headspace at -10° to -20°C. Stock standards for gases must be replaced at least every week. Other stock standards must be replaced at least every 6 months.

7.2.1.2. Secondary dilution standards: A working solution containing the compounds of interest prepared from the stock solution(s) in methanol. These standards are stored with minimal headspace and monitored for signs of degradation or evaporation, especially just prior to preparing calibration standards from them. The standards are stored for a maximum of one week.

7.2.1.3. Aqueous Calibration Standards are prepared in reagent water using the secondary dilution standards. These aqueous standards must be prepared daily.

7.2.1.4. If stock or secondary dilution standards are purchased in sealed ampoules they may be used up to the manufacturers expiration date. Once the ampoule is opened the expiration dates in Section 7.2.1.1 become effective.

7.2.2. Internal Standards: Internal standards are added to all samples, standards, and blank analyses. Refer to Table 7 for internal standard components.

7.2.3. Surrogate Standards: Refer to Table 8 for surrogate standard components and spiking levels.

7.2.4. Laboratory Control Sample Spiking Solutions: Refer to Table 9 for LCS components and spiking levels.

7.2.5. Matrix Spiking Solutions: The matrix spike contains the same components as the LCS. Refer to Table 9.

7.2.6. Tuning Standard: A standard is made up that will deliver 50 ng on column upon injection. A recommended concentration of 25 ng/µL of 4-Bromofluorobenzene in methanol is prepared as described in Sections 7.2.1.1 and 7.2.1.2.

8. SAMPLE COLLECTION, PRESERVATION AND STORAGE

8.1. Standard 40 mL glass screw-cap VOA vials with Teflon-faced silicone septa may be used for both liquid and solid matrices. Solid samples may also be collected in wide-mouth glass jars with Teflon-lined caps. Samples should be introduced into the containers with minimum agitation to avoid loss of volatile compounds. For liquid samples, each VOA vial should be filled without introduction of bubbles. Fill until there is a meniscus over the lip of the vial. The lid with septum (Teflon side toward the sample) should be tightened onto the vial. After tightening the lid, the vial should be inverted and tapped to check for air bubbles. If there are any air bubbles present the sample must be retaken. Sample containers for solid samples should be filled as completely as possible with minimum air space.

8.2. Water samples are preserved with HCl. Aromatic compounds are particularly susceptible to biodegradation at normal pH. The pH of the sample should be adjusted to less than 2 with HCl in the field at the time of sampling.

8.2.1. If the sample is unpreserved every reasonable effort should be made to analyze the sample within 7 days from sampling. The condition should be documented as an anomaly and the normal holding time applied. If the sample requires analysis of aromatic compounds the potential impact on the data must be documented.

8.3. All samples must be iced or refrigerated at 4° ± 2°C from the time of collection until analysis or extraction.

8.4. For shipping information, see the facility Sample Procurement Protocol SOP.

8.5. The holding time is fourteen days from sampling to the completion of analysis.

9. QUALITY CONTROL

9.1. Initial Demonstration of Capability

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

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

9.2. Control Limits

In-house historical control limits must be determined for surrogates, matrix spikes, and laboratory control samples (LCS). These limits must be determined at least annually. The recovery limits are mean recovery +/- 3 standard deviations for surrogates and LCS, and mean recovery +/- 2 standard deviations for matrix spikes. Precision limits for matrix spikes / matrix spike duplicates are 0 to mean relative percent difference + 2 standard deviations.

9.2.1. For medium level soils only, these limits do not apply to dilutions. Surrogate and matrix spike recoveries for medium level soils will be reported unless the dilution is more than 5X.

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

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

9.3. Surrogates

Every sample, blank, and QC sample is spiked with surrogates. Surrogate recoveries in samples, blanks, and QC samples must be assessed to ensure that recoveries are within established limits. The compounds included in the surrogate spiking solutions are listed in Tables 8 and 16. If any surrogates are outside limits, the following corrective actions must take place (except for dilutions):

- Check all calculations for error.

- Ensure that instrument performance is acceptable.
- Recalculate the data and/or reanalyze if either of the above checks reveal a problem.
- Reprepare and reanalyze the sample or flag the data as "Estimated Concentration" if neither of the above resolves the problem.

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

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

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

9.4. Method Blanks

For each batch of samples, analyze a method blank. The method blank is normally analyzed immediately after the calibration standards. For low-level volatiles, the method blank consists of reagent water. For medium-level volatiles, the method blank consists of 9.0 mL of methanol. Surrogates are added and the method blank is carried through the entire analytical procedure. The method blank must not contain any analyte of interest at or above the reporting limit (except common laboratory contaminants, see below) or at or above 5% of the measured concentration of that analyte in the associated samples, whichever is higher.

- If the analyte is a common laboratory contaminant (methylene chloride, acetone, 2-butanone) the data may be reported with qualifiers if the concentration of the analyte is less than five times the reporting limit. Such action must be taken in consultation with the client.
- Reanalysis of samples associated with an unacceptable method blank is required when reportable concentrations are determined in the samples.
- If there is no target analyte greater than the RL in the samples associated with an unacceptable method blank, the data may be reported with qualifiers. Such action should be done in consultation with the client.

9.4.1. The method blank must have acceptable surrogate recoveries. If surrogate recoveries are not acceptable, the data must be evaluated to determine if the method blank has served the purpose of demonstrating that the analysis is free of contamination. If surrogate recoveries are low and there are reportable analytes in the associated samples re-extraction of the blank and affected samples will normally be required. Consultation with the client should take place.

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

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

9.5. Laboratory Control Samples (LCS)

For each batch of samples, analyze a LCS. The LCS is normally analyzed immediately after the method blank. The LCS contains a representative subset of the analytes of interest (See Table 9), and must contain the same analytes as the matrix spike. If any analyte or surrogate is outside established control limits, the system is out of control and corrective action must occur. Corrective action will normally be repreparation and reanalysis of the batch.

- If the batch is not re-extracted and reanalyzed, the reasons for accepting the batch must be clearly presented in the project records and the report. (An example of acceptable reasons for not reanalyzing might be that the matrix spike and matrix spike duplicate are acceptable, and sample surrogate recoveries are good, demonstrating that the problem was confined to the LCS.)

- If re-extraction and reanalysis of the batch is not possible due to limited sample volume or other constraints, the LCS is reported, all associated samples are flagged, and appropriate comments are made in a narrative to provide further documentation.

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

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

9.6. Matrix Spikes

For each QC batch, analyze a matrix spike and matrix spike duplicate. Spiking compounds and levels are given in Table 9. Compare the percent recovery and relative percent difference (RPD) to that in the laboratory specific historically generated limits.

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

- If the recovery for any component is outside QC limits for both the matrix spike/ spike duplicate and the LCS, the laboratory is out of control and corrective action must be taken. Corrective action will normally include reanalysis of the batch.

- If a MS/MSD is not possible due to limited sample, then a LCS duplicate should be analyzed. RPD of the LCS and LCSD are compared to the matrix spike limits.

- The matrix spike/duplicate must be analyzed at the same dilution as the unspiked sample, even if the matrix spike compounds will be diluted out.

9.7. Nonconformance and Corrective Action

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

9.8. Quality Assurance Summaries

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

9.9. Quanterra QC Program

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

10. CALIBRATION AND STANDARDIZATION

10.1. Summary

10.1.1. Prior to the analysis of samples and blanks, each GC/MS system must be tuned and calibrated. Hardware tuning is checked through the analysis of the 4-Bromofluorobenzene (BFB) to establish that a given GC/MS system meets the standard mass spectral abundance criteria. The GC/MS system must be calibrated initially at a minimum of five concentrations (analyzed under the same BFB tune), to determine the linearity of the response utilizing target calibration standards. Once the system has been calibrated, the calibration must be verified each twelve hour time period for each GC/MS system. The use of separate calibrations is required for water and low soil matrices.

10.2. Recommended Instrument Conditions

10.2.1. General

Electron Energy:	70 volts (nominal)
Mass Range:	35–300 AMU
Scan Time:	to give at least 5 scans/peak, but not to exceed 1 second/scan
Injector Temperature:	200–250°C
Source Temperature:	According to manufacturer's specifications
Transfer Line	Temperature: 250–300°C
Purge Flow:	40 mL/minute
Carrier Gas	Flow: 15 mL/minute
Make-up Gas Flow:	25–30 mL/minute

10.2.2. Gas chromatograph suggested temperature program

10.2.2.1. BFB Analysis

Isothermal:	170°C

10.2.2.2. Sample Analysis

Initial Temperature:	40°C
Initial Hold Time:	4 minutes
Temperature Program:	8°C/minute
Final Temperature:	184°C
Second Temperature	Program: 40°C/minute
Final Temperature:	240°C

Final Hold Time: 2.6 minutes

10.3. Instrument Tuning

10.3.1. Each GC/MS system must be hardware-tuned to meet the abundance criteria listed in Table 10 for a maximum of a 50 ng injection or purging of BFB. Analysis must not begin until these criteria are met. These criteria must be met for each twelve-hour time period. The twelve-hour time period begins at the moment of injection of BFB.

10.4. Initial Calibration

10.4.1. A series of five initial calibration standards is prepared and analyzed for the target compounds and each surrogate compound. The calibration levels for a 5 mL purge are: 10, 20, 50, 100, and 200 µg/L. Certain analytes are prepared at higher concentrations due to poor purge performance. Calibration levels for a 25 mL purge are 2, 5, 10, 30, and 60 µg/L. Again, some analytes are prepared at higher levels. Tables 2, 4, and 17 list the calibration levels for each analyte.

10.4.2. It may be necessary to analyze more than one set of calibration standards to encompass all of the analytes required for same tests. For example, the Appendix IX list requires the Primary standard (Table 5) and the Appendix IX standard (Table 6).

10.4.3. Internal standard calibration is used. The internal standards are listed in Tables 7 and 15. Target compounds should reference the nearest internal standard. In particular, the SPCC compounds bromoform and 1,1,2,2-tetrachloroethane reference chlorobenzene-d5, not 1,4-difluorobenzene, which was appropriate for packed column analysis. Note different internal standards are used for the method 8260A drinking water analyte list (Appendix A). Each calibration standard is analyzed and the response factor (RF) for each compound is calculated using the area response of the characteristic ions against the concentration for each compound and internal standard. See equation 1, Section 12, for calculation of response factor.

10.4.4. The % RSD of the calibration check compounds (CCC) must be less than 30%. Refer to Table 12 for the CCCs.

10.4.4.1. If none of the CCCs are required analytes, project specific calibration specifications must be agreed with the client. Otherwise, all CCCs must meet the 30% criterion.

10.4.5. The average RF must be calculated for each compound. A system performance check is made prior to using the calibration curve. The five system performance check compounds (SPCC) are checked for a minimum average response factor. Refer to Table 11 for the SPCC compounds and required minimum response factors.

10.4.6. In general, a curve is created from the calibration points. However, if the %RSD of the response factors is less than or equal to 15%, the average response factor may be used in place of a curve (see Section 12 for calculations).

10.4.7. If the %RSD for a compound is greater than 15%, a calibration curve must be used. Linear (first order) or quadratic (second order) curves may be used.

10.4.8. Weighting of data points

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

10.4.9. Forcing through zero

Forcing a calibration curve through zero introduces unnecessary error and should not be used. The intercept at zero response must be less than the reporting limit for the analyte.

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

10.4.11. A separate five point calibration must be prepared for analysis of low level soils. Each standard is prepared as in Section 10.4.1, except that the standards are heated to 40°C for purging. All low-level soil samples, standards, and blanks must also be heated to 40°C for purging. Medium soil extracts should be analyzed using the water (unheated) calibration curve.

10.5. Continuing Calibration: The initial calibration must be verified every twelve hours.

10.5.1. Continuing calibration begins with analysis of BFB as described in Section 10.3. If the system tune is acceptable, the continuing calibration standard(s) are analyzed. The level 3 calibration standard is used as the continuing calibration.

10.5.2. The RF data from the standards are compared with the average RF from the initial five-point calibration to determine the percent drift of the CCC compounds. The calculation is given in equation 4, Section 12.3.4.

10.5.3. The % drift of the CCCs must be < 20% for the continuing calibration to be valid. The SPCCs are also monitored. The SPCCs must meet the criteria described in Table 11. In addition, the % drift of all anlalytes must be ≤ 50% with allowance for up to six target analytes to have % drift > 50%.

10.5.3.1. If none of the CCCs are required analytes, project specific calibration specifications must be agreed with the client. Otherwise, all CCCs must meet the 20% criterion.

10.5.3.2. Cyclohexanone, one of the components of the Appendix IX standard, is unstable in the calibration solution, forming 1,1-dimethoxycyclohexane. No calibration criteria are applied to cyclohexanone and quantitation is tentative. Cyclohexanone is included on the Universal Treatment Standard and FO-39 regulatory lists (but not on Appendix IX).

10.5.4. If the CCCs and or the SPCCs do not meet the criteria in Sections 10.5.3 and 10.5.4, the system must be evaluated and corrective action must be taken. The BFB tune and continuing calibration must be acceptable before analysis begins. Extensive corrective action such as column replacement or source cleaning will require a new initial calibration.

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

11. PROCEDURE

11.1. Procedural Variations

11.1.1. One time procedural variations are allowed only if deemed necessary in the professional judgment of supervision to accommodate variation in sample matrix, radioactivity, chemistry, sample size, or other parameters. Any variation shall be completely documented using a Nonconformance Memo and approved by a Supervisor or group leader and QA Manager. If contractually required, the client shall be notified. The Nonconformance Memo shall be filed in the project file.

11.1.2. Any unauthorized deviations from this procedure must also be documented as a nonconformance, with a cause and corrective action described.

11.2. Preliminary Evaluation

11.2.1. Where possible, samples are screened by headspace or GC/MS off-tune analysis to determine the correct aliquot for analysis. Alternatively, an appropriate aliquot can be determined from sample histories.

11.2.2. Dilutions should be done just prior to the GC/MS analysis of the sample. Dilutions are made in volumetric flasks or in a 5 or 25 mL Luerlok syringe. Calculate the volume of reagent water required for the dilution. Fill the 5/25 mL syringe with reagent water, compress the water to vent any residual air and adjust the water volume to the desired amount. Adjust the plunger to the 5/25 mL mark and inject the proper aliquot of sample into the syringe. If the dilution required would use less than 5 µL of sample then serial dilutions must be made in volumetric flasks.

11.2.2.1. The diluted concentration is to be estimated to be in the upper half of the calibration range.

11.3. Sample Analysis Procedure

11.3.1. All analysis conditions for samples must be the same as for the calibration standards (including purge time and flow, desorb time and temperature, column temperatures, multiplier setting etc.).

11.3.2. Sample Preparation Procedure

11.3.2.1. Samples fall into three general categories: waters, low-level soils, and medium-level soils or wastes. For waters and low-level soils, no sample preparation is necessary. For those soils which contain greater than 1 mg/kg of individual purgeable compounds, a medium-level preparation is necessary.

11.3.3. All samples must be analyzed as part of a batch. The batch is a set of up to 20 samples of the same matrix processed using the same procedures and reagents within the same time period. The batch also must contain a MS/MSD, a LCS, and a method blank.

11.3.3.1. If there is insufficient time in the 12-hour tune period to analyze 20 samples, the batch may be continued into the next tune period. However, if any re-tuning of the instrument is necessary, or if a period of greater than 14 hours from the preceding continuing calibration has passed, a new batch must be started. For medium level soils the batch is defined at the sample preparation stage.

11.3.3.2. One MS/MSD pair does not count towards the maximum 20 samples in the batch. Additional client requested MS/MSD samples do count towards the maximum 20 samples.

11.3.3.3. It is not necessary to reanalyze batch QC with reanalyses of samples. However, any reruns must be as part of a valid batch.

11.3.4. Water Samples

11.3.4.1. All samples and standard solutions must be at ambient temperature before analysis.

11.3.4.2. Fill a 5 or 25 mL syringe with the sample. If a dilution is necessary it may be made in the syringe if the sample aliquot is ≥ 5 µL. Check and document the pH of the remaining sample.

11.3.4.3. Add 250 ng of each internal and surrogate standard (10 µL of a 25 µg/mL solution, refer to Tables 7, 8, 15 and 16). The internal standards and the surrogate standards may be mixed and added as one spiking solution (this results in a 50 µg/L solution for a 5 mL sample, and a 10 µg/L solution for a 25 mL sample). Inject the sample into the purging chamber.

11.3.4.4. Purge the sample for eleven minutes (the trap must be below 35°C).

11.3.4.5. After purging is complete, desorb the sample, start the GC temperature program, and begin data acquisition. After desorption, bake the trap for 5-10 minutes to condition it for the next analysis. When the trap is cool, it is ready for the next sample.

11.3.4.6. Desorb and bake time and temperature are optimized for the type of trap in use. The same conditions must be used for samples and standards.

11.3.5. Medium-Level Soil/Sediment and Waste Samples

11.3.5.1. Sediments/soils and waste that are insoluble in methanol.

11.3.5.1.1. Gently mix the contents of the sample container with a narrow metal or wood spatula. Weigh 4 g (wet weight) into a tared vial. Use a top-loading balance. Record the weight to 0.1 gram. Do not discard any supernatant liquids.

11.3.5.1.2. Quickly add 9 mL of methanol, and 1 mL of surrogate spiking solution to bring the final volume of methanol to 10 mL. For an LCS or MS/MSD

sample add 8 mL of methanol, 1 mL of surrogate spike solution, and 1 mL of matrix spike solution. Cap the vial and vortex to mix thoroughly.

NOTE: Sections 11.3.5.1.1 and 11.3.5.1.2 must be performed rapidly and without interruption to avoid the loss of volatile organics.

11.3.5.2. Liquid wastes that are soluble in methanol and insoluble in water.

11.3.5.2.1. Pipet 2 mL of the sample into a tared vial. Use a top-loading balance. Record the weight to the nearest 0.1 gram.

11.3.5.2.2. Quickly add 7 mL of methanol, then add 1 mL of surrogate spiking solution to bring the final volume to 10 mL. Cap the vial and shake for 2 minutes to mix thoroughly. For a MS/MSD or LCS, 6 mL of methanol, 1 mL of surrogate solution, and 1 mL of matrix spike solution is used.

11.3.5.3. Fill a 5 mL syringe with 5 mL of reagent water. Add 100 µL (or less if a dilution is required) of methanol extract from the sample preparation in Section 11.3.5.1 or 11.3.5.2. If less than 5 µL of the methanol extract is required, then an intermediate dilution is required. Add 10 µL of the 25 µg/L internal standard solution. (Note that the combined internal standard/surrogate standard solution is not used since surrogates have been added previously.) Inject the sample into the purging chamber and proceed with the analysis as per Sections 11.3.4.4 and 11.3.4.5.

11.3.6. Low-Level Soils

11.3.6.1. This is designed for samples containing individual purgeable compounds of < 2 mg/kg. It is limited to soil/sediment samples and waste that is of a similar consistency (granular and porous). Weigh 5 g of the sample into a tared purge vessel. If a dilution is required, a smaller sample amount can be analyzed, down to a minimum of 1.0 g. Any soil sample requiring further dilution must be run as a medium-level soil. Add 5 mL of reagent water to which 10 µL of the 25 µg/L internal standard/surrogate standard solution has been added. Proceed with the analysis as per Sections 11.3.4.4 and 11.3.4.5.

11.4. Initial review and corrective actions

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

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

11.4.3. Internal standard response in each sample should be within 50% to 200% of the response in the preceding continuing calibration standard. If more than one calibration standard is used, the internal standard response for the samples is compared to the last calibration standard analyzed.

11.4.3.1. Any samples that do not meet the internal standard criteria must be evaluated for validity. If the change in sensitivity is a matrix effect confined to an individual sample reanalysis may not be necessary. If the change in sensitivity is due to instrumental problems all affected samples must be reanalyzed after the problem is corrected. In any event, the reason for accepting the sample analysis must be documented. Some clients may require reanalysis of all samples with internal standard criteria outside the 50-200% criteria. Consideration should be given to reanalyzing at a dilution to reduce matrix effects. It is only necessary to reanalyze once to confirm matrix effect.

11.4.4. The surrogate standard recoveries are evaluated to ensure that they are within limits. Corrective action for surrogates out of control will normally be to reanalyze the affected samples. However, if the surrogate standard response is out high and there are no target analytes or tentatively identified compounds, reanalysis may not be necessary. Out of control surrogate standard response may be a matrix effect. It is only necessary to reanalyze a sample once to demonstrate matrix effect, but reanalysis at a dilution should be considered.

11.5. Dilutions

If the response for any compound exceeds the working range of the GC/MS system, a dilution of the extract is prepared and analyzed. An appropriate dilution should be in the upper half of the calibration range. Samples may be screened to determine the appropriate dilution for the initial run. If the initial diluted run has no hits or hits below 20% of the calibration range and the matrix allows for analysis at a lesser dilution, then the sample must be reanalyzed at a dilution targeted to bring the largest hit above 50% of the calibration range.

11.5.1. Guidance for Dilutions Due to Matrix

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

11.5.2. Reporting Dilutions

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

12. DATA ANALYSIS AND CALCULATIONS

12.1. Qualitative identification

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

- The sample component retention time must compare to within ± 0.2 min. of the retention time of the standard component. For reference, the standard must be run within the same twelve hours as the sample.
- All ions present in the standard mass spectra at a relative intensity greater than 10% (most abundant ion in the spectrum equals 100%) should be present in the sample spectrum.
- The relative intensities of ions should agree to within ±30% between the standard and sample spectra. (Example: For an ion with an abundance of 50% in the standard spectra, the corresponding sample abundance must be between 20 and 80 percent.)

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

12.1.2. Structural isomers with very similar or identical mass spectra are identified as isomer pairs if resolution between the two is < 25%.

12.2. Tentatively Identified Compounds (TICs)

12.2.1. If the client requests components not associated with the calibration standards, a search of the NIST library may be made for the purpose of tentative identification. Guidelines are:

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

12.2.1.2. The relative intensities of the major ions should agree to within 20%. (Example: If an ion shows an abundance of 50% in the standard spectrum, the corresponding sample ion abundance must be between 30% and 70%).

12.2.1.3. Molecular ions present in the reference spectrum should be present in the sample spectrum.

12.2.1.4. Ions present in the sample spectrum but not in the reference spectrum should be reviewed for possible background contamination or presence of coeluting compounds.

12.2.1.5. Ions present in the reference spectrum but not in the sample spectrum should be reviewed for possible subtraction from the spectrum because of background contamination or coeluting peaks. (Data system reduction programs can sometimes create these discrepancies.)

12.2.1.6. Computer-generated library search routines should not use normalization routines that would misrepresent the library or unknown spectra when compared to each other. Only after visual inspection of the sample with the nearest library searches should the analyst assign a tentative identification.

12.3. Calculations.

12.3.1. Response factor (RF):

Equation 1

$$RF = \frac{A_x C_{is}}{A_{is} C_x}$$

Where:

A_x = Area of the characteristic ion for the compound to be measured

A_{is} = Area of the characteristic ion for the specific internal standard

C_{is} = Concentration of the specific internal standard, ng

C_x = Concentration of the compound being measured, ng

12.3.2. Standard deviation (SD):

Equation 2

$$SD = \sqrt{\sum_{i=1}^{N} \frac{(Xi - X)^2}{N - 1}}$$

X_i = Value of X at i through N

N = Number of points

X = Average value of X_i

12.3.3. Percent relative standard deviation (%RSD):

Equation 3

$$\%RSD = \frac{\textbf{Standard Deviation}}{\overline{RFi}} \times 100$$

$\overline{RFi}$ = **Mean of RF values in the curve**

12.3.4. Percent drift between the initial calibration and the continuing calibration:

Equation 4

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

Where

$C_{expected}$ = Known concentration in standard

C_{found} = Measured concentration using selected quantitation method

12.3.5. Target compound and surrogate concentrations:

Concentrations in the sample may be determined from linear or second order (quadratic) curve fitted to the initial calibration points, or from the average response factor of the initial calibration points. Average response factor may only be used when the % RSD of the response factors in the initial calibration is ≤ 15%. In general, use of quadratic fit is recommended to reduce quantitation errors.

12.3.5.1. Calculation of concentration using Quadratic fit

Equation 5

$$\textbf{Concentration } \mu g / L = A + Bx + Cx^2$$

x is defined in equations 8, 9 and 10

A is a constant defined by the intercept

B is the slope of the curve

C is the curvature

12.3.5.2. Calculation of concentration using Linear fit

Equation 6

$$\textbf{Concentration } \mu g / L = A + Bx$$

12.3.5.3. Calculation of concentration using Average Response Factors

Equation 7

$$\textbf{Concentration } \mu g / L = \frac{x}{RF}$$

12.3.5.4. Calculation of x for Water and water-miscible waste:

Equation 8

$$x = \frac{(A_x)(I_s)(D_f)}{(A_{is})(V_o)}$$

Where:

A_x = Area of characteristic ion for the compound being measured (secondary ion quantitation is allowed only when there are sample interferences with the primary ion)

A_{is} = Area of the characteristic ion for the internal standard

I_s = Amount of internal standard added in ng

$$\text{Dilution Factor} = D_f = \frac{\text{Total volume purged (mL)}}{\text{Volume of original sample used (mL)}}$$

V_o = Volume of water purged, mL

12.3.5.5. Calculation of x for Medium level soils:

Equation 9

$$x = \frac{(A_x)(I_s)(V_t)(1000)(D_f)}{(A_{is})(V_a)(W_s)(D)}$$

Where:

A_x, I_s, D_f, A_{is}, same as for water.

V_t = Volume of total extract, mL (Typically 10 mL)

V_a = Volume of extract added for purging, µL

W_s = Weight of sample extracted, g

$$D = \frac{100 - \%moisture}{100}$$

12.3.5.6. Calculation of x for Low level soils:

Equation 10

$$x = \frac{(A_x)(I_s)}{(A_{is})(W_s)(D)}$$

Where:

A_x, I_s, A_{is}, same as for water.

D is as for medium level soils

W_s = Weight of sample added to the purge vessel, g

12.3.5.7. Calculation of TICs: The calculation of TICs (tentatively identified compounds) is identical to the above calculations with the following exceptions:

A_x = Area in the total ion chromatogram for the compound being measured

A_{is} = Area of the total ion chromatogram for the nearest internal standard without interference

RF = 1

In other words, the concentration is equal to x as defined in equations 8, 9 and 10.

12.3.6. MS/MSD Recovery

Equation 11

$$\textbf{Matrix Spike Recovery, \%} = \frac{SSR - SR}{SA} \times 100$$

SSR = Spike sample result
SR = Sample result
SA = Spike added

12.3.7. Relative % Difference calculation for the MS/MSD

Equation 12

$$\mathbf{RPD} = \frac{|\mathbf{MSR} - \mathbf{MSDR}|}{\frac{1}{2}(\mathbf{MSR} + \mathbf{MSDR})} \times 100$$

Where:

RPD = Relative percent difference

MSR = Matrix spike result

MSDR = Matrix spike duplicate result

13. METHOD PERFORMANCE

13.1. Method Detection Limit

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

13.2. Initial Demonstration

Each laboratory must make a one time initial demonstration of capability for each individual method. Demonstration of capability for both soil and water matrices is required. This requires analysis of QC check samples containing all of the standard analytes for the method. For some tests it may be necessary to use more than one QC check mix to cover all analytes of interest. The QC check sample is made up at 20 µg/L. (Some compounds will be at higher levels, refer to the calibration standard levels for guidance.)

13.2.1. Four aliquots of the QC check sample are analyzed using the same procedures used to analyze samples, including sample preparation.

13.2.2. Calculate the average recovery and standard deviation of the recovery for each analyte of interest. The %RSD should be ≤ 15% for each analyte, and the % recovery should be within 80-120%.

13.2.3. If any analyte does not meet the acceptance criteria, check the acceptance limits in the reference methods (Table 6 of method 8240B, paragraph 8.3.5 of method 8260A). If the recovery or precision is outside the limits in the reference methods, the test must be repeated. Only those analytes that did not meet criteria in the first test need to be evaluated. Repeated failure for any analyte indicates the need for the laboratory to evaluate the analytical procedure and take corrective action.

13.3. Training Qualification

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

14. POLLUTION PREVENTION

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

15. WASTE MANAGEMENT

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

16. REFERENCES

16.1. SW846, *Test Methods for Evaluating Solid Waste*, Third Edition, Gas Chromatography/Mass Spectrometry for Volatile Organics, Method 8240B, Update II, September 1994.

16.2. SW846, *Test Methods for Evaluating Solid Waste,* Third Edition, Gas Chromatography/Mass Spectrometry for Volatile Organics, Method 8260A, Update II, September 1994.

17. MISCELLANEOUS

17.1. Modifications from the reference method

17.1.1. Method 8240B has been modified in this SOP to use capillary columns rather than the packed columns listed in the reference method. This provides improved separation of the target analytes and improved detection limits.

17.1.2. Ion 119 is used as the quantitation ion for chlorobenzene-d5 for 25 mL purge tests.

17.1.3. The internal standard control criteria of 50% to 200% is applied to each sample, rather than the subsequent continuing calibration standard as listed in the reference method.

17.1.4. A retention time window of 0.2 minutes is used for all components, since some data systems do not have the capability of using the relative retention time units specified in the reference method.

17.1.5. The quantitation and qualifier ions for some compounds have been changed from those recommended in SW-846 in order to improve the reliability of qualitative identification.

17.1.6. Method 8260A requires that the purge vessel is run through an additional purge cycle after 25 mL sample analysis to remove carryover. Instead, purge vessels are oven baked between analyses or disposable vessels are used one time only.

17.1.7. Matrix spikes and surrogares are spiked at the levels specified in methods 3500 and 5030.

17.2. Modifications from previous revisions

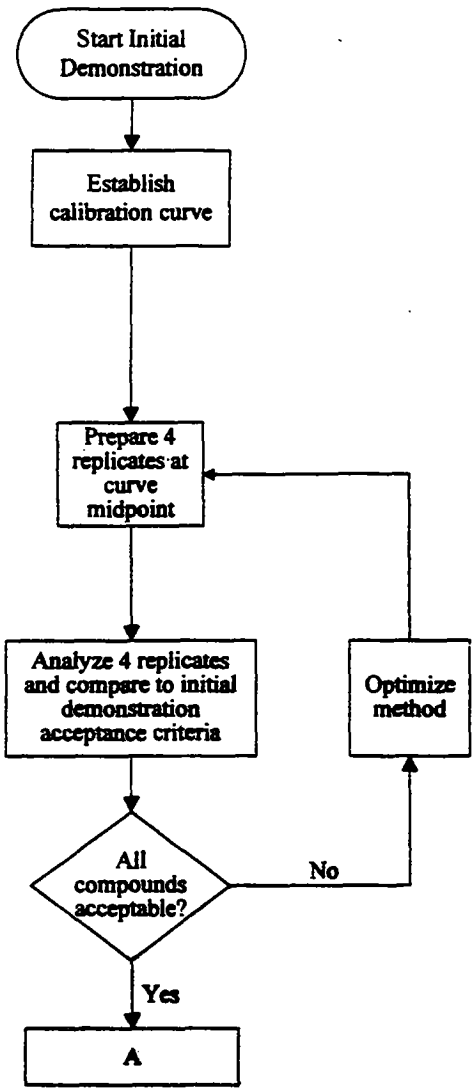
None

17.3. Facility specific SOPs

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

17.4. Flow diagrams

17.4.1. Initial Demonstration and MDL



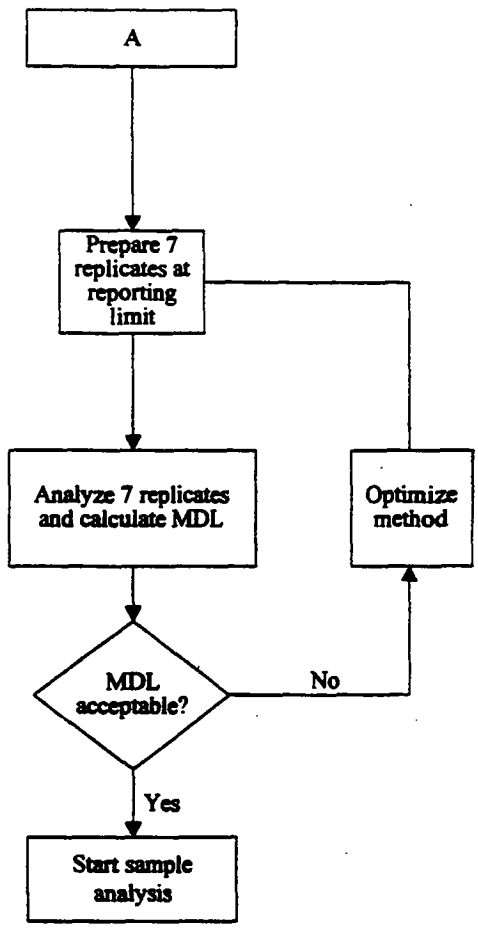


Table 1

Quanterra Primary Standard and Reporting Limits

Compound	CAS Number	Reporting Limits[1]			
		5 mL Water µg/L	25 mL water µg/L	Low soil µg/kg	Med. Soil µg/kg
Dichlorodifluoromethane	75-71-8	10	2	10	1200
Chloromethane	74-87-3	10	2	10	1200
Bromomethane	74-83-9	10	2	10	1200
Vinyl chloride	75-01-4	10	2	10	1200
Chloroethane	75-00-3	10	2	10	1200
Trichlorofluoromethane	75-69-4	10	2	10	1200
Acrolein	107-02-8	100	20	100	12000
Acetone	67-64-1	20	10	20	2500
Trichlorotrifluoroethane	76-13-1	5	1	5	620
Ethanol	64-17-5	500	200	500	62,000
Iodomethane	74-88-4	5	1	5	620
Carbon disulfide	75-15-0	5	1	5	620
Methylene chloride	75-09-2	5	1	5	620
tert-Butyl alcohol	75-65-0	200	50	200	25,000
1,1-Dichloroethene	75-35-4	5	1	5	620
1,1-Dichloroethane	75-34-3	5	1	5	620
trans-1,2-Dichloroethene	156-60-5	2.5	0.5	2.5	310
Acrylonitrile	107-13-1	100	20	100	12000
Methyl *tert*-butyl ether (MTBE)	1634-04-4	20	5	20	2500
Hexane	110-54-3	5	1	5	620
cis-1,2-Dichloroethene	156-59-2	2.5	0.5	2.5	310
1,2-Dichloroethene (Total)	540-59-0	5	1	5	620
Tetrahydrofuran	109-99-9	20	5	20	2500
Chloroform	67-66-3	5	1	5	620
1,2-Dichloroethane	107-06-2	5	1	5	620
Dibromomethane	74-95-3	5	1	5	620
2-Butanone	78-93-3	20	5	20	2500
1,4-Dioxane	123-91-1	500	200	500	62000
1,1,1-Trichloroethane	71-55-6	5	1	5	620
Carbon tetrachloride	56-23-5	5	1	5	620
Bromodichloromethane	75-27-4	5	1	5	620
1,2-Dichloropropane	78-87-5	5	1	5	620
cis-1,3-Dichloropropene	10061-01-5	5	1	5	620
Trichloroethene	79-01-6	5	1	5	620

Table 1

Quanterra Primary Standard and Reporting Limits

Compound	CAS Number	Reporting Limits[1]			
		5 mL Water µg/L	25 mL water µg/L	Low soil µg/kg	Med. Soil µg/kg
Dibromochloromethane	124-48-1	5	1	5	620
1,2-Dibromoethane	106-93-4	5	1	5	620
1,2,3-Trichloropropane	96-18-4	5	1	5	620
1,1,2-Trichloroethane	79-00-5	5	1	5	620
Benzene	71-43-2	5	1	5	620
Ethylmethacrylate	97-63-2	5	1	5	620
trans-1,3-Dichloropropene	10061-02-6	5	1	5	620
Bromoform	75-25-2	5	1	5	620
4-Methyl-2-pentanone	108-10-1	20	5	20	2500
2-Hexanone	591-78-6	20	5	20	2500
Tetrachloroethene	127-18-4	5	1	5	620
Toluene	108-88-3	5	1	5	620
1,1,2,2-Tetrachloroethane	79-34-5	5	1	5	620
2-Chloroethyl vinyl ether	110-75-8	N/A[2]	N/A	50	6200
Vinyl acetate	108-05-4	10	2	10	1200
Chlorobenzene	108-90-7	5	1	5	620
Ethylbenzene	100-41-4	5	1	5	620
Styrene	100-42-5	5	1	5	620
t-1,4-Dichloro-2-butene	110-57-6	5	1	5	620
m and p Xylenes		2.5	0.5	2.5	310
o-xylene	95-47-6	2.5	0.5	2.5	310
Total xylenes	1330-20-7	5	1	5	620
1,3-Dichlorobenzene	541-73-1	5	1	5	620
1,4-Dichlorobenzene	106-46-7	5	1	5	620
1,2-Dichlorobenzene	95-50-1	5	1	5	620

[1] Reporting limits listed for soil/sediment are based on wet weight. The reporting limits calculated by the laboratory for soil/sediment calculated on dry weight basis, will be higher.

[2] 2-Chloroethyl vinyl ether cannot be reliably recovered from acid preserved samples

Table 2

Quanterra Primary Standard Calibration Levels, 5 mL purge[1]

Compound	Calibration Level ug/L				
	Level 1	Level 2	Level 3	Level 4	Level 5
1,2-Dichloroethane-d4 (Surrogate)	10	20	50	100	200
Toluene-d8 (Surrogate)	10	20	50	100	200
4-Bromofluorobenzene (Surrogate)	10	20	50	100	200
Dichlorodifluoromethane	10	20	50	100	200
Chloromethane	10	20	50	100	200
Bromomethane	10	20	50	100	200
Vinyl chloride	10	20	50	100	200
Chloroethane	10	20	50	100	200
Trichlorofluoromethane	10	20	50	100	200
Acrolein	100	200	500	1000	2000
Acetone	10	20	50	100	200
Trichlorotrifluoroethane	10	20	50	100	200
Ethanol	500	1000	5000	10,000	20,000
Iodomethane	10	20	50	100	200
Carbon disulfide	10	20	50	100	200
Methylene chloride	10	20	50	100	200
tert-Butyl alcohol	200	400	1,000	2,000	4,000
1,1-Dichloroethene	10	20	50	100	200
1,1-Dichloroethane	10	20	50	100	200
trans-1,2-Dichloroethene	10	20	50	100	200
Acrylonitrile	100	200	500	1,000	2,000
Methyl *tert*-butyl ether (MTBE)	10	20	50	100	200
Hexane	10	20	50	100	200
cis-1,2-Dichloroethene	10	20	50	100	200
Tetrahydrofuran	10	20	50	100	200
Chloroform	10	20	50	100	200
1,2-Dichloroethane	10	20	50	100	200
Dibromomethane	10	20	50	100	200
2-Butanone	10	20	50	100	200
1,4-Dioxane	500	1000	2,500	5,000	10,000
1,1,1-Trichloroethane	10	20	50	100	200
Carbon tetrachloride	10	20	50	100	200
Bromodichloromethane	10	20	50	100	200
1,2-Dichloropropane	10	20	50	100	200
cis-1,3-Dichloropropene	10	20	50	100	200

Table 2

Quanterra Primary Standard Calibration Levels, 5 mL purge[1]

Compound	Calibration Level ug/L				
	Level 1	Level 2	Level 3	Level 4	Level 5
Trichloroethene	10	20	50	100	200
Dibromochloromethane	10	20	50	100	200
1,2-Dibromoethane	10	20	50	100	200
1,2,3-Trichloropropane	10	20	50	100	200
1,1,2-Trichloroethane	10	20	50	100	200
Benzene	10	20	50	100	200
Ethylmethacrylate	10	20	50	100	200
trans-1,3-Dichloropropene	10	20	50	100	200
Bromoform	10	20	50	100	200
4-Methyl-2-pentanone	10	20	50	100	200
2-Hexanone	10	20	50	100	200
Tetrachloroethene	10	20	50	100	200
Toluene	10	20	50	100	200
1,1,2,2-Tetrachloroethane	10	20	50	100	200
2-Chloroethyl vinyl ether	20	40	100	200	400
Vinyl acetate	10	20	50	100	200
Chlorobenzene	10	20	50	100	200
Ethylbenzene	10	20	50	100	200
Styrene	10	20	50	100	200
t-1,4-Dichloro-2-butene	10	20	50	100	200
m and p Xylenes	20	40	100	200	400
o-xylene	10	20	50	100	200
1,3-Dichlorobenzene	10	20	50	100	200
1,4-Dichlorobenzene	10	20	50	100	200
1,2-Dichlorobenzene	10	20	50	100	200

[1] **Levels for 25 mL purge are 5 times lower in all cases**

Table 3

Quanterra Appendix IX Standard and Reporting Limits, 5 mL purge[1]

Compound	CAS Number	Reporting Limits			
		5 mL Water µg/L	25 mL water µg/L	Low Soil µg/kg	Medium Soil µg/mL
Allyl Chloride	107-05-1	10	2	10	1,200
Acetonitrile	75-05-8	100	20	100	12,000
Dichlorofluoromethane		10	2	10	1,200
Isopropyl ether	108-20-3	50	10	50	6,200
Chloroprene	126-99-8	5	1	5	620
n-Butanol	71-36-3	200	50	200	25,000
Propionitrile	107-12-0	20	4	20	2500
Methacrylonitrile	126-98-7	5	1	5	620
Isobutanol	78-83-1	200	50	200	25,000
Methyl methacrylate	80-62-6	5	1	5	620
1,1,1,2-Tetrachloroethane	630-20-6	5	1	5	620
1,2-Dibromo-3-chloropropane	96-12-8	10	2	10	1,200
Ethyl ether	60-29-7	10	2	10	1,200
Ethyl Acetate	141-78-6	20	4	20	2500
2-Nitropropane	79-46-9	10	2	10	1,200
Cyclohexanone	108-94-1	N/A[2]	N/A[2]	N/A[2]	N/A[2]
Isopropylbenzene	98-82-8	5	1	5	620

[1] **Levels for 25 mL purge are 5 times lower in all cases**

[2] Cyclohexanone decomposes to 1,1-dimethoxycyclohexane in methanolic solution. Reporting limits cannot be accurately determined.

Table 4

Quanterra Appendix IX Standard Calibration Levels, µg/L

Compound	Level 1	Level 2	Level 3	Level 4	Level 5
Allyl Chloride	10	20	50	100	200
Acetonitrile	100	200	500	1,000	2,000
Dichlorofluoromethane	10	20	50	100	200
Isopropyl ether	50	100	250	500	1,000
Chloroprene	10	20	50	100	200
n-Butanol	200	400	1,000	2,000	4,000
Propionitrile	20	40	100	200	400
Methacrylonitrile	10	20	50	100	200
Isobutanol	200	400	1,000	2,000	4,000
Methyl methacrylate	10	20	50	100	200
1,1,1,2-Tetrachloroethane	10	20	50	100	200
1,2-Dibromo-3-chloropropane	20	40	100	200	400
Ethyl ether	10	20	50	100	200
Ethyl Acetate	20	40	100	200	400
2-Nitropropane	20	40	100	200	400
Cyclohexanone	100	200	500	1,000	2,000
Isopropylbenzene	10	20	50	100	200

Table 5

Reportable Analytes for Quanterra Standard Tests, Primary Standard

Compound	CAS Number	Quanterra Standard List	TCLP	TCL	Appendix IX	UTS
Dichlorodifluoromethane	75-71-8				X	X
Chloromethane	74-87-3	X		X	X	X
Bromomethane	74-83-9	X		X	X	X
Vinyl chloride	75-01-4	X	X	X	X	X
Chloroethane	75-00-3	X		X	X	X
Trichlorofluoromethane	75-69-4				X	X
Acrolein	107-02-8				X	X
Acetone	67-64-1	X		X	X	X
Trichlorotrifluoroethane	76-13-1					X
Ethanol	64-17-5					
Iodomethane	74-88-4				X	X
Carbon disulfide	75-15-0	X		X	X	X
Methylene chloride	75-09-2	X		X	X	X
tert-Butly alcohol	75-65-0					
1,1-Dichloroethene	75-35-4	X	X	X	X	X
1,1-Dichloroethane	75-34-3	X		X	X	X
trans-1,2-Dichloroethene	156-60-5	X		X	X	X
Total dichloroethene		X		X	X	X
Acrylonitrile	107-13-1					X
Methyl *tert*-butyl ether (MTBE)	1634-04-4					
Hexane	110-54-3					
cis-1,2-Dichloroethene	156-59-2	X		X		
Tetrahydrofuran	109-99-9					
Chloroform	67-66-3	X	X	X	X	X
1,2-Dichloroethane	107-06-2	X	X	X	X	X
Dibromomethane	74-95-3				X	X
2-Butanone	78-93-3	X	X	X	X	X
1,4-Dioxane	123-91-1				X	X
1,1,1-Trichloroethane	71-55-6	X		X	X	X
Carbon tetrachloride	56-23-5	X	X	X	X	X
Bromodichloromethane	75-27-4	X		X	X	X
1,2-Dichloropropane	78-87-5	X		X	X	X
cis-1,3-Dichloropropene	10061-01-	X		X	X	X

Table 5

Reportable Analytes for Quanterra Standard Tests, Primary Standard

Compound	CAS Number	Quanterra Standard List	TCLP	TCL	Appendix IX	UTS
	5					
Trichloroethene	79-01-6	X	X	X	X	X
Dibromochloromethane	124-48-1	X		X	X	X
1,2-Dibromoethane	106-93-4				X	X
1,2,3-Trichloropropane	96-18-4				X	X
1,1,2-Trichloroethane	79-00-5	X		X	X	X
Benzene	71-43-2	X	X	X	X	X
Ethylmethacrylate	97-63-2				X	X
trans-1,3-Dichloropropene	10061-02-6	X		X	X	X
Bromoform	75-25-2	X		X	X	X
4-Methyl-2-pentanone	108-10-1	X		X	X	X
2-Hexanone	591-78-6	X		X	X	
Tetrachloroethene	127-18-4	X	X	X	X	X
Toluene	108-88-3	X		X	X	X
1,1,2,2-Tetrachloroethane	79-34-5	X		X	X	X
2-Chloroethyl vinyl ether	110-75-8					
Vinyl acetate	108-05-4				X	
Chlorobenzene	108-90-7	X	X	X	X	X
Ethylbenzene	100-41-4	X		X	X	X
Styrene	100-42-5	X		X	X	
t-1,4-Dichloro-2-butene	110-57-6				X	
m and p Xylenes		X		X	X	X
o-xylene	95-47-6	X		X	X	X
Total xylenes	1330-20-7	X		X	X	X
1,3-Dichlorobenzene	541-73-1					
1,4-Dichlorobenzene	106-46-7					
1,2-Dichlorobenzene	95-50-1					

Table 6

Reportable Analytes for Quanterra Standard Tests, Appendix IX standard

Compound	Number	Quanterra Standard List	TCLP	TCL	Appendix IX	UTS
Allyl Chloride	107-05-1				X	
Acetonitrile	75-05-8				X	X
Dichlorofluoromethane	75-43-4					
Isopropyl ether	108-20-3					
Chloroprene	126-99-8				X	
n-Butanol	71-36-3					
Propionitrile	107-12-0				X	
Methacrylonitrile	126-98-7				X	X
Isobutanol	78-83-1				X	X
Methyl methacrylate	80-62-6				X	X
1,1,1,2-Tetrachloroethane	630-20-6				X	X
1,2-Dibromo-3-chloropropane	96-12-8				X	X
Ethyl ether	60-29-7					X
Ethyl Acetate	141-78-6					X
2-Nitropropane	79-46-9					
Cyclohexanone	108-94-1					X
Isopropylbenzene	98-82-8					

Table 7

Internal Standards

	Standard Concentration µg/mL	Quantitation ion (5 mL purge)	Quantitation ion (25 mL purge)
Bromochloromethane	25	128	128
1,4-Difluorobenzene	25	114	114
Chlorobenzene d5	25	117	119

Notes:

1) 10 µL of the internal standard is added to the sample. This results in a concentration of each internal in the sample of 50µg/L for a 5 mL purge or 10 µg/L for a 25 mL purge.

2) Except for medium level soils, the surrogate and internal standards may be combined in one solution.

Table 8

Surrogate Standards

Surrogate Compounds	Standard Concentration µg/mL
1,2-Dichloroethane-d_4	25
Toluene-d_8	25
4-Bromofluorobenzene	25

Notes:

1) 10 µL of the surrogate standard is added to the sample. This results in a concentration of each surrogate in the sample of 50µg/L for a 5 mL purge or 10 µg/L for a 25 mL purge.

2) Except for medium level soils, the surrogate and internal standards may be combined in one solution.

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

Table 9

Matrix Spike / LCS Compounds

Compound	Standard Concentration µg /mL
1,1-Dichloroethene	25
Trichloroethene	25
Toluene	25
Benzene	25
Chlorobenzene	25

Notes:

1) 10 µL of the standard is added to the LCS or matrix spiked sample. This results in a concentration of each spike analyte in the sample of 50µg/L for a 5 mL purge or 10 µg/L for a 25 mL purge.

2) Recovery and precision limits for LCS and MS/MSD are generated from historical data and are maintained by the QA dedartment.

Table 10

BFB Key Ion Abundance Criteria

Mass	Ion Abundance Criteria
50	15% to 40% of Mass 95
75	30% to 60% of Mass 95
95	Base Peak, 100% Relative Abundance
96	5% to 9% of Mass 95
173	Less Than 2% of Mass 174
174	Greater Than 50% of Mass 95
175	5% to 9% of Mass 174
176	Greater Than 95%, But Less Than 101% of Mass 174
177	5% to 9% of Mass 176

Table 11

SPCC Compounds and Minimum Response Factors

Compound	8240B Min. RF	8260A Min. RF
Chloromethane	0.300	0.100
1,1-Dichloroethane	0.300	0.100
Bromoform	>0.100	>0.100
1,1,2,2-Tetrachloroethane	0.300	0.300
Chlorobenzene	0.300	0.300

Table 12

CCC compounds

Compound	Max. %RSD from Initial Calibration	Max. %D for continuing calibration
Vinyl Chloride	<30.0	<20.0
1,1-Dichloroethene	<30.0	<20.0
Chloroform	<30.0	<20.0
1,2-Dichloropropane	<30.0	<20.0
Toluene	<30.0	<20.0
Ethylbenzene	<30.0	<20.0

Table 13

Characteristic ions

Compound	Primary*	Secondary	Tertiary
Bromochloromethane (Internal Standard)	128	49	130, 51
1,2-Dichloroethane-d_4 (Surrogate)	65	102	
Dichlorodifluoromethane	85	87	50, 101,103
Chloromethane	50	52	49
Vinyl chloride	62	64	61
Bromomethane	94	96	79
Chloroethane	64	66	49
Trichlorofluoromethane	101	103	66
1,1-Dichloroethene	96	61	98
Acrolein	56	55	58

Table 13

Characteristic ions

Compound	Primary*	Secondary	Tertiary
Iodomethane	142	127	141
Carbon disulfide	76	78	
Trichlorotrifluoroethane	151	101	153
Ethanol	45	46	
Acetone	43	58	
Methylene chloride	84	49	51, 86
tert-Butyl alcohol	59	74	
trans-1,2-Dichloroethene	96	61	98
Acrylonitrile	53	52	51
Methyl *tert* butyl ether	73		
Hexane	57	43	
1,1-Dichloroethane	63	65	83
cis-1,2-Dichloroethene	96	61	98
2-Butanone	43	72**	
Tetrahydrofuran	42	71	
Chloroform	83	85	47
1,2-Dichloroethane	62	64	98
Dibromomethane	93	174	95, 172, 176
1,4-Dioxane	88	58	
1,4-Difluorobenzene (Internal Standard)	114	63	88
Vinyl acetate	43	86	
1,1,1-Trichloroethane	97	99	117
Carbon tetrachloride	117	119	121
Benzene	78	52	77
Trichloroethene	130	95	97, 132
1,2-Dichloropropane	63	65	41
Bromodichloromethane	83	85	129
2-Chloroethyl vinyl ether	63	65	106
cis-1,3-Dichloropropene	75	77	39
trans-1,3-Dichloropropene	75	77	39
1,1,2-Trichloroethane	97	83	85, 99
Chlorodibromomethane	129	127	131
Bromoform	173	171	175, 252
1,2,3-Trichloropropane	75	110	77, 112, 97
Chlorobenzene-d_5 (Internal Standard)	117,119	117,119	
Toluene-d_8 (Surrogate)	98	70	100
4-Bromofluorobenzene (Surrogate)	95	174	176
Toluene	91	92	65

Table 13

Characteristic ions

Compound	Primary*	Secondary	Tertiary
4-Methyl-2-pentanone	43	58	57, 100
Tetrachloroethene	164	166	131
Ethyl methacrylate	69	41	99, 86, 114
2-Hexanone	43	58	57, 100
Chlorobenzene	112	114	77
Ethylbenzene	106	91	
Xylenes	106	91	
Styrene	104	103	78, 51, 77
Dichlorobenzene (all isomers)	146	148	111
trans 1,4-Dichloro-2-butene	53	75	89, 77, 124
1,1,2,2-Tetrachloroethane	83	85	131, 133
Allyl Chloride	76	41	78
Acetonitrile	40	41	
Dichlorofluoromethane	67	69	
Isopropyl ether	87	59	45
Chloroprene	53	88	90
n-Butanol	56	41	42
Propionitrile	54	52	55
Methacrylonitrile	41	67	52
Isobutanol	41	43	74
Methyl methacrylate	41	69	100
1,1,1,2-Tetrachloroethane	131	133	119
1,2-Dibromo-3-chloropropane	157	155	75
Ethyl ether	59	74	
Ethyl Acetate	43	88	61
2-Nitropropane	41	43	46
Cyclohexanone	55	42	98
Isopropylbenzene	105	120	

* The primary ion should be used for quantitation unless interferences are present, in which case a secondary ion may be used.

** m/z 43 may be used for quantitation of 2-Butanone, but m/z 72 must be present for positive identification.

1. SUMMARY

A target analyte list based on the list in method 524.2 is frequently requested for analysis by method 8260A. Quanterra's standard analyte list for this test, and the internal and surrogate standards used, are listed in Tables below.. In all other respects the method is as described in the main body of this SOP. Note that this SOP is ***not*** appropriate for drinking water analysis by method 524.2.

Table 14

Quanterra 8260 Drinking Water List Standard and Reporting Limits

Compound	CAS Number	Reporting Limits[1]			
		5 mL water µg/L	25 mL water µg/L	Low soil µg/kg	Med. Soil µg/kg
Dichlorodifluoromethane	75-71-8	10	2	10	1200
Chloromethane	74-87-3	10	2	10	1200
Bromomethane	74-83-9	10	2	10	1200
Vinyl chloride	75-01-4	10	2	10	1200
Chloroethane	75-00-3	10	2	10	1200
Trichlorofluoromethane	75-69-4	10	2	10	1200
Acetone[1]	67-64-1	20	10	20	2500
Methylene chloride	75-09-2	5	2	5	620
1,1-Dichloroethene	75-35-4	5	1	5	620
1,1-Dichloroethane	75-34-3	5	1	5	620
trans-1,2-Dichloroethene	156-60-5	2.5	0.5	2.5	310
Methyl *tert*-butyl ether (MTBE)	1634-04-4	20	5	20	620
2,2-Dichloropropane	590-20-7	5	1	5	620
cis-1,2-Dichloroethene	156-59-2	2.5	0.5	2.5	310
1,2-Dichloroethene (Total)	540-59-0	5	1	5	620
Chloroform	67-66-3	5	1	5	620
Bromochloromethane	74-97-5	5	1	5	620
1,2-Dichloroethane	107-06-2	5	1	5	620
Dibromomethane	74-95-3	5	1	5	620
2-Butanone[1]	78-93-3	20	5	20	2500
1,1,1-Trichloroethane	71-55-6	5	1	5	620
Carbon tetrachloride	56-23-5	5	1	5	620
Bromodichloromethane	75-27-4	5	1	5	620
1,2-Dichloropropane	78-87-5	5	1	5	620
cis-1,3-Dichloropropene	10061-01-5	5	1	5	620

Table 14

Quanterra 8260 Drinking Water List Standard and Reporting Limits

Compound	CAS Number	Reporting Limits[1]			
		5 mL water µg/L	25 mL water µg/L	Low soil µg/kg	Med. Soil µg/kg
Trichloroethene	79-01-6	5	1	5	620
Dibromochloromethane	124-48-1	5	1	5	620
1,2-Dibromoethane	106-93-4	5	1	5	620
1,2,3-Trichloropropane	96-18-4	5	1	5	620
1,1,2-Trichloroethane	79-00-5	5	1	5	620
Benzene	71-43-2	5	1	5	620
trans-1,3-Dichloropropene	10061-02-6	5	1	5	620
Bromoform	75-25-2	5	1	5	620
4-Methyl-2-pentanone[1]	108-10-1	20	5	20	2500
2-Hexanone[1]	591-78-6	20	5	20	2500
Tetrachloroethene	127-18-4	5	1	5	620
Toluene	108-88-3	5	1	5	620
1,1,2,2-Tetrachloroethane	79-34-5	5	1	5	620
Chlorobenzene	108-90-7	5	1	5	620
Ethylbenzene	100-41-4	5	1	5	620
Styrene	100-42-5	5	1	5	620
m and p Xylenes		2.5	0.5	2.5	310
o-xylene	95-47-6	2.5	0.5	2.5	310
Total xylenes	1330-20-7	5	1	5	620
Isopropylbenzene	98-82-8	5	1	5	620
Bromobenzene	108-86-1	5	1	5	620
n-Propylbenzene	103-65-1	5	1	5	620
2-Chlorotoluene	95-49-8	5	1	5	620
4-Chlorotoluene	106-43-4	5	1	5	620
1,3,5-Trimethylbenzene	108-67-8	5	1	5	620
tert-Butylbenzene	98-06-6	5	1	5	620
1,2,4-Trimethylbenzene	95-63-6	5	1	5	620
sec-butylbenzene	135-98-8	5	1	5	620
1,3-Dichlorobenzene	541-73-1	5	1	5	620
1,4-Dichlorobenzene	106-46-7	5	1	5	620
1,2-Dichlorobenzene	95-50-1	5	1	5	620
4-Isopropyltoluene	99-87-6	5	1	5	620
n-Butylbenzene	104-51-8	5	1	5	620
1,2-Dibromo-3-chloropropane	96-12-8	5	1	5	620
1,2,4-Trichlorobenzene	120-82-1	5	1	5	620

Table 14

Quanterra 8260 Drinking Water List Standard and Reporting Limits

Compound	CAS Number	Reporting Limits[1]			
		5 mL water µg/L	25 mL water µg/L	Low soil µg/kg	Med. Soil µg/kg
Napthalene	91-20-3	5	1	5	620
Hexachlorobutadiene	87-68-3	5	1	5	620
1,2,3-Trichlorobenzene	87-61-6	5	1	5	620

1 Not included on the method 524.2 analyte list, but includes in the calibration standard as an add on frequently requested by method 8260A.

Table 15

Internal Standards, Method 8260A Drinking water list

	Standard Concentration µg/mL	Quantitation ion
Fluorobenzene	25	128
Chlorobenzene-d5	25	114
1,4-Dichlorobenzene-d4	25	119

Notes:

1) 10 µL of the internal standard is added to the sample. This results in a concentration of each internal in the sample of 50µg/L for a 5 mL purge or 10 µg/L for a 25 mL purge.

2) Except for medium level soils, the surrogate and internal standards may be combined in one solution.

Table 16

Surrogate Standards, Method 8260B Drinking water list

Surrogate Compounds	Standard Concentration µg/mL
1,2-Dichloroethane-d_4	25
Toluene-d_8	25
4-Bromofluorobenzene	25

Notes:

1) 10 µL of the surrogate standard is added to the sample. This results in a concentration of each surrogate in the sample of 50µg/L for a 5 mL purge or 10 µg/L for a 25 mL purge.

2) Except for medium level soils, the surrogate and internal standards may be combined in one solution.

Table 17

Quanterra 8260 Drinking water list Standard: Calibration Levels

Compound	Level 1		Level 2		Level 3		Level 4		Level 5	
	5 mL	25 mL	5 mL	25 mL	5 mL	25 mL	5 mL	25 mL	5 mL	25 mL
Dichlorodifluoromethane	20	4	40	10	100	20	200	60	400	120
Chloromethane	20	4	40	10	100	20	200	60	400	120
Bromomethane	20	4	40	10	100	20	200	60	400	120
Vinyl chloride	20	4	40	10	100	20	200	60	400	120
Chloroethane	20	4	40	10	100	20	200	60	400	120
Trichlorofluoromethane	20	4	40	10	100	20	200	60	400	120
Acetone[1]	20	4	40	10	100	20	200	60	400	120
Methylene chloride	10	2	20	5	50	10	100	30	200	60
1,1-Dichloroethene	10	2	20	5	50	10	100	30	200	60
1,1-Dichloroethane	10	2	20	5	50	10	100	30	200	60
trans-1,2-Dichloroethene	10	2	20	5	50	10	100	30	200	60
Methyl *tert*-butyl ether (MTBE)	20	4	40	10	100	20	200	60	400	120
2,2-Dichloropropane	10	2	20	5	50	10	100	30	200	60
cis-1,2-Dichloroethene	10	2	20	5	50	10	100	30	200	60
Chloroform	10	2	20	5	50	10	100	30	200	60
Bromochloromethane	10	2	20	5	50	10	100	30	200	60
1,2-Dichloroethane	10	2	20	5	50	10	100	30	200	60
Dibromomethane	10	2	20	5	50	10	100	30	200	60
2-Butanone[1]	20	4	40	10	100	20	200	60	400	120
1,1,1-Trichloroethane	10	2	20	5	50	10	100	30	200	60
Carbon tetrachloride	10	2	20	5	50	10	100	30	200	60
Bromodichloromethane	10	2	20	5	50	10	100	30	200	60
1,2-Dichloropropane	10	2	20	5	50	10	100	30	200	60
cis-1,3-Dichloropropene	10	2	20	5	50	10	100	30	200	60
Trichloroethene	10	2	20	5	50	10	100	30	200	60

Table 17

Quanterra 8260 Drinking water list Standard: Calibration Levels

Compound	Level 1		Level 2		Level 3		Level 4		Level 5	
Dibromochloromethane	10	2	20	5	50	10	100	30	200	60
1,2-Dibromoethane	10	2	20	5	50	10	100	30	200	60
1,2,3-Trichloropropane	10	2	20	5	50	10	100	30	200	60
1,1,2-Trichloroethane	10	2	20	5	50	10	100	30	200	60
Benzene	10	2	20	5	50	10	100	30	200	60
trans-1,3-Dichloropropene	10	2	20	5	50	10	100	30	200	60
Bromoform	10	2	20	5	50	10	100	30	200	60
4-Methyl-2-pentanone[1]	20	4	40	10	100	20	200	60	400	120
2-Hexanone[1]	20	4	40	10	100	20	200	60	400	120
Tetrachloroethene	10	2	20	5	50	10	100	30	200	60
Toluene	10	2	20	5	50	10	100	30	200	60
1,1,2,2-Tetrachloroethane	10	2	20	5	50	10	100	30	200	60
Chlorobenzene	10	2	20	5	50	10	100	30	200	60
Ethylbenzene	10	2	20	5	50	10	100	30	200	60
Styrene	10	2	20	5	50	10	100	30	200	60
m and p Xylenes	10	2	20	5	50	10	100	30	200	60
o-xylene	10	2	20	5	50	10	100	30	200	60
Isopropylbenzene	10	2	20	5	50	10	100	30	200	60
Bromobenzene	10	2	20	5	50	10	100	30	200	60
n-Propylbenzene	10	2	20	5	50	10	100	30	200	60
2-Chlorotoluene	10	2	20	5	50	10	100	30	200	60
4-Chlorotoluene	10	2	20	5	50	10	100	30	200	60
1,3,5-Trimethylbenzene	10	2	20	5	50	10	100	30	200	60
tert-Butylbenzene	10	2	20	5	50	10	100	30	200	60
1,2,4-Trimethylbenzene	10	2	20	5	50	10	100	30	200	60
sec-butylbenzene	10	2	20	5	50	10	100	30	200	60
1,3-Dichlorobenzene	10	2	20	5	50	10	100	30	200	60
1,4-Dichlorobenzene	10	2	20	5	50	10	100	30	200	60

Table 17

Quanterra 8260 Drinking water list Standard: Calibration Levels

Compound	Level 1		Level 2		Level 3		Level 4		Level 5	
1,2-Dichlorobenzene	10	2	20	5	50	10	100	30	200	60
4-Isopropyltoluene	10	2	20	5	50	10	100	30	200	60
n-Butylbenzene	10	2	20	5	50	10	100	30	200	60
1,2-Dibromo-3-chloropropane	10	2	20	5	50	10	100	30	200	60
1,2,4-Trichlorobenzene	10	2	20	5	50	10	100	30	200	60
Napthalene	10	2	20	5	50	10	100	30	200	60
Hexachlorobutadiene	10	2	20	5	50	10	100	30	200	60
1,2,3-Trichlorobenzene	10	2	20	5	50	10	100	30	200	60

[1] Not included in the Quanterra Standard test, but included in the standard as a frequently requested add-on.

QUANTERRA STANDARD OPERATING PROCEDURE

TITLE: **EXTRACTION AND CLEANUP OF ORGANIC COMPOUNDS FROM WATERS AND SOILS, BASED ON SW-846 3500 SERIES, 3600 SERIES, 8150 AND 600 SERIES METHODS.**

(SUPERSEDES: Revision 0)

Prepared by: Richard Burrows

Reviewed by: _____
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Director, Quality Assurance

Approved by: _____
Director, Health and Safety

Approved by: _____
Management

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TABLE OF CONTENTS

1. Scope and Application 4
2. Summary of Method 4
3. Definitions 5
4. Interferences 5
5. Safety 5
6. Equipment and Supplies 6
7. Reagents and Standards 8
8. Sample Preservation and Storage 10
9. Quality Control 11
10. Calibration and Standardization 13
11. Procedure 13
    11.1 Procedural Variations 13
    11.2 Separatory Funnel Extraction from Aqueous Samples 14
    11.3 Continuous Liquid/Liquid Extraction from Aqueous Samples 18
    11.4 Sonication Extraction from Soil Samples 23
    11.5 Soxhlet Extraction from soil samples 28
    11.6 Waste dilution 30
    11.6 Concentration 31
    11.7 Cleanup Techniques 35
12. Data Analysis and Calculations 52
13. Method Performance 52
14. Pollution Prevention 52
15. Waste Management 52
16. References 53
17. Miscellaneous 53

List of Appendices:

Appendix A Extraction Procedure for Chlorinated Acid Herbicides 68

List of Tables

Table 1 Liquid / liquid extraction conditions
Table 2 Exchange solvents and final volumes
Table 3 Surrogate spiking solutions
Table 4 Matrix spike and LCS solutions
Table 5 Surrogate spike components
Table 6 Matrix spike components
Table A1 Herbicide surrogate spiking solutions

Table A2 Herbicide martix spike and LCS solutions
Table A3 Herbicide surrogate spike components
Table A4 Herbicide matrix spike and LCS components

1. SCOPE AND APPLICATION

This SOP describes procedures for preparation (extraction and cleanup) of semivolatile organic analytes in aqueous, TCLP leachate, and soil matrices for analysis by Gas Chromatography (GC) and Gas Chromatography / Mass Spectrometry (GC/MS). The procedures are based on SW-846 and 600 series methodology and are applicable for measurements made to comply with the Resource Conservation and Recovery Act (RCRA) and for wastewater testing.

1.1. Extraction procedures for the following determinitive methods are covered:
8080A, 8100, 8140, 8141A, 8150B, 8270B, 8310
608, 610, and 625

1.2. The extraction procedures here may be appropriate for other determinitive methods when appropriate spiking mixtures are used.

2. SUMMARY OF METHOD

2.1. Separatory Funnel Extraction

A measured volume of sample, typically 1 liter, is adjusted, if necessary, to a specified pH and serially extracted with methylene chloride using a separatory funnel.

2.2. Continuous Liquid/Liquid Extraction

A measured volume of sample, typically 1 litre, is placed into a continuous liquid/liquid extractor, adjusted, if necessary, to a specific pH and extracted with methylene chloride for 18-24 hours.

2.3. Sonication Extraction

A measured weight of sample, typically 30 g, is mixed with anhydrous sodium sulfate to form a free flowing powder. This is solvent extracted three times using an ultrasonic horn.

2.4. Soxhlet Extraction

A 30 g sample is mixed with anhydrous sodium sulfate to form a free flowing powder. This is extracted with refluxing solvent.

2.5. Cleanup and Concentration

Procedures are presented for removing interferents from sample extracts, and for drying and concentration of the extract to final volume for analysis.

2.6. Phenoxy acid herbicide extractions

Procedures for the extraction and cleanup of phenoxy acid herbicides are presented in Appendix A.

3. DEFINITIONS

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

4. INTERFERENCES

4.1. Method interferences may be caused by contaminants in solvents, reagents, glassware, and other processing apparatus that lead to discrete artifacts. All these materials must be routinely demonstrated to be free from interferences under conditions of the analysis by running laboratory method blanks as described in the Quality Control section. Specific selection of reagents may be required to avoid introduction of contaminants.

4.2. Visual interferences or anomalies (such as foaming, emulsions, odor, etc.) must be documented.

5. SAFETY

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

5.2. Eye protection that satisfies ANSI Z87.1 (as per the Chemical Hygiene Plan), laboratory coat and appropriate gloves must be worn while samples, standards, solvents and reagents are being handled. Disposable gloves that have become contaminated will be removed and discarded, other gloves will be cleaned immediately. Viton gloves may be worn when halogenated solvents are used for extractions or sample preparation. Nitrile gloves may be used when other solvents are handled. [Note: VITON is readily degraded by acetone]. When good manual dexterity is needed, for example, when handling small quantities/containers, disposable gloves (such as latex or N-DEX®) shall be used. While these gloves protect against splashes, they give little or no protection against contact with large quantities of solvent, and no protection against spills or immersion.

5.3. The health and safety hazards of many of the chemicals used in this procedure have not been fully defined. Additional health and safety information can be obtained from the MSDS files maintained in the laboratory. The following specific hazards are known:

Chemicals that have been classified as **carcinogens** or **potential carcinogens** under OSHA include: Methylene chloride, PCBs, certain pesticides.

The following materials are known to be **corrosive**: Concentrated sulfuric acid, sodium hydroxide.
Sulfuric acid cleanup must not be performed on any matrix that may have water present as a violent reaction between the acid and water may result in acid exploding out of the vessel.

Chemicals known to be **flammable** are: Diethyl ether, acetone, hexane.

Mercury is a highly toxic compound that must be handled with care. The analyst must be aware of the handling and clean-up techniques before handling this material.

5.4. Exposure to hazardous chemicals must be maintained **as low as reasonably achievable**, therefore, unless they are known to be non-hazardous, all samples should be opened, transferred and prepared in a fume hood, or under other means of mechanical ventilation. Solvent and waste containers will be kept closed unless transfers are being made.

5.5. The preparation of standards and reagents and glassware cleaning procedures that involve solvents such as methylene chloride will be conducted in a fume hood with the sash closed as far as the operations will permit.

5.6. All work must be stopped in the event of a known or potential compromise to the health and safety of a Quanterra associate. The situation must be reported **immediately** to a laboratory supervisor.

6. EQUIPMENT AND SUPPLIES

6.1. Glassware should be cleaned with soap and water, rinsed with water and dried in an oven at 400°C for at least 2 hours. Alternatively the glassware can be solvent rinsed with acetone or methanol followed by methylene chloride after the water rinse.

6.2. Equipment and supplies for extraction procedures

EQUIPMENT AND SUPPLIES	Sep fun.	CLLE	Soni.	Sox	Conc
Separatory Funnel: 2 L	√				
Separatory Funnel Rack	√				
Balance: >1400 g capacity, accurate ±1 g	√	√			
pH indicator paper, wide-range: covers extraction pH	√	√			
Graduated cylinder: 1 liter. (other sizes may be used)	√	√			
Erlenmeyer Flask or Fleaker: 125 & 300 mL (other sizes optional)	√		√		

EQUIPMENT AND SUPPLIES	Sep fun.	CLLE	Soni	Sox	Conc
Solvent Dispenser Pump or 100 mL Graduated Cylinder	√		√		
Continuous Liquid/Liquid Extractor		√			
Round or flat Bottom: 250, 500 mL or 1 L		√			
Boiling Chips: Contaminant free, approximately 10/40 mesh (Teflon® PTFE, carbide or equivalent).		√		√	√
Cooling Condensers		√		√	
Heating Mantle: Rheostat controlled		√		√	
Auto-timer for heating mantle		√		√	
Beakers: 250 & 400 mL, graduated			√		
Balance: >100 g capacity, accurate ±0.1 g			√	√	
Soxhlet Extractor				√	
Sonicator (at least 300 watts)			√		
Sonicator horn, 3/4 inch			√		
Kuderna-Danish (K-D) Apparatus: 500 mL					√
Concentrator Tube: 10 mL, attached to K-D with clips					√
Snyder Column: Three-ball macro					√
Water Bath: Heated, with concentric ring cover, capable of temperature control (± 5°C) up to 95°C. The bath must be used in a hood or with a solvent recovery system.					√
Vials: Glass, 2 mL, 4 mL, and 10 mL capacity with Teflon®-lined screw-cap					√
Nitrogen Blowdown Apparatus					√
Nitrogen: reagent grade.					√
Culture tubes: 10 mL, 16 mmx100 mm					√
Syringe: 1 mL	√	√	√	√	
Phase Separation Paper	√	√	√	√	
Glass Wool	√	√	√	√	
Glass Funnel: 75 X 75 mm	√	√	√	√	√
Disposable Pipets	√	√	√	√	√
Aluminum foil	√	√	√	√	√
Paper Towels	√	√	√	√	√

6.3. Equipment and Supplies for Cleanup Procedures

EQUIPMENT AND SUPPLIES	GPC	Florosil	Sulfur	Acid	A/B
Gel permeation chromatography system (GPC Autoprep Model 1002A or 1002B Analytical Biochemical Laboratories, Inc. or Zymark Benchmate or equivalent).	√				
Bio Beads: (S-X3) -200-400 mesh, 70 gm (Bio-Rad Laboratories, Richmond, CA, Catalog 152-2750 or equivalent).	√				
Chromatographic column: 700 mm x 25 mm ID glass column. Flow is upward.	√				

EQUIPMENT AND SUPPLIES	GPC	Florosil	Sulfur	Acid	A/B
Ultraviolet detector: Fixed wavelength (254 nm) and a semi-prep flow-through cell.	√				
Strip chart recorder, recording integrator, or laboratory data system.	√				
Syringe: 10 mL with Luerlok fitting.	√				
Syringe filter assembly, with disposable 5 um filter discs, Millipore No. LSWP 01300 or equivalent.	√				
Chromatographic column: 250 mm long x 10 mm ID; with Pyrex glass wool at the bottom and a Teflon stopcock (for silica gel cleanup).	√				
Vacuum system for eluting multiple cleanup cartridges. Vac Elute Manifold - Analytichem International, J.T. Baker, or Supelco (or equivalent). The manifold design must ensure that there is no contact between plastics containing phthalates and sample extracts.		√			
Vacuum trap made from a 500 mL sidearm flask fitted with a one-hole stopper and glass tubing.		√			
Vacuum pressure gauge.		√			
Rack for holding 10 mL volumetric flasks in the manifold.		√			
Mechanical shaker or mixer: Vortex Genie or equivalent.			√	√	
Separatory Funnels with Ground-Glass Stoppers: 250 mL					√
Erlenmeyer Flasks: 125 mL					√
Disposable Pipets		√	√	√	√
Culture tubes: 10 mL, 16 mmx100 mm	√	√	√	√	√

7. REAGENTS AND STANDARDS

7.1. Reagents for Extraction Procedures

All reagents must be ACS reagent grade or better unless otherwise specified.

REAGENTS	Sep fun.	CLLE	Soni	Sox	Conc
Sodium hydroxide (NaOH), Pellets: Reagent Grade	√	√			
Sodium hydroxide solution, 10 N: Dissolve 40 g of NaOH in reagent water and dilute to 100 mL.	√	√			
Sulfuric acid (H_2SO_4), Concentrated: Reagent Grade	√	√			
Sulfuric acid (1:1): Carefully add 500 mL of H_2SO_4 to 500 mL of reagent water. Mix well.	√	√			
Organic free reagent water.	√	√			
Sodium sulfate (Na_2SO_4), Granular, Anhydrous: Purify by heating at 400°C a minimum of two hours.	√	√	√	√	
Extraction/Exchange Solvents: Methylene chloride, hexane, acetonitrile, acetone, pesticide quality or equivalent	√	√	√	√	√
Acetone: Used for cleaning	√	√	√	√	√

7.2. Reagents for Cleanup Procedures

REAGENTS	GPC	Florosil	Sulfur	Acid	A/B
Florisil: 500 mg or 1 g cartridges with stainless steel or Teflon frits (calalog 694-313, Analytichem, 24201 Frampton Ave., Harbor City, CA, or equivalent.)		√			
Mercury: triple distilled			√		
Tetrabutylammonium hydrogen sulfate			√		
Sodium sulfite			√		
Tetrabutylammonium (TBA) sulfite reagent: Prepare reagent by dissolving 3.39 g of Tetrabutylammonium hydrogen sulfate in 100 mL organic-free reagent water. Extract this solution 3 times with 20 mL portions of hexane. Discard the hexane extracts. Add 25 g sodium sulfite to the water solution.			√		
2-Propanol			√		
Nitric acid: 1N			√		
Copper powder: remove oxides (if powder is dark) by treating with 1N nitric acid, rinse with organic-free reagent water to remove all traces of acid, rinse with acetone, and dry under a stream of nitrogen.			√		
Sulfuric acid, Concentrated				√	
Sodium hydroxide, Pellets					√
Sodium hydroxide, 10N: Dissolve 40 g of NaOH in 100 mL of reagent water					√
Sulfuric acid (H_2SO_4), Concentrated: Reagent Grade					√
Sulfuric acid (1:1): Carefully add 500 mL of H_2SO_4 to 500 mL of reagent water. Mix well.					√

7.3. Standards

7.3.1. Stock Standards

Stock standards are purchased as certified solutions or prepared from neats. Semivolatile stock standards are stored at $\leq 6^{\circ}C$. All stock standards must be protected from light. Stock standard solutions must be replaced after six months (from the time of preparation, if prepared in house, or from the time the ampule is opened if purchased.) Standards must be allowed to come to room temperature before use.

7.3.2. Surrogate Spiking Standards

Prepare or purchase surrogate spiking standards at the concentrations listed in Table 5. Surrogate spiking standards are prepared as dilutions

of the stock standards. Surrogate spiking solutions must be refrigerated and protected from light. The standards must be replaced at least every six months or sooner if there is reason to believe that the standard has degraded or concentrated.

7.3.3. Matrix Spiking and Laboratory Control Spiking Standards.

The same spiking solution is used for the matrix spike and the Laboratory Control Sample. Prepare MS/LCS spiking standards at the concentrations listed in Table 6. Spiking standards are purchased or prepared as dilutions of the stock standards. Surrogate spiking solutions must be refrigerated and protected from light. The standards must be replaced at least every six months or sooner if there is reason to believe that the standard has degraded or concentrated.

7.3.4. GPC calibration solution - prepare or purchase a solution in methylene chloride that contains the following analytes in the concentrations listed below:

Analyte	mg/mL
Corn Oil	25.0
Bis (2-ethylhexyl) phthalate	1.0
Methoxychlor	0.2
Perylene	0.02
Sulfur	0.08

NOTE: Sulfur is not very soluble in methylene chloride, however, it is soluble in warm corn oil. Therefore, one approach is to weigh out the corn oil, warm it, and transfer the weighed amount of sulfur into the warm corn oil. Mix it and then transfer into a volumetric flask with methylene chloride, along with the other calibration compounds. This standard has a lifetime of 6 months.

8. SAMPLE COLLECTION PRESERVATION AND STORAGE

8.1. Samples are not chemically preserved.

8.2. Samples are stored at 4°C ± 2°C in glass containers with Teflon®-lined caps.

8.3. Holding Times

8.3.1. Extraction is initiated within 7 days of the sampling date for aqueous samples, 14 days for solid and waste samples.

8.3.2. For TCLP leachates, extraction is initiated within seven days from when the leaching procedure is completed.

8.3.3. Analysis of the extracts is completed within forty days of extraction.

9. QUALITY CONTROL

9.1. Quality Control Batch

The batch is a set of up to 20 samples that are of the same matrix and are processed together using the same procedures and reagents. The batch must contain a method blank, an LCS and a matrix spike / matrix spike duplicate. (In some cases, at client request, it may be appropriate to process a matrix spike and sample duplicate in place of the MS / MSD). If clients specify specific samples for MS/MSD, the batch may contain multiple MS/MSD.

9.2. Definition of matrix

The possible matrix types are aqueous, soil, waste and TCLP leachate.

9.3. Insufficient Sample

If insufficient sample is available to process a MS/MSD, then a second LCS must be processed. The LCS pair is then evaluated according to the MS/MSD criteria. Use of a LCS pair in place of a MS/MSD must be documented as a nonconformance.

9.4. Sample count

Laboratory generated QC samples (method blanks, LCS, MS/MSD) are not included in the sample count. Field QC samples and multiple dilutions of samples are included.

9.5. Method Blank

A method blank consisting of all reagents added to the samples must be prepared and analyzed with each batch of samples. Surrogates are spiked into the method blank at the same level as the samples. The method blank is used to identify any background interference or contamination of the analytical system which may lead to the reporting of elevated concentration levels or false positive data.

9.5.1. Aqueous Method Blanks use 1000 mL of reagent water spiked with the surrogates. The method blank goes through the entire analytical procedure, including any cleanup steps.

9.5.2. Solid method blanks use 90 g of sodium sulfate spiked with the surrogates. The method blank goes through the entire analytical procedure, including any cleanup steps.

9.5.3. TCLP method blanks use 200 mL of leachate fluid (100 mL for pesticides or herbicides) spiked with the surrogates. The leachate may optionally be diluted to 1000 mL with reagent water. The method blank goes through the entire analytical procedure, including any cleanup steps.

9.6. Laboratory Control Sample (LCS)

Laboratory Control Samples are well-characterized, laboratory generated samples used to monitor the laboratory's day to day performance of routine analytical methods. The LCS, spiked with a group of target compounds representative of the method analytes, is used to monitor the accuracy of the analytical process, independent of matrix effects. On-going monitoring of the LCS results provides evidence that the laboratory is performing the method within accepted QC guidelines for accuracy and precision. The LCS goes through the entire analytical procedure, including any cleanup steps.

9.6.1. The LCS is made up in the same way as the method blank, (except that soil LCS consist of 30 g of reagent sand and 60 g of sodium sulfate) (See sections 9.5.1 - 9.5.3) but spiked with the LCS standard and the surrogates.

9.7. Surrogates

9.7.1. Surrogates are organic compounds which are similar to the target analyte(s) in chemical composition and behavior in the analytical process, but which are not normally found in environmental samples.

9.7.2. Each applicable sample, blank, LCS and MS/MSD is spiked with surrogate standards. Surrogate spike recoveries must be evaluated by determining whether the concentration (measured as percent recovery) falls within the required recovery limits.

9.8. Matrix Spike/Matrix Spike Duplicate (MS/MSD)

A matrix spike is an environmental sample to which known concentrations of target analytes have been added. A matrix spike duplicate is a second spiked aliquot of the same sample which is prepared and analyzed along with the sample and matrix spike.

9.9. Initial Demonstration of Capability

The initial demonstration and method detection limit studies described in section 13 must be acceptable before analysis of samples may begin.

9.10. Quality Assurance Summaries

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

9.11. Quanterra QC Program

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

10. CALIBRATION AND STANDARDIZATION

Refer to section 11.8.1.2 for calibration of the GPC. Otherwise this section is not applicable.

11. PROCEDURE

Procedures for separatory funnel liquid/liquid extraction (11.2), continuous liquid/liquid extraction (11.3), sonication extraction (11.4), soxhlet extraction (11.5), waste dilution (11.6), extract concentration (11.7), and extract cleanup (11.8) are presented in this section.

11.1. Procedural Variations

Procedural variations are allowed only if deemed necessary in the professional judgment of the supervisor to accommodate variation in sample matrix, radioactivity, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using a Nonconformance memo and approved by a supervisor and QA/QC manager. If contractually required, the client will be notified. The Nonconformance memo will be filed in the project file.

Any deviations from this procedure identified after the work has been completed must be documented as a nonconformance, with a cause and corrective action described. A Nonconformance memo shall be used for this documentation.

11.2. Separatory Funnel Liquid/Liquid Extraction of Water Samples.

A flow chart for this procedure is included in Section 17. There are slight differences in the initial steps for SW-846 and 600 series methods,as defined below.

11.2.1. Sample prepararion for SW-846 methods

11.2.1.1. For pesticides and TCLP leachates, measure the initial sample pH with wide-range pH paper and record on the extraction benchsheet. If sample is a leachate (e.g. TCLP), compare the current pH against leachate log, note on the benchsheet if there is any discrepancy.

11.2.1.2. If the entire sample will be used (about 1 L), weigh the sample container on a balance (± 1 g), taring the sample and container. Transfer the sample to the separatory funnel. Decant the water and leave the sediment on the bottom of the container. Reweigh the container. Assume a density of 1 g/mL and record the difference as the sample volume on the benchsheet to the nearest milliliter. Add 60 mL of methylene chloride directly to the separatory funnel. If the entire sample is not used the sample volume is measured using a graduated cylinder.

11.2.1.3. Prepare a method blank, LCS and MS/MSD for each batch as specified in section 9 of this SOP. Use 1 L of reagent water for method blanks and LCS.

11.2.1.4. Use 100 mL of leachate for TCLP pesticides, and 200 mL of leachate for TCLP semivolatiles, measured in a graduated cylinder. The leachate should be made up to 1 L in volume with reagent water.

11.2.1.5. For a TCLP method blank, measure 100 mL (200 mL for semivolatiles) of the buffer solution used in the leaching procedure and transfer to the separatory funnel. Dilute to about 1 liter with reagent water. Add 60 mL of methylene chloride to the separatory funnel.

- The sample volume may also be determined by marking the meniscus level on the sample container. Mark the meniscus and transfer the sample to the separatory funnel. Fill the sample container with water up to the marked level. Transfer the measured amount of water to a graduated

cylinder and record the volume on the benchsheet to the nearest 5 mL.

- Less than one liter of sample may be used. If necessary, dilute the sample to about 1 liter with reagent water.

11.2.1.6. Proceed to section 11.2.3 (SW-846 and 600 series).

11.2.2. 600 Series methods

11.2.2.1. Mix the sample well by shaking the container.

11.2.2.2. Measure the initial sample pH with wide-range pH paper and record on the extraction benchsheet.

11.2.2.3. If the entire sample will be used (about 1 L), weigh the sample container on a balance (± 1 g), taring the sample and container. Transfer the sample to the separatory funnel. Empty the container completely. Reweigh the container. Assume a density of 1 g/mL and record the difference as the sample volume on the benchsheet to the nearest milliliter. Add 60 mL of methylene chloride to the sample container, seal and shake to rinse the inner surface and remove residual analytes adhering to the glass walls. Transfer this solvent to the separatory funnel.

- **Warning:** Dichloromethane creates excessive pressure very rapidly! Therefore, initial venting should be done immediately after the sample container has been sealed and inverted. Vent into hood away from analysts and other samples. The sample volume may also be determined by marking the meniscus level on the sample container. Transfer the sample to the extractor (see 11.1.3). Fill the sample container with water up to the marked level. Transfer the measured amount of water to a graduated cylinder and record the volume on the benchsheet to the nearest 5 mL.
- Less than one liter of sample may be used. Dilute the sample to about 1 liter with reagent water.

11.2.2.4. Prepare a method blank, LCS and MS/MSD for each batch as specified in section 9 of this SOP. Use 1 L of reagent water for method blanks and LCS.

11.2.2.5. Proceed to section 11.2.3 (SW-846 and 600 series)

11.2.3. Add 1 mL of the surrogate spiking solution (Table 3) to each sample, method blank, Laboratory Control Sample (LCS), and Matrix Spike/Matrix Spike Duplicate (MS/MSD). Add 1 mL of the matrix spiking solution (Table 4) to each LCS and MS/MSD. Note that different matrix spiking solutions are used for 600 series, 8000 series and TCLP batches. Swirl liquid in separatory funnel gently to homogenize sample and surrogate/spiking compounds. Note that the surrogate and matrix spiking solutions should be brought to room temperature before spiking the samples.

11.2.4. Adjust sample pH as indicated in Table 1 for the initial extraction. Use the minimum amount of 1:1 H_2SO_4 or 10 N NaOH necessary. Recheck the sample with pH paper by dipping a disposable pipette into the sample and wetting the pH paper. Record adjusted pH, spiking volumes and standard numbers on the benchsheet. Return spiking solutions to the refrigerator as soon as possible.

11.2.5. Seal and shake the separatory funnel vigorously for 2 minutes with periodic venting to release excess pressure.

Warning: Dichloromethane creates excessive pressure very rapidly! Therefore, initial venting should be done immediately after the separatory funnel has been sealed and inverted. Vent into hood away from analysts and other samples.

11.2.6. Allow the organic layer to separate from the water phase until complete visible separation has been achieved (approximately 10 minutes). If the emulsion interface between layers is more than one-third the size of the solvent layer, the analyst must employ mechanical techniques to complete the phase separation. The optimum technique depends upon the sample and may include stirring, filtration of the emulsion through glass wool, centrifugation, or other physical methods. If the emulsion cannot be broken (recovery of <80% of the methylene chloride*), transfer the sample, solvent, and emulsion into the extraction chamber of a continuous extractor and proceed as described in continuous liquid-liquid extraction (Section 11.2.). If this is done, the sample must be extracted as part of a valid CLLE batch.

*Note: 15 - 20 mL of methylene chloride is expected to dissolve in 1 L of water. Thus, solvent recovery could be as low as 35 mL from the first shake and still be acceptable. Subsequent shakes should recover at least 50 mL of solvent.

11.2.7. Fill a funnel with 10-20 g of anhydrous sodium sulfate. The funnel can be plugged with glass wool or phase separation filter paper may be used to hold the sodium sulfate. Drain the solvent extract from the separatory

funnel through the prepared filtration funnel into a clean glass container. The extract may be drained directly into the KD flask. Close the stopcock just before the water level begins draining out of the separatory funnel. If the sodium sulfate becomes saturated with water add more to the funnel or replace the existing sodium sulfate with fresh drying agent.

11.2.8. Repeat the extraction process two more times using fresh 60 mL portions of solvent, combining the three solvent extracts in the collection container.

11.2.9. If extraction at a secondary pH is required, adjust the pH of the sample in the separatory funnel to the pH indicated in Table 1 with a minimum amount of 10 N NaOH or 1:1 H_2SO_4. Measure with pH paper and record the adjusted pH on the benchsheet. Serially extract with three 60 mL portions of methylene chloride, as outlined in Steps 11.1.7 to 11.1.9. Collect these three extracts in the same container used for the acid fraction.

Note: Alternatively, the acid and base fractions may be kept separate. This may be required for method 625. Separate analysis of the acid and base fractions may also be required for method 625. Individual client requirements must be checked before starting the extraction.

11.2.10. Rinse the extract residue from the sodium sulfate by pouring 20-30 mL of clean methylene chloride through the funnel and into the collection container.

11.2.11. Dispose of solvent and water remaining in the extractor into the appropriate waste container.

11.2.12. Cover with aluminum foil if the extract is not concentrated immediately. Refer to Section 11.7 for concentration and Section 11.8 for cleanup.

11.3. Continuous Liquid/Liquid Extraction from Water Samples.

A flow chart for this procedure is included in Section 17. There are slight differences in the initial steps for SW-846 and 600 series methods,as defined below.

11.3.1. Sample preparation for SW-846 methods.

11.3.1.1. Assemble the apparatus. Add 450 mL of methylene chloride to the extractor body. Add 3 to 5 boiling chips to the round bottom distilling flask.

11.3.1.2. Measure the initial sample pH with wide-range pH paper and record on the extraction benchsheet. If sample is a leachate (e.g. TCLP), compare the current pH against leachate log, note on the benchsheet if there is any discrepancy.

11.3.1.3. If the entire sample will be used (about 1 L), weigh the sample container on a balance (± 1 g), taring the sample and container. Transfer the sample to the continuous extractor body. Decant the water and leave the sediment on the bottom of the container. Reweigh the container. Assume a density of 1 g/mL and record the difference as the sample volume on the benchsheet to the nearest milliliter.

11.3.1.4. Prepare a method blank, LCS and MS/MSD for each batch as specified in section 9 of this SOP. Use 1 L of reagent water for method blanks and LCS.

11.3.1.5. Use 100 mL of leachate for TCLP pesticides, and 200 mL of leachate for TCLP semivolatiles, measured in a graduated cylinder. The leachate may be made up to 1 L in volume with reagent water.

11.3.1.6. For a TCLP method blank, measure 100 mL (200 mL for semivolatiles) of the buffer solution used in the leaching procedure and transfer to the separatory funnel. Dilute to about 1 liter with reagent water.

11.3.1.7. The sample volume may also be determined by marking the meniscus level on the sample container. Transfer the sample to the extractor (see 11.1.3). Fill the sample container with water up to the marked level. Transfer the measured amount of water

to a graduated cylinder and record the volume on the benchsheet to the nearest 5 mL.

11.3.1.8. Less than one liter of sample may be used, for highly contaminated samples. or if the reporting limit can be achieved with less than one liter of sample. In this event dilute the sample to about 1 liter with reagent water.

11.3.1.9. Proceed to section 11.3.4 (SW-846 and 600 series methods).

11.3.2. Sample preparation for 600 series methods.

11.3.2.1. Assemble the apparatus. Add 400 mL of methylene chloride to the extractor body. Add 3 to 5 boiling chips to the round bottom distilling flask.

11.3.2.2. Mix the sample well by shaking the container.

11.3.2.3. Measure the initial sample pH with wide-range pH paper and record on the extraction benchsheet.

11.3.2.4. If the entire sample will be used (about 1 L), weigh the sample container on a balance (± 1 g), taring the sample and container. Transfer the sample to the continuous extractor body. Empty the container completely. Reweigh the container. Assume a density of 1 g/mL and record the difference as the sample volume on the benchsheet to the nearest milliliter. Add 50 mL of methylene chloride to the sample container, seal and shake to rinse the inner surface and remove residual analytes adhering to the glass walls. Transfer this solvent to the continuous extractor body. If the entire sample is not used the sample volume is measured using a graduated cylinder.

Warning: Dichloromethane creates excessive pressure very rapidly! Therefore, initial venting should be done immediately after the sample container has been sealed and inverted. Vent into hood away from analysts and other samples.

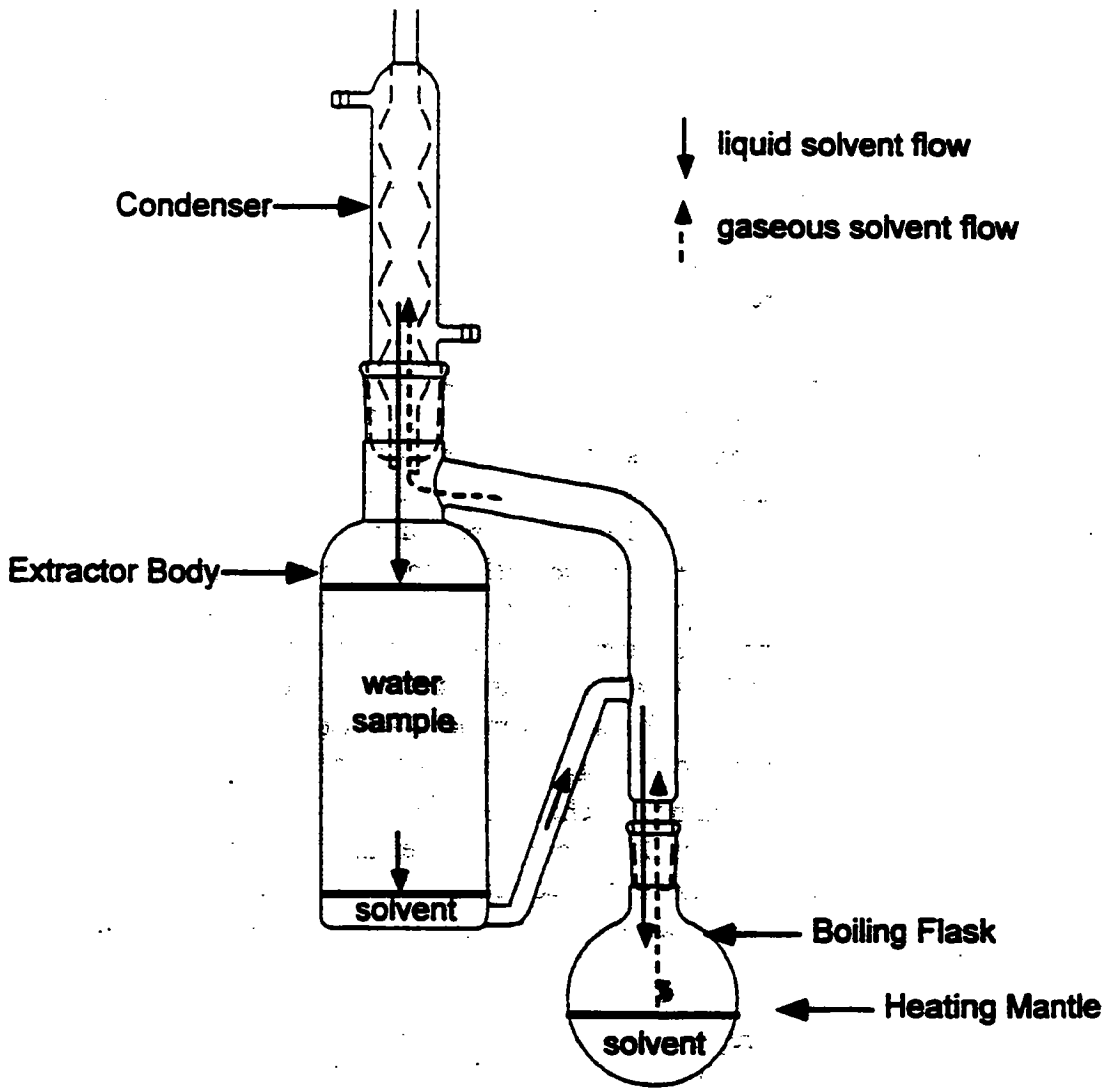
11.3.2.5. Prepare a method blank, LCS and MS/MSD for each batch as specified in section 9 of this SOP. Use 1 L of reagent water for method blanks and LCS.

11.3.2.6. The sample volume may also be determined by marking the meniscus level on the sample container. Transfer the sample to

the extractor (see 11.1.3). Fill the sample container with water up to the marked level. Transfer the measured amount of water to a graduated cylinder and record the volume on the benchsheet to the nearest 5 mL.

11.3.2.7. Less than one liter of sample may be used, for highly contaminated samples, or if the reporting limit can be achieved with less than one liter of sample. In this event dilute the sample to about 1 liter with reagent water.

11.3.2.8. Proceed to section 11.3.4 (SW-846 and 600 series methods).



11.3.3. Add 1 mL of the surrogate spiking solution (Table 3) to each sample, method blank, Laboratory Control Sample (LCS), and Matrix

Spike/Matrix Spike Duplicate (MS/MSD). Add 1 mL of the matrix spiking solution (Table 4) to each LCS and MS/MSD.

- **Different matrix spiking solutions are used for 600 series, 8000 series and TCLP batches.**
- Surrogate and matrix spiking solutions should be brought to room temperature before spiking the samples.

11.3.4. Adjust sample pH as indicated in Table 1 for the initial extraction. Use the minimum amount of 1:1 H_2SO_4 or 10 N NaOH necessary. Recheck the sample with pH paper. Record adjusted pH, spiking volumes and standard numbers on the benchsheet. Return spiking solutions to the refrigerator as soon as possible.

11.3.5. Add reagent water to the extractor body until approximately 250 mL of methylene chloride is pushed over into the round bottomed flask to ensure proper operation and solvent cycling. Attach cold condenser (about 10°C). Turn on heating mantle. Inspect joints for leaks once solvent has begun cycling. Extract for 18-24 hours. (24 hours required for 600 series)

11.3.6. If extraction at a secondary pH is required, (see Table 1) turn off the heating mantle and allow the extractor to cool. Detach the condenser and adjust the pH of the sample in the extractor body to the pH indicated in Table 1 with a minimum amount of 10 N NaOH or 1:1 H_2SO_4. Measure with pH paper and record the adjusted pH on the benchsheet. If desired, the acid and base fractions may be kept separate by replacing the boiling flask with a clean flask and fresh solvent. Reattach the condenser and turn on heating mantle. Extract for 18-24 hours.

Note: Alternatively, the acid and base fractions may be kept separate. This may be required for method 625. Separate analysis of the acid and base fractions may also be required for method 625. Individual client requirements must be checked before starting the extraction.

11.3.7. Turn off the heating mantle and allow the extractor to cool.

11.3.8. Place a funnel containing 10-20 g of anhydrous sodium sulfate on the Kuderna-Danish (K-D) apparatus or other glass container. The funnel can be plugged with glass wool enabling it to hold the granular anhydrous sodium sulfate or phase separation filter paper may be used.

11.3.9. Dry the extract in the round bottom flask by filtering it through the sodium sulfate filled funnel. Note that it is not necessary nor advisable to attempt to add the solvent remaining in the continuous extractor body to the extract.

11.3.10. Collect the dried extract in a K-D or other glass container . Rinse the flask which contained the solvent extract with 20-30 mL of methylene chloride and add it to the funnel to complete the quantitative transfer. Dispose of solvent and water remaining in the extractor in the appropriate waste container.

Note: Some types of CLLE apparatus have built in drying columns. If this type of apparatus is used then a drying step subsequent to the extraction may not be necessary.

11.3.11. Cover with aluminum foil if the extract is not concentrated immediately. Refer to Section 11.7 for concentration and Section 11.8 for cleanup.

11.4. Sonication

11.4.1. A flow chart for this procedure is included in Section 17.

11.4.2. Determination of percent moisture (Optional - if a different group performs this test, refer to the facility SOP.)

In some cases, sample results are needed on a dry weight basis. If this is the case, weigh 5-10 g of sample into a suitable tared container (typically an aluminum weigh pan. Determine the % moisture by drying overnight (at least 12 hours) at 105°C. Allow to cool in a desiccator before weighing.

$$\% \text{ Moisture} = \frac{(\text{weight of wet sample} - \text{weight of dry sample})}{\text{weight of wet sample}} \times 100$$

11.4.3. Determination of pH (Optional - if a different group performs this test, refer to the facility SOP.)

If pH determination is required, transfer 10 g of soil to a beaker. Add 10 mL of water. Stir for 1 hour. Determine the pH of the sample with a glass electrode and pH meter.

11.4.4. Sonication Procedure

11.4.4.1. Tune the sonicator at the start of each day. Refer to Section 11.4.5 for tuning instructions.

11.4.4.2. Decant and discard any water layer on a sediment/soil sample. Homogenize the sample by mixing thoroughly. Discard any foreign objects such as sticks, leaves and rocks, unless extraction of this material is required by the client. If the sample consists primarily of foreign materials consult with the client (via the Project Manager or Administrator). Document if a water layer was discarded.

11.4.4.3. Remove surrogate and matrix spiking solutions from refrigerator and allow to warm to room temperature.

11.4.4.4. Weigh 30 g of sample ± 1.0 g into a 250 or 400 mL beaker. Record the weight to the nearest 0. 1 g in the appropriate column on the benchsheet. Use 90 g of sodium sulfate for the method blank and 60 g of sodium sulfate with 30 g of reagent sand for the LCS.

11.4.4.5. Mix weighed sample with a spatula adding enough anhydrous sodium sulfate (approximately 60 g) to be free flowing. (If the sample is not free flowing extraction efficiency may be reduced)

11.4.4.6. Prepare a method blank, LCS and MS/MSD for each batch as specified in section 9 of this SOP.

11.4.4.7. Add 1 mL of the surrogate spiking solution to each sample, method blank, Laboratory Control Sample (LCS), and matrix spikes. Add 1 mL of the appropriate matrix spiking solution to each Matrix Spike/Matrix Spike Duplicate (MS/MSD) and LCS. Refer to Table 3 and 5 for details of the spiking solutions. Record spiking volumes and standard numbers on the benchsheet. Return spiking solutions promptly to refrigerator.

Note: The same volume of surrogate and matrix spiking solution is used if GPC is indicated since the final volume would be reduced to compensate for loss of extract during the GPC procedure.

11.4.4.8. Immediately add a minimum of 100 mL of 1:1 methylene chloride:acetone to the beaker.

Note: Steps 11.4.4.2 - 11.4.4.6 should be performed rapidly to avoid loss of the more volatile extractables.

11.4.4.9. Place the bottom surface of the appropriate disrupter horn tip approximately ½ inch below the surface of the solvent, but above the sediment layer.

11.4.4.10. Sonicate for 3 minutes, making sure the entire sample is agitated. If the W-380 or W-385 sonicator is used the output should be set at 6 for the 3/4 inch high gain (Q) horn or 10 for the 3/4 inch standard horn. with mode switch on pulse, and percent-duty cycle knob set at 50%.

Note: Do *not* use *Microtip* probe.

11.4.4.11. Loosely plug the stem of a 75 mm x 75 mm glass funnel with glass wool and/or line the funnel with filter paper. Add 10-20 g of anhydrous sodium sulfate to the funnel cup.

11.4.4.12. Place the prepared funnel on a collection apparatus (beaker or K-D Apparatus.

11.4.4.13. Decant and filter extracts through the prepared funnel into a clean beaker or K-D Apparatus.

11.4.4.14. Repeat the extraction two more times with additional 100 mL minimum portions of methylene chloride / acetone each time. Decant off extraction solvent after each sonication. On the final sonication pour the entire sample (sediment and solvent) into the funnel and rinse with an additional 10 mL-20 mL of the methylene chloride/acetone.

Note: Alternatively, the three extracts may be collected together and then filtered through the sodium sulfate.

11.4.4.15. Cover with aluminum foil if the extract is not concentrated immediately. Refer to Section 11.7 for concentration and Section 11.8 for cleanup.

11.4.5. Sonicator Tuning.

11.4.5.1. The tuning procedure should be conducted each day of use to assure optimum operation. The generator should also be tuned each time a horn or horntip is changed or after extended idle time.

11.4.5.2. Before proceeding, familiarize yourself with the operating instructions and control locations on your generator.

11.4.5.3. Tuning is the means of assuring optimum operation at the highest efficiency and thus the lowest power transistor temperatures. This occurs when the highest output control known setting (highest tip amplitude) is achieved at the lowest possible input power. The tuning control serves this critical function by matching the impedance of the generator to that of the converter, horn, and tip to allow these mechanical parts to resonate at 20 kHz. At maximum output control setting (10 with the flat tip, and 5 with the MICROTIP probe) adjust the tuning control to the phase angle required to maintain resonance as indicated by the minimum reading on the meter.

Note: Any change in horn or tip length by replacement, erosion, smoothing, elongation or contraction may require tuning.

11.4.5.4. Tuning standard ½", ¾", 1" and larger disrupter horns.

11.4.5.4.1. Place power (on/off/tune) switch in the "OFF" position.

11.4.5.4.2. Place the tip of the horn into a beaker of methylene chloride or methylene chloride / acetone.

11.4.5.4.3. Turn the output control knob fully counter-clockwise to the setting of "1" for minimum amplitude.

11.4.5.4.4. Turn the cycle time switch to continuous.

11.4.5.4.5. Push and hold power switch down in the "TUNE" position.

11.4.5.4.6. Turn the tuning knob in the direction that will cause the meter needle to move towards zero. Continue turning the tuning knob in the same direction until the needle stops moving and then reverses direction. Reverse the tuning knob very slowly to return the meter needle to its lowest reading (the point at which any motion of the tuning knob in either direction will cause the needle to deflect away from zero).

11.4.5.4.7. Release the power switch after the tuning criteria has been met.

11.4.5.4.8. Repeat step 11.4.5.5.5 and 11.3.5.4.6 with the output control set at 5, then 10.

Note: See tuning the MICROTIP for proper tuning of this horn type.

11.4.5.4.9. After step 11.4.5.4.8 is completed, leave output control knob on maximum setting and turn cycle time switch back to 1 second. The generator is now tuned properly and ready for use. Document the tuning of the sonicator.

11.4.5.5. Tuning the MICROTIP.

Note: These instructions are necessary to prevent fatigue failure of these very high amplitude accessories or their connecting studs.

Note: Practice tuning with standard horns before attempting to tune a MICRO-TIP

Caution: The maximum output control knob setting for these probes should ***never*** be exceeded and only approached momentarily while tuning.

Caution: If a high-pitched squeal emanates from the probe, shut off the machine, remove the tip, clean and re-tighten the joint and tune again. Do ***not*** alter the mating joint surfaces of the tip or horn.

11.4.5.5.1. The maximum output control knob setting for the MICROTIP probe is 5.

11.4.5.6. Follow procedures 11.4.5.4.1 through 11.4.5.4.9 with the exception as stated in 11.4.5.5.1

11.5. Soxhlet

11.5.1. Determination of % moisture

In some cases, sample results are needed on a dry weight basis. If this is the case, weigh 5-10 g of sample into a suitable tared container (typically an aluminum weigh pan. Determine the % moisture by drying overnight at 105°C. Allow to cool in a desiccator before weighing.

$$\% \text{ Moisture} = \frac{(\text{weight of wet sample} - \text{weight of dry sample})}{\text{weight of wet sample}} \times 100$$

11.5.2. Determination of pH

If pH determination is required, transfer 10 g of soil to a beaker. Add 10 mL of water. Stir for 1 hour. Determine the pH of the sample with a glass electrode and pH meter.

11.5.3. Decant and discard any water layer on a sediment/soil sample. Homogenize the sample by mixing thoroughly. Discard any foreign objects such as sticks, leaves and rocks, unless extraction of this material is required by the client. If the sample consists primarily of foreign materials consult with the client. Document on benchsheet if a water layer was discarded.

11.5.4. Remove surrogate and matrix spiking solutions from refrigerator and allow to warm to room temperature.

11.5.5. Weigh 30 g of sample ± 1.0 g into a beaker, recording the weight to the nearest 0.1 g on the benchsheet.. Use 30 g of sodium sulfate for the method blank and LCS. Add 30 g of anhydrous sodium sulfate and mix well. The mixture should have a free flowing texture. If not, add more sodium sulfate. Add the sample/sodium sulfate mixture to a soxhlet thimble, but do not pack the thimble tightly. The extraction thimble must drain freely for the duration of the extraction period. A glass wool plug above and below the sample in the soxhlet extractor is an acceptable alternative for the thimble.

11.5.5.1. Sample weights less than 30 g but over 5 g may be used if the appropriate reporting limits can be met.

11.5.6. Prepare a method blank, LCS and MS/MSD for each batch as specified in section 9 of this SOP, using sodium sulfate as the matrix. The weight of sodium sulfate used should be approximately the weight of soil used in each sample.

11.5.7. Add 1 mL of the surrogate spiking solution to each sample, method blank, Laboratory Control Sample (LCS), and matrix spikes. Add 1 mL of the appropriate matrix spiking solution to each Matrix Spike/Matrix Spike Duplicate (MS/MSD) and LCS. Refer to Tables 3 - 6 for details of the spiking solutions. Record spiking volumes and standard numbers on the benchsheet. Return spiking solutions promptly to refrigerator.

Note: The same volume of surrogates and matrix spiking compounds is used if GPC is indicated since the final volume would be reduced to compensate for loss of extract during the GPC procedure.

11.5.8. Place 250 mL of 1:1 methylene chloride:acetone into a 500 mL flat bottom flask containing one or two clean boiling chips. Attach the flask to the extractor and extract the sample for 16-24 hours at 4-6 cycles per hour. Check the system for leaks at the ground glass joints after it has warmed up.

11.5.9. Allow the extract to cool after the extraction is complete, then disassemble by gently twisting the soxhlet from the flask. Dry the extract in the flask by filtering it through a sodium sulfate filled funnel.

11.5.10. Collect the dried extract in a K-D or other glass container. Rinse the flask which contained the solvent extract with 20-30 mL of methylene chloride and add it to the funnel to complete the quantitative transfer.

11.5.11. Cover with aluminum foil if the extract is not concentrated immediately. Refer to Section 11.7 for concentration and Section 11.8 for cleanup.

11.6. Waste Dilution

11.6.1. This method is used for materials that are soluble in an organic solvent.

11.6.2. Transfer 10 mL of the solvent to be used for dilution into a teflon capped vial. Mark the meniscus on the vial, then discard the solvent.

11.6.3. Tare the vial, then transfer 1.0 g $\pm$ 0.1 g of sample to the vial. Record the weight to the nearest 0.1 g.

11.6.4. Add 1 mL of surrogate solution (Table 3) For GC/MS analysis, the surrogate solution should be 10x more concentrated than the normal solution (i.e. 1000/1500 ppm). For pesticides the normal surrogate solution is appropriate.

11.6.5. Dilute to 10 mL (make up to the meniscus mark) with the appropriate solvent. (Methylene Chloride for GC/MS analysis, hexane for pesticides analysis.)

11.6.6. Add 2 g + 0.1 g sodium sulfate to the sample. Cap and shake for 2 minutes.

11.6.7. Add 4-5 g sodium sulfate to a small funnel. The funnel can be plugged with glass wool or phase separation filter paper may be used to hold the sodium sulfate.

11.6.8. Pour the sample through the funnel, collecting as much as possible in a clean vial. Do NOT rinse the funnel with additional solvent, and do NOT concentrate the sample. The final volume is defined as 10 mL.

11.6.9. Label the sample, which is now ready for cleanup or analysis.

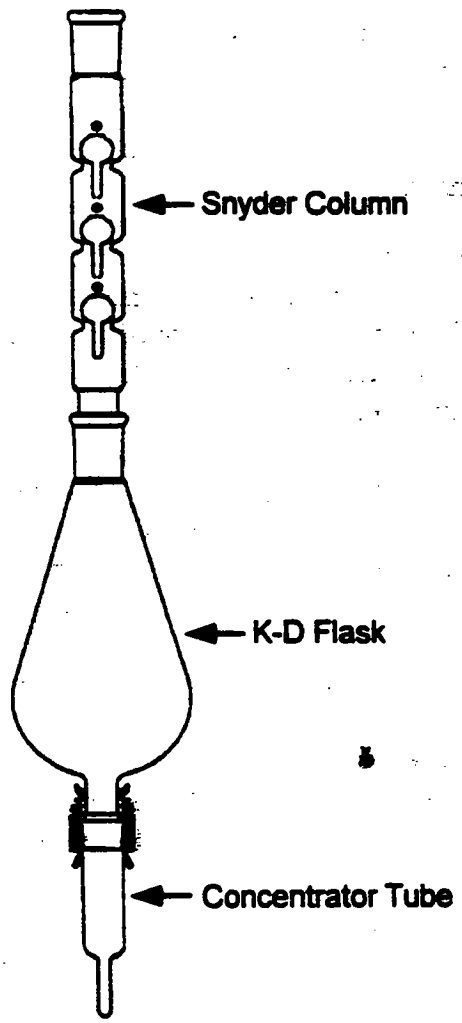
11.7. Concentration

According to the type of sample and any cleanup procedures needed, different final solvents and volumes will be required. Refer to Table 2 for the appropriate final volumes and concentrations.

11.7.1. Kuderna-Danish (KD) Method:

11.7.1.1. Assemble a Kuderna-Danish concentrator by attaching a 10 mL concentrator tube to the 500 mL KD flask. Transfer the sample to the K-D flask.

11.7.1.2. Add one or two clean boiling chips and the extract to be concentrated to the KD flask and attach a three ball Snyder Column. Add approximately 1 mL of clean methylene chloride to the top of the Snyder column (this is important to ensure that the balls are not stuck and that the column will work properly).



11.7.1.3. Place the KD apparatus on a water bath (80-90°C) so that the tip of the concentrator tube is submerged. The water level should not reach the joint between the concentrator and the KD flask. At the proper rate of distillation, the balls will actively chatter but the chambers should not flood.

11.7.1.4. Concentrate to 5-15 mL. If the determinative method requires a solvent exchange add the appropriate exchange solvent (50 mL hexane, 50 mL cyclohexane, 2 mL acetonitrile or 10 mL toluene) to the top of the Snyder Column, and then continue the water bath concentration back down to 1-4 mL. Refer to Table 2 for details of exchange solvents and final volumes. The Snyder column may be insulated if necessary to maintain the correct rate of distillation.

Note: Add an additional boiling chip with the addition of exchange solvent.
An alternative technique for solvent exchange is to replace the macro Snyder column and KD flask with a micro Snyder column, concentrate to approximately 1 mL, add 10 mL of hexane, and concentrate back down to 1 mL. The extract must be cool before the macro Snyder assembly is removed.
Note: It is very important not to concentrate to dryness as analytes will be lost.

11.7.1.5. Remove the KD apparatus from the water bath and allow to cool for a minimum of 10 minutes. If the level of the extract is above the level of the concentrator tube joint, continue to distill the solvent as necessary. Again, allow the KD flask to cool for a minimum of 10 minutes.

11.7.1.6. If the final volume is 5 or 10 mL the extract may be made up to volume in the graduated KD tube or transferred to a 12 mL vial previously marked at the appropriate volume level. Document the final volume. Otherwise proceed to section 11.7.2

11.7.2. Nitrogen Evaporation to Final Concentration

11.7.2.1. Transfer the entire extract to a calibrated evaporation tube. Rinse the concentrator tube with 1-2 mL of the appropriate solvent and transfer the solvent rinsate to the evaporation tube.

11.7.2.2. Place the tube in a warm water bath that is at least 5°C below the boiling temperature of the solvent being evaporated and evaporate the solvent using a gentle stream of nitrogen. The

nitrogen flow will form a slight depression on the surface of the solvent, but should not create splattering of the extract.

Boiling points of commonly used solvents are:

Methylene chloride	40°C
Acetone	56°C
Hexane	69°C
Acetonitrile	82°C
Toluene	111°C

11.7.2.3. During the course of the evaporation rinse the sides of the evaporation tube twice with approximately 1 mL of clean solvent. The first rinse should be about half way through the process, with the second rinse when the solvent volume gets close to 1 mL. Concentrate the solvent accurately to the calibrated volume line and transfer the extract to the appropriate storage vial.

Note: It is very important not to concentrate to dryness as analytes will be lost.

11.7.2.4. An alternative technique is to follow the previous steps concentrating the solvent to slightly below the required final volume and then drawing the extract into a syringe. Rinse the evaporation tube with a small amount of solvent and draw additional solvent into the syringe to make up the accurate final volume.

11.7.2.5. *Note:* The final concentration and volume measurement steps are critical. Use care when concentrating and make certain that the final volume measurement is accurate.

11.7.3. Turbovap Method

11.7.3.1. Turn on the Turbovap and adjust the water temperature to 5-10°C less than the boiling point of the solvent to be evaporated.

11.7.3.2. Switch all endpoint sensors to the "NO" position.

11.7.3.3. Adjust the water bath level

11.7.3.4. Adjust the nitrogen gas pressure to approximately 12 psi.

11.7.3.5. Transfer the extract into the Turbovap tube and load into the Turbovap. Do not fill the Turbovap tubes over approximately 3/4 full.

11.7.3.6. Reset the sensor and close the lid.

11.7.3.7. *Note:* If the extract splashes when the nitrogen flow starts, reduce the nitrogen flow or transfer a portion of the extract back into the original extract container.

11.7.3.8. As the extract concentrates, transfer the remainder of the extract into the appropriate Turbovap tube. After all of the extract has been transferred, rinse the flask with a few mL of methylene chloride and add to the Turbovap tube.

11.7.3.9. During the concentration rinse the Turbovap tube walls with a few mL of solvent 1 or 2 times.

11.7.3.10. Concentrate the extract to slightly less than the required final volume.

11.7.3.11. If solvent exchange is required, concentrate to 1-4 mL and add 50 mL of the exchange solvent. Concentrate back down to the appropriate volume. Refer to table 2 for details of exchange solvents and final volumes.

11.7.3.12. Transfer the concentrated extract to volumetric glassware for adjustment of final volume, using a small amount of solvent to rinse the tube and complete the transfer.

Note: Water contamination from condensation during concentration is not acceptable. If water is present, remove the Turbovap tube and filter the extract through sodium sulfate. Transfer to a clean Turbovap tube and continue the concentration.

Note 2: Dark, opaque or turbid samples may not concentrate. If this occurs, set the concentrator sensor to dryness and supervise the entire concentration procedure.

11.8. Cleanup Techniques

The following techniques may be used to remove interfering peaks, and /or to remove materials that may cause column deterioration and/ or loss of detector sensitivity.

Gel Permeation Chromatography (Section 11.8.1) is a generally applicable technique which can be used to prepare extracts for Semivolatiles (8270), PAH (8100) and pesticides (8080) analysis. It is capable of separating high molecular weight material from the sample analytes, and so is particularly useful if tissue or vegetable matter is part of the sample, and for many soil samples.

Florisil column cleanup (Section 11.8.2) is particularly useful for cleanup of pesticides for analysis by method 8080 and should normally be applied to these samples unless the matrix is clean. It separates compounds with a different polarity from the target analytes.

Gel Permeation Chromatography and Florisil column cleanup may both be applied to samples for analysis by method 8080. In this case the GPC should be performed first.

Sulfur cleanup (Section 11.8.3) is generally applied to samples for analysis by method 8080, since the Electron Capture Detector responds strongly to sulfur. It is performed after GPC and Florisil cleanup.

Sulfuric acid cleanup (Section 11.8.4) is applied to samples requiring analysis for Polychlorinated Biphenyls (PCBs) only. Most organic matter is destroyed by the sulfuric acid.

Acid Base Partition Cleanup (Section 11.8.5) is useful for separating organic acids and phenols from basic and neutral organics.

Silica gel cleanup (Section 11.8.6) may be used to separate polynuclear aromatic hydrocarbons from interfering aliphatic hydrocarbons prior to analysis by GC/FID.

11.8.1. Gel Permeation Chromatography (GPC)

Note: GPC systems include the GPC Autoprep Model 1002A or 1002B Analytical Biochemical Laboratories, Inc., Zymark Benchmate, or equivalent.

11.8.1.1. GPC Column Preparation

11.8.1.1.1. Weigh out 70 g of Bio Beads (SX-3) into a 400-mL beaker.

11.8.1.1.2. Add approximately 300 mL of methylene chloride and stir gently.

11.8.1.1.3. Cover with aluminum foil and allow the beads to swell for a minimum of two hours. Maintain enough solvent to sufficiently cover the beads at all times.

11.8.1.1.4. Position and tighten the outlet bed support (top) plunger assembly in the tube by inserting the plunger and turning it clockwise until snug. Install the plunger near the column end but no closer than 5 cm (measured from the gel packing to the collar).

11.8.1.1.5. Turn the column upside down from its normal position with the open end up. Place the tubing from the top plunger assembly into a waste beaker below the column.

11.8.1.1.6. Swirl the bead/solvent slurry to get a homogeneous mixture and pour the mixture into the open end of the column. Transfer as much as possible with one continuous pour trying to minimize bubble formation. Pour enough to fill the column. Wait for the excess solvent to drain out before pouring in the rest. Add additional methylene chloride to transfer the remaining beads and to rinse the beaker and the sides of the column. If the top of the gel begins to look dry, add more methylene chloride to rewet the beads.

11.8.1.1.7. Wipe any remaining beads and solvent from the inner walls of the column with a laboratory tissue. Loosen the seal slightly on the other plunger assembly (long plunger) and insert it into the column. Make the seal just tight enough so that any beads on the glass surface will be pushed forward, but loose enough so that the plunger can be pushed forward.

CAUTION: Do not tighten the seal if beads are between the seal and the glass surface because this can damage the seal and cause leakage.

11.8.1.1.8. Push the plunger until it meets the gel, then compress the column bed about 4 cm.

11.8.1.1.9. Connect the column inlet to the solvent reservoir and place the column outlet tube in a waste container. Pump methylene chloride through the column at a rate of 5 mL/min. for one hour.

11.8.1.1.10. After washing the column for at least one hour, connect the column outlet tube to the inlet side of the UV detector. Connect the system outlet to the outlet side of the UV detector. Placing a restrictor (made from a piece of capillary tubing of 1/16"OD x 10/1000"ID x 2") in the outlet tube from the UV detector will prevent bubble formation which causes a noisy UV baseline. The restrictor will not effect the flow rate. After pumping methylene chloride through the column for an additional 1-2 hours, adjust the inlet bed support plunger until approximately 6-10 psi back-pressure is achieved. Push the plunger in to increase pressure or slowly pull outward to reduce pressure.

11.8.1.1.11. When the GPC column is not to be used for several days, connect the column inlet and outlet lines to prevent column drying and/or channeling. If channeling occurs, the gel must be removed from the column, re-swelled, and re-poured as described above. If drying occurs, pump methylene chloride through the column until the observed column pressure is constant and the column appears wet. Always recalibrate after column drying has occurred to verify that retention volumes have not changed.

11.8.1.2. Initial Calibration of the GPC Column

11.8.1.2.1. Before use, the GPC must be calibrated based on monitoring the elution of standards with a UV detector connected to the GPC column.

11.8.1.2.2. Pump solvent through the GPC column for 2 hours. Verify that the flow rate is 4.5-5.5 mL/min. Corrective action must be taken if the flow rate is outside this range. Record the column pressure (should be 6-10 psi) and room temperature (22°C is ideal).

Note: Changes in pressure, solvent flow rate, and temperature conditions can affect analyte retention

times and must be monitored. If the flow rate and/or column pressure do not fall within the above ranges, a new column should be prepared.

11.8.1.2.3. Inject the calibration solution and retain a UV trace that meets the following requirements (See resolution calculation in section 11.8.1.6):

- Peaks must be observed and should be symmetrical for all compounds in the calibration solution.
- Corn oil and phthalate peaks must exhibit >85% resolution.
- Phthalate and methoxychlor peaks must exhibit >85% resolution.
- Methoxychlor and perylene peaks must exhibit >85% resolution.
- Perylene and sulfur peaks must not be saturated and must exhibit >90% baseline resolution.

11.8.1.2.4. A UV trace that does not meet the criteria in paragraph 11.8.1.2.3 indicates the need for system maintenance and/or the need for a new column.

11.8.1.2.5. Determine appropriate collect and dump cycles.

11.8.1.2.6. The calibrated GPC program for pesticides/PCB should dump >85% of the phthalate and should collect >95% of the methoxychlor and perylene. Use a wash time of 10 minutes.

11.8.1.2.7. For semivolatile extracts, initiate a column eluate collection just before the elution of bis (2-ethylhexyl) phthalate and after the elution of the corn oil. Stop eluate collection shortly after the elution of perylene. Stop collection before sulfur elutes. Use a wash time of 10 minutes after the elution of sulfur.

11.8.1.2.8. Reinject the calibration solution after appropriate dump and collect cycles have been set.

11.8.1.2.9. Measure and record the volume of collected GPC eluate in a graduated cylinder.

11.8.1.2.10. The retention times for both bis(2-ethylhexyl) phthalate and perylene must not vary more than +/- 5% between calibrations.

11.8.1.3. GPC calibration check

Check the calibration of the GPC immediately after the initial calibration and at least every 7 days thereafter, while the column is in use.

11.8.1.3.1. Inject the calibration solution, and obtain a UV trace. If the retention times of bis(2-ethylhexyl)phthalate or perylene have changed by more than ± 5% use this run as the start of a new initial calibration. Otherwise, proceed with the recovery check. Excessive retention time shifts may be caused by poor laboratory temperature control or system leaks, an unstabilized column, or high laboratory temperature causing outgassing of methylene chloride. Pump methylene chloride through the system and check the retention times each day until stabilized.

11.8.1.4.. GPC Recovery Check for Pesticides/ PCBs

11.8.1.4.1. The recovery from the GPC must be verified immediately after the initial calibration and at least every 7 days, when the instrument is in use. Two recovery check solutions are used. The first mixture is prepared by diluting 1.0 mL of the pesticide matrix spiking solution (Table 6) to 10 mL in methylene chloride. The second mixture is prepared by diluting 1 mL of the PCB only matrix spiking solution (Table 6) to 10 mL with methylene chloride.

11.8.1.4.2. Load the pesticide matrix spike mixture, the PCB mixture, and a methylene chloride blank onto the GPC using the GC dump and collect values.

Note: If the analysis is for PCBs only, then the pesticide recovery check is not necessary.

11.8.1.4.3. After collecting the GPC calibration check fraction, concentrate, solvent exchanging to hexane. Adjust the final volume to 5.0 mL, and analyze by GC/EC. Refer to concentration, section 11.7.

11.8.1.4.4. The methylene chloride blank may not exceed more than one half the reporting limit of any analyte. And if the recovery of each of the single component analytes is 80-110% and if the Aroclor pattern is the same as previously run standards, then the analyst may use the column. If the above criteria are not met, there may be a need for system maintenance.

11.8.1.5. GPC Recovery Check for Semivolatiles

11.8.1.5.1. The recovery from the GPC must be verified immediately after the initial calibration and at least every 7 days, when the instrument is in use. Dilute 1.0 mL of the semivolatiles matrix spiking solution (Table 6) to 10 mL in methylene chloride.

11.8.1.5.2. Load the matrix spike mixture and a methylene chloride blank onto the GPC using the semivolatiles dump and collect values.

11.8.1.5.3. After collecting the GPC recovery check fraction, concentrate to 0.5 mL, and analyze by GC/MS. Refer to the concentration section 11.7.

11.8.1.5.4. Recovery of the matrix spike analytes should be at least 85%. The blank should not contain any analytes at or above the reporting limit. If these conditions are met the column may be used for sample analysis. Otherwise correct the contamination problem, or extend the collect time to improve recovery of target analytes.

11.8.1.6. Sample Extract Cleanup

11.8.1.6.1. Reduce the sample extract volume to 1-2 mL, then adjust to 10 mL with methylene chloride prior to cleanup. This reduces the amount of acetone in the extract. Refer to section 11.7.

11.8.1.6.2. Start the pump and let the flow stabilize for 2 hours. The solvent flow rate should be 4.5-5.5 mL/min. The ideal laboratory temperature to prevent outgassing of the methylene chloride is 22°C. The normal backpressure is 6-10 psi.

11.8.1.6.3. In order to prevent overloading of the GPC column, highly viscous sample extracts must be diluted prior to cleanup. Any sample extract with a viscosity greater than that of a 1:1 glycerol:water solution (by visual comparison) must be diluted and loaded into several loops.

11.8.1.6.4. Samples being loaded onto the GPC should be filtered with a 5 micron (or less) filter disk. Attach a filter to a 10 mL Luerlok syringe and filter the 10 mL sample extract into the sample tube.

Note: The Zymark Benchmate automatically filters the sample extracts, but manual prefiltration of very dirty extracts may be necessary.

11.8.1.6.5. Load the filtered (or unfiltered if using the Benchmate) samples into the proper sample tubes and place on the GPC.

Note: For the GPC Autoprep Model 1002A, wash the loading port with methylene chloride after loading each sample loop in order to minimize cross contamination. This step is automated on the GPC Autoprep 1002B and on the Benchmate.

11.8.1.6.6. Set the collect, dump, and wash times determined by the calibration procedure.

11.8.1.6.7. Switch to the run mode and start the automated sequence. Process each sample using the collect and dump cycle times established by the calibration procedure.

11.8.1.6.8. Collect each sample in a suitable glass container. Monitor sample volumes collected.

11.8.1.6.9. Any samples that were loaded into 2 or more positions must be recombined.

11.8.1.6.10. Concentrate semivolatile sample extracts to 0.5 mL in methylene chloride. Refer to the concentration section 11.7.

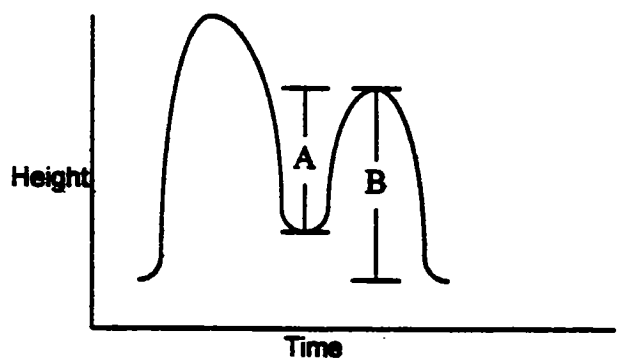
11.8.1.6.11. Solvent exchange pesticide/PCB sample extracts into hexane and concentrate to 5.0 mL. Refer to the concentration section 11.7.

11.8.1.7. Calculations

11.8.1.7.1. Resolution

To calculate the resolution between two peaks on a chromatograph, divide the depth of the valley between the peaks by the peak height of the smaller peak being resolved and multiply by 100.

Resolution Calculation



$$\% \text{ Resolution} = \frac{A}{B} \times 100$$

Where: A = depth of valley to height of smaller peak
B = peak height of smaller peak

11.8.1.7.2. Dump Time

Mark on the chromatograph the point where collection is to begin. Measure the distance from the injection point. Divide the distance by the chart speed. Alternatively the collect and dump times may be measured by means of an integrator or data system.

$$\text{Dump time (min)} = \frac{\text{Distance (cm) from injection to collection start}}{\text{Chart speed (cm / min)}}$$

11.8.1.7.3. Collection Time

$$\text{Collection time (min)} = \frac{\text{Distance (cm) between collection start and stop}}{\text{Chart speed (cm / min)}}$$

11.8.2. Florisil Cartridge Cleanup

Note 1: Systems for eluting multiple cleanup cartridges include the Supelco, Inc. Solid Phase Extraction (SPE) assembly, Zymark Benchmate, or equivalent.

Note 2: Follow the lab specific procedure when using the Zymark benchmate.

11.8.2.1. Before florisil cleanup sample volume must be reduced to 10 mL (5 mL if GPC cleanup was used) and the solvent must be hexane. Refer to Section 11.7 for details of concentration.

11.8.2.2. Attach a vacuum manifold to a vacuum pump or water aspirator with a trap installed between the manifold and the vacuum. Adjust the vacuum in the manifold to 5-10 psi.

11.8.2.3. Place one Florisil cartridge into the vacuum manifold for each sample extract. Prior to cleanup of samples, pre-elute each cartridge with 5 mL of hexane/acetone (9:1). Adjust the vacuum applied to each cartridge so that the flow through each cartridge is approximately 2 mL/min. Do not allow the cartridges to go dry.

11.8.2.4. Just before the cartridges go dry, release the vacuum to the manifold and remove the manifold top.

11.8.2.5. Place a rack of clean labeled 12 mL concentrator tubes into the manifold and replace the manifold top. Make sure that the solvent line from each cartridge is placed inside the appropriate tube.

11.8.2.6. After the clean tubes are in place, vacuum to the manifold is restored and 2.0 mL of the extract is added to the appropriate Florisil cartridge.

11.8.2.7. The pesticides/aroclors in the extract concentrates are then eluted through the column with 8 mL of hexane/acetone (90:10) and are collected into the 10 mL culture tube or concentrator tube held in the rack inside the vacuum manifold.

11.8.2.8. Transfer the extract to a graduated concentrator tube and concentrate the extract to 2 mL. Transfer the extract to a labeled vial. Refer to the concentration (Section 11.7)

Note 1: A cartridge performance standard must be run with each lot of Florisil cartridges.

Note 2: Florisil cartridge performance check--every lot number of Florisil must be tested before use. Add 0.5 ug/mL of 2,4,5-trichlorophenol solution and 0.5 mL of GC Standard Mix A (midpoint concentration) to 4 mL hexane. Reduce volume to 0.5 mL. Add the concentrate to a pre-washed Florisil cartridge and elute with 9 mL hexane/acetone [(90:10)(v/v)]. Rinse cartridge with 1.0 mL hexane two additional times. Concentrate eluate to 1.0 mL final volume and transfer to vial. Analyze the solution by GC/EC. The test sample must show 80 to 120% recovery of all pesticide analytes with <5% trichlorophenol recovery, and no peaks interfering with target compounds can be detected. This standard has a lifetime of six months. Alternatively, this standard may be purchased as a stock solution.

11.8.3. **Sulfur Removal**

11.8.3.1. Sulfur can be removed by one of three methods: mercury, copper, or tetrabutylammonium sulfite (TBA) according to laboratory preference. If the sulfur concentration is such that crystallization occurs in the concentrated extract, centrifuge the extract to settle the crystals, and carefully draw off the sample extract with a disposable pipet, leaving the excess sulfur in the centrifuge tube. Transfer the extract to a clean concentrator tube before proceeding with further sulfur cleanup.

11.8.3.2. Sulfur Removal with Elemental Mercury

Note: Use Mercury sparingly in order to minimize exposure and disposal costs.

11.8.3.2.1. Transfer 2 mL of sample extract into a clean concentrator tube or Teflon sealed vial.

11.8.3.2.2. Add one to three drops of mercury to the extract vial and seal.

11.8.3.2.3. Shake well for 15-30 seconds. If prolonged shaking is required, use a mechanical shaker.

11.8.3.2.4. Remove the extract from the mercury using a disposable pipet and transfer to a clean vial.

11.8.3.2.5. If black precipitate forms, sulfur was present. Shake again, then centrifuge. After centrifugation, transfer the supernate to a clean test tube and repeat. Do this until relatively little precipitate remains, or the screens indicate that cleanup is complete.

11.8.3.2.6. Properly dispose of the mercury waste.

11.8.3.3. Sulfur Removal with Copper

11.8.3.3.1. Transfer 1.0 mL of sample extract into a centrifuge or concentrator tube.

11.8.3.3.2. Add approximately 2 g of cleaned copper powder (see 7.1.15.4 for copper cleaning procedure) to the sample extract tube.

11.8.3.3.3. Mix for one minute on a mechanical shaker.

11.8.3.3.4. If the copper changes color, sulfur was present. Repeat the sulfur removal procedure until the copper remains shiny.

11.8.3.3.5. Transfer the supernate to a clean vial.

11.8.3.4. Sulfur Removal with Tetrabutylammonium (TBA) Sulfite Reagent

11.8.3.4.1. Transfer 1.0 mL of sample extract into a culture tube.

11.8.3.4.2. Add 1.0 mL TBA sulfite reagent and 2 mL 2-propanol to the sample extract. Cap and shake for 1 minute. If clear crystals (precipitated sodium sulfite) form, sufficient sodium sulfite is present.

11.8.3.4.3. If a precipitate does not form, add sodium sulfite in approximately 0.1 g portions until a solid residue remains after repeated shaking.

11.8.3.4.4. Add 5 mL organic free reagent water and shake for 1 minute. Allow sample to stand for 5-10 minutes. (Centrifuge if necessary to separate the layers). Transfer the sample extract (top layer) to a vial. The final volume is defined as 1.0 mL in section 11.8.3.4.1.

11.8.4. Sulfuric Acid Cleanup

11.8.4.1. Add approximately 10 mL of concentrated sulfuric acid to 2 mL of sample extract in a Teflon capped vial.

Caution: There must be no water present in the extract or the reaction may shatter the sample container.

11.8.4.2. Shake for about thirty seconds and allow to settle. (Centrifuge if necessary)

11.8.4.3. Remove the sample extract (top layer) from the acid using a Pasteur pipet and transfer to a clean vial. ***CAUTION:*** It is not necessary to remove all the extract since the final volume is already determined. Transfer of small amounts of sulfuric acid along with the extract will result in extremely rapid degradation of the chromatographic column.

11.8.4.4. If the sulfuric acid layer becomes highly colored after shaking with the sample extract, transfer the hexane extract to a clean vial and repeat the cleanup procedure until color is no longer being removed by the acid, or a maximum of 5 acid cleanups.

11.8.4.5. Properly dispose of the acid waste.

11.8.5. Acid/Base Partition Cleanup

11.8.5.1. Place 10 mL of the solvent extract from a prior extraction procedure into a 125 mL separatory funnel.

11.8.5.2. Add 20 mL of methylene chloride to the separatory funnel.

11.8.5.3. Slowly add 20 mL of DI water which has been previously adjusted to a pH of 12 to 13 with 10 N sodium hydroxide.

11.8.5.4. Seal and shake the separatory funnel for at least two minutes with periodic venting to release excess pressure.

CAUTION: Methylene chloride creates excessive pressure very rapidly. Initial venting should be done immediately after the separatory funnel has been sealed.

11.8.5.5. Allow the organic layer to separate from the aqueous phase for a minimum of ten minutes.

11.8.5.6. If an emulsion interface between layers is more than one-third the size of the solvent layer, the analyst must employ mechanical techniques to complete the phase separation. The optimum technique depends upon the sample, and may include stirring, filtration of the emulsion through glass wool, centrifugation, or other physical methods.

11.8.5.7. Separate the aqueous phase and transfer it to a 125 mL Erlenmeyer flask. Repeat the extraction two more times using fresh 20 mL aliquots of dilute sodium hydroxide (pH 12-13). Combine the aqueous extracts.

11.8.5.8. Water soluble organic acids and phenols will be primarily in the aqueous phase. Base/neutral analytes will be in the methylene chloride. If the analytes of interest are only in the aqueous phase discard the methylene chloride and proceed to Section 11.8.5.9. If the analytes of interest are only in the methylene chloride, discard the aqueous phase and proceed to Section 11.8.5.11.

11.8.5.9. Adjust the aqueous phase to a pH of 1-2 with sulfuric acid (1:1). Allow to cool and transfer the cool aqueous phase to a clean 125 mL separatory funnel.

11.8.5.10. Add 20 mL of methylene chloride to the separatory funnel and shake for at least two minutes. Allow the methyene chloride to separate from the aqueous phase and collect the methylene chloride in an Erlenmeyer flask. Repeat the extraction two more times using fresh methylene chloride and extracting at pH 1-2. Combine the three extracts.

11.8.5.11. Dry the extract by passing through a funnel containing 10-20 g anhydrous sodium sulfate. Rinse the funnel with an additional 20-30 mL of clean methylene chloride

11.8.5.12. Cover with aluminum foil if the extract is not concentrated immediately. Refer to section 11.7 for concentration.

11.8.5.13. Dispose of solvent and water remaining in the separatory funnel into the appropriate waste container.

11.8.6. Silica Gel Cleanup

This procedure is appropriate for cleanup of extracts prior to determination of polynuclear aromatic hydrocarbons by GC/FID.

11.8.6.1. Exchange the extract solvent to cyclohexane and reduce the volume to 2 mL prior to cleanup.

11.8.6.2. Prepare a slurry of approximately 10 g of activated silica gel in about 15 mL of methylene chloride. Place this into a 10 mm ID chromatographic column. Tap the column to settle the silica gel. Add 1-2 cm of anhydrous sodium sulfate to the top of the silica gel.

11.8.6.3. Preelute the column with 40 mL of pentane. Elution rate should be approximately 2 mL/min. Discard the eluate and just prior to exposure of the sodium sulfate to air, transfer the 2 mL cyclohexane sample extract onto the column using an additional 2 mL of cyclohexane to complete the transfer. Just prior to exposure of the sodium sulfate layer to the air, add 25 mL of pentane and continue the elution of the column. Discard this pentane elute.

11.8.6.4. Elute the column with 25 mL of methylene chloride/pentane (2:3 v/v). Since the polynuclear aromatic hydrocarbons elute in this fraction, collect the eluate and concentrate to the required volume.

12. DATA ANALYSIS AND CALCULATIONS

Not applicable

13. METHOD PERFORMANCE

13.1. Method detection limit

Each laboratory must generate a valid method detection limit for each analyte of interest. The procedure for the determination of the method detection limit is given in Quanterra QA Policy #: QA-005

13.2. Initial demonstration

Each laboratory must make an initial demonstration of capability for each individual method. This requires analysis of four QC Check samples. The QC check sample is a well characterized laboratory generated sample used to monitor method performance, which should contain all the analytes of interest. The spiking level should be equivalent to a mid level calibration. (For certain tests more than one set of QC check samples may be necessary in order to demonstrate capability for the full analyte list.)

13.2.1. Four aliquots of the QC check sample are analyzed using the same procedures used to analyze samples, including sample preparation.

13.2.2. Calculations and acceptance criteria for the QC check samples are given in the determinative SOPs. (CORP-GC-0001, CORP-MS-0001, 0002)

13.3. Training Qualification

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

14. POLLUTION PREVENTION

Within the constraints of following the methodology in this SOP, use of organic solvents should be minimized.

15. WASTE MANAGEMENT

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

15.1. Solvent waste must be disposed of in the clearly labeled waste cans.

15.2. Acid waste must be collected in the clearly labeled acid waste containers.

15.3. Solid materials (soil, gloves, soiled paper products) are placed in the solid debris container. Do not put liquids in the solid waste container.

15.4. Methylene chloride saturated water and remaining sample after the extraction is dumped into Methylene chloride waste cans.

16. REFERENCES

Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW846, 3rd Edition, Final Update II (September 1994). Sections 3500A, 3510B, 3520B, 3540B, 3550A, 3600B, 3610A, 3620A, 3640A, 3650A, and 3660A.

17. MISCELLANEOUS

17.1. Modifications from Reference method

17.1.1. Some surrogate spiking concentrations are modified from those recommended in SW-846, in order to make the concentrations more consistent with the calibration levels in the determinative methods.

17.1.2. Aqueous sample volumes may be determined by weight.

17.1.3. Toluene is used as the final solvent for analysis of organophosphorous pesticides by method 8140 / 8141, since these compounds have limited solubility in hexane.

17.1.4. In the CLLE procedure the same solvent is used for the acid and base neutral extractions.

17.1.5. Spiking levels for method 608 have been reduced by a factor of ten to bring the levels within the nrmal calibration range of the instrument.

17.1.6. 10 g of soil is used for pH determination, rather than the 50 g suggested in the reference method. The volume of water is also adjusted to maintain the sample / water ratio specified in the method.

17.2. Modifications from previous revisions

17.2.1. Directions for GPC calibration have been clarified

17.2.2. The spiking amount for semivolatiles has been changed from 1 mL to 0.5 mL to allow for a change from 1µL to 2µL injections in the analysis procedure.

17.2.3. A number of typographical errors have been corrected.

17.3. Facility Specific SOPs

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

17.4. Tables

Table 1
Liquid /Liquid Extraction Conditions

Determinative Method	Initial Ext. pH	Secondary Ext. pH
BNA: 8270	1-2	11-12
625	11-12	1-2
Pest/PCB: 8080 & 608	5-9	None
OPP: 8140	6-8	None
OPP: 8141	as received	None
Hydrocarbons: 8015	as received	None
PAH: 8310, 8100 & 610	as received	None

Table 2
Exchange Solvents and Final Volumes

Final Volumes and Exchange Solvents if no cleanup is used		
Type	Exchange Solvent for Analysis	Final Volume for Analysis
Semivolatiles	N/A	1.0
Pesticides	Hexane	10.0
PAH by HPLC	Acetonitrile	Refer to facility SOP for HPLC
PAH by GC	N/A	1.0
TPH	N/A	1.0
OPP	Toluene	10.0

Final Volumes and exchange solvents if GPC cleanup is used			
Type	Exchange Solvent for GPC	Final Volume for GPC	Final Volume and solvent for Analysis
Semivolatiles	CH_2Cl_2	10 mL[1]	0.5 mL CH_2Cl_2
Pesticides	CH_2Cl_2	10 mL[1]	5 mL, hexane
OPP	CH_2Cl_2	10 mL	5 mL, toluene

[1] Final volume for GPC may be 4 mL if a 2 mL sample loop is used

Final volumes and exchange solvents if Florisil cleanup is used			
Type	Exchange Solvent for Florisil	Final Volume for Florisil	Final Volume and solvent for Analysis
Pesticides	Hexane	10 mL (2 mL aliquot used)	2 mL, hexane

Final volumes and exchange solvents if both GPC and Florisil cleanup are used					
Type	Exchange Solvent for GPC	Final Volume for GPC	Exchange Solvent for Florisil	Final Volume for Florisil	Final volume for analysis
Pesticides	Methylene Chloride	10 mL	Hexane	5 mL (2 mL aliquot used)	2 mL, hexane

Note: Different final volumes may be necessary to meet special client reporting limit requirements.

Note: If silica gel cleanup is performed, exchange solvent to cyclohexane prior to cleanup.

Table 3		
Surrogate Spiking Solutions		
Analyte Group	Surrogate Spike Solution ID	Volume (mL)
BNA	100/150 ppm BNA	0.5
Pest	0.2 ppm DCB/TCX	1.0
Pest/PCB	0.2 ppm DCB/TCX	1.0
PCB	0.2 ppm DCB/TCX	1.0

Table 4		
Matrix Spike and LCS Solutions		
Analyte Group	Matrix Spike Solution ID	Volume (mL)
BNA	100/150 ppm BNA	0.5
BNA TCLP	BNA TCLP Spike	0.5
BNA NPDES	BNA NPDES Spike	0.5
Pest	0.2/0.5 ppm Pest	1.0
Pest TCLP	Pest TCLP Spike	1.0
Pest NPDES	Pest NPDES Spike	1.0
Pest/PCB	0.5 ppm pest spike	1.0
PCB	10 ppm PCB Spike	1.0

Table 5			
Surrogate Spike Components			
Type	Compounds	Solvent	Conc. (µg/mL)
BNA	2-Fluorobiphenyl	Methanol	100
	Nitrobenzene-d5		100
	p-Terphenyl-d14		100
	2-Fluorophenol		150
	Phenol-d6		150
	2,4,6-Tribromophenol		150
	1,2-Dichlorobenzene-d4		100
	2-Chlorophenol-d4		150
Pest/PCB	Decachlorobiphenyl	Acetone	0.2
	Tetrachloro-m-xylene		0.2

Table 6			
Matrix Spike Components			
Type	Compounds	Solvent	Conc. (µg/mL)
TCL BNA	Acenaphthene	Methanol	100
	4-Chloro-3-Methylphenol		150
	2-Chlorophenol		150
	1,4-Dichlorobenzene		100
	2,4-Dinitrotoluene		100
	4-Nitrophenol		150
	N-Nitroso-Di-n-Propylamine		100
	Pentachlorophenol		150

Table 6

Matrix Spike Components

Type	Compounds	Solvent	Conc. (µg/mL)
	Phenol		150
	Pyrene		100
	1,2,4-Trichlorobenzene		100
BNA TCLP	2,4-Dinitrotoluene	Methanol	100
	Hexachlorobutadiene		100
	Hexachloroethane		100
	2-Methylphenol		100
	3-Methylphenol		100
	4-Methylphenol		100
	Nitrobenzene		100
	Pentachlorophenol		100
	Pyridine		100
	2,4,5-Trichlorophenol		100
	2,4,6-Trichlorophenol		100
BNA NPDES		Methanol	
	Acenaphthene		100
	Acenaphthylene		100
	Anthracene		100
	Benzo(a)anthracene		100
	Benzo(b)fluoranthene		100
	Benzo(k)fluoranthene		100
	Benzo(a)pyrene		100
	Benzo(ghi)perylene		100
	Benzyl butyl phthalate		100

Table 6

Matrix Spike Components

Type	Compounds	Solvent	Conc. (µg/mL)
	Bis(2-chloroethyl)ether		100
	Bis(2-chloroethoxy)methane		100
	Bis(2-ethylhexyl)phthalate		100
	Bis(2-chloroisopropyl)ether		100
	4-Bromophenyl phenyl ether		100
	2-Chloronaphthalene		100
	4-Chlorophenyl phenyl ether		100
	Chrysene		100
	Dibenzo(a,h)anthracene		100
	Di-n-butylphthalate		100
	1,3-Dichlorobenzene		100
	1,2-Dichlorobenzene		100
	1,4-Dichlorobenzene		100
	3,3'-Dichlorobenzidine		100
	Diethyl phthalate		100
	Dimethyl phthalate		100
	2,4-Dinitrotoluene		100
	2,6-Dinitrotoluene		100
	Di-n-octylphthalate		100
	Fluoranthene		100
	Fluorene		100
	Hexachlorobenzene		100
	Hexachlorobutadiene		100
	Hexachloroethane		100

Table 6

Matrix Spike Components

Type	Compounds	Solvent	Conc. (µg/mL)
	Indeno(1,2,3-cd)pyrene		100
	Isophorone		100
	Naphthalene		100
	Nitrobenzene		100
	N-Nitrosodi-n-propylamine		100
	Phenanthrene		100
	Pyrene		100
	1,2,4-Trichlorobenzene		100
	4-Chloro-3-methylphenol		100
	2-Chlorophenol		100
	2,4-Dichlorophenol		100
	2,4-Dimethylphenol		100
	2,4-Dinitrophenol		100
	2-Methyl-4,6-dinitrophenol		100
	2-Nitrophenol		100
	4-Nitrophenol		100
	Pentachlorophenol		100
	Phenol		100
	2,4,6-Trichlorophenol		100
Pest	Aldrin	Acetone	0.2
	gamma-BHC (Lindane)		0.2
	4,4'-DDT		0.5
	Dieldrin		0.5

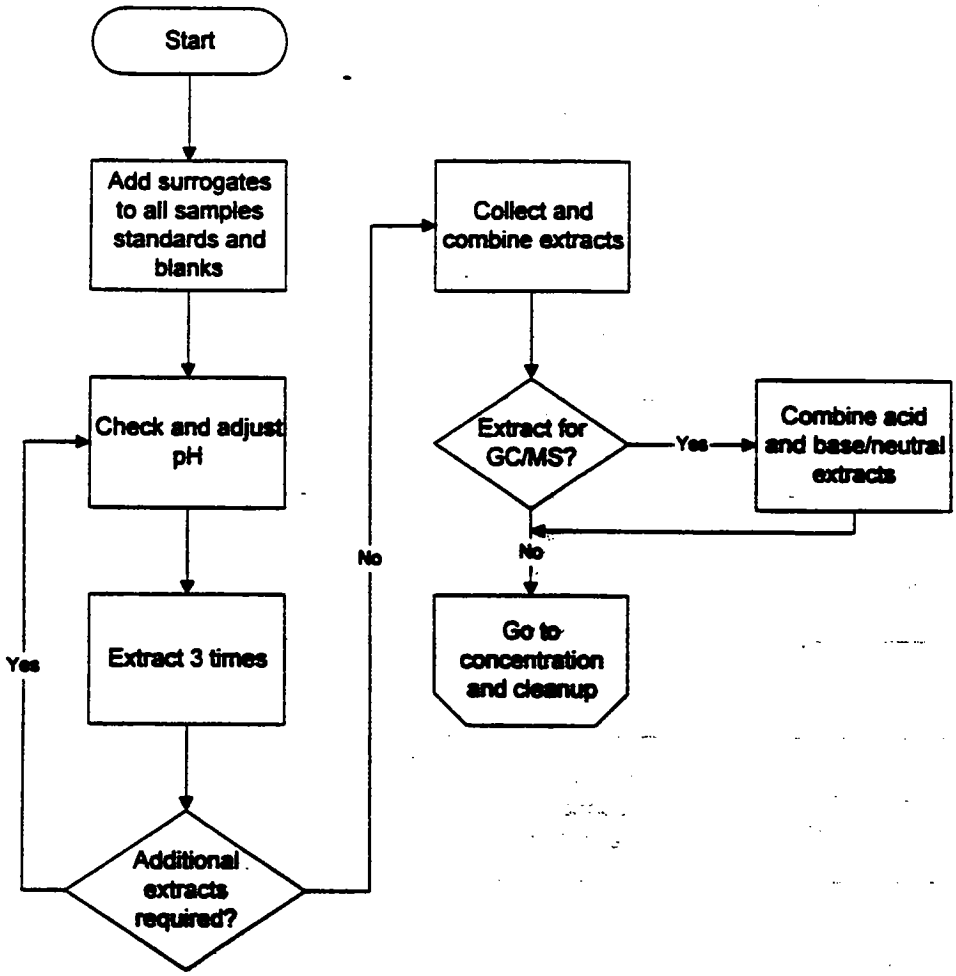
Table 6

Matrix Spike Components

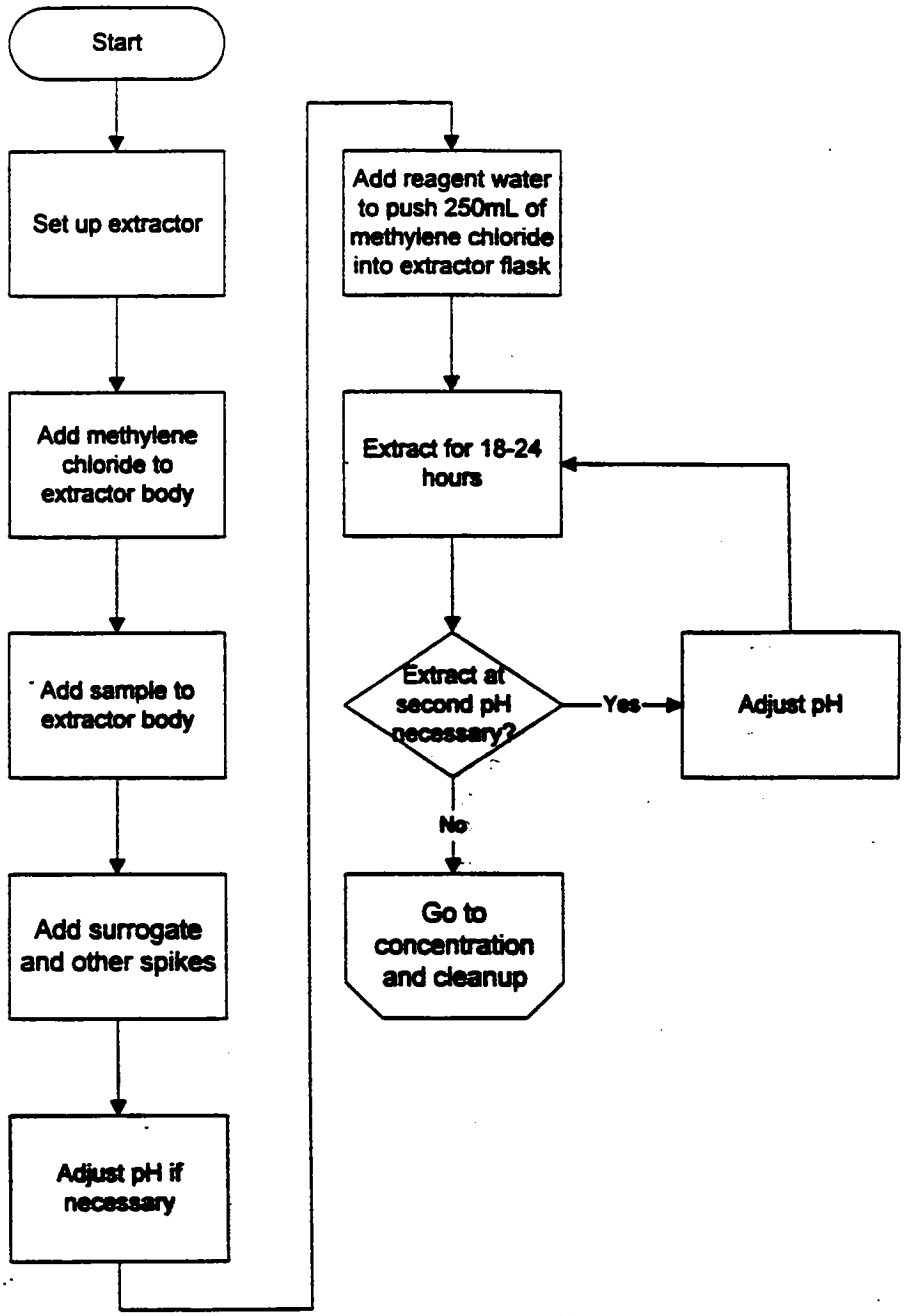
Type	Compounds	Solvent	Conc. (µg/mL)
	Endrin		0.5
	Heptachlor		0.2
Pest TCLP	Heptachlor	Acetone	0.5
	Lindane		0.5
	Endrin		0.5
	Methoxychlor		1.0
Pest NPDES	Aldrin	Acetone	0.2
	alpha-BHC		0.2
	beta-BHC		0.2
	delta-BHC		0.2
	gamma-BHC (Lindane)		0.2
	4,4'-DDD		1.0
	4,4'-DDE		0.2
	4,4'-DDT		1.0
	Dieldrin		0.2
	alpha-Endosulfan		0.2
	beta-Endosulfan		1.0
	Endosulfan Sulfate		1.0
	Endrin		1.0
	Heptachlor		0.2
	Heptachlor Epoxide		0.2
PCB only	PCB 1016/1260	Acetone	10

17.5 Flow diagrams

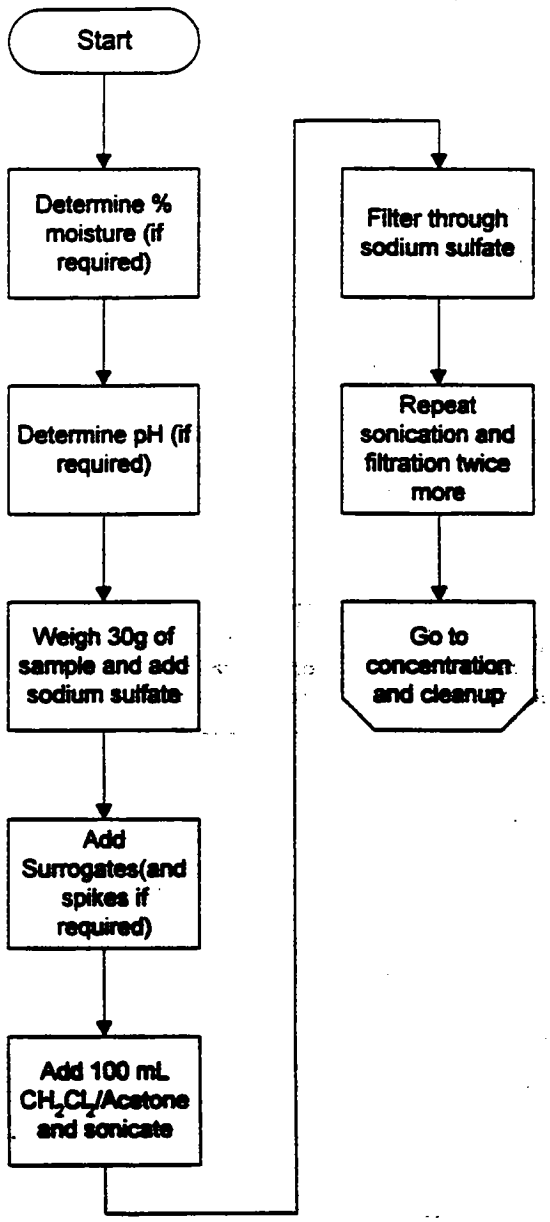
17.4.1. Separatory funnel extraction



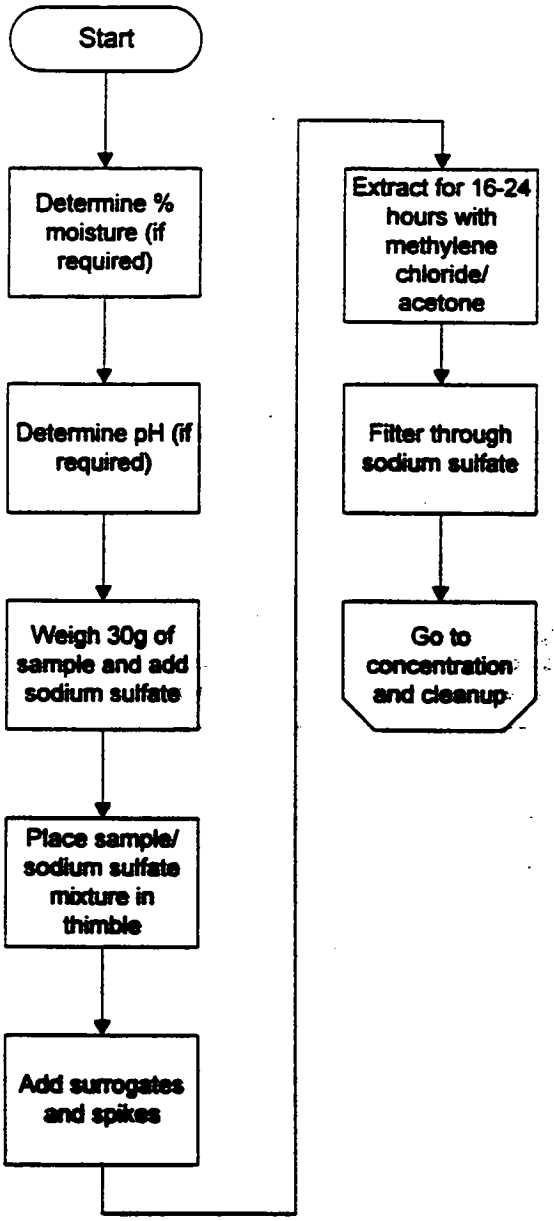
17.4.2. Continuous liquid/liquid extraction



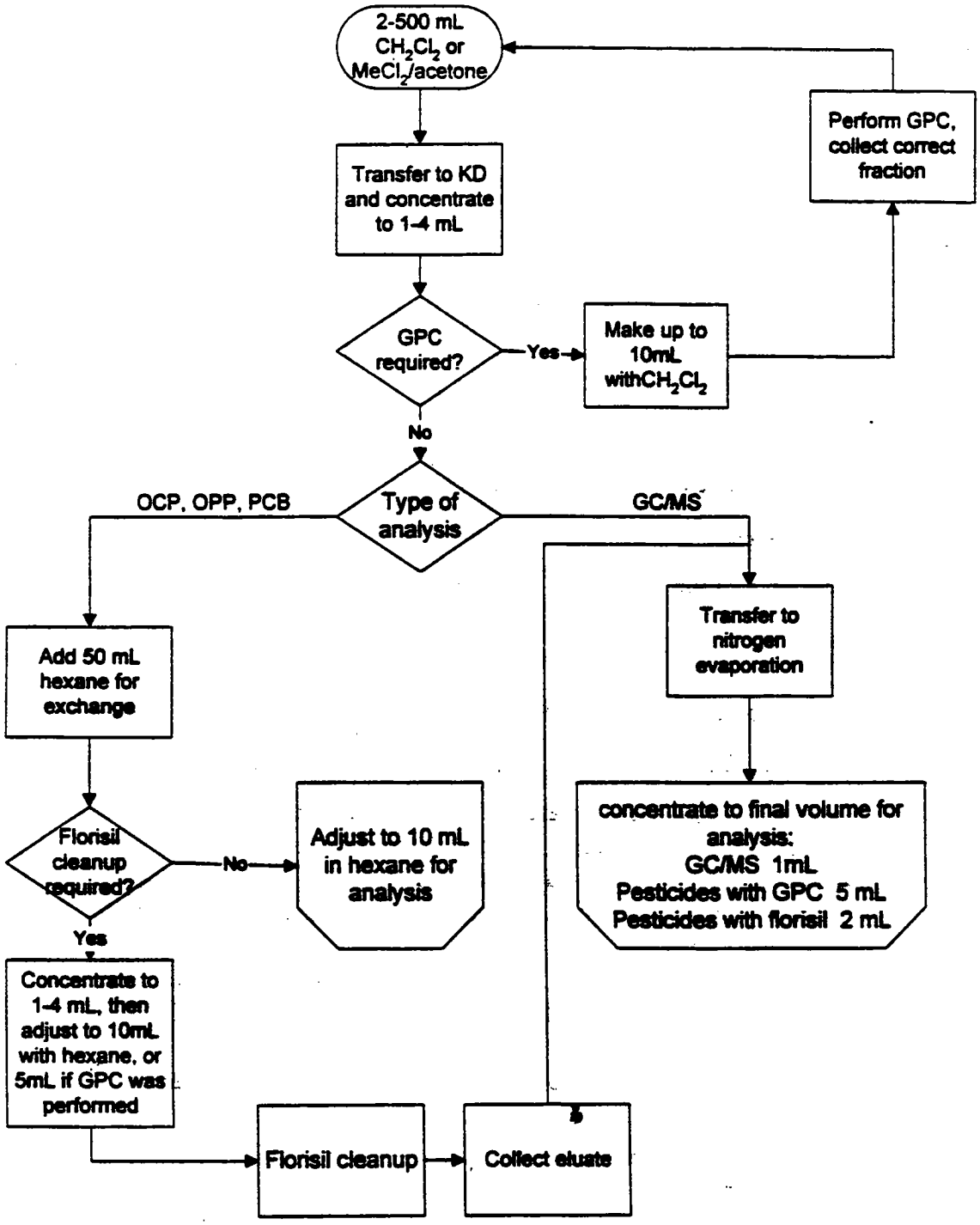
17.4.3. Sonication Extraction



17.4.4. Soxhlet extraction



17.4.5. Concentration and cleanup



Note: Exchange solvent is toluene for organophosphorous pesticides.

1. SCOPE AND APPLICATION

This method is applicable to the extraction of chlorinated herbicides in waters, solids, oils, and TCLP extracts. Appropriate compounds for extraction by this method are listed in CORP-GC-0001, Appendix D, Gas Chromatography of Phenoxy Acid Herbicides based on Method 8150A.

2. SUMMARY OF METHOD

This method is based on SW846 method 8150A. Aqueous samples are extracted into diethyl ether by a separatory funnel extraction. Solids are extracted into diethyl ether by shaking. Phenoxy acid herbicides in the extract are hydrolyzed with potassium hydroxide and the aqueous solution is washed with diethyl ether to remove interferences. The aqueous solution is then acidified and the herbicides are extracted with diethyl ether. The ether solution is dried and the herbicides are esterified using diazomethane. The final volume is adjusted to prepare the extract for gas chromatography.

3. DEFINITIONS

Refer to section 3 of the main body of this SOP.

4. INTERFERENCES

Refer to section 4 of the main body of this SOP.

5. SAFETY

5.1. Refer to section 5 of the main body this SOP for basic safety information.

5.2. DIAZOMETHANE is an extremely toxic gas with an explosion potential. Since the explosion potential is catalyzed by imperfections in glass, generation of diazomethane must be carried out in glassware free of scratches, cracks, chips and which does not have ground glass joints. Solutions of diazomethane will be kept at temperatures below 90°C. Diazomethane must be generated and handled in a fume hood.

5.3. Diethyl ether is extremely flammable

5.4. Diethyl ether must be free of peroxides as demonstrated by EM Quant test strips.

5.5. Concentrated potassium hydroxide solution is highly caustic.

6. EQUIPMENT AND SUPPLIES

6.1. Refer to Section 6 of the main body of this SOP for basic extraction equipment and supplies. Additional equipment and supplies needed for this procedure are listed below.

6.2. Diazomethane generation apparatus

6.3. EM Peroxide test strips

7. REAGENTS AND STANDARDS

7.1. Reagents are listed in Section 7 of the main body of this SOP. Additional Reagents and standards needed for this procedure are listed below.

7.2. Reagents

7.2.1. Potassium hydroxide solution, 37% aqueous solution, (w/v): Dissolve 37 g of potassium hydroxide pellets in reagent water and dilute to 100 mL. CAUTION: Considerable heat will be generated.

7.2.2. Diazald, 99% purity

7.2.3. Sodium sulfate, Na_2SO_4, Anhydrous, granular, acidified: Heat sodium sulfate in a shallow tray at 400°C for a minimum of 4 hours to remove phthalates and other interfering organic substances. In a 250 mL beaker, acidify by slurrying 150 g sodium sulfate with just enough diethyl ether to cover. Add 5.0 mL of concentrated sulfuric acid and mix thoroughly. Place the mixture on a steam bath in a hood to evaporate the ether, or allow the ether to evaporate overnight. Larger batches of acidified sodium sulfate may be prepared using the reagents in the same proportions.

7.2.4. Acidified 5% sodium sulfate solution

Add 50 g of sodium sulfate to one liter of reagent water. Add 10 mL of concentrated H_2SO_4. (This reagent may be prepared in different quantities if the proportions are kept the same).

7.2.5. Diethyl ether, reagent grade.

7.2.6. Methanol, reagent grade.

7.2.7. Silicic acid

7.3. Standards

7.3.1. Surrogate Standard
See Table A3.

7.3.2. Matrix Spike and LCS standard
See Table A4.

8. SAMPLE COLLECTION PRESERVATION AND STORAGE

8.1. Sample collection and storage is described in Section 8 of the main body of this SOP.

9. QUALITY CONTROL

9.1. Refer to Section 9 of the main body of this SOP for Quality control procedures.

10. CALIBRATION AND STANDARDIZATION

Not applicable

11. PROCEDURE

11.1. Preparation of soil, sediment and other solid samples

11.1.1. Weigh 50.0 g of moist solid sample into an 8 oz. glass jar. Use 50 g of sodium sulfate for the Method Blank and LCS. Acidify the sample with 5 mL of concentrated HCl.

11.1.2. There should be a small amount of liquid phase. If not, add reagent water until there is. Stir well with a spatula. (Note: This is not necessary for the method blank or LCS)

11.1.3. After 15 minutes, stir with a spatula and check the pH of the liquid phase. Add more acid if necessary to bring the pH to <2, repeating the stirring and standing time after each acid addition. (Note: The pH of the method blank and LCS is not determined.)

11.1.4. Add 60 g of acidified sodium sulfate and mix well. The sample should be free flowing. If not, add more sodium sulfate.

11.1.5. Spike with 1.0 mL of DCAA surrogate solution. Spike matrix spikes and LCS with 1 mL of herbicide matrix spiking solution. (Refer to tables A1 and A2)

11.1.6. Add 100 mL 20% acetone in ethyl ether.

Note: If dinoseb is a target, acetone should not be used. Instead, use 100 mL of ethyl ether.

11.1.7. Mix contents on orbital shaker for 20 minutes. Decant extract through glass wool plugged funnel.

11.1.8. Extract sample twice more, using 100 mL 20% acetone in ether (or 100% ether if dinoseb is a target). After each addition shake for 10 minutes and decant the extract.

11.1.9. Combine the extracts in a 1 or 2 liter separatory funnel containing 250 mL of acidified 5% sodium sulfate solution.

11.1.10. Check the pH of the extract. If it is not at or below pH 2, add more concentrated HCl until stabilized at the desired pH. Gently mix the contents of the separatory funnel for one minute to extract interferences into the aqueous layer. Allow the layers to separate. Collect the aqueous phase in a clean beaker and the extract phase in a 500 mL ground glass Erlenmeyer flask. Place the aqueous phase back in the separatory funnel and re-extract using 25 mL of diethyl ether. Allow the layers to separate and discard the aqueous layer. Combine the ether extracts in the 500 mL Erlenmeyer flask containing one or two boiling chips. Proceed to step 11.3, Hydrolysis.

11.2. Preparation of Aqueous Samples

11.2.1. Weigh the sample bottle and pour approximately 1 liter (100 mL for TCLP leachates) into a 2 liter separatory funnel. The sample should be decanted off any sediment. Reweigh the bottle and record the sample volume on the benchsheet, assuming a density of 1.0. Alternatively, measure 1 liter in a graduated cylinder. If less than 1 liter was used, add reagent water to make the volume up to 1 liter.

11.2.2. Adjust pH to 2 with 1:1 sulfuric acid. Spike with 1 mL of surrogate solution. Spike MS/MSD and LCS samples with 1 mL of matrix spiking solution. (Refer to Tables A1 and A2). Add 150 mL diethyl ether and shake funnel for 2 minutes with frequent venting to release excess pressure. ***Caution:*** Diethyl ether will generate pressure rapidly. Vent

the funnel immediately after it is first sealed and inverted, and vent frequently thereafter.

11.2.3. Allow layers to separate for at least 10 minutes and drain the aqueous phase into a clean beaker. If an emulsion forms break by centrifuge or mechanically. Collect the solvent extract (upper layer) in a 500 mL ground glass Erlenmeyer containing 2 mL of 37% potassium hydroxide. Use 5 mL of potassium hydroxide for soil and TCLP samples.

11.2.4. Repeat the extraction two more times using 50 mL of diethyl ether each time. Combine the extracts in the Erlenmeyer flask. Rinse the beaker with each additional aliquot of extraction solvent. Proceed to section 11.3, Hydrolysis.

11.3. Hydrolysis

11.3.1. Add one or two clean boiling chips, the sample extract, and 15 mL of water for water samples (30 mL for soil samples) to the 500 mL flask. Attach a three-ball Snyder column.

11.3.2. Place the apparatus on a hot water bath (approx 60°C) so that the bottom of the flask is bathed with hot water vapor. Although the diethyl ether will evaporate in about 15 minutes, continue heating for a total of 60 minutes), beginning from the time the flask is placed in the water bath. Remove the apparatus and let stand at room temperature for at least 10 minutes. Check the pH of the solution. If not at or above 11, add additional KOH to bring the pH above 11 and hydrolyze for an additional 60 minutes.

11.4. Solvent Clean-up

11.4.1. Transfer the solution to an acid-rinsed 125 or 250 mL separatory funnel using 5-10 mL of reagent water to rinse out the flask. Wash the basic solution by shaking for 1 minute with a 20 mL portion of diethyl ether. Drain the aqueous (bottom) layer back into the flask and discard the ether layer. Pour the aqueous layer back into the separatory funnel and repeat the wash with a second 20 mL of ether. Pour the aqueous phase back into the separatory funnel. The herbicides remain in the aqueous phase. Additional washes may be used if the sample appears dirty.

11.5. Solvent Extraction

11.5.1. Acidify the contents of the separatory funnel to pH 2 by adding 1:1 sulfuric acid (2 mL for aqueous, 5 mL for soils). Test with pH indicator paper. Add 40 mL ether and shake vigorously for 1 minute. Drain and collect the aqueous phase and pour the ether phase into a 250 mL Erlenmeyer containing 5-7 g of acidified sodium sulfate.

11.5.2. Pour the aqueous phase back into the separatory funnel and repeat the extraction twice more with 20 mL aliquots of ether, combining all solvent in the 125 mL flask. Allow the extract to remain in contact with the sodium sulfate for at least 2 hours.

NOTE: The drying step is very critical to ensure complete esterfication. Any moisture remaining in the ether will result in low herbicide recoveries. The amount of sodium sulfate is adequate if some free flowing crystals are visible when swirling the flask. If all the sodium sulfate solidifies in a cake, add a few additional grams of sodium sulfate and again test by swirling. The 2 hour drying time is a minimum, however, the extracts may be held in contact with the sodium sulfate overnight.

11.6. Concentration

11.6.1. Transfer the ether extract by decanting, or through a funnel plugged with acid washed glass wool, into a Turbovap concentrator tube or a 500 mL K-D flask equipped with a 10 mL concentrator tube. Use a stirring rod to crush the caked sodium sulfate during transfer. Rinse the Erlenmeyer with 20-30 mL ether to complete transfer.

11.6.2. Attach a three ball Snyder column to the K-D apparatus, prewet the column with a few mL of ether from the top, and place the apparatus on a water bath at approximately 60°C. At the proper rate of distillation, the balls of the column will chatter, but the chambers will not flood. When the apparent volume reaches 2-5 mL, remove from the water bath and allow to completely cool.

11.6.3. Carefully disassemble the concentrator tube and rinse the lower glass joint with a small amount of diethyl ether. Transfer the concentrator tube to the nitrogen blow down apparatus. Concentrate to approximately 1 mL under a gentle stream of nitrogen. The temperature of the water bath should be 30 - 40°C. If the extract is highly colored, forms a precipitate or stops concentrating, inform the extraction laboratory supervisor or group leader.

11.6.4. Add 0.1 mL of methanol.

11.6.5. The extract is now ready for esterification by either the diazomethane solution method (11.7) or the bubbler method (11.8)

11.7. Esterification (diazomethane solution method)

11.7.1. Preparation of Diazomethane solution

- **CAUTION: Diazomethane is potentially explosive.**
- **A temporary shield or the sash of the hood must protect the face and body of the analyst.**
- **Never heat solutions of diazomethane above 90°C, due to the explosive potential.**
- **Do not use glass stirring rods or any glassware with ground glass joints, as this can initiate violent reaction or explosion.**
- **All glassware must be scrupulously cleaned and free from scratches, to avoid potential initiation of violent reaction or explosion.**

11.7.1.1. Weigh out 10.0 g potassium hydroxide in a 125 mL beaker. Add 16 mL water and 20 mL ethanol. Mix well until the potassium hydroxide is dissolved and pour into the reaction vessel.

11.7.1.2. Attach a 100 mL receiving flask to the condenser and cool the receiver in an ice bath.

11.7.1.3. Fill the condenser with dry ice, then add acetone slowly until the cold finger is about one third full.

11.7.1.4. Mix 10.0 g diazald and 90 mL ether. Place in a separatory funnel over the reaction vessel.

11.7.1.5. Warm the reaction vessel on the water bath to 50-60°C or until the ether begins to reflux. CAUTION: Do not allow the temperature to exceed 70°C. Add diazald solution over a period of 40 minutes. The rate of distillation should approximate the rate of addition. Replenish the cold finger with dry ice as necessary.

11.7.1.6. When the diazald solution has been used up, slowly add 10 mL of ether and continue distillation until the distillate is colorless.

11.7.1.7. The diazomethane solution is documented with a lot number. It is stored in a freezer at -10 to -20°C and has an expiration date of six months.

11.7.2. Disposal of outdated diazomethane solutions

11.7.2.1. Add 20 mL of ether to 2 mL of glacial acetic acid in a large beaker in the hood.

11.7.2.2. Add the diazomethane solution slowly to the acetic acid. The yellow color of the diazomethane disappears as it reacts with the acetic acid. If the yellow color persists at any time during the addition, and does not disappear with gentle swirling of the beaker, add additional acetic acid in ether before continuing the diazomethane addition.

11.7.2.3. Dispose of the ether solution in the non-chlorinated wastes bottle.

11.8. Esterification (Diazomethane solution method)

11.8.1. Add approximately 2 mL of diazomethane solution and let sit for 10-15 minutes.

11.8.2. Add approximately 0.2 g of silicic acid to the extract. Allow to spontaneously evaporate to about 1.0 mL, then make up to 10 mL with hexane.

11.8.3. Extract is ready for analysis by gas chromatography.

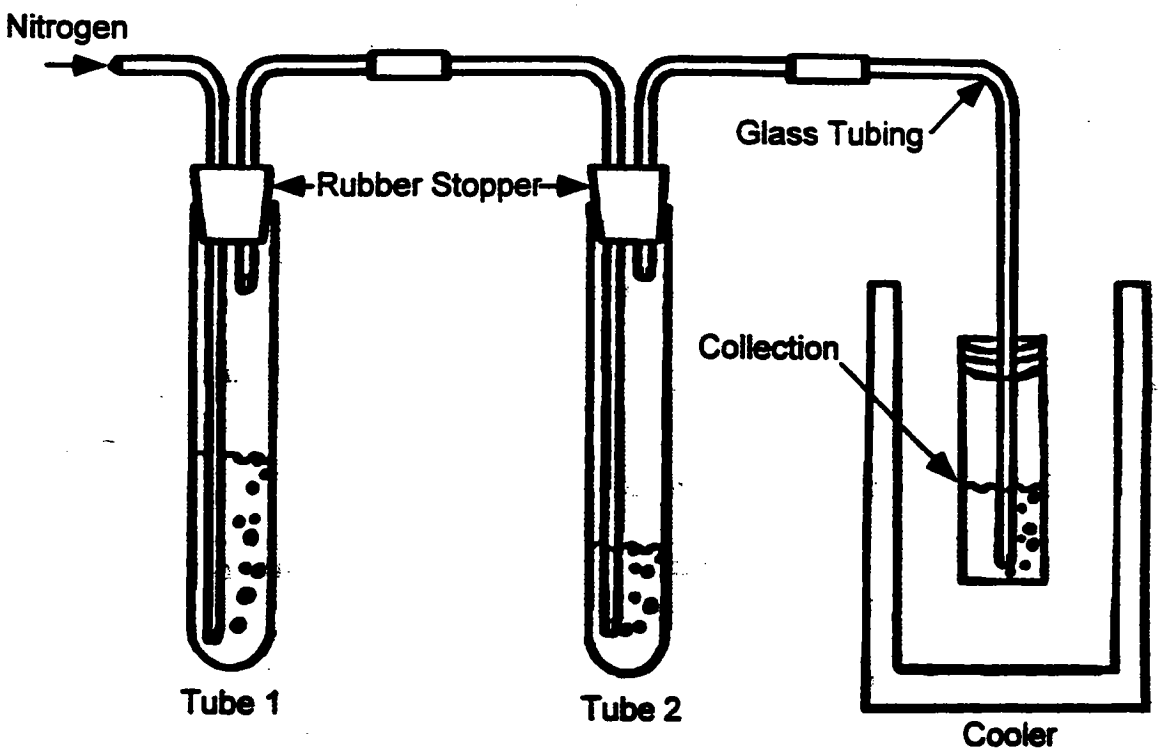
11.9. Esterification (Bubbler Method)

11.9.1. Assemble the diazomethane apparatus (Figure A1) in a hood. Add 10 mL of diethyl ether to tube 1. Add 5 mL of 2% methanolic KOH, 3 mL of ether and 0.5-1 g of diazald to tube 2.

11.9.2. Place the tip of the disposable pipet into the vial containing the first sample extract. Apply nitrogen flow (approx. 10 mL/min) to bubble diazomethane through the sample extract for about 1 minute, or until the yellow color persists. Replace the disposable pipet and place the tip into the vial containing the second extract. Continue until the diazald is consumed. (An additional 0.1-0.5 g diazald may be added to extend the generation of diazomethane).

11.9.3. Allow the extracts to stand for 20 minutes, then add approximately 0.2 g of silicic acid to each extract. Allow to stand for an additional 20 minutes.

11.9.4. Adjust the volume to 10 mL with hexane. The sample is now ready for gas chromatography.



12. DATA ANALYSIS AND CALCULATIONS

Not applicable

13. METHOD PERFORMANCE

Refer to CORP-GC-0001 for details of method performance.

14. POLLUTION PREVENTION

Refer to section 14 of the main body of this SOP.

15. WASTE MANAGEMENT

Refer to section 15 of the main body of this SOP.

16. REFERENCES

16.1. SW846, Test Methods for Evaluating Solid Waste, Third Edition, Update II, September 1994, Chlorinated Herbicides, Method 8150B.

17. MISCELLANEOUS

17.1. Modifications from Reference Method

17.1.1. Directions to add sufficient reagent water to the soil sample so that the pH can be measured have been added (Section 11.1.2)

17.1.2. Directions to add sodium sulfate to the soil sample until a free flowing texture is achieved have been added. In common with other SW-846 extraction procedures, this improves the extraction efficiency. (Section 11.1.4)

17.1.3. For the soil extraction, the acetone and ether are added together rather than separately. (Section 11.1.6)

17.1.4. The requirement for the sulfuric acid added in the solvent extraction to be cold has been removed. Since a small quantity of acid is added to a large quantity of extract, nothing is gained by having the acid cold. (Section 11.5.1)

17.1.5. Silicic acid is stored at room temperature.

17.1.6. The bubbler esterification method uses methanolic KOH in place of the aqueous KOH / carbitol mixture recommended in method 8150B. This has been found to provide a more effective and reliable exterification.

17.2. Modifications from previous revisions

In the hydrolysis procedure, the extract is added to the potassium hydroxide solution rather than visa versa.

17.3. Tables

Table A1
Herbicide Surrogate Spiking Solutions

Analyte Group	Surrogate Spike Solution ID	Volume (mL)
Herbicides	Herbicides SS	1

Table A2
Herbicide Matrix Spike and LCS Solutions

Analyte Group	Matrix Spike Solution ID	Volume (mL)
Herbicides	Herbicides MS	1

Table A3
Herbicide Surrogate Spike Components

Type	Compounds[1]	Solvent	Conc. (ug/mL)
Herbicides SS	2,4-DCAA	Methanol	16

[1]The surrogate is spiked as the free acid

Table A4			
Herbicide Matrix Spike Components			
Type	Compounds[1]	Solvent	Conc. (ug/mL)
Herbicides MS	2,4-D	Methanol	16
	2,4-DB		8
	2,4,5-Tp (Silvex)		4
	Dalapon		16
	Dicamba		8

[1]The herbicide spiking solution contains the herbicides as the free acids.

QUANTERRA STANDARD OPERATING PROCEDURE

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

(SUPERSEDES: None)

Prepared by: Richard Burrows

Reviewed by:
Technology Standardization Committee

Approved by:
Director, Quality Assurance

Approved by:
Director, Environmental Health and Safety

Approved by:
Management

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TABLE OF CONTENTS

1. SCOPE AND APPLICATION 4

2. SUMMARY OF METHOD 5

3. DEFINITIONS 5

4. INTERFERENCES 6

5. SAFETY PRECAUTIONS 6

6. EQUIPMENT AND SUPPLIES 7

7. REAGENTS AND STANDARDS 8

8. SAMPLE PRESERVATION AND STORAGE 9

9. QUALITY CONTROL 9

10. CALIBRATION AND STANDARDIZATION 14

11. PROCEDURE 17

12. DATA ANALYSIS AND CALCULATIONS 21

13. METHOD PERFORMANCE 27

14. POLLUTION PREVENTION 28

15. WASTE MANAGEMENT 28

16. REFERENCES 28

17. MISCELLANEOUS 29

TABLE 1 Quanterra Primary Standard and Standard Reporting Limits

TABLE 2 Quanterra Appendix IX Standard and Standard Reporting Limits

TABLE 3 Reportable Analytes for Quanterra Standard Tests, Primary Standard

TABLE 4 Reportable analytes for Quanterra Standard Tests, Appendix IX Standard

TABLE 5 Recommended Instrument Conditions

TABLE 6 DFTPP Ion Abundance Criteria

TABLE 7 Characteristic Ions, Primary Standard

TABLE 8 Characteristic Ions, Appendix IX Standard

TABLE 9 8270B LCS Compounds

TABLE 10 TCLP LCS Compounds

TABLE 11 8270B Surrogate Compounds

TABLE 12 Calibration Levels, Primary Standard

TABLE 13 Calibration Levels, Appendix IX Standard

TABLE 14 Initial Demonstration Accuracy and Precision Limits

1. SCOPE AND APPLICATION

1.1. This method is based upon SW846 8270B, and is applicable to the determination of the concentration of semivolatile organic compounds in extracts prepared from solid and aqueous matrices. Direct injection of a sample may be used in limited applications. Refer to Tables 1, 2, 3 and 4 for the list of compounds applicable for this method. Note that the compounds are listed in approximate retention time order. Additional compounds may be amenable to this method. If non-standard analytes are required, they must be validated by the procedures described in section 13 before sample analysis.

1.2. The following compounds may require special treatment when being determined by this method:

- Benzidine can be subject to oxidative losses during solvent concentration and exhibits poor chromatography. Neutral extraction should be performed if this compound is expected.
- Hexachlorocyclopentadiene is subject to thermal decomposition in the inlet of the gas chromatograph, chemical reaction in acetone solution, and photochemical decomposition.
- N-Nitrosodiphenylamine decomposes in the gas chromatographic inlet and cannot be distinguished from diphenylamine.
- Pentachlorophenol, 2,4-dinitrophenol, 4-nitrophenol, 4,6-dinitro-2-methylphenol, 4-chloro-3-methylphenol, benzoic acid, 2-nitroaniline, 3-nitroaniline, 4-chloroaniline, and benzyl alcohol are subject to erratic chromatographic behavior, especially if the GC system is contaminated with high boiling material.
- Hexachlorophene is not amenable to analysis by this method.
- 3-Methylphenol cannot be separated from 4-methylphenol by the conditions specified in this method.

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

2. SUMMARY OF METHOD

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

3. DEFINITIONS

3.1. CCC (Calibration Check Compounds) - A subset of target compounds used to evaluate the calibration stability of the GC/MS system. A maximum percent deviation of the CCC's is specified for calibration acceptance.

3.2. SPCC (System Performance Check Compounds) - Target compounds designated to monitor chromatographic performance, sensitivity, and compound instability or degradation on active sites. Minimum response factors are specified for acceptable performance.

3.3. Batch - The batch is a set of up to 20 samples of the same matrix processed using the same procedures and reagents within the same time period. The Quality Control batch must contain a matrix spike / spike duplicate (MS/MSD), a Laboratory Control Sample (LCS), and a method blank. In some cases, at client request, the MS/MSD may be replaced with a matrix spike and sample duplicate. Batches are defined at the sample preparation stage. Batches should be kept together through the whole analytical process to the extent possible, but it is not mandatory to analyze prepared extracts on the same instrument or in the same sequence. Refer to the Quanterra QC Program document (QA-003) for further details of the batch definition.

3.4. Method Blank - An analytical control consisting of all reagents, internal standards and surrogate standards, that is carried through the entire analytical procedure. The method blank is used to define the level of laboratory background and reagent contamination.

3.5. LCS (Laboratory Control Sample) - A blank spiked with the parameters of interest that is carried through the entire analytical procedure. Analysis of this sample with acceptable recoveries of the spiked materials demonstrates that the laboratory techniques for this method are acceptable.

3.6. MS (Matrix Spike)- aliquot of a matrix (water or soil) fortified (spiked) with known quantities of specific compounds and subjected to the entire analytical procedure in order to indicate the appropriateness of the method for the matrix by measuring recovery.

3.7. MSD (Matrix Spike Duplicate)- a second aliquot of the same sample as the matrix spike (above) that is spiked in order to determine the precision of the method.

4. INTERFERENCES

4.1. Method interferences may be caused by contaminants in solvents, reagents, glassware, and other processing apparatus that lead to discrete artifacts. All of these materials must be routinely demonstrated to be free from interferences under conditions of the analysis by running laboratory method blanks as described in the Quality Control section. Raw GC/MS data from all blanks, samples, and spikes must be evaluated for interferences. If an interference is detected it is necessary to determine if the source of interference is in the preparation and/or cleanup of the samples; then take corrective action to eliminate the problem.

4.2. The use of high purity reagents, solvents, and gases helps to minimize interference problems.

4.3. Matrix interferences may be caused by contaminants that are coextracted from the sample. The extent of matrix interferences will vary considerably from source to source, depending upon the nature of the sample.

4.4. Contamination by carryover can occur whenever high-level and low-level samples are sequentially analyzed. To reduce carryover, the sample syringe must be rinsed with solvent between samples. Whenever an unusually concentrated sample is encountered, it should be followed by the analysis of solvent to check for cross contamination.

4.5. Phthalate contamination is commonly observed in this analysis and its occurrence should be carefully evaluated as an indicator of a contamination problem in the sample preparation step of the analysis.

5. SAFETY PRECAUTIONS

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

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

5.1.2. The health and safety hazards of many of the chemicals used in this procedure have not been fully defined. Additional health and safety information can be obtained from the MSDS files maintained in the laboratory. The following specific hazards are known:

5.1.2.1. Chemicals that have been classified as carcinogens, or potential carcinogens, under OSHA include: Benzo(a)anthracene, benzidine, 3,3'-dichlorobenzidine, benzo(a)pyrene, dibenzo(a,h)anthracene, and n-nitrosodimethylamine. Primary standards should be purchaced in solution. If neat materials must be obtained, they shall be handled in a hood.

5.1.3. Exposure to chemicals must be maintained as low as reasonably achievable; therefore, unless they are known to be non-hazardous, all samples should be opened, transferred, and prepared in a fume hood, or under other means of mechanical ventilation. Solvent and waste containers should be kept closed unless transfers are being made.

5.1.4. All work must be stopped in the event of a known or potential compromise to the health and safety of a Quanterra associate. The situation must be reported immediately to a laboratory supervisor.

6. EQUIPMENT AND SUPPLIES

6.1.1. Gas Chromatograph/Mass Spectrometer System: An analytical system complete with a temperature-programmable gas chromatograph suitable for split/splitless injection and all required accessories, including syringes, analytical columns, and gases. The capillary column should be directly coupled to the source.

6.1.2. Column: 30 m x 0.32 mm I.D. (or 0.25 mm I.D.) 0.5-µm film thickness silicon-coated fused-silica capillary column (J & W Scientific DB-5.625 or equivalent). Alternate columns are acceptable if they provide acceptable performance.

6.1.3. Mass Spectrometer: Capable of scanning from 35 to 500 AMU every one second or less, using 70 volts (nominal) electron energy in the electron impact ionization mode. The mass spectrometer must be capable of producing a mass spectrum for

decafluorotriphenylphosphine (DFTPP) which meets all of the criteria in Table 6 when 50 ng of the GC/MS tuning standard is injected through the GC.

6.1.4. GC/MS Interface: Any GC-to-MS interface that gives acceptable calibration points and achieves acceptable tuning performance criteria may be used.

6.1.5. Data System: A computer system must be interfaced to the mass spectrometer. The system must allow the continuous acquisition and storage on machine-readable media of all mass spectra obtained throughout the duration of the chromatographic program. The computer must have software that can search any GC/MS data file for ions of a specific mass and that can plot such ion abundances versus time or scan number. This type of plot is defined as the Extracted Ion Current Profile (EICP). Software must also be available that allows integrating the abundances in any EICP between specified time or scan-number limits. The most recent version of the EPA/NIH Mass Spectral Library is recommended.

6.2. Syringe: 10 µL Hamilton Laboratory grade syringes or equivalent. A selection of syringe sizes should be on hand so that the minimum volume dispensed is never less than 10% of total syringe volume (i.e., 10 mL syringe for 1 mL measured volume).

6.3. Carrier gas: Ultra high purity helium.

7. REAGENTS AND STANDARDS

7.1. A minimum five point calibration curve is prepared at nominal concentrations of 10, 25, 40, 60 and 80 µg/mL for each parameter of interest. Some compounds are prepared at higher levels due to chromatographic performance, stability, and sensitivity. Refer to Tables 12 and 13 for calibration levels for all analytes.

7.2. A 400 µg/mL Internal Standard solution is prepared. Compounds in the I.S. Mix are: acenaphthene-d10, chrysene-d12, 1,4-dichlorobenzene-d4, naphthalene-d8, perylene-d12, and phenanthrene-d10

7.2.1. Internal Standards are added to all standards and extracts at 20 µg per mL of extract. For example, if the volume of an extract used was 200 µL, 10 µL of the 400 µg/mL internal standard solution would be added.

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

7.4. GC/MS Tuning Standard: A methylene chloride solution containing 25 µg/mL of decafluorotriphenylphosphine (DFTPP) is prepared. Pentachlorophenol, benzidine, and DDT, should also be included in the Tuning Standard at 25 µg/mL.

7.5. Laboratory Control Spiking Solution: Prepare as indicated in the preparative methods. See appropriate preparation SOP. LCS compounds and levels are listed in Tables 9 and 10.

7.6. Matrix Spike Solution: Prepare as indicated in the preparative methods. See preparation SOP. The matrix spike compounds and levels are the same as the LCS compounds.

7.7. The working standards listed in 7.1 to 7.6 should be refrigerated at -10°C to -20°C when not in use. The standards must be replaced at least once a year. The continuing calibration standard must be replaced every week and is stored at 4 ± 2°C.

8. SAMPLE PRESERVATION AND STORAGE

8.1. Reference appropriate facility SOP for sample bottle preservation and storage.

8.2. Samples are stored at 4 ± 2°C. Samples and extracts should be stored in suitable glass containers with Teflon lined caps. (Extracts will normally be stored for 30 days after invoicing.)

8.3. Water samples are extracted within seven days of sampling and the extracts are analyzed within forty days of extraction. Solids, sludges, and organic liquids are extracted within fourteen days of sampling and the extracts are analyzed within forty days of extraction.

9. QUALITY CONTROL

9.1. Initial Demonstration of Capability

9.1.1. For the standard analyte list, the initial demonstration and method detection limit (MDL) studies described in section 13 must be acceptable before analysis of samples may begin. Refer to the flow chart in section 17.4.1.

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

9.2. Control Limits

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

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

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

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

9.3. Method Blank

A method blank is prepared and analyzed with each batch of samples. The method blank consists of reagent water for aqueous samples, and sodium sulfate for soil samples (Refer to SOP No. CORP-OP-0001 for details). Surrogates are added and the method blank is carried through the entire analytical procedure. The method blank must not contain any analyte of interest at or above the reporting limit (except common laboratory contaminants, see below) or at or above 5% of the measured concentration of that analyte in the associated samples, whichever is higher.

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

- Reanalysis of all samples associated with an unacceptable method blank is required when concentrations above the reporting limit are determined in the samples.

- If there is no target analyte greater than the RL in the samples associated with an unacceptable method blank, the data may be reported with qualifiers. Such action should be taken in consultation with the client.

9.3.1. The method blank must have acceptable surrogate recoveries. If surrogate recoveries are not acceptable, the data must be evaluated to determine if the method blank has served the purpose of demonstrating that the analysis is free of contamination. If surrogate recoveries are low and there are reportable analytes in the associated samples, re-extraction of the blank and affected samples will normally be required. Consultation with the client should take place.

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

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

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

9.4. Instrument Blank

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

9.5. Laboratory Control Sample (LCS)

9.5.1. A laboratory control sample (LCS) is prepared and analyzed with every batch of samples. All analytes must be within established control limits. The LCS is spiked with the compounds listed in Tables 9 and 10 unless specified by a client or agency. The compounds must be spiked at a concentration equivalent to 100 or 150 ng on-column depending on the analyte.

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

- If the batch is not re-extracted and reanalyzed, the reasons for accepting the batch must be clearly presented in the project records and the report. (An example of acceptable reasons for not reanalyzing might be that the matrix spike and matrix spike duplicate are acceptable, and sample surrogate

recoveries are good, demonstrating that the problem was confined to the LCS).

- If re-extraction and reanalysis of the batch is not possible due to limited sample volume or other constraints, the LCS is reported, all associated samples are flagged, and appropriate comments are made in a narrative to provide further documentation.

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

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

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

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

- If the recovery for any component is outside QC limits for both the Matrix spike / spike duplicate and the LCS, the laboratory is out of control and corrective action must be taken. Corrective action will normally include repreparation and reanalysis of the batch.

- If a MS/MSD is not possible due to limited sample, then a LCS duplicate should be analyzed. RPD of the LCS and LCSD are compared to the matrix spike limits.

- The matrix spike / duplicate must be analyzed at the same dilution as the unspiked sample, even if the matrix spike compounds will be diluted out.

9.7. Surrogates

9.7.1. Every sample, blank, and QC sample is spiked with surrogate standards. Surrogate spike recoveries must be evaluated by determining whether the concentration (measured as percent recovery) falls within the required recovery limits. Surrogate compounds must be spiked at either 100 or 150 ng on-column, depending on the surrogate. The compounds routinely included in the surrogate

spiking solution, along with recommended standard concentrations, are listed in Table 11.

9.7.2. If any surrogates are outside limits the following corrective actions must take place (except for dilutions):

- Check all calculations for error.
- Ensure that instrument performance is acceptable.
- Recalculate the data and/or reanalyze the extract if either of the above checks reveal a problem.
- 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.

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

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

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

9.8. Nonconformance and Corrective Action

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

9.9. Quality Assurance Summaries

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

9.10. Quanterra QC Program

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

10. CALIBRATION AND STANDARDIZATION

10.1. Summary

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

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

10.3. Instrument Tuning

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

10.3.1. Inject 50 ng of the GC/MS tuning standard (Section 7.4) into the GC/MS system. (2µL of the 25µg/mL solution). Obtain a background-corrected mass spectra of DFTPP and confirm that all the key m/z criteria in Table 6 are achieved. If all the criteria are not achieved, the analyst must retune the mass spectrometer and repeat the test until all criteria are achieved. The performance criteria must be achieved before any samples, blanks, or standards are analyzed.

10.3.2. The GC/MS tuning standard should also be used to evaluate the inertness of the chromatographic system. Breakdown of DDT should be less than 20%, and benzidine and pentachlorophenol should not exhibit excessive tailing. Refer to section 12 for the appropriate calculations.

10.4. Initial Calibration

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

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

10.4.3. Prepare calibration standards at a minimum of five concentration levels for each parameter of interest. Add the internal standard mixture at 20 µg per mL of calibration standard. (For example, if the volume of the calibration standard used is 1 mL, add 50 µL of the 400 µg/mL internal standard solution). The working range of the GC/MS system is defined by calibration standards at concentrations of 10 to 80 µg/mL. The exact concentrations of all analytes are listed in tables 12 and 13.

10.4.4. Using injections of 2 µL, analyze each calibration standard and tabulate the area of the primary characteristic m/z against concentration for each compound and internal standard. Calculate response factors (RF), average response factors, and the percent RSD of the response factors for each compound using the equations in section 12 and verify that the CCC and SPCC criteria in section 10.4.5 and 10.4.6 are met. **No sample analysis may be performed unless these criteria are met.**

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

SPCC Compounds:

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

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

10.4.6.1. If none of the CCCs are required analytes, project specific calibration specifications must be agreed with the client. Otherwise, all CCCs must meet the 30% criterion.

CCC Compounds:

Phenol
Acenaphthene
1,4-Dichlorobenzene
N-nitrosodiphenylamine
2-Nitrophenol
Pentachlorophenol
2,4-Dichlorophenol
Fluoranthene
Hexachlorobutadiene
Di-n-octylphthalate
4-Chloro-3-methylphenol
Benzo(a)pyrene
2,4,6-Trichlorophenol

10.4.7. If the %RSD of any compound (including the CCCs) is > 15% then a linear or quadratic calibration curve must be used for that compound. (Rather than the average response factor which is permitted if the %RSD is ≤ 15%). It is recommended that quadratic curves be used for all analytes.

10.4.8. Weighting of data points

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

10.4.9. Forcing through zero

If a curve is not forced through zero, the intercept at zero response must be less than the reporting limit for the analyte.

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

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

10.5. Continuing Calibration

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

10.5.2. Following a successful DFTPP analysis the continuing calibration standard(s) are analyzed. The standards must contain all semivolatile analytes, including all required surrogates. The level 2 calibration standard is used for the continuing calibration.

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

- The SPCC compounds must have a response factor of ≥ 0.05.
- The percent drift of the CCC compounds from the initial calibration must be $\leq$ 20%. (see section 12 for calculations) In addition, the percent drift of all analytes must be $\leq$ 50%, with allowance being made for up to six target compounds to have percent drift greater than 50%.

10.5.3.1. If none of the CCCs are required analytes, project specific calibration specifications must be agreed with the client. Otherwise, all CCCs must meet the 30% criterion.

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

11. PROCEDURE

11.1. Sample Preparation

Samples are prepared following SOP CORP-OP-0001.

11.2. Sample Analysis Procedure

11.2.1. Calibrate the instrument as described in section 10. Depending on the target compounds required by the client, it may be necessary to use more than one calibration standard.

11.2.2. All samples must be analyzed using the same instrument conditions as the preceeding continuing calibration standard.

11.2.3. Add internal standard at 20 µg per mL of extract (for example, 25 µL internal standard solution in 0.5 mL of extract) to the sample extract and mix thoroughly before injection into the instrument.

11.2.4. Inject 2 µL of the sample extract into the GC/MS system using the same injection technique as used for the standards.

11.2.5. The data system will determine the concentration of each analyte in the extract using calculations equivalent to those in section 12. Quantitation is based on the initial calibration, not the continuing calibration.

11.2.6. Identified compounds are reviewed for proper integration. Manual integrations are performed if necessary and are documented by the analyst or automatically by the data system.

11.2.7. Target compounds identified by the data system are evaluated using the criteria listed in section 12.1.

11.2.8. Library searches of peaks present in the chromatogram that are not target compounds (Tentatively Identified Compounds, TIC) may be performed if required by the client. They are evaluated using the criteria in section 12.3.

11.3. Dilutions

If the response for any compound exceeds the working range of the GC/MS system, a dilution of the extract is prepared and analyzed. An appropriate dilution should be in the upper half of the calibration range. Samples may be screened to determine the appropriate dilution for the initial run. If the initial diluted run has no hits or hits below 20% of the calibration range and the matrix allows for analysis at a lesser dilution, the sample must be reanalyzed at a dilution targeted to bring the largest hit above 50% of the calibration range.

11.3.1. Guidance for Dilutions Due to Matrix

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

11.3.2. Reporting Dilutions

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

11.4. Perform all qualitative and quantitative measurements. When the extracts are not being used for analyses, refrigerate them at 4 ± 2°C, protected from light in screw cap vials equipped with unpierced Teflon lined septa.

11.5. Internal Standard Criteria for Samples

Internal standard response in each sample should be within 50% to 200% of the response in the preceding continuing calibration standard. If more than one calibration standard is used in the sequence, the internal standard response for the samples is compared to the last calibration standard analyzed.

11.5.1. Any samples that do not meet the internal standard criteria must be evaluated for validity. If the change in sensitivity is a matrix effect confined to an individual sample reanalysis may not be necessary. If the change in sensitivity is due to instrumental problems all affected samples must be reanalyzed after the problem is corrected. In any event, the reason for accepting the sample analysis must be documented. Some clients may require reanalysis of all samples with internal standard criteria outside the 50-200% criteria. Consideration should be given to reanalyzing at a dilution to reduce matrix effects. It is only necessary to reanalyze once to confirm matrix effect.

11.6. Retention time criteria for samples

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

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

11.7. Percent Moisture

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

11.8. Procedural Variations

11.8.1. One-time procedural variations are allowed only if deemed necessary in the professional judgment of supervision to accommodate variation in sample matrix, radioactivity, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using a Nonconformance Memo and approved by a Technical Specialist and QA Manager. If contractually required, the client shall be notified. The Nonconformance Memo shall be filed in the project file. Any unauthorized deviations from this procedure must also be documented as a non-conformance, with a cause and corrective action described.

11.9. Troubleshooting Guide

11.9.1. Daily Instrument Maintenance

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

11.9.1.1. Clip Column as necessary.

11.9.1.2. Install new or cleaned injection port liner as necessary.

11.9.1.3. Install new septum as necessary.

11.9.1.4. Perform mass calibration as necessary.

11.9.2. Major Maintenance

11.9.2.1. If minor maintenance does not result in acceptable chromatography, it may be necessary to change the column.

11.9.2.2. Clean ion volume or repeller. Cleaning is indicated when DFTPP does not pass spectral criteria.

11.9.2.3. Clean source. If after cleaning ion volume or repeller, tune still does not meet criteria, or if overall sensitivity is poor, the source is removed and cleaned.

11.9.2.4. Replace filaments, replace filters and change pump oil. All of these may be done at the same time as source cleaning. The frequency of changing filters and pump oil is about every 6-12 months.

11.9.2.5. A multiplier gain check is performed if sensitivity is still poor and/or analyst suspects that the multiplier is going bad.

11.9.2.6. Mass calibration is performed if the analyst notices mass misassignments.

11.9.2.7. Refer to the manufacturer's manual for specific guidance.

12. DATA ANALYSIS AND CALCULATIONS

12.1. Qualitative identification

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

- The sample component retention time must compare to within ± 0.2 min. of the retention time of the standard component. For reference, the standard must be run within the same twelve hours as the sample.
- All ions present in the standard mass spectra at a relative intensity greater than 10% (most abundant ion in the spectrum equals 100%) should be present in the sample spectrum.
- The characteristic ions of a compound must maximize in the same scan or within one scan of each other.
- The relative intensities of ions should agree to within ±30% between the standard and sample spectra. (Example: For an ion with an abundance of 50% in the standard spectra, the corresponding sample abundance must be between 20% and 80%.)

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

12.2. Mass chromatogram searches.

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

12.2.1. Hexachlorophene

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

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

- Relative intensities of major ions in the reference spectrum (ions >10% of the most abundant ion) should be present in the sample spectrum.
- The relative intensities of the major ions should agree within ±20%. (Example: For an ion with an abundance of 50% in the standard spectrum, the corresponding sample ion abundance should be between 30%and 70%.)
- Molecular ions present in the reference spectrum should be present in the sample spectrum.
- Ions present in the sample spectrum, but not in the reference spectrum, should be reviewed for possible background contamination or presence of coeluting compounds.
- Ions present in the reference spectrum, but not in the sample spectrum, should be reviewed for possible subtraction from the sample spectrum because of background contamination or coeluting peaks. Data system library reduction programs can sometimes create these discrepancies.
- Automatic background subtraction can severely distort spectra from samples with unresolved hydrocarbons.

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

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

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

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

Benzoic acid
Chloroanilines
Nitroanilines
2,4-Dinitrophenol
4-Nitrophenol
Pentachlorophenol
3,3'-Dichlorobenzidine
Benzyl alcohol
4,6-Dinitro-2-methylphenol

Manually checking the integrations would be appropriate for these compounds.

12.5. Calculations

12.5.1. Percent Relative Standard Deviation for Initial Calibration

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

$\overline{RF}$ = Mean of RFs from initial calibration for a compound

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

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

RF_i = RF for each of the calibration levels

N = Number of RF values

12.5.2. Continuing calibration percent drift

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

$C_{expected}$ = Known concentration in standard

C_{found} = Measured concentration using selected quantitation method

12.5.3. Concentration in the extract

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

12.5.3.1. Linear fit

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

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

R_x = Response for analyte

R_{is} = Response for internal standard

C_{is} = Concentration of internal standard

A = Intercept

B = Slope

12.5.3.2. Quadratic fit

$$C_{ex} = A + B\left(\frac{R_x C_{is}}{R_{is}}\right) + C\left(\frac{R_x C_{is}}{R_{is}}\right)^2$$

Where: C = Curvature

12.5.3.3. Average response factor

Alternatively, if the %RSD of the initial calibration response factors is < 15%, the average response factor from the initial calibration may be used.

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

Where: $\overline{RF}$ = Average response factor

12.5.4. The concentration in the sample is then calculated.

12.5.4.1. Aqueous Calculation

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

Where:

V_t = Volume of total extract, µL, taking into account dilutions (i.e., a 1-to-10 dilution of a 1 mL extract will mean V_t = 10,000 µL. If half of the base/neutral extract and half of the acid extract are combined, V_t = 2,000.)

V_o = Volume of water extracted (mL)

12.5.5. Sediment/Soil, Sludge (on a dry-weight basis) and Waste (normally on a wet-weight basis:

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

Where:

W_s = Weight of sample extracted or diluted in grams.

D = (100 - % moisture in sample)/100, for a dry weight basis or 1 for a wet weight basis

12.6. MS/MSD percent recovery calculation.

$$\text{Matrix Spike recovery} = \frac{SSR - SR}{SA} \times 100$$

Where:

SSR = Spike sample result

SR = Sample result

SA = Spike added

12.7. Relative % Difference calculation for the MS/MSD

$$\text{RPD} = \frac{|\text{MSR - MSDR}|}{\frac{1}{2}(\text{MSR + MSDR})} \times 100$$

Where:

RPD = Relative percent difference

MSR = Matrix spike result

MSDR = Matrix spike duplicate result

12.8. Relative response factor calculation.

$$\text{RF} = \frac{A_x C_{is}}{A_{is} C_x}$$

Where:

A_x=Area of the characteristic ion for the compound being measured

A_{is}=Area of the characteristic ion for the specific internal standard

C_x=Concentration of the compound being measured (µg/L)

C_{is}=Concentration of the specific internal standard (µg/L)

12.9. Calculation of TICs: The calculation of TICs (tentatively identified compounds) is identical to the above calculations with the following exceptions:

A_x=Area of the total ion chromatogram for the compound being measured

A_{is}=Area of the total ion chromatogram for the nearest internal standard without interference

RF=1

12.10. Percent DDT breakdown

$$\% \text{ DDT breakdown} = \frac{\text{DDE area} + \text{DDD area}}{\text{DDT area} + \text{DDE area} + \text{DDD area}} \times 100$$

The total ion current areas are used for this calculation

13. METHOD PERFORMANCE

13.1. Method Detection Limit

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

13.2. Initial Demonstration

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

13.2.1. Four aliquots of the QC check sample are analyzed using the same procedures used to analyze samples, including sample preparation. The concentration of the QC check sample should be equivalent to the level 4 calibration standard.

13.2.2. Calculate the average recovery and standard deviation of the recovery for each analyte of interest. Compare these results with the acceptance criteria given in table 14.

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

13.3. Non-standard analytes

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

13.4. Training Qualification

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

13.5. Data Quality Objectives (DQO). Refer to project-specific Quality Assurance plans for DQO information.

14. POLLUTION PREVENTION

14.1. This section is not applicable to this procedure.

15. WASTE MANAGEMENT

15.1. Waste generated during aliquotting and from used vials must be disposed of in accordance with the facility hazardous waste procedures. The Health and Safety Director should be contacted if additional information is required.

16. REFERENCES

16.1. SW846, Test Methods for Evaluating Solid Waste, Third Edition, Update II, October 1994, Semivolatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS): Capillary Column Technique, Method 8270B.

16.2. J. W. Eichelberger, L. E. Harris, and W. L. Budde, "Reference Compound to Calibrate Ion Abundance Measurement in Gas Chromatography/Mass Spectrometry," Analytical Chemistry, 47, 995 (1975)

17. MISCELLANEOUS

17.1. Modifications from Reference Method

17.1.1. Spike concentrations and Injection Volume

As allowed in section 7.3 of the reference method, a 2 µL injection volume is used. Some of the recommended spike concentrations in the reference method have been changed to bring them within the calibration range of the instrument.

17.1.2. The concentration of the internal standard spiking solution is more dilute than that specified in the reference method. This eliminates the risk of precipitation of the internal standards from solution when the standard is stored in the freezer, and also allows more accurate internal standard addition to small volumes of extract.

17.1.3. The internal standard control criteria of 50% to 200% is applied to each sample, rather than the subsequent continuing calibration standard as listed in the reference method. This is a more rigorous criterion than that in the reference method.

17.1.4. A retention time window of 0.2 minutes is used for all components, since some data systems do not have the capability of using the relative retention time units specified in the reference method.

17.1.5. The quantitation and qualifier ions for some compounds have been changed from those recommended in SW-846 in order to improve the reliability of qualitative identification.

17.2. Modifications from Previous Revisions

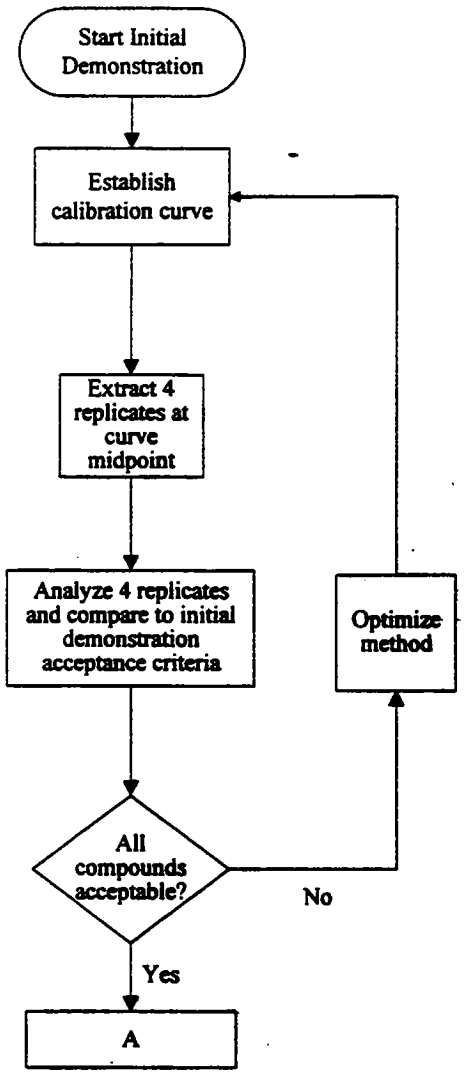
None

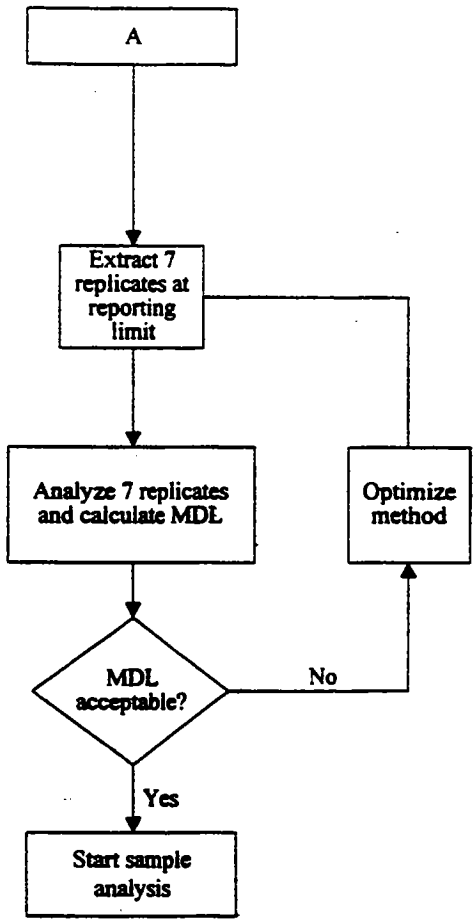
17.3. Facility Specific SOPs

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

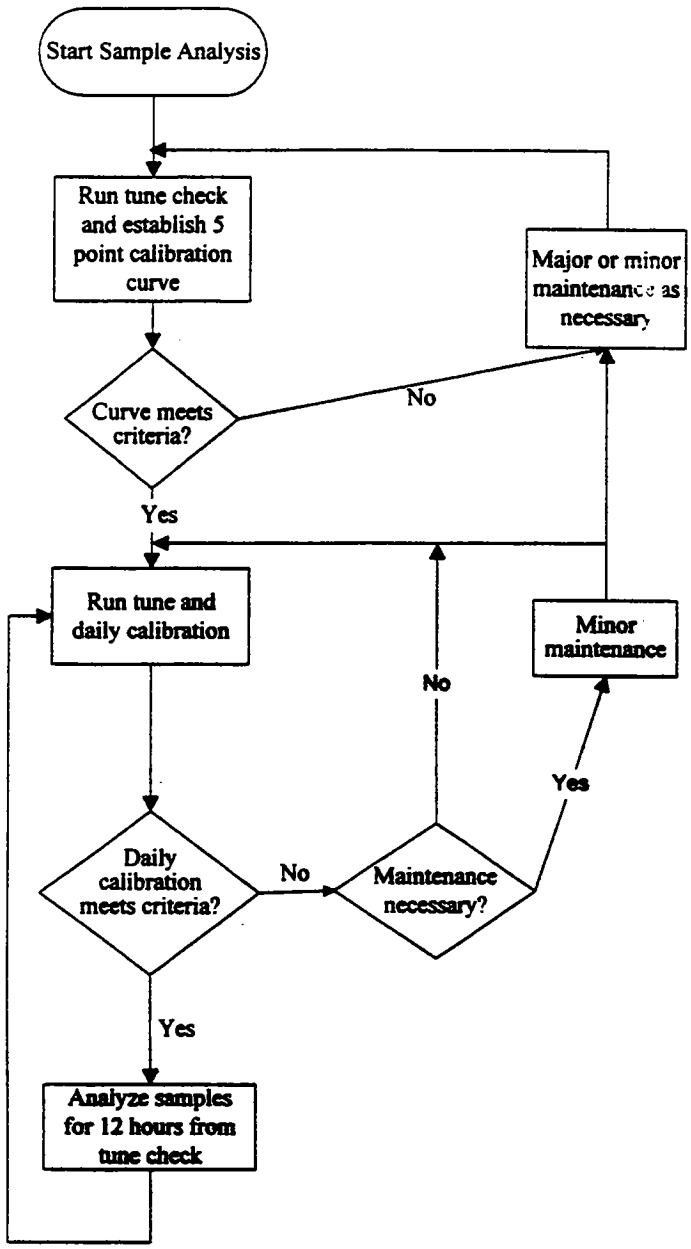
17.4. Flow Diagrams

17.4.1. Initial demonstration and MDL





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.

17.5. Tables

Table 1

Quanterra Primary Standard[1] and Standard Reporting Limits

Analytes	CAS Number	Standard Reporting Limits	
		Aqueous µg/L	Low Soil/Sediment µg/kg
Pyridine	110-86-1	20	660
N-nitrosodimethylamine	62-75-9	10	330
Aniline	62-53-3	10	330
Phenol	108-95-2	10	330
Bis(2-chloroethyl)ether	111-44-4	10	330
2-Chlorophenol	95-57-8	10	330
1,3-Dichlorobenzene	541-73-1	10	330
1,4-Dichlorobenzene	106-46-7	10	330
Benzyl alcohol	100-51-6	10	330
1,2-Dichlorobenzene	95-50-1	10	330
2-Methylphenol	95-48-7	10	330
2,2'-oxybis(1-chloropropane)[2]	180-60-1	10	330
4-Methylphenol	106-44-5	10	330
N-Nitroso-di-n-propylamine	621-64-7	10	330
Hexachloroethane	67-72-1	10	330
Nitrobenzene	98-95-3	10	330
Isophorone	78-59-1	10	330
2-Nitrophenol	88-75-5	10	330
2,4-Dimethylphenol	105-67-9	10	330
Benzoic acid	65-85-0	50	1600
Bis(2-chloroethoxy)methane	111-91-1	10	330
2,4-Dichlorophenol	120-83-2	10	330
1,2,4-Trichlorobenzene	120-82-1	10	330
Naphthalene	91-20-3	10	330
4-Chloroaniline	106-47-8	10	330
Hexachlorobutadiene	87-68-3	10	330
4-Chloro-3-methylphenol	59-50-7	10	330
2-Methylnaphthalene	91-57-6	10	330
Hexachlorocyclopentadiene	77-47-4	50	1600
2,4,6-Trichlorophenol	88-06-2	10	330
2,4,5-Trichlorophenol	95-95-4	10	330
2-Chloronaphthalene	91-58-7	10	330
2-Nitroaniline	88-74-4	50	1600
Dimethyl phthalate	131-11-3	10	330
Acenaphthylene	208-96-8	10	330
3-Nitroaniline	99-09-2	50	1600
Acenaphthene	83-32-9	10	330
2,4-Dinitrophenol	51-28-5	50	1600
4-Nitrophenol	100-02-7	50	1600

Table 1

Quanterra Primary Standard[1] and Standard Reporting Limits

Analytes	CAS Number	Standard Reporting Limits	
		Aqueous µg/L	Low Soil/Sediment µg/kg
Dibenzofuran	132-64-9	10	330
2,4-Dinitrotoluene	121-14-2	10	330
2,6-Dinitrotoluene	606-20-2	10	330
Diethylphthalate	84-66-2	10	330
4-Chlorophenyl phenyl ether	7005-72-3	10	330
Fluorene	86-73-7	10	330
4-Nitroaniline	100-01-6	50	1600
4,6-Dinitro-2-methylphenol	534-52-1	50	1600
N-Nitrosodiphenylamine	86-30-6	10	330
Azobenzene	103-33-3	10	330
4-Bromophenyl phenyl ether	101-55-3	10	330
Hexachlorobenzene	118-74-1	10	330
Pentachlorophenol	87-86-5	50	1600
Phenanthrene	85-01-8	10	330
Anthracene	120-12-7	10	330
Carbazole	86-74-8	10	330
Di-n-butyl phthalate	84-74-2	10	330
Fluoranthene	206-44-0	10	330
Benzidine	92-87-5	100	3300
Pyrene	129-00-0	10	330
Butyl benzyl phthalate	85-68-7	10	330
3,3'-Dichlorobenzidine	91-94-1	50	1600
Benzo(a)anthracene	56-55-3	10	330
Bis(2-ethylhexyl)phthalate	117-81-7	10	330
Chrysene	218-01-9	10	330
Di-n-octylphthalate	117-84-0	10	330
Benzo(b)fluoranthene	205-99-2	10	330
Benzo(k)fluoranthene	207-08-9	10	330
Benzo(a)pyrene	50-32-8	10	330
Indeno(1,2,3-cd)pyrene	193-39-5	10	330
Dibenz(a,h)anthracene	53-70-3	10	330
Benzo(g,h,i)perylene	191-24-2	10	330

[1] The Quanterra primary standard is the standard normally used at Quanterra. Additional standards, such as the Appendix IX standard may be necessary to include all target analytes required for some clients.

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

Table 2

Quanterra Appendix IX[1] Standard Reporting Limits

Semivolatiles	CAS Number	Standard Reporting Limits	
		Aqueous µg/L	Low Soil/Sediment µg/kg
2-Picoline	109-06-8	20	660
N-Nitrosomethylethylamine	10595-95-6	10	330
Methyl methanesulfonate	66-27-3	10	330
N-Nitrosodiethylamine	55-18-5	10	330
Ethyl methanesulfonate	62-50-0	10	330
Pentachloroethane	76-01-7	50	1600
Acetophenone	98-86-2	10	330
N-Nitrosopyrrolidine	930-55-2	10	330
N-Nitrosomorpholine	59-89-2	10	330
o-Toluidine	95-53-4	20	660
3-Methylphenol	108-39-4	10	330
N-Nitrosopiperidine	100-75-4	10	330
o,o,o-Triethyl-Phosphorothioate[2]	126-68-1	50	1600
a,a-Dimethyl-phenethylamine	122-09-8	50	1600
2,6-Dichlorophenol	87-65-0	10	330
Hexachloropropene	1888-71-7	100	3300
p-Phenylenediamine	106-50-3	100	3300
n-Nitrosodi-n-butylamine	924-16-3	10	330
Safrole	94-59-7	20	660
1,2,4,5-Tetrachlorobenzene	95-94-3	10	330
Isosafrole	120-58-1	20	660
1,4-Dinitrobenzene	100-25-4	10	330
1,4-Naphthoquinone	130-15-4	50	1600
1,3-Dinitrobenzene	99-65-0	10	330
Pentachlorobenzene	608-93-5	10	330
1-Naphthylamine	134-32-7	10	330
2-Naphthylamine	91-59-8	10	330
2,3,4,6-Tetrachlorophenol	58-90-2	50	1600
5-Nitro-o-toluidine	99-55-8	20	660
Thionazin[2]	297-97-2	50	1600
1,3,5-Trinitrobenzene	99-35-4	50	1600
Sulfotepp[2]	3689-24-5	50	1600
Phorate[2]	298-02-2	50	1600
Phenacetin	62-44-2	20	660
Diallate[3]	2303-16-4	20	660
Dimethoate[2]	60-51-5	20	660
4-Aminobiphenyl	92-67-1	50	1600
Pentachloronitrobenzene	82-68-8	50	1600
Pronamide	23950-58-5	20	660
Disulfoton[2]	298-04-4	50	1600
2-secbutyl-4,6-dinitrophenol (Dinoseb)	88-85-7	20	660
Methyl Parathion[2]	298-00-0	50	1600

Table 2

Quanterra Appendix IX[1] Standard Reporting Limits

Semivolatiles	CAS Number	Standard Reporting Limits	
		Aqueous µg/L	Low Soil/Sediment µg/kg
4-Nitroquinoline-1-oxide	56-57-5	100	3300
Parathion[2]	56-38-2	50	1600
Methapyrilene	91-80-5	50	1600
Aramite	140-57-8	20	660
Isodrin[3]	465-73-6	10	330
Kepone[2]	143-50-0	100	3300
Famphur[3]	52-85-7	100	3300
p-(Dimethylamino)azobenzene	60-11-7	20	660
p-Chlorobenzilate[3]	510-15-6	10	330
3,3'-Dimethylbenzidine	119-93-7	50	1600
2-Acetylaminofluorene	53-96-3	100	3300
Dibenz(a,j)acridine	224-42-0	20	660
7,12-Dimethylbenz(a)anthracene	57-97-6	20	660
3-Methylcholanthrene	56-49-5	20	660

1 The Appendix IX standard contains additional analytes required for the Appendix IX list. The Quanterra primary standard must also be analyzed to include all of the Appendix IX list.

2 May also be analyzed by method 8140 or 8141, which can acheive lower reporting limits.

3 May also be analyzed by method 8080 or 8081, which can achieve lower reporting limits

Table 3

Reportable Analytes for Quanterra Standard Tests, Primary Standard

Analyte	CAS Number	Quanterra Standard List	TCLP	TCL	Appendix IX
Pyridine	110-86-1		X		X
N-nitrosodimethylamine	62-75-9				X
Aniline	62-53-3				X
Phenol	108-95-2	X		X	X
Bis(2-chloroethyl)ether	111-44-4	X		X	X
2-Chlorophenol	95-57-8	X		X	X
1,3-Dichlorobenzene	541-73-1	X		X	X
1,4-Dichlorobenzene	106-46-7	X		X	X
Benzyl alcohol	100-51-6				X
1,2-Dichlorobenzene	95-50-1	X		X	X
2-Methylphenol	95-48-7	X	X	X	X
2,2'-oxybis(1-chloropropane)[1]	180-60-1	X		X	
4-Methylphenol	106-44-5	X	X	X	X
N-Nitroso-di-n-propylamine	621-64-7	X		X	X
Hexachloroethane	67-72-1	X	X	X	X
Nitrobenzene	98-95-3	X	X	X	X
Isophorone	78-59-1	X		X	X
2-Nitrophenol	88-75-5	X		X	X
2,4-Dimethylphenol	105-67-9	X		X	X
Benzoic acid	65-85-0				
Bis(2-chloroethoxy)methane	111-91-1	X		X	X
2,4-Dichlorophenol	120-83-2	X		X	X
1,2,4-Trichlorobenzene	120-82-1	X		X	X
Naphthalene	91-20-3	X		X	X
4-Chloroaniline	106-47-8	X		X	X
Hexachlorobutadiene	87-68-3	X	X	X	X
4-Chloro-3-methylphenol	59-50-7	X		X	X
2-Methylnaphthalene	91-57-6	X		X	X
Hexachlorocyclopentadiene	77-47-4	X		X	X
2,4,6-Trichlorophenol	88-06-2	X		X	X
2,4,5-Trichlorophenol	95-95-4	X		X	X
2-Chloronaphthalene	91-58-7	X		X	X
2-Nitroaniline	88-74-4	X		X	X
Dimethyl phthalate	131-11-3	X		X	X
Acenaphthylene	208-96-8	X		X	X
3-Nitroaniline	99-09-2	X		X	X
Acenaphthene	83-32-9	X		X	X
2,4-Dinitrophenol	51-28-5	X		X	X
4-Nitrophenol	100-02-7	X		X	X
Dibenzofuran	132-64-9	X		X	X
2,4-Dinitrotoluene	121-14-2	X	X	X	X
2,6-Dinitrotoluene	606-20-2	X		X	X
Diethylphthalate	84-66-2	X		X	X

Table 3

Reportable Analytes for Quanterra Standard Tests, Primary Standard

Analyte	CAS Number	Quanterra Standard List	TCLP	TCL	Appendix IX
4-Chlorophenyl phenyl ether	7005-72-3	X		X	X
Fluorene	86-73-7	X		X	X
4-Nitroaniline	100-01-6	X		X	X
4,6-Dinitro-2-methylphenol	534-52-1	X		X	X
N-Nitrosodiphenylamine	86-30-6	X		X	X
Azobenzene[4]	103-33-3				
4-Bromophenyl phenyl ether	101-55-3	X		X	X
Hexachlorobenzene	118-74-1	X	X	X	X
Pentachlorophenol	87-86-5	X	X	X	X
Phenanthrene	85-01-8	X		X	X
Anthracene	120-12-7	X		X	X
Carbazole	86-74-8	X		X	
Di-n-butyl phthalate	84-74-2	X		X	X
Fluoranthene	206-44-0	X		X	X
Benzidine	92-87-5				
Pyrene	129-00-0	X		X	X
Butyl benzyl phthalate	85-68-7	X		X	X
3,3'-Dichlorobenzidine	91-94-1	X		X	X
Benzo(a)anthracene	56-55-3	X		X	X
Bis(2-ethylhexyl)phthalate	117-81-7			X	X
Chrysene	218-01-9	X		X	X
Di-n-octylphthalate	117-84-0	X		X	X
Benzo(b)fluoranthene	205-99-2	X		X	X
Benzo(k)fluoranthene	207-08-9	X		X	X
Benzo(a)pyrene	50-32-8	X		X	X
Indeno(1,2,3-cd)pyrene	193-39-5	X		X	X
Dibenz(a,h)anthracene	53-70-3	X		X	X
Benzo(g,h,i)perylene	191-24-2	X		X	X

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

[2] Azobenzene is formed by decomposition of 1,2-diphenlyhydrazine. If 1,2-diphenylhydrazine is requested, it will be analyzed as azobenzene.

Table 4

Reportable analytes for Quanterra Standard Tests, Appendix IX Standard

Semivolatiles	CAS Number	Quanterra Standard List	TCLP	TCL	Appendix IX
2-Picoline	109-06-8				X
N-Nitrosomethylethylamine	10595-95-6				X
Methyl methanesulfonate	66-27-3				X
N-Nitrosodiethylamine	55-18-5				X
Ethyl methanesulfonate	62-50-0				X
Pentachloroethane	76-01-7				X
Acetophenone	98-86-2				X
N-Nitrosopyrrolidine	930-55-2				X
N-Nitrosomorpholine	59-89-2				X
o-Toluidine	95-53-4				X
3-Methylphenol	108-39-4				X
N-Nitrosopiperidine	100-75-4				X
o,o,o-Triethyl-Phosphorothioate[2]	126-68-1				X
a,a-Dimethyl-phenethylamine	122-09-8				X
2,6-Dichlorophenol	87-65-0				X
Hexachloropropene	1888-71-7				X
p-Phenylenediamine	106-50-3				X
n-Nitrosodi-n-butylamine	924-16-3				X
Safrole	94-59-7				X
1,2,4,5-Tetrachlorobenzene	95-94-3				X
Isosafrole	120-58-1				X
1,4-Dinitrobenzene	100-25-4				
1,4-Naphthoquinone	130-15-4				X
1,3-Dinitrobenzene	99-65-0				X
Pentachlorobenzene	608-93-5				X
1-Naphthylamine	134-32-7				X
2-Naphthylamine	91-59-8				X
2,3,4,6-Tetrachlorophenol	58-90-2				X
5-Nitro-o-toluidine	99-55-8				X
Thionazin[2]	297-97-2				X
1,3,5-Trinitrobenzene	99-35-4				X
Sulfotepp[2]	3689-24-5				X
Phorate[2]	298-02-2				X
Phenacetin	62-44-2				X
Diallate	2303-16-4				X
Dimethoate[2]	60-51-5				X
4-Aminobiphenyl	92-67-1				X
Pentachloronitrobenzene	82-68-8				X
Pronamide	23950-58-5				X
Disulfoton[2]	298-04-4				X
2-secbutyl-4,6-dinitrophenol (Dinoseb)[2]	88-85-7				X
Methyl parathion[2]	298-00-0				X

Table 4

Reportable analytes for Quanterra Standard Tests, Appendix IX Standard

Semivolatiles	CAS Number	Quanterra Standard List	TCLP	TCL	Appendix IX
4-Nitroquinoline-1-oxide	56-57-5				X
Parathion[2]	56-38-2				X
Isodrin[3]	465-73-6				X
Kepone [2]	143-50-0				
Famphur[2]	52-85-7				X
Methapyrilene	91-80-5				X
Aramite	140-57-8				X
p-(Dimethylamino)azobenzene	60-11-7				X
p-Chlorobenzilate[3]	510-15-6				X
3,3'-Dimethylbenzidine	119-93-7				X
2-Acetylaminofluorene	53-96-3				X
Dibenz(a,j)acridine	224-42-0				
7,12-Dimethylbenz(a)anthracene	57-97-6				X
3-Methylcholanthrene	56-49-5				X
Hexachlorophene[4]	70-30-4				X
Diphenylamine[5]	122-39-4				X

[2] May also be analyzed by method 8140 or 8141, which can acheive lower reporting limits.

[3] May also be analyzed by method 8080 or 8081, which can achieve lower reporting limits

[4] Hexachlorophene is a required analyte for Appendix IX. This compound is not stable, and therefore not included in the calibration standard. The characteristic ions for hexachlorophene is searched for in the chromatogram. (See section 12.2.1)

[5] Diphenylamine is a required compound for Appendix IX. N-nitrosodiphenylamine decomposes in the injection port to form diphenylamine. Therefore these two compounds cannot be distinguished. Diphenylamine is not included in the calibration standard.

Table 5

Recommended Instrumental Conditions

Mass Range	35-500 amu
Scan Time	≤1 second/scan
Initial Column Temperature/Hold Time	40°C for 2 minutes
Column Temperature Program	40 - 320°C at 11.5°C/min
Final Column Temperature/Hold Time	320°C (until at least one minute after benzo(g,h,i)perylene has eluted)
Injector Temperature	250 - 300°C
Transfer Line Temperature	250 - 300°C
Source Temperature	According to manufacturer's specifications
Injector	Grob-type, split / splitless
Sample Volume	2 µl
Carrier Gas	Helium at 30 cm/sec

Table 6

DFTPP Key Ions and Ion Abundance Criteria

Mass	Ion Abundance Criteria
51	30 - 60% of mass 198
68	<2% of mass 69
70	<2% of mass 69
127	40 - 60% of mass 198
197	<1% of mass 198
198	Base peak, 100% relative abundance
199	5 - 9% of mass 198
275	10 - 30% of mass 198
365	>1% of mass 198
441	Present, but less than mass 443
442	>40% of mass 198
443	17 - 23% of mass 442

Table 7

Analytes in Approximate Retention Time Order and Characteristic Ions, Primary Standard

Analyte	Primary	Secondary	Tertiary
N-nitrosodimethylamine	74	42	
Pyridine	79	52	
2-Fluorophenol (Surrogate Standard)	112	64	63
Phenol-d5 (Surrogate Standard)	99	42	71
Aniline	93	66	
Phenol	94	65	66
Bis(2-chloroethyl)ether	93	63	95
2-Chlorophenol	128	64	130
1,3-Dichlorobenzene	146	148	113
1,4-Dichlorobenzene-d4 (Internal Standard)	152	150	115
1,4-Dichlorobenzene	146	148	113
Benzyl Alcohol	108	79	77
1,2-Dichlorobenzene	146	148	113
2-Methylphenol	108	107	79
2,2'-oxybis(1-chloropropane)[1]	45	77	79
4-Methylphenol	108	107	79
N-Nitroso-di-n-propylamine	70	42	101,130
Hexachloroethane	117	201	199
Nitrobenzene-d5 (Surrogate Standard)	82	128	54
Nitrobenzene	77	123	65
Isophorone	82	95	138
2-Nitrophenol	139	65	109
2,4-Dimethylphenol	107	121	122
Benzoic Acid	122	105	77
Bis(2-chloroethoxy)methane	93	95	123
2,4-Dichlorophenol	162	164	98
1,2,4-Trichlorobenzene	180	182	145
Naphthalene-d8 (Internal Standard)	136	68	54
Naphthalene	128	129	127
4-Chloroaniline	127	129	65
Hexachlorobutadiene	225	223	227
4-Chloro-3-methylphenol	107	144	142
2-Methylnaphthalene	142	141	115
Hexachlorocyclopentadiene	237	235	272
2,4,6-Trichlorophenol	196	198	200
2,4,5-Trichlorophenol	196	198	200
2-Fluorobiphenyl (Surrogate Standard)	172	171	170
2-Chloronaphthalene	162	164	127
2-Nitroaniline	65	92	138
Dimethylphthalate	163	194	164
Acenaphthylene	152	151	153

Table 7

Analytes in Approximate Retention Time Order and Characteristic Ions, Primary Standard

Analyte	Primary	Secondary	Tertiary
2,6-Dinitrotoluene	165	63	89
Acenaphthene-d10 (Internal Standard)	164	162	160
3-Nitroaniline	138	108	92
Acenaphthene	153	152	154
2,4-Dinitrophenol	184	63	154
Dibenzofuran	168	139	84
4-Nitrophenol	109	139	65
2,4-Dinitrotoluene	165	63	89
Diethylphthalate	149	177	150
Fluorene	166	165	167
4-Chlorophenylphenylether	204	206	141
4-Nitroaniline	138	92	108
4,6-Dinitro-2-methylphenol	198	182	77
N-Nitrosodiphenylamine	169	168	167
2,4,6-Tribromophenol (Surrogate Standard)	330	332	141
Azobenzene	77	182	105
4-Bromophenylphenylether	248	250	141
Hexachlorobenzene	284	142	249
Pentachlorophenol	266	264	268
Phenanthrene-d10 (Internal Standard)	188	94	80
Phenanthrene	178	179	176
Anthracene	178	179	176
Carbazole	167	166	139
Di-n-butylphthalate	149	150	104
Fluoranthene	202	101	100
Benzidine	184	92	185
Pyrene	202	101	100
Terphenyl-d14 (Surrogate Standard)	244	122	212
Butylbenzylphthalate	149	91	206
Benzo(a)Anthracene	228	229	226
Chrysene-d12 (Internal Standard)	240	120	236
3,3'-Dichlorobenzidine	252	254	126
Chrysene	228	226	229
Bis(2-ethylhexyl)phthalate	149	167	279
Di-n-octylphthalate	149	167	43
Benzo(b)fluoranthene	252	253	125
Benzo(k)fluoranthene	252	253	125
Benzo(a)pyrene	252	253	125
Perylene-d12 (Internal Standard)	264	260	265
Indeno(1,2,3-cd)pyrene	276	138	227
Dibenz(a,h)anthracene	278	139	279
Benzo(g,h,i)perylene	276	138	277

Table 8

Analytes in Approximate Retention Time Order and Characteristic Ions, Appendix IX Standard

Analyte	Primary	Secondary	Tertiary
2-Picoline	93	66	92
N-Nitrosomethylethylamine	88	42	43
Methyl methanesulfonate	80	79	65
N-Nitrosodiethylamine	102	44	57
Ethyl methanesulfonate	79	109	97
Pentachloroethane	117	119	167
Acetophenone	105	77	120
N-Nitrosopyrrolidine	100	41	42
N-Nitrosomorpholine	116	56	86
o-Toluidine	106	107	
3-Methylphenol	108	107	77
N-Nitrosopiperidine	114	42	55
o,o,o-Triethyl-Phosphorothioate	198	121	93
a,a-Dimethyl-phenethylamine	58	91	
2,6-Dichlorophenol	162	164	63
Hexachloropropene	213	215	211
p-Phenylenediamine	108	80	
n-Nitrosodi-n-butylamine	84	57	41
Safrole	162	104	77
1,2,4,5-Tetrachlorobenzene	216	214	218
Isosafrole 1	162	104	131
Isosafrole 2	162	104	131
1,4-Dinitrobenzene	168	75	122
1,4-Naphthoquinone	158	104	102
1,3-Dinitrobenzene	168	75	76
Pentachlorobenzene	250	248	252
1-Naphthylamine	143	115	
2-Naphthylamine	143	115	
2,3,4,6-Tetrachlorophenol	232	230	131
5-Nitro-o-toluidine	152	77	106
Thionazin	97	96	143
1,3,5-Trinitrobenzene	213	75	120
Sulfotepp	97	322	202
Phorate	75	179	121
Phenacetin	108	180	109
Diallate	86	234	
Dimethoate	87	93	125
4-Aminobiphenyl	169		
Pentachloronitrobenzene	237	142	214
Pronamide	173	175	255
Disulfoton	88	97	89
2-secbutyl-4,6-dinitrophenol (Dinoseb)	211	163	147
Methyl parathion	109	125	263
4-Nitroquinoline-1-oxide	190	128	160

Table 8

Analytes in Approximate Retention Time Order and Characteristic Ions, Appendix IX Standard

Analyte	Primary	Secondary	Tertiary
Parathion	109	97	291
Isodrin	193	66	195
Kepone	272	274	237
Famphur	218	125	93
Methapyrilene	97	50	
Aramite 1	185	319	
Aramite 2	185	319	
p-(Dimethylamino)azobenzene	120	225	77
p-Chlorobenzilate	251	139	253
3,3'-Dimethylbenzidine	212	106	
2-Acetylaminofluorene	181	180	223
Dibenz(a,j)acridine	279	280	
7,12-Dimethylbenz(a)anthracene	256	241	120
3-Methylcholanthrene	268	252	253

Table 8

Analytes in Approximate Retention Time Order and Characteristic Ions, Appendix IX Standard

Analyte	Primary	Secondary	Tertiary
Parathion	109	97	291
Isodrin	193	66	195
Kepone	272	274	237
Famphur	218	125	93
Methapyrilene	97	50	
Aramite 1	185	319	
Aramite 2	185	319	
p-(Dimethylamino)azobenzene	120	225	77
p-Chlorobenzilate	251	139	253
3,3'-Dimethylbenzidine	212	106	
2-Acetylaminofluorene	181	180	223
Dibenz(a,j)acridine	279	280	
7,12-Dimethylbenz(a)anthracene	256	241	120
3-Methylcholanthrene	268	252	253

Table 9

8270B LCS Compounds

LCS Compounds	Spiking Level, ng/µL in extract
1,2,4-Trichlorobenzene	50
Acenaphthene	50
2,4-Dinitrotoluene	50
Pyrene	50
N-Nitroso-di-n-propylamine	50
1,4-Dichlorobenzene	50
Pentachlorophenol	75
Phenol	75
2-Chlorophenol	75
4-Chloro-3-methylphenol	75
4-Nitrophenol	75

Table 10

TCLP LCS Compounds

LCS Compounds	Spiking Level, ng/µL in extract
2,4-Dinitrotoluene	50
Hexachlorobutadiene	50
Hexachloroethane	50
2-Methylphenol	50
3-Methylphenol	50
4-Methylphenol	50
Nitrobenzene	50
Pentachlorophenol	50
Pyridine	50
2,4,5-Trichlorophenol	50
2,4,6-Trichlorophenol	50

Recovery limits for the LCS and for matrix spikes are generated from historical data and are maintained by the QA department.

Table 11

8270B Surrogate Compounds

Surrogate Compounds	Spiking Level, ng/µL in extract
Nitrobenzene-d5	50
2-Fluorobiphenyl	50
Terphenyl-d14	50
1,2-Dichlorobenzene-d4[1]	50
Phenol-d5	75
2-Fluorophenol	75
2,4,6-Tribromophenol	75
2-Chlorophenol-d4[1]	75

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

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

Table 12

Calibration Levels, Primary Standard, µg/mL

Analyte	Level 1	Level 2	Level 3	Level 4	Level 5
Pyridine	10	25	40	60	80
N-nitrosodimethylamine	10	25	40	60	80
Aniline	10	25	40	60	80
Phenol	10	25	40	60	80
Bis(2-chloroethyl)ether	10	25	40	60	80
2-Chlorophenol	10	25	40	60	80
1,3-Dichlorobenzene	10	25	40	60	80
1,4-Dichlorobenzene	10	25	40	60	80
Benzyl alcohol	10	25	40	60	80
1,2-Dichlorobenzene	10	25	40	60	80
2-Methylphenol	10	25	40	60	80
2,2'-oxybis(1-chloropropane)[1]	10	25	40	60	80
4-Methylphenol	10	25	40	60	80
N-Nitroso-di-n-propylamine	10	25	40	60	80
Hexachloroethane	10	25	40	60	80
Nitrobenzene	10	25	40	60	80
Isophorone	10	25	40	60	80
2-Nitrophenol	10	25	40	60	80
2,4-Dimethylphenol	10	25	40	60	80
Benzoic acid	10	25	40	60	80
Bis(2-chloroethoxy)methane	10	25	40	60	80
2,4-Dichlorophenol	10	25	40	60	80
1,2,4-Trichlorobenzene	10	25	40	60	80
Naphthalene	10	25	40	60	80
4-Chloroaniline	10	25	40	60	80
Hexachlorobutadiene	10	25	40	60	80

Table 12

Calibration Levels, Primary Standard, µg/mL

Analyte	Level 1	Level 2	Level 3	Level 4	Level 5
4-Chloro-3-methylphenol	10	25	40	60	80
2-Methylnaphthalene	10	25	40	60	80
Hexachlorocyclopentadiene	10	25	40	60	80
2,4,6-Trichlorophenol	10	25	40	60	80
2,4,5-Trichlorophenol	10	25	40	60	80
2-Chloronaphthalene	10	25	40	60	80
2-Nitroaniline	10	25	40	60	80
Dimethyl phthalate	10	25	40	60	80
Acenaphthylene	10	25	40	60	80
3-Nitroaniline	10	25	40	60	80
Acenaphthene	10	25	40	60	80
2,4-Dinitrophenol	10	25	40	60	80
4-Nitrophenol	10	25	40	60	80
Dibenzofuran	10	25	40	60	80
2,4-Dinitrotoluene	10	25	40	60	80
2,6-Dinitrotoluene	10	25	40	60	80
Diethylphthalate	10	25	40	60	80
4-Chlorophenyl phenyl ether	10	25	40	60	80
Fluorene	10	25	40	60	80
4-Nitroaniline	10	25	40	60	80
4,6-Dinitro-2-methylphenol	10	25	40	60	80
N-Nitrosodiphenylamine	10	25	40	60	80
Azobenzene[2]	10	25	40	60	80
4-Bromophenyl phenyl ether	10	25	40	60	80
Hexachlorobenzene	10	25	40	60	80
Pentachlorophenol	10	25	40	60	80
Phenanthrene	10	25	40	60	80
Anthracene	10	25	40	60	80
Carbazole	10	25	40	60	80
Di-n-butyl phthalate	10	25	40	60	80
Fluoranthene	10	25	40	60	80
Benzidine	10	25	40	60	80
Pyrene	10	25	40	60	80
Butyl benzyl phthalate	10	25	40	60	80
3,3'-Dichlorobenzidine	10	25	40	60	80
Benzo(a)anthracene	10	25	40	60	80
Bis(2-ethylhexyl)phthalate	10	25	40	60	80
Chrysene	10	25	40	60	80
Di-n-octylphthalate	10	25	40	60	80
Benzo(b)fluoranthene	10	25	40	60	80
Benzo(k)fluoranthene	10	25	40	60	80
Benzo(a)pyrene	10	25	40	60	80
Indeno(1,2,3-cd)pyrene	10	25	40	60	80
Dibenz(a,h)anthracene	10	25	40	60	80
Benzo(g,h,i)perylene	10	25	40	60	80

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

[2] Azobenzene is formed by decomposition of 1,2-diphenlyhydrazine. If 1,2-diphenylhydrazine is requested, it will be analyzed as azobenzene.

Table 13

Calibration Levels, Appendix IX Standard, µg/mL

Semivolatiles	Level 1	Level 2	Level 3	Level 4	Level 5
2-Picoline	10	25	40	60	80
N-Nitrosomethylethylamine	10	25	40	60	80
Methyl methanesulfonate	10	25	40	60	80
N-Nitrosodiethylamine	10	25	40	60	80
Ethyl methanesulfonate	10	25	40	60	80
Pentachloroethane	10	25	40	60	80
Acetophenone	10	25	40	60	80
N-Nitrosopyrrolidine	10	25	40	60	80
N-Nitrosomorpholine	10	25	40	60	80
o-Toluidine	10	25	40	60	80
3-Methylphenol	10	25	40	60	80
N-Nitrosopiperidine	10	25	40	60	80
o,o,o-Triethyl-Phosphorothioate	20	50	80	120	160
a,a-Dimethyl-phenethylamine	10	25	40	60	80
2,6-Dichlorophenol	10	25	40	60	80
Hexachloropropene	20	50	80	120	160
p-Phenylenediamine	10	25	40	60	80
n-Nitrosodi-n-butylamine	10	25	40	60	80
Safrole	10	25	40	60	80
1,2,4,5-Tetrachlorobenzene	10	25	40	60	80
Isosafrole 1 + 2	20	50	80	120	160
1,4-Dinitrobenzene	10	25	40	60	80
1,4-Naphthoquinone	10	25	40	60	80
1,3-Dinitrobenzene	10	25	40	60	80
Pentachlorobenzene	10	25	40	60	80
1-Naphthylamine	10	25	40	60	80
2-Naphthylamine	10	25	40	60	80
2,3,4,6-Tetrachlorophenol	10	25	40	60	80
5-Nitro-o-toluidine	10	25	40	60	80
Thionazin	10	25	40	60	80
1,3,5-Trinitrobenzene	20	50	80	120	160
Sulfotepp	10	25	40	60	80
Phorate	10	25	40	60	80
Phenacetin	10	25	40	60	80
Diallate 1 + 2	20	50	80	120	160
Dimethoate	10	25	40	60	80
4-Aminobiphenyl	10	25	40	60	80
Pentachloronitrobenzene	20	50	80	120	160
Pronamide	10	25	40	60	80
Disulfoton	10	25	40	60	80

Table 13

Calibration Levels, Appendix IX Standard, µg/mL

Semivolatiles	Level 1	Level 2	Level 3	Level 4	Level 5
2-secbutyl-4,6-dinitrophenol (Dinoseb)	20	50	80	120	160
Methyl parathion	10	25	40	60	80
4-Nitroquinoline-1-oxide	20	50	80	120	160
Parathion	10	25	40	60	80
Isodrin	10	25	40	60	80
Kepone	40	100	160	240	320
Famphur	40	100	160	240	320
Methapyrilene	10	25	40	60	80
Aramite 1 and 2	20	50	80	120	160
p-(Dimethylamino)azobenzene	10	25	40	60	80
p-Chlorobenzilate	10	25	40	60	80
3,3'-Dimethylbenzidine	10	25	40	60	80
2-Acetylaminofluorene	10	25	40	60	80
Dibenz (a,j)acridine	10	25	40	60	80
7,12-Dimethylbenz(a)anthracene	10	25	40	60	80
3-Methylcholanthrene	10	25	40	60	80

Table 14

Initial demonstration recovery and precision limits

Compound	Spiking concentration µg/L	Limit for Relative Standard Deviation	Limit for average recovery, %
Acenaphthene	60	27.6	60.1-132.3
Acenaphthylene	60	40.2	53.5-126.0
Aldrin[1]	60	39.0	7.2-152.2
Anthracene	60	32.0	43.4-118.0
Benz(a)anthracene	60	27.6	41.8-133.0
Benzo(b)fluoranthene	60	38.8	42.0-140.4
Benzo(k)fluoranthene	60	32.3	25.2-145.7
Benzo(a)pyrene	60	39.0	31.7-148.0
Benzo(ghi)perylene	60	58.9	D-195.0
Benzylbutyl phthalate	60	23.4	D-139.9
B-BHC[1]	60	31.5	41.5-130.6
d-BHC[1]	60	21.6	D-100.0
Bis(2-chloroethyl) ether	60	55.0	42.9-126.0
Bis(2-chloroethoxy)methane	60	34.5	49.2-164.7
Bis(2-chloroisopropyl) ether	60	46.3	62.8-138.6
Bis(2-ethylhexyl) phthalate	60	41.1	28.9-136.8
4-Bromophenyl phenyl ether	60	23.0	64.9-114.4
2-Chloronaphthalene	60	13.0	64.5-113.5
4-Chlorophenyl phenyl ether	60	33.4	38.4-144.7
Chrysene	60	48.3	44.1-139.9
4,4'-DDD[1]	60	31.0	D-134.5
4,4'-DDE[1]	60	32.0	19.2-119.7
4,4'-DDT[1]	60	61.6	D-170.6
Dibenzo(a,h)anthracene	60	70.0	D-199.7
Di-n-butyl phthalate	60	16.7	8.4-111.0
1,2-Dichlorobenzene	60	30.9	48.6-112.0
1,3-Dichlorobenzene	60	41.7	16.7-153.9
1,4-Dichlorobenzene	60	32.1	37.3-105.7
3,3'-Dichlorobenzidine	60	71.4	8.2-212.5
Dieldrin[1]	60	30.7	44.3-119.3
Diethyl phthalate	60	26.5	D-100.0
Dimethyl phthalate	60	23.2	D-100.0
2,4-Dinitrotoluene	60	21.8	47.5-126.9
2,6-Dinitrotoluene	60	29.6	68.1-136.7
Di-n-octylphthalate	60	31.4	18.6-131.8
Endosulfan sulfate[1]	60	16.7	D-103.5
Endrin aldehyde	60	32.5	D-188.8
Fluoranthene	60	32.8	42.9-121.3
Fluorene	60	20.7	71.6-108.4
Heptachlor[1]	60	37.2	D-172.2
Heptachlor epoxide[1]	60	54.7	70.9-109.4
Hexachlorobenzene	60	24.9	7.8-141.5

Table 14

Initial demonstration recovery and precision limits

Compound	Spiking concentration µg/L	Limit for Relative Standard Deviation	Limit for average recovery, %
Hexachlorobutadiene	60	26.3	37.8-102.2
Hexachloroethane	60	24.5	55.2-100.0
Indeno(1,2,3-cd)pyrene	60	44.6	D-150.9
Isophorone	60	63.3	46.6-180.2
Naphthalene	60	30.1	35.6-119.6
Nitrobenzene	60	39.3	54.3-157.6
N-Nitrosodi-n-propylamine	60	55.4	13.6-197.9
PCB-1260[1]	60	54.2	19.3-121.0
Phenanthrene	60	20.6	65.2-108.7
Pyrene	60	25.2	69.6-100.0
1,2,4-Trichlorobenzene	60	28.1	57.3-129.2
4-Chloro-3-methylphenol	60	37.2	40.8-127.9
2-Chlorophenol	60	28.7	36.2-120.4
2,4-Chlorophenol	60	26.4	52.5-121.7
2,4-Dimethylphenol	60	26.1	41.8-109.0
2,4-Dinitrophenol	60	49.8	D-172.9
2-Methyl-4,6-dinitrophenol	60	93.2	53.0-100.0
2-Nitrophenol	60	35.2	45.0-166.7
4-Nitrophenol	60	47.2	13.0-106.5
Pentachlorophenol	60	48.9	38.1-151.8
Phenol	60	22.6	16.6-100.0
2,4,6-Trichlorophenol	60	31.7	52.4-129.2

[1] Since the organochlorine pesticides and PCBs are normally determined by method 8080 at Quanterra, they will not be included in the initial demonstration of capability for method 8270B.

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QUANTERRA STANDARD OPERATING PROCEDURE

TITLE: GAS CHROMATOGRAPHIC ANALYSIS BASED ON METHOD 8000A, 8010B, 8020A, 8021A, 8080A and 8150B, SW-846

(SUPERSEDES: CORP-GC-0001, Revision 0)

Prepared by: Richard Burrows

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Technology Standardization Committee

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TABLE OF CONTENTS

1. Scope and Application 4
2. Summary of Method 4
3. Definitions 4
4. Interferences 4
5. Safety 4
6. Equipment and Supplies 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 based on Method 8080A

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

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	Stan ırd 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 nd 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 ≤6°C. All stock standards must be protected from light. Stock standard solutions should be brought to room temperature before using.

Semivolatile stock standard solutions must be replaced after one year. (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 ≤6°C and protected from light. The standards must be replaced at least every six months or sooner if comparison with check standards indicates a problem.

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

7.4. Quality control (QC) Standards

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

8. SAMPLE PRESERVATION AND STORAGE

Semivolatile extracts must be refrigerated at ≤6°C and analyzed within 40 days of the 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. Quadratic fit is normally used since it will minimize the quantitation errors. 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

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

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

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

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

- Response must increase with increasing concentration.
- 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, 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 c 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 : an 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

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

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 requires 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 requires 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 Revisions

17.2.1. Instrument blank guidance (section 9.6) has been added.

17.2.2. The recommendation that calibration curves be forced through zero has been removed.

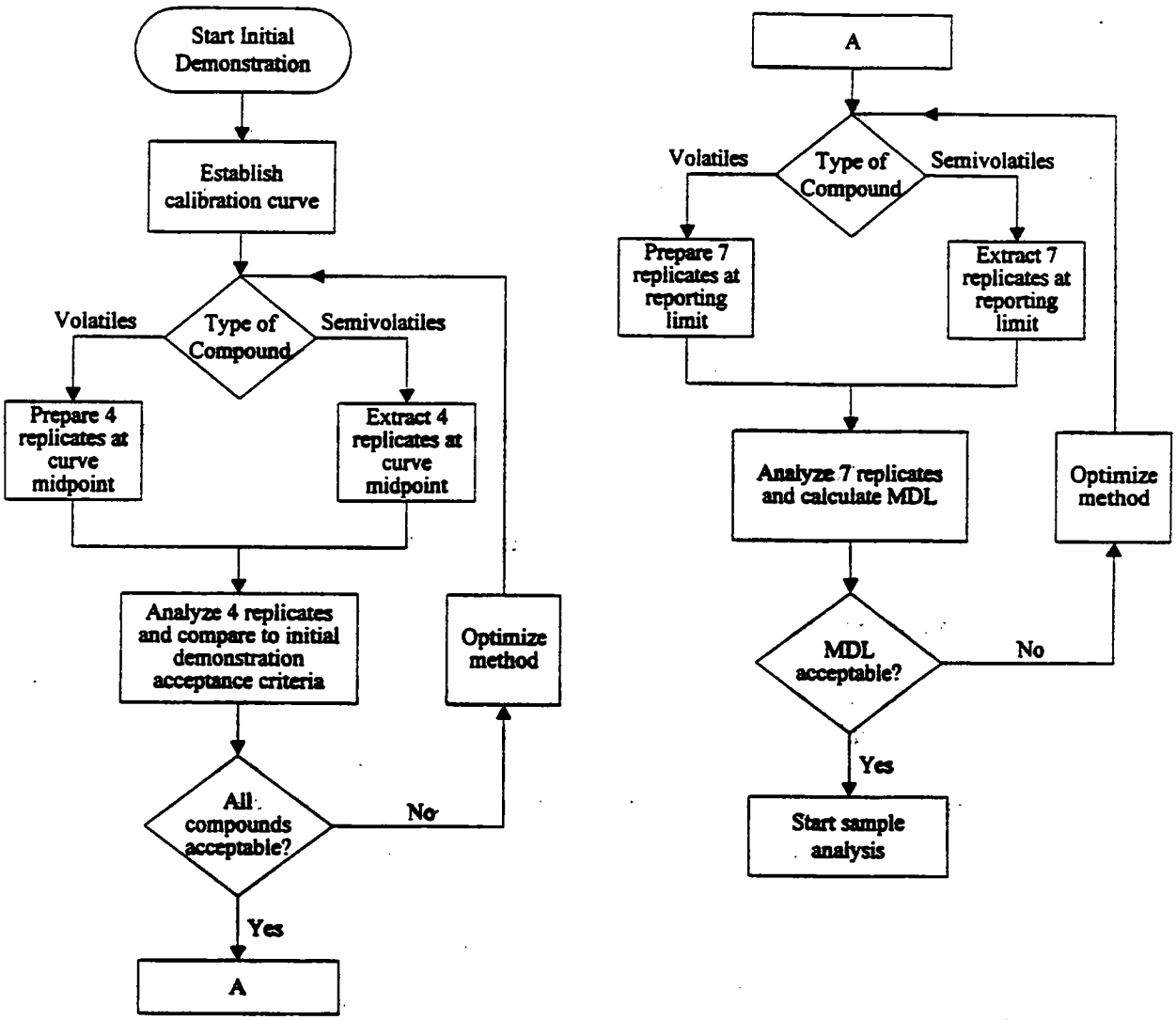
17.2.3. A number of typographical errors have been corrected.

17.3. Facility Specific SOPs

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

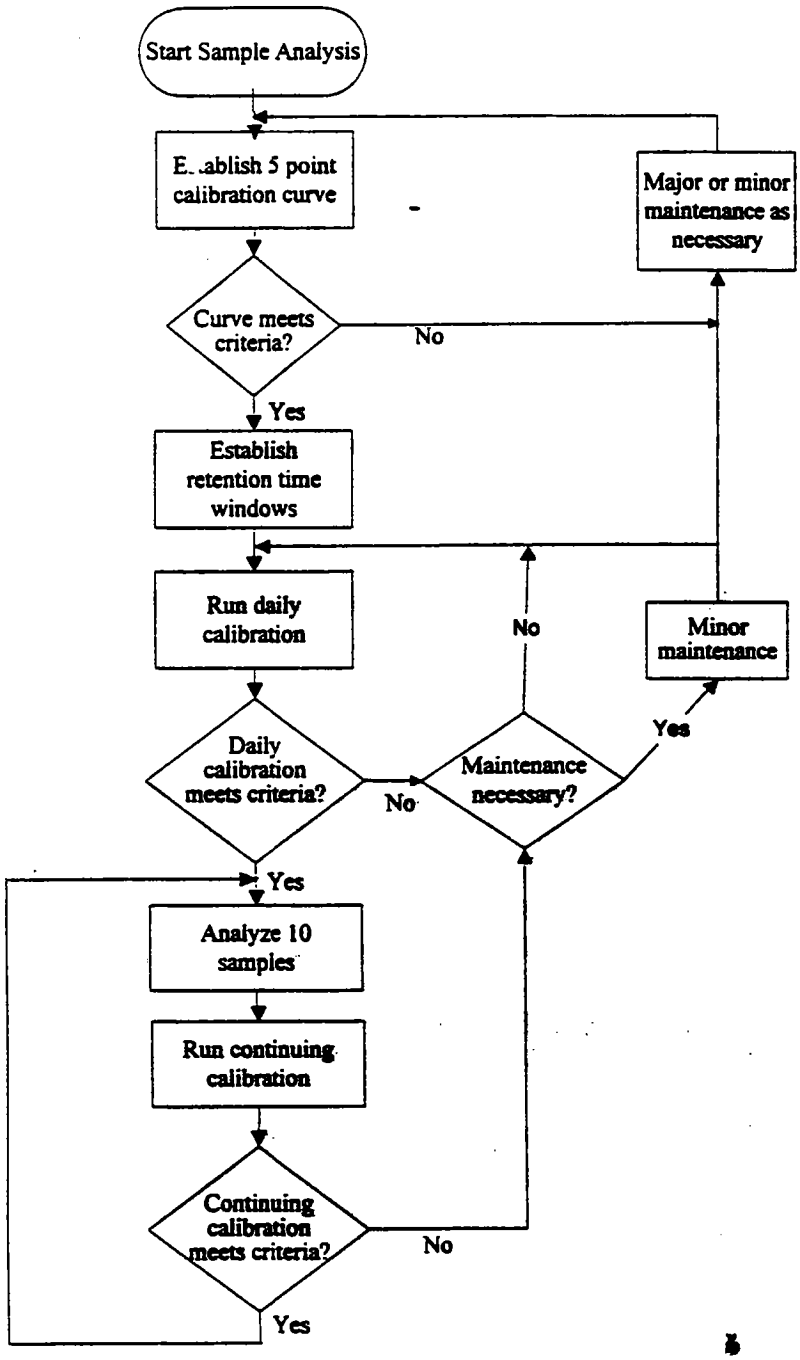
17.4. Flow Diagrams

17.4.1. Initial demonstration and MDL



[1] 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. This procedure invalidates the contents of the VOA vial for further analysis, unless an aliquot is transferred to a smaller VOA vial with no headspace (e.g., 20 mL) at the same time the analysis aliquot is removed.

11.4.5. Spike with the appropriate volume of surrogate/internal standard solution and 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.

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

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

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

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

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

11.5. Aqueous Sample Analysis (Purge and Trap units that sample directly from the VOA vial)

See also section 11.1 for cautions and corrective actions.

11.5.1. Units which sample from the VOA vial should be equipped with a module which automatically adds surrogate and internal standard solution to the sample prior to purging the sample.

11.5.2. If the autosampler uses automatic IS/SS injection, no further preparation of the VOA vial is needed. Otherwise the internal and surrogate standards must be added to the vial. *Note:* Samples with high amounts of sediment present in the vial may not be suitable for analysis on this instrumentation.

11.5.3. Sample remaining in the vial after sampling with one of these mechanisms is no longer valid for further analysis. A fresh VOA vial must be used for further sample analysis.

11.5.4. Check the pH of the sample remaining in the VOA vial after analysis is completed.

11.6. 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.

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

11.6.2. Weigh out 5 g (or other appropriate aliquot) of sample into a disposable culture tube or other purge vessel. Record the weight to the nearest 0.1 g. If method sensitivity is demonstrated, a smaller aliquot may be used. Do not use aliquots less than 1.0 g. If the sample is contaminated with analytes such that a purge amount less than 1.0 g is appropriate, use the medium level method described in section 11.7.

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

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

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

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

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

11.7. 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.

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

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

11.7.3. 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.

11.7.4. Shake the vial for 2 min.

11.7.5. 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.

11.7.6. 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. 1,4-Dichlorobutane has been substituted for bromochloromethane and fluorobenzene as the method 8021A surrogate.

17.2.2. 1-Chloro-3-fluorobenzene has been substituted for 1-chloro-2-fluorobenzene as the method 8021A internal standard.

17.3. Facility Specific SOPs

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

17.4. Tables

Table A-1					
Standard Analyte List					
Test	Compound	CAS number	Reporting Limit, 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	a,a,a,-TFT (SS)	20	5	20
	1-Chloro-3-fluorobenzene (IS)	40		40
8010B IS/SS	Bromochloromethane (SS)	20	5	20
	1-Chloro-3-fluorobenzene (IS)	40		40
8021A IS/SS	a,a,a-TFT (SS)	20	5	20
	1,4-Dichlorobutane (SS)	20	5	20
	1-Chloro-3-fluorobenzene (IS)	40	10	40

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	a,a,a,-TFT (SS)	20	500	2500	20
8020A IS	1-chloro-2-fluorobenzene	20	5	N/A	20
8010B SS	Bromochloromethane (SS)	20	500	2500	20
8010B IS	1-chloro-2-fluorobenzene	20	5	N/A	20
8021A SS	a,a,a,-TFT	20	500	2500	20
	1-4-Dichlorobutane	20	500	2500	20
8021A IS	1-chloro-3-fluorobenzene	20	5	N/A	20

2-Bromo-1-Chloropropane and 1,4-Dichlorobutane may also be used as surrogates for 8010B and 8021A
Bromofluorobenzene may be used as a surrogate for 8010B and 8020A.

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
	1,3-Dichlorobenzene	10		10
	Toluene	10		10
	Chlorobenzene	10		10
8010B MS	Chlorobenzene	10	500	10
	Chloroform	10		10
	1,3-Dichlorobenzene	10		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.

5. SAFETY

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

5.2. Aroclors have been classified as a potential carcinogen under OSHA. Concentrated solutions of Aroclors must be handled with extreme care to avoid excess exposure. Contaminated gloves and clothing must be removed immediately. Contaminated skin surfaces must be washed thoroughly.

6. EQUIPMENT AND SUPPLIES

6.1. Refer to Section 6 of the 8000A section of this SOP. A ^{63}Ni electron capture detector is required.

6.2. Refer to Table B-2 for analytical columns.

6.3. Microsyringes, various sizes, for standards preparation, sample injection, and extract dilution.

7. REAGENTS AND STANDARDS

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

7.2. Refer to Table B-3 for details of calibration standards.

7.3. Surrogate Standards

Tetrachloro-m-xylene and decachlorobiphenyl are the surrogate standards. Dibutylchlorendate may be used at client request. Refer to tables B-5, B-6, and B-7 for details of surrogate standards.

7.4. Column Degradation Evaluation Mix

A mid-level standard containing 4,4'-DDT and Endrin and not containing any of their breakdown products must be prepared for evaluation of degradation of these compounds by the GC column and injection port. This mix must be replaced after one year, or whenever corrective action to columns fails to eliminate the breakdown of the compounds, whichever is shorter. This solution also contains the surrogates. Refer to Table B-4 for details of the column degradation evaluation mix.

8. SAMPLE COLLECTION, PRESERVATION AND STORAGE

Refer to Section 8 of the 8000A section of this SOP.

9. QUALITY CONTROL

Refer to Section 9 of the 8000A section of this SOP.

10. CALIBRATION AND STANDARDIZATION

10.1. Refer to Section 10 of the 8000A section of this SOP for general calibration requirements.

10.2. External standard calibration is used for this method.

10.3. Refer to Table B-2 for details of GC operating conditions. The conditions listed should result in resolution of all analytes listed in Table B-1 on both columns. Closely eluting pairs are DDE and Dieldrin on the Rtx-5 or DB-5 column and Endosulfan II and DDD on the 1701 column.

10.4. Column Degradation Evaluation

Before any calibration runs, either initial or daily, the column evaluation mix must be injected. The degradation of DDT and endrin must be calculated (see equations 9 and 10) and each shown to be less than 20% before calibration can proceed. This is only necessary if the target compound list includes DDT, Endrin, or any of their degradation products.

If the breakdown of DDT and/or endrin exceeds the limits given above, corrective action must be taken. This action may include:

- Replacement of the injection port liner or the glass wool.
- Cutting off a portion of the injection end of a capillary column.
- Replacing the GC column.

10.5. Initial Calibration

Refer to Section 10 of the 8000A section of this SOP for details of calibration procedures.

10.5.1. Refer to Table B-8 for the initial calibration analytical sequence.

10.5.2. The response for each single-peak analyte will be calculated by the external standard procedure described in the general method for GC analysis.

10.5.3. The surrogate calibration curve is calculated from the Individual AB mix (or from the Aroclor 1016/1260 mix if the analysis is for acid cleaned

Aroclor only). Surrogates in the other calibration standards are used only as retention time markers.

10.5.4. For multi-component pesticides and Aroclors:

Two options are possible for quantitation of multicomponent pesticides and Aroclors. The same quantitation option must be used for standards and samples.

10.5.4.1. Multiple peak option

Select 3-10 major peaks in the analyte pattern. Calculate the response using the total area or total height of these peaks. Alternatively, find the response of each of the 3-10 peaks per multi-peak pesticide or Aroclor, and use these responses independently, averaging the resultant concentrations found in samples for a final concentration result. When using this option, it is appropriate to remove peaks that appear to be coeluting with contaminant peaks from the quantitation. (i.e. peaks which are significantly larger than would be expected from the rest of the pattern.)

10.5.4.2. Total area option

The total area of the standards and samples may be used for quantitation of multicomponent analytes. Any surrogate or extraneous peaks within the envelope must be subtracted from the total area. This option should not be used if there are significant interference peaks within the multicomponent pattern in the samples. The retention time window for total area measurement must contain at least 90% of the area of the analyte.

10.5.5. For multicomponent analytes, the low level standard must be analyzed as part of the initial calibration. If multicomponent analytes are present above half the reporting limit in any of the samples, these samples must be reanalyzed and quantitated against a valid five point calibration (unless a 5 point calibration for multicomponent was included as part of the initial calibration).

10.5.6. The analyst may include a full 5 point calibration for any of the multicomponent analytes with the initial calibration.

10.5.7. For Aroclor only analysis, a five point calibration of the 1016/1260 mix is generated with at least low level single points for the other Aroclor mixes. If multicomponent analytes are present above half the reporting limit in any of the samples, these samples must be reanalyzed and quantitated against a valid five point calibration.

10.6. Daily Calibration Verification

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

10.6.1. At a minimum, the daily calibration includes analysis of the breakdown mix followed by mid level standards of any single component analytes. (The individual mix AB and any additional single component analytes.)

10.6.2. If any multicomponent analytes are to be quantitated, then the daily calibration must include mid level standards of the appropriate multicomponents.

10.6.3. If multicomponent analytes do not have a full five point initial calibration or are not included with the daily calibration, then any multicomponent analytes found at greater than half the reporting limit must be reanalyzed and quantitated against a five point. Note that, at a minimum, all multicomponent analytes of interest must have a low level standard analyzed as part of the initial calibration to ensure that the sensitivity of the instrument is sufficient for pattern recognition.

10.6.4. Multicomponent analytes may be quantitated if there is a valid 5 point initial calibration on the instrument for the multicomponent analyte and a mid level standard of the multicomponent is analyzed within 24 hours and prior to the sample. The mid level standard must meet the 15% calibration criteria for quantitation.

10.6.5. The retention time windows for any analytes included in the daily calibration are updated.

10.7. Continuing Calibration

The AB calibration mix is analyzed as the continuing calibration standard. This is analyzed after every 10 samples, including matrix spikes, LCS, and method blanks. The continuing calibration standard need not include multicomponent analytes.

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

10.7.2. If only Aroclors are requested, the continuing calibration standard will consist of the mid level 1016/1260 Aroclor standard with the surrogates added.

11. PROCEDURE

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

11.2. Extraction

The extraction procedure is described in SOP No. CORP-OP-0001.

11.3. Cleanup

Cleanup procedures are described in SOP No. CORP-OP-0001.

11.4. Suggested gas chromatographic conditions are given in Table B-2.

11.5. Allow extracts to warm to ambient temperature before injection.

11.6. The suggested analytical sequence is given in Table B-8.

12. DATA ANALYSIS AND CALCULATIONS

12.1. Refer to the 8000A section of this SOP for identification and quantitation of single component analytes.

12.2. Identification of Multicomponent Analytes

Retention time windows are also used for identification of multi-component analytes, but the "fingerprint" produced by major peaks of those compounds in the standard is used in tandem with the retention times to identify the compounds. The ratios of the areas of the major peaks are also taken into consideration. Identification of these compounds may be made even if the retention times of the peaks in the sample fall outside of the retention time windows of the standard, if in the analyst's judgment the fingerprint (retention time and peak ratios) resembles the standard chromatogram.

12.2.1. The chromatogram is evaluated for technical chlordane if alpha and gamma chlordane are present. If technical chlordane is present it must be quantitated against a 5 point technical chlordane curve.

12.3. Quantitation of Multicomponent Analytes

Use 3-10 major peaks for quantitation as described in section 10.5.4, initial calibration of multicomponent analytes.

If the analyst believes that a combination of Aroclor 1254 and 1260, or a combination of 1242, 1248 and 1232 is present, then only the predominant aroclor is quantitated and reported, but the suspicion of multiple aroclors is discussed in the narrative. If well separated Aroclor patterns are present, then both aroclors are quantitated and reported.

12.3.1. If there are no interfering peaks within the envelope of the multicomponent analyte, the total area of the standards and samples may be used for quantitation. Any surrogate or extraneous peaks within the envelope must be subtracted from the total area.

12.4. Second column confirmation of Aroclors and other multi-component analytes will only be performed when requested by the client, because the appearance of the multiple peaks in the sample usually serves as a confirmation of Aroclor presence.

12.5. Surrogate recovery results are calculated and reported for decachlorobiphenyl (DCB) unless it is determined that sample interference has adversely affected the quantitation of that surrogate. Tetrachloro-m-xylene (TCMX) recovery is reported in lieu of DCB in samples having the interference, and also may be used in samples in which DCB recovery is low. Corrective action is only necessary if DCB and TCMX are both outside of acceptance limits.

12.6. Calculation of Column Degradation/% Breakdown (%B)

Equation 15

$$DDT\,\%B = \frac{A_{DDD} + A_{DDE}}{A_{DDD} + A_{DDE} + A_{DDT}} \times 100$$

where:
A_{DDD}, A_{DDE}, and A_{DDT} = the response of the peaks for 4,4'-DDD, 4,4'-DDE, and 4,4'-DDT in the column degradation evaluation mix.

Equation 16

$$Endrin\,\%B = \frac{A_{EK} + A_{EA}}{A_{EK} + A_{EA} + A_{E}} \times 100$$

where:
A_{EK}, A_{EA}, and A_E = the response of endrin ketone, endrin aldehyde, and endrin in the column degradation evaluation mix.

13. METHOD PERFORMANCE

13.1. Performance limits for the four replicate initial demonstration of capability required under Section 13.1 of the main body of this SOP are presented in Table B-7. The spiking level should be equivalent to a mid level calibration.

14. POLLUTION PREVENTION

Refer to section 14 of the 8000A section of this SOP.

15. WASTE MANAGEMENT

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

16. REFERENCES

SW846, Update II, September 1994, Method 8080A

17. MISCELLANEOUS

17.1. Modifications from Reference Method

17.1.1. Standards are made up in hexane, not isooctane. Use of standards in the same solvent as the sample extracts (hexane) ensures that analysis of the standard is consistent with analysis of the sample.

17.1.2. Only a single point initial calibration is required for multicomponent analytes (Aroclors, toxaphene and technical chlordane). However, if any of these analytes are found, they must be quantitated against a valid 5 point initial calibration.

17.1.3. Endrin ketone, alpha-chlordane and gamma-chlordane are included in the calibration standards. These are not standard 8080A analytes.

17.2. Modifications from Previous Revisions

None

17.3. Tables

Table B-1
Standard Analyte list and Reporting Limits

Compound	Reporting Limit, ug/L or ug/kg		
	water	soil	high level soil
Aldrin	0.05	1.7	50
a-BHC	0.05	1.7	50
b-BHC	0.05	1.7	50
d-BHC	0.05	1.7	50
g-BHC (Lindane)	0.05	1.7	50
Chlordane (technical)	0.5	17	500
4,4'-DDD	0.05	1.7	50
4,4'-DDE	0.05	1.7	50
4,4'-DDT	0.05	1.7	50
Dieldrin	0.05	1.7	50
Endosulfan I	0.05	1.7	50
Endosulfan II	0.05	1.7	50
Endosulfan Sulfate	0.05	1.7	50
Endrin	0.05	1.7	50
Endrin Aldehyde	0.05	1.7	50
Heptachlor	0.05	1.7	50
Heptachlor Epoxide	0.05	1.7	50
Methoxychlor	0.1	3.3	100
Toxaphene	2.0	67	2000
Aroclor-1016	1.0	33	1000
Aroclor-1221	1.0	33	1000
Aroclor-1232	1.0	33	1000
Aroclor 1242	1.0	33	1000
Aroclor-1248	1.0	33	1000
Aroclor-1254	1.0	33	1000
Aroclor-1260	1.0	33	1000
APPENDIX IX ADD ONs			
Diallate	1.0	33	1000
Isodrin	0.1	3.3	100
Chlorobenzillate	0.1	3.3	100
Kepone	1.0	33	1000

Note: alpha chlordane, gamma chlordane, and endrin ketone are not 8080A compounds.

The following concentration factors are assumed in calculating the Reporting Limits:

	Extraction Vol.	Final Vol.
Ground water	1000 mL	10 mL
Low-level Soil	30 g	10 mL
High-level soil / waste	1 g	10 mL

Table B-2

Parameter	Recommended Conditions
Injection port temp	220°C
Detector temp	325°C
Temperature program	70°C for 0.5min, 30°C/min to 190°C, 2.5°C/min to 225, 18°C/min to 280°C, 3 min hold
Column 1	DB-5 or Rtx-5 30m x 0.32mm id, 0.5µm
Column 2	DB-1701 or Rtx-1701 30m x 0.32 mm id, 0.25µm
Column 3	DB-608, 30m X 0.32 mm, 0.25µm
Injection	2µL
Carrier gas	Helium or Hydrogen
Make up gas	Nitrogen
Y splitter	Restek or J&W or Supelco glass tee

Table B-3						
Calibration Levels ng/mL						
	Level 1	Level 2	Level 3	Level 4	Level 5	Level 6[2]
Individual Mix AB[1]						
Aldrin	5	10	25	50	100	200
g-BHC (Lindane)	5	10	25	50	100	200
Heptachlor	5	10	25	50	100	200
Methoxychlor	10	20	50	100	200	400
Dieldrin	5	10	25	50	100	200
Endosulfan I	5	10	25	50	100	200
Endosulfan II	5	10	25	50	100	200
4,4'-DDT	5	10	25	50	100	200
Endrin Aldehyde	5	10	25	50	100	200
Endrin Ketone	5	10	25	50	100	200
b-BHC	5	10	25	50	100	200
d-BHC	5	10	25	50	100	200
a-BHC	5	10	25	50	100	200
4,4'-DDD	5	10	25	50	100	200
4,4'-DDE	5	10	25	50	100	200
Endosulfan Sulfate	5	10	25	50	100	200
Endrin	5	10	25	50	100	200
a-Chlordane[3]	5	10	25	50	100	200
g-Chlordane[3]	5	10	25	50	100	200
Multicomponent Standards						
Chlordane (Technical)	50	100	250	500	1000	2000
Toxaphene	200	400	1000	2000	4000	8000
Aroclor 1016/1260	100	200	500	1000	2000	4000
Aroclor 1242	100	200	500	1000	2000	4000
Aroclor 1221 +1254	100	200	500	1000	2000	4000
Aroclor 1232	100	200	500	1000	2000	4000
Aroclor 1248	100	200	500	1000	2000	4000
Surrogates are included with all the calibration mixes at the following levels:						
Tetrachloro-m-xylene	5	10	25	50	100	200
Decachlorobiphenyl[4]	5	10	25	50	100	200

[1] Standards may be split into an A and B mix if resolution of all compounds on both columns is not obtained.

[2] Level 6 is optional and should only be used if linearity can be maintained on the instrument to this level.

[3] Compounds are not 8080A target compounds but may be used in lieu of running a daily technical Chlordane standard for samples that are non-detect for technical Chlordane.

[4] Dibutylchlorendate may be includes as a surrogate at the same concentration as decachlorobiphenyl if needed for specific client requests.

Table B-4	
Column Degradation Evaluation Mix	
Component	Concentration
4,4'-DDT	25
Endrin	25
Tetrachloro-m-xylene (Surrogate)	20
Decachlorobiphenyl (Surrogate)	20

Table B-5			
LCS/Matrix Spike and Surrogate Spike levels ug/L or ug/kg			
	Aqueous	Soil	Waste
gamma BHC (Lindane)	0.20	6.67	200
Aldrin	0.20	6.67	200
Heptachlor	0.20	6.67	200
Dieldrin	0.50	16.7	500
Endrin	0.50	16.7	500
4,4'DDT	0.50	16.7	500
Tetrachloro-m-xylene (Surrogate)	0.20	6.67	200
Decachlorobiphenyl (Surrogate)	0.20	6.67	200

Table B-6			
LCS/Matrix Spike and Surrogate Spike levels for Aroclor analysis with Acid Cleanup ug/L or ug/kg			
	Aqueous	Soil	Waste
Aroclor 1016/1260	10	333	10,000
Tetrachloro-m-xylene (Surrogate)	0.20	6.67	200
Decachlorobiphenyl (Surrogate)	0.20	6.67	200

Table B-7		
LCS/Matrix Spike and Surrogate Spike levels for TCLP ug/L or ug/kg		
	Aqueous	Waste
Heptachlor	5	500
Lindane	5	500
Endrin	5	500
Methoxychlor	10	1000

Table B-8
Suggested Analytical Sequence

Initial Calibration

Injection #		
1	Solvent blank (optional)	
2	Breakdown Mix	
3-7	Individual mix AB	All levels
8	Aroclor 1016/1260	Level 1[1]
9	Aroclor 1232	Level 1[1]
10	Aroclor 1242	Level 1[1]
9	Aroclor 1248	Level 1[1]
10	Aroclor 1221/1254	Level 1[1]
12	Technical Chlordane	Level 1[1]
13	Toxaphene	Level 1[1]
15	Solvent blank	
16-25	Sample 1-10	
	Solvent blank (optional)	
26	Individual mix AB	Mid level (Continuing calibration)
etc	Sample 11-20	
	Solvent blank (optional)	
	Individual mix AB	Mid level (Continuing calibration)
	Sample 21-30	
	After 24 hours:	
	Breakdown mix	
	Individual mix AB	
	Any other single component analytes	
	Any multicomponent analytes for quantitation	

[1] A five point curve for any of the multicomponent analytes may be included, and is required for quantitation.

Daily Calibration

At least every 24 hours, counting from the start of the initial calibration, or from the start of the last daily calibration, the retention time windows must be updated using the Individual mix AB, and the breakdown mix must be run before the continuing calibration.

- All samples that have a positive hit for a multicomponent must be run after and within 24 hours of the standard that contains that multicomponent. The standard mix that contains this multicomponent must meet all the calibration criteria.

Table B-9		
Performance limits, four replicate initial demonstration of capability		
Compound	Initial demonstration, mean recovery limits	Initial demonstration, RSD limits
Aldrin	46-112	21
alpha-BHC	51-122	24
beta-BHC	61-120	32
delta-BHC	49.5-118.5	36
gamma-BHC	57-116	23
Chlordane	44.8-108.6	20
4,4'-DDD	52-126	28
4,4'-DDE	46-120	27.5
4,4'-DDT	54-137	36
Dieldrin	42.5-124.5	38
Endosulfan I	43-141	24.5
Endosulfan II	78-171	61
Endosulfan Sulfate	62-132	27
Endrin	49-126	37
Heptachlor	57-100	20
Heptachlor Epoxide	43.5-131.5	25.4
Toxaphene	44.4-111.2	20
Aroclor 1016 + 1260	62.5-110	20

1. SCOPE AND APPLICATION

This method is applicable to the gas chromatographic determination of Chlorinated phenoxy acid herbicides in extracts prepared by SOP CORP-OP-0001: The herbicides listed in Table C1 are routinely analyzed. Other chlorinated acids may be analyzed by this method if the quality control criteria in section 9 and the initial demonstration of method performance in section 13 are met.

2. SUMMARY OF METHOD

This method presents conditions for the analysis of prepared extracts of phenoxy acid herbicides by gas chromatography. The herbicides, as their methyl esters, are injected onto the column, separated, and detected by electron capture detectors. Quantitation is by the external standard method.

3. DEFINITIONS

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

4. INTERFERENCES

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

4.2. Chlorinated acids and phenols cause the most direct interference with this method.

4.3. Sulfur may interfere and may be removed by the procedure described in SOP#CORP-OP-0001.

5. SAFETY

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

6. EQUIPMENT AND SUPPLIES

6.1. Refer to Section 6 of the 8000A section of this SOP. A Ni_{63} electron capture detector is required.

6.2. Refer to Table C2 for analytical columns.

6.3. Microsyringes, various sizes, for standards preparation, sample injection, and extract dilution.

7. REAGENTS AND STANDARDS

7.1. Refer to section 7 of the 8000A section of this SOP for general information on reagents and standards.

7.2. Refer to Table C-3 and C-4 for details of calibration and other standards.

8. SAMPLE PREPARATION, PRESERVATION AND STORAGE

Refer to section 8 of the 8000A section of this SOP.

9. QUALITY CONTROL

9.1. Refer to Section 9 of the 8000A section of this SOP for quality control requirements, including the initial demonstration of capability, definition of a batch, surrogate limits, method blanks, laboratory control samples (LCS), and matrix spikes (MS).

9.2. Refer to Table C-6 for minimum performance criteria for the initial demonstration of capability.

9.3. Refer to Table C-5 for the components and levels of the LCS and MS mixes.

10. CALIBRATION AND STANDARDIZATION

10.1. Refer to Section 10 of the 8000A section of this SOP for general calibration requirements.

10.2. External standard calibration is used for this method.

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

10.4. Refer to Table C-2, for details of GC operating conditions.

11. PROCEDURE

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

11.2. Extraction

The extraction procedure is described in SOP #CORP-OP-0001.

11.3. Cleanup

The alkaline hydrolysis and subsequent extraction of the basic solution described in the extraction procedure provides an effective cleanup.

11.4. Analytical Sequence

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

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

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

11.4.3. After every 10 samples (including LCS, method blank, and MS samples), a continuing calibration is analyzed. The continuing calibration consists of mid level standards of all analytes of interest. It is not necessary but it is permissible to update retention time windows with continuing calibrations.

11.5. Gas Chromatography

Chromatographic conditions are listed in Table C-2.

12. DATA ANALYSIS AND CALCULATIONS

12.1. Refer to the 8000A section of this SOP for identification and quantitation of single component analytes.

12.2. The herbicides are analyzed as their methyl esters, but reported as the free acid. For this reason it is necessary to correct the results for the molecular weight of the ester versus the free acid. This is achieved through the concentrations of the calibration standards. For example the 20ug/L calibration standard for 2,4-D contains 21.3 ug/L of the methyl ester. No further correction is necessary.

13. METHOD PERFORMANCE

13.1. Multiple laboratory performance data has not been published by the EPA for this method. Table C-5 lists minimum performance standards required by Quanterra for the four replicate initial demonstration or capability (required by Section 13.2 of the 8000A part of this SOP) for this method. The spiking level should be equivalent to a mid level calibration.

14. POLLUTION PREVENTION

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

15. WASTE MANAGEMENT

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

16. REFERENCES

Method 8150B, SW-846, Update II, September 1994

17. MISCELLANEOUS

17.1. Modifications from Reference Method

Refer to the method 8000A section of this SOP for modifications from the reference method.

17.2. Modifications from Previous Revision

None

17.3. Tables

Table C-1				
Standard Analyte list				
Compound	CAS Number	Reporting Limit, ug/L or ug/kg		
		Aqueous	Soil	Waste
2,4-D	94-75-7	4	80	4000
2,4-DB	94-82-6	4	80	4000
2,4,5-TP (Silvex)	93-72-1	1	20	1000
2,4,5-T	93-76-5	1	20	1000
Dalapon	75-99-0	2	40	2000
Dicamba	1918-00-9	2	40	2000
Dichloroprop	120-36-5	4	80	4000
Dinoseb	88-85-7	0.6	12	600
MCPA	94-74-6	400	8000	400,000
MCPP	93-65-2	400	8000	400,000

The following concentration factors are assumed in calculating the Reporting Limits:

	Extraction Vol.	Final Vol.	Dilution Factor
Ground water	1000 mL	10 mL	20
Low-level Soil without GPC	50 g	10 mL	20
High-level soil / waste	1 g	10 mL	20

Specific reporting limits are highly matrix dependent. The reporting limits listed above are provided for guidance only and may not always be achievable.

Table C-2	
Instrumental Conditions	
PARAMETER	Recommended conditions
Injection port temp	220°C
Detector temp	325°C
Temperature program	80,2/30/170,0/1/180,1
Column 1	DB-5MS or RTX 5 30x0.32, 0.5um
Column 2	DB-1701 or Rtx-1701
Injection	2uL
Carrier gas	Helium / Hydrogen
Make up gas	Nitrogen

Recommended conditions should result in resolution of all analytes listed in Table C-1.

Table C-3						
Calibration Standards, free acid equivalent (methyl ester)[1] ng/mL						
Compound	Level 1	Level 2	Level 3	Level 4	Level 5	Level 6[2]
2,4-D	20 (21.3)	40 (42.5)	80 (85.1)	160 (170)	320 (340)	640 (681)
2,4-DB	20 (21.1)	40 (42.2)	80 (84.5)	160 (169)	320 (338)	640 (676)
2,4,5-TP [Silvex]	5 (5.25)	10 (10.5)	20 (21.1)	40 (42.1)	80 (84)	160 (168)
2,4,5-T	5 (5.27)	10 (10.5)	20 (21.1)	40 (42.2)	80 (84.4)	160 (169)
Dalapon	10 (11.0)	20 (22.0)	40 (43.9)	80 (87.8)	160 (176)	20 (351)
Dicamba	10 (10.6)	20 (21.3)	40 (42.5)	80 (85.1)	160 (170)	320 (340)
Dichloroprop	20 (21.2)	40 (42.4)	80 (84.8)	160 (170)	320 (339)	640 (678)
Dinoseb	3 (3.17)	6 (6.35)	12 (12.7)	24 (25.4)	48 (50.8)	96 (102)
MCPA	2,000 (2,140)	4,000 (4,280)	8,000 (8,560)	16,000 (17,100)	32,000 (34000)	64,000 (68,000)
MCPP	2,000 (2,120)	4,000 (4,260)	8,000 (8,520)	16,000 (17,000)	32,000 (34,100)	64,000 (68,200)
2,4-DCAA [Surrogate]	20	40	80	160	320	640

[1]The calibration standard solutions contain the herbicides as their methyl esters at the levels in parentheses above. These levels are equivalent to the free acid levels also listed above. The herbicides are reported as the free acids.

[2] Level 6 is optional

The reporting limits listed in Table C-1 will be achieved with these calibration levels and a 20 fold dilution of the sample extract. Lower reporting limits can be achieved with lesser dilutions of the sample extract.

Table C-4			
LCS/Matrix Spike and Surrogate Spike levels ug/L or ug/kg[1]			
	Aqueous	Soil	Waste
2,4-D	16	800	16000
Silvex	4	200	4000
2,4,5-T	4	200	4000
2,4-DB	16	800	16000
Dalapon	8	400	8000
DCAA (surrogate)	16	800	16000

[1] LCS, MS and SS spikes are as the free acid.

Table C-5		
Performance limits, four replicate initial demonstration of capability		
Compound	Initial demonstration, mean recovery limits	Initial demonstration, RSD limits
2,4-D	50-150	25
2,4-DB	50-150	25
2,4,5-TP (Silvex)	50-150	25
2,4,5-T	50-150	25
Dalapon	50-150	25
Dicamba	50-150	25
Dichloroprop	50-150	25
Dinoseb	25-120	40
MCPA	50-150	25
MCPP	50-150	25

INTERNATIONAL TECHNOLOGY CORPORATION

IT Analytical Services

Knoxville Laboratory Standard Operating Procedure

Title: **Analysis of Explosives Residues by HPLC/UV Detection (Method 8330)**

Prepared by: _____ Date: 4/13/94

Reviewed by: _____ Date: 4/14/94
Technical Specialist

_____ Date: 4/14/94
Quality Control Coordinator

_____ Date: 4/18/94
Director, Quality and Compliance, ITAS

_____ Date: 4-20-94
Director, Health and Safety, ITAS

Approved by _____ Date: 4/14/94
Laboratory Director

Controlled Copy No: UNCONTROLLED COPY

Key Words: **Explosives, HPLC, Analysis**

Revision #	0	1				
Date:	11/01/93	04/13/94				

HISTORY OF REVISION PAGE

REVISION NO	DATE	PAGES AFFECTED
01	04/13/94	ALL

REASON FOR REVISION

The purpose of this revision is to incorporate the requirements of the ITAS Division SOP No. ITAS-IT-QC-0004, "Use and Purpose of Laboratory Control Samples". In addition, the following changes were made: EQLs for HMX and RDX were lowered; requirements for method detection limit studies were added; instructions for sample preparation were corrected; and the definition of a QC batch was changed from a 30 day period to a 14 day period.

Title: Analysis of Explosives Residues by HPLC/UV Detection (Method 8330)

1. Purpose, Application, and Responsibility

1.1 This procedure provides instructions for the analysis of explosives residues (nitroaromatic and nitroamine compounds) by high performance liquid chromatography/UV detection.

1.2 This procedure is an implementation of EPA SW-846 Method 8330. It is applicable to the analysis of water samples that are amenable to direct aqueous injection and to soil/sediment samples.

1.3 Table 1 identifies compounds for which this method has been applied along with estimated quantitation limits for each matrix.

1.4 It is the responsibility of the group/team leader, or his/her designee, to delegate the performance of this procedure to analysts who are qualified to perform this procedure and operate associated equipment.

1.5 It is the responsibility of the qualified analyst to follow this procedure, to report deviations from this procedure, and to perform this procedure independently only when properly qualified.

2. References

2.1 "Test Methods for Evaluating Solid Waste; Physical/Chemical Methods", SW-846, Third Edition, USEPA, "Nitroaromatics and Nitramines by High Performance Liquid Chromatography (HPLC)", Method 8330, Revision 1, December 1990.

2.2 ANSI Z87.1 - American National Standard Institute, "Standard for Occupational and Educational Eye and Face Protection".

3. Associated Standard Operating Procedures

3.1 AD910312R1, "General Health and Safety Practices for Tasks Performed in the ITAS-Knoxville Laboratory".

3.2 KN-QC-0005, "Nonconformance and Corrective Action".

3.3 ITAS-IT-QC-0003, "ITAS Data and Report Review Responsibilities".

3.4 ITAS-IT-QC-0004, "Use and Purpose of Laboratory Control Samples".

4. Definitions

4.1 Extraction Batch: An extraction batch consists of a group of samples of the same matrix that is prepared at the same time using the same reagents. An extraction batch cannot exceed 20 samples, not including method blanks, spikes, and other laboratory QC samples.

4.2 QC Batch: A QC batch consists of one or more extraction batches, prepared by the same method within the same 14-day period. A QC batch cannot exceed 20 samples, not including method blanks, spikes, and other QC samples.

4.3 Method Blank: A method blank is a laboratory initiated sample consisting of blank water or blank solid matrix (e.g. sand) which is carried through all of the steps of an analysis. This sample serves to monitor the introduction of artifacts into the process.

4.4 Laboratory Control Sample (LCS): A LCS is a blank sample that has been spiked with known quantities of target analytes to monitor system performance. The LCS is analyzed exactly as other samples

carried through the method. The LCS is prepared using the same matrix medium as the method blank.

4.5 Matrix Spike (MS): A matrix spike is a sample aliquot that has been spiked with known quantities of target analytes before being subjected to the entire analytical procedure. The recovery of the analytes is determined after the analysis of both the sample and its spiked aliquot.

4.6 Matrix Spike Duplicate (MSD): A matrix spike duplicate is a matrix spike performed on a second aliquot of the same sample used for the MS. The MSD must be spiked at the same levels as the MS.

4.7 Surrogate: A surrogate is a compound having similar chemical/physical properties as some or all of the compounds of interest which is added to all samples, including QC samples, prior to sample preparation. The percent recovery of the surrogate is expected to give an indication of the extraction efficiency for the sample. The surrogate should be a compound not expected to be found in the sample.

4.8 MSDS: Material Safety Data Sheet

4.9 Method Detection Limit (MDL): The minimum concentration of a substance that can be measured and reported with 99% confidence that the analyte concentration is greater than zero. The MDL is determined by multiplying the appropriate one-sided 99% t-statistic by the standard deviation obtained from a minimum of three analyses of a blank matrix spike containing the analyte of interest at a concentration three to five times the estimated MDL.

4.10 Estimated Quantitation Limit (EQL): The lowest concentration that can be reliably achieved within specified limits of precision and accuracy during routine laboratory operating conditions. The EQL is generally 5 to 10 times the MDL. However, it may be nominally chosen within these guidelines to simplify data reporting. For many analytes, the EQL analyte concentration is selected as the lowest non-zero standard in the calibration curve. Sample EQLs are highly matrix-dependent. The EQLs are provided for guidance and may not always be achievable.

5. Procedure

5.1 Summary

5.1.1 This method provides high performance liquid chromatographic (HPLC) conditions for the detection of part per billion (ppb) levels of certain explosives residues in water, soil and sediment samples. Prior to use of this method, appropriate sample preparation techniques must be used.

5.1.2 High-Level Direct Injection Method: Aqueous samples of high concentration can be diluted 1/1 (v/v) with methanol or acetonitrile, filtered, separated on a C-18 reverse phase column, determined at 255 nm, and confirmed on a CN reverse phase column. Unless HMX is an important target analyte, acetonitrile is preferred.

5.1.3 Soil and sediment samples are extracted using acetonitrile in an ultrasonic bath, filtered and chromatographed as in Section 5.1.2

5.1.4 Qualifications

5.1.4.1 The analyst must have at a minimum a Bachelor's degree in chemistry or equivalent scientific discipline or four or more years work experience in an analytical laboratory in a related area.

5.1.5 Training

5.1.5.1 Before training begins, the instructor shall have the analyst trainee read this procedure.

5.1.5.2 Initial training on this procedure will consist, at a minimum, of an oral review followed by at least one manual demonstration. Training will be given by personnel already qualified to perform the procedure.

5.1.5.3 The analyst trainee will conduct several analyses of known or unknown qualification spikes and blank samples under direct oversight of the qualified personnel.

5.1.5.4 After correct results are achieved with direct oversight, the analyst will proceed to perform analyses of a minimum of two qualification samples without assistance of qualified personnel.

5.1.5.5 Qualification is achieved by performance of analyses of qualification samples to within the appropriate acceptance limits (method limits, certified limits, or laboratory control limits).

5.1.5.6 Requalification must be performed annually thereafter for this procedure.

5.2 Safety

5.2.1 All procedures shall be carried out in a manner that protects the health and safety of all IT associates.

5.2.2 Eye protection that satisfies ANSI Z87.1, laboratory coat, and appropriate gloves must be worn while samples, standards, solvents and reagents are being handled. Disposable gloves that have become contaminated will be removed and discarded; other gloves will be cleaned immediately.

5.2.3 The health and safety hazards of many of the chemicals used in this procedure have not been fully defined. Additional health and safety information can be obtained from the MSDS files maintained in the laboratory. The following sections list specific hazards that are known:

5.2.3.1 Chemicals known to be flammable include methanol and acetonitrile.

5.2.3.2 The method analytes are explosive and highly toxic compounds that must be handled with care. The analyst must be aware of the handling and clean-up techniques before using these materials. Refer to the appropriate MSDS.

5.2.4 Standard precautionary measures used for handling other organic compounds should be sufficient for the safe handling of the analytes targeted by Method 8330. The only extra caution that should be taken is when handling the analytical standard neat material for the explosives themselves and in rare cases where soil or waste samples are highly contaminated with the explosives. Follow the requirements for drying the neat materials at ambient temperatures (see section 5.6.6.1).

5.2.5 246TNT is the analyte most often detected in high concentrations in soil samples. Since soil samples as high as 2% 246TNT have been safely ground, the project manager or client must provide information as to whether the samples are suspected to contain explosives at a level greater than 2%. Samples containing higher concentrations should not be ground. Visual observation of soil samples taken from a site expected to contain explosives is also important. Lumps of material that have a chemical appearance should be suspect and not ground. Explosives are generally a very finely ground grayish-white material.

5.2.6 Sample preparation will be performed in a fume hood with the sash lowered or

with a safety shield in place for protection. The preparation of all standards and reagents will be conducted in a fume hood with the sash closed as far as the operations will permit.

5.2.7 Exposure to chemicals will be maintained as low as reasonably achievable, therefore, unless they are known to be non-hazardous, all samples will be opened, transferred and prepared in a fume hood, or under other means of mechanical ventilation. Solvent, and waste containers will be kept closed unless transfers are being made.

5.2.8 All work must be stopped in the event of a known, or potential compromise to the health or safety of any IT associate. The situation must be reported immediately to a laboratory supervisor.

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

5.2.10 Arrangements shall be made, to return to the client, samples suspected to contain high levels (> 2%) of explosives.

5.3 Interferences

5.3.1 Solvents, reagents, glassware and other sample processing hardware may yield discrete artifacts and/or elevated baselines, causing misinterpretation of the chromatograms. All of these materials must be demonstrated to be free from interferences, under the conditions of the analysis, by running method blanks.

5.3.2 24DNT and 26DNT elute at similar retention times (retention time difference of ~0.2 minutes). A large concentration of one isomer may mask the response of the other isomer. If these isomers cannot be resolved, an isomeric mixture should be reported.

5.3.3 Tetryl decomposes rapidly in methanol/water solutions, as well as with heat. All aqueous samples expected to contain Tetryl should be diluted with acetonitrile prior to filtration. SAMPLES EXPECTED TO CONTAIN TETRYL SHOULD NOT BE EXPOSED TO TEMPERATURES ABOVE ROOM TEMPERATURE.

5.3.4 Degradation products of Tetryl appear as a shoulder on the 246TNT peak. Peak heights rather than peak areas should be used when Tetryl is present in concentrations that are significant relative to the concentration of 246TNT.

5.4 Preservation and Holding Times

5.4.1 Samples must be collected and stored in glass containers.

5.4.2 Samples must be kept below 4° C and in the dark from the time of collection through analysis, except during drying.

5.4.3 Soil and sediment samples should be air dried at room temperature or colder after collection.

5.4.4 All water samples must be prepared within 7 days of collection and analyzed within 40 days after preparation. All soil and sediment samples must be extracted within 14 days of collection and analyzed within 40 days after extraction.

5.5 Required Equipment

5.5.1 HPLC system: Analytical system complete with an HPLC equipped with a pump capable of achieving 4000 psi, a 100-µL loop injector and a 255-nm UV detector, as well as all required

accessories, including analytical columns, column supplies, gases, glassware, and vials. A data system for measuring peak height and/or peak area is required.

5.5.1.2 C-18 Reverse phase HPLC column, 25-cm x 4.6-mm (5-µm), (Supelco LC-18 or equivalent).

5.5.1.3 CN Reverse phase HPLC column, 25-cm x 4.6-mm (5-µm), (Supelco LC-CN or equivalent).

5.5.2 Temperature controlled ultrasonic bath.

5.5.3 Vortex mixer.

5.5.4 Analytical Balance.

5.5.5 High pressure injection syringes - 10-µl, 25-µl, 50-µl, 100-µl, 500-µl, (Hamilton liquid syringe or equivalent).

5.5.6 Disposable cartridge filters - 0.45-µm Teflon filter.

5.5.7 Vacuum desiccator - Glass.

5.6. Reagents and Standards

5.6.1 Reagent water - All references to reagent water in this method refer to water in which an interference is not observed at the estimated quantitation limits of the compounds of interest. Reagent water can be generated by passing tap water through a carbon filter bed containing about 1 pound of activated carbon. A water purification system may be used to generate organic-free deionized water.

5.6.2 Acetonitrile - HPLC grade.

5.6.3 Methanol - HPLC grade.

5.6.4 Calcium chloride - Reagent grade. Prepare an aqueous solution of 5 g/L.

5.6.5 HPLC Mobile Phase - 50/50 methanol/ reagent water.

5.6.6 Stock Standard Solutions:

5.6.6.1 Stock standard solutions are prepared using Standard Analytical Reference Materials (SARMs) obtained from USATHAMA. HMX and RDX reference materials are shipped under water; dry ~ 11 mg of these standards to a constant weight in a vacuum desiccator (in the dark) prior to preparing stock standard solutions.

NOTE: HMX, RDX, Tetryl, and 246TNT are explosives and the neat material must be handled carefully. See SAFETY section for guidance. Drying at ambient temperature requires several days. DO NOT DRY AT HEATED TEMPERATURES!

5.6.6.2 Place about 10 mg (weighed to the nearest 0.1 mg) of a single analyte into a 10 mL volumetric flask and dilute to volume with acetonitrile. Invert the flask several times until dissolved. Store in a refrigerator at 4°C in the dark. Calculate the concentration of the stock solution from the actual weight used (nominal concentration = 1,000 mg/L). Stock solutions may be used for up to one year.

5.6.7 Intermediate Stock Standard Solutions:

5.6.7.1 Prepare an intermediate stock standard solution at 13 mg/L per analyte in acetonitrile.

NOTE: If the analytical column cannot achieve separation of 24DNT and 26DNT, it will be necessary to prepare a separate intermediate stock solution of 26DNT.

5.6.8 Calibration Standards:

5.6.8.1 Prepare calibration standards at a minimum of five concentration levels

through dilution of the intermediate standard solutions by 50% (v/v) with 5 g/L calcium chloride solution. These solutions must be refrigerated and stored in the dark, and prepared fresh on the day of calibration. Refer to Table 2 for suggested concentration levels.

5.6.9 Spiking Solutions:

5.6.9.1 Table 3 presents a list of suggested concentrations for spiking solutions for surrogates, LCSs, and MS/MSDs. The surrogate typically used is 4-nitrotoluene.

NOTE: If 4-nitrotoluene is expected to be present in the samples, another analyte must be chosen as a surrogate.

5.7 Calibration

5.7.1 Initial Calibration

5.7.1.1 Initial calibration of the instrument must be performed whenever the current column is replaced, instrument operation parameters are significantly changed, when major instrument maintenance is performed (e.g., UV lamp replacement), or when the current calibration no longer meets criteria.

5.7.1.2 Initial calibration consists of the analysis of triplicate injections of a minimum of five calibration levels for each analyte, including the surrogate. The lowest level should be at or below the estimated quantitation limit.

NOTE: Triplicate injection of each calibration standard is not necessary if single injection results in an acceptable calibration curve (see 5.7.1.3 for acceptance criteria).

5.7.1.3 Calibration is performed using an external standard technique. Calculate response factors (RF) for each analyte at each level, then calculate the %RSD of these RFs. For each analyte, if the %RSD is $\leq$ 25%, the average RF may be used for calculation of sample concentrations. Alternatively, the calibration curve may be determined using linear or non-linear regression with a non-zero intercept.

NOTE: Tetryl breaks down readily in the presence of water. Therefore, an acceptance criteria of $\leq$ 30% RSD is used for Tetryl.

5.7.1.4 Up to ten (10) samples, including QC samples, may be analyzed following the analysis of the initial calibration standards.

5.7.2 Daily Calibration

5.7.2.1 Analyte response factors must be verified at the beginning of each analytical sequence and every 24 hours thereafter through the analysis of triplicate injections of a mid-level calibration standard. The percent difference (%D) between the analyzed concentrations and the initial calibration must be $\leq$ 25% otherwise corrective action or new initial calibration must be performed.

NOTE: Tetryl breaks down readily in the presence of water. Therefore, an acceptance criteria of $\leq$ 30% D is used for Tetryl.

5.7.2.2 The retention times of analytes in the daily calibration are to be used as the center of the retention time window for qualitative identification. A retention time window of $\pm$ 2% shall be used.

5.7.3 Continuing Calibration

5.7.3.1 Analyte response factors must be verified after the analysis of no more than ten (10) samples (including QC samples) through the analysis of a mid-level calibration standard. The %D between the analyzed concentrations and the

ITAS - KNOXVILLE LABORATORY

PROCEDURE CHANGE APPROVAL

Number	Title	Revision
ITAS-KN-LC-0002	Analysis of Explosive Residues by HPLC/UV Detection	Ø 1 10/4/94

Sections Affected	Pages Affected:
Addition of section 5.8.2.2	Page 7

CHANGE: Section 5.8.2.2 Low level solid phase extraction

1. Measure 1000mL of sample and spike with 50uL of a 70ppm surrogate solution of 4-nitro toluene (50ul of 60ppm spike solution if necessary for LCS and matrix spikes.)

2. Adjust flow rate through a Porapak RDX column (500mg Vac 6cc) to 10mL/min with the vacuum pressure.

3. Precondition the column with 10mL of acetonitrile.

4. Condition the column with 30mL of DI water.

5. Slowly add 1000mL of sample to the solid phase column (do not prefilter the sample). Do not let the solid phase extraction tube go completely dry. If the tube clogs after sample loading, measure the amount of sample successfully filtered and use that volume for the extraction constant.

6. If the tube is not clogged, rinse the column with 10mL of DI water.

7. Shut off the vacuum and dry the vacuum manifold.

8. Put sample collection vials in rack and elute with 1mL of acetonitrile.

9. Elute with 4mL of additional acetonitrile.

10. Add 5mL of DI water to each sample (50/50 sample/water).

11. Inject 100uL of sample for analysis.

Use Additional Sheets If Necessary

REASON FOR CHANGE: To allow solid phase extraction for water samples to achieve low level detection limits.

Submitted By: _____ Date: 10/6/94

Approved By: Robyn M. Wagner Date: 10/6/94
Technical Director

_____ Date: 10/6/94
Quality Control Coordinator

ITAS-K-QA038.R1

initial calibration must be ≤ 25%. In addition, the analyte retention times must fall within the retention time windows established by the daily calibration standard. If any analyte fails either of these criteria, the analysis for that analyte is out-of-control. Any samples analyzed after an out-of-control continuing calibration standard must be re-analyzed under a valid calibration; however, samples analyzed prior to an out-of-control standard are considered acceptable.

NOTE: Tetryl breaks down readily in the presence of water. Therefore, an acceptance criteria of ≤ 30% D is used for Tetryl.

5.8 Analysis/Operation

5.8.1 Extraction of Soil/Sediment Samples

5.8.1.1 Sample homogenization: Dry soil/sediment samples in air at room temperature or colder, being careful not to expose the samples to direct sunlight. Grind and homogenize the dried sample thoroughly.

NOTE: DO NOT GRIND SUSPECTED HIGH LEVEL SAMPLES! CONTACT THE PROJECT MANAGER FOR INFORMATION RE. SUSPECTED EXPLOSIVES CONCENTRATIONS. ADDITIONALLY, VISUALLY INSPECT SAMPLES FOR LUMPS OF GRAYISH-WHITE MATERIAL. SEE SAFETY SECTION 5.2.5.

5.8.1.2 Place a 2.0 g subsample of each soil sample in a 12 mL glass vial. Add the appropriate amount of surrogate spike mix, and if the sample is an LCS or MS/MSD, add the appropriate amount of the spike mix (see Table 3). Allow the LCS, MS/MSD to dry for ~1 hour before adding acetonitrile. Add 10.0 mL of acetonitrile, cap with Teflon-lined cap, vortex swirl for one minute, and place in a cooled ultrasonic bath for 18 hours.

5.8.1.3 After sonication, allow sample to settle for 30 minutes. Remove 5.0 mL of supernatant, and combine with 5.0 mL of 5g/L calcium chloride solution in a 12 mL vial. Shake, and let stand for 15 minutes.

5.8.1.4 Place supernatant in a disposable syringe and filter through a 0.45-μm Teflon filter. Discard first 3 mL and retain remainder in a Teflon-capped vial for HPLC analysis.

5.8.2 Preparation of Aqueous Samples (High Level)

5.8.2.1 Place a 5 mL aliquot of each water sample in a 10 ml volumetric flask; add 2.5 ml of acetonitrile. Add the appropriate amount of surrogate spike mix, and if the sample is an LCS or MS/MSD, add the appropriate amount of the spike mix (see Table 3). Bring to volume with acetonitrile. Shake thoroughly, and filter through a 0.45-μm Teflon filter. Discard the first 3 mL of filtrate, and retain the remainder in a Teflon-capped vial for HPLC analysis.

5.8.3 HPLC Analysis

5.8.3.1 Chromatographic Conditions

Primary Column:	C-18 reverse phase HPLC column, 25-cm x 4.6-mm, 5-μm, (Supelco LC-18 or equivalent)
Guard Column:	LC-18 2-cm x 4.6-mm (Supelco)
Secondary Column:	CN reverse phase HPLC column, 25-cm x 4.6-mm, 5-μm, (Supelco LC-CN or equivalent)
Guard Column:	LC-CN 2-cm x 4.6-mm (Supelco)

Mobile Phase:	50/50 (v/v) methanol/organic-free reagent water
Flow Rate:	1 mL/min
Injection Volume:	100-µL
UV Detector:	255-nm

5.8.3.2 Perform the calibration procedures as described in section 5.7.

5.8.3.3 Using the same instrument conditions as were used for the calibration standards, analyze the samples with the appropriate daily and continuing calibration standards as described in section 5.7.

5.8.3.4 A qualitative identification of a target analyte is achieved if the retention time of a peak is within the retention time window established by the daily calibration standard.

5.8.3.5 Quantitate any positive hits by calculating the concentration of the analyte using either the average RF or the regression curve, as determined by the initial calibration.

5.8.3.6 All positive measurements observed on the C-18 column must be confirmed by injection onto the CN column. Correct soil samples for percent moisture if requested by the client.

5.9 Calculations

5.9.1 Aqueous Sample Concentration Using Response Factors

$$Conc(\mu g/L) = \overline{RF} \times PkHt \times DF$$

Where:

$\overline{RF}$ = Average response factor (Conc in ng/ml ÷ peak height or area)

PkHt = Peak height or area of an analyte from sample analysis

DF = Sample dilution factor

5.9.2 Solid Sample Concentration Using Response Factors

$$Conc\ (mg/kg) = \frac{\overline{RF} \times PkHt \times DF \times V_{ext}}{W_s}$$

Where:

V_{ext} = Volume of the total extract, in ml

W_s = Weight of sample extracted, in mg

5.9.3 Aqueous Sample Concentration Using Linear Regression

$$Conc(\mu g/L) = (Bx + A)\ (DF)$$

Where:

B = Slope

x = Peak height or area of an analyte from sample analysis

A = Intercept

5.9.4 Solid Sample Concentration Using Linear Regression

$$Conc\ (mg/Kg) = \frac{(Bx + A)\ (DF)\ (V_{ext})}{W_s}$$

5.10 Quality Control

5.10.1 Initial demonstration of precision and accuracy: Before processing samples, the laboratory must demonstrate capability to perform HPLC analyses for explosives that conform to the precision and accuracy requirements of SW-846

Method 8330. The procedure for performing this demonstration is given in Section 8.6 of Method 8000 (SW-846 Third Edition, Final Update I).

5.10.2 Method detection limit studies shall be performed for each matrix on an annual basis. The procedure is given in Chapter One, page 26 of SW-846 Third Edition, Final Update I.

5.10.3 Table 4 summarizes the frequency of analysis of QC samples.

5.10.4 Method blanks must be free of contamination and interference at least to the estimated quantitation limit (EQL). A contaminated method blank requires the re-extraction of all samples extracted with the affected blank. An exception to this requirement is allowed for samples associated with the blank which have no detected analytes above the EQL and/or have analytes with concentrations > five times the level in the blank.

5.10.5 LCSs shall be analyzed in accordance with ITAS Division SOP, ITAS-IT-QC-0004, "Use and Purpose of Laboratory Control Samples". If any LCS exceeds the laboratory acceptance limits determined by control charting, look for calculation errors; if none are found, reanalyze the LCS. If results are still noncompliant, reprepare and reanalyze the LCS and all samples associated with the LCS.

5.10.6 Surrogate recoveries shall be evaluated against the laboratory acceptance limits determined by control charting. If recoveries are not within limits, the following actions shall be taken:

5.10.6.1 Check to be sure there are no errors in calculations or surrogate solutions. Also, check instrument performance. If any of the above checks reveal a problem, recalculate the data and/or reanalyze the sample.

5.10.6.2 If no errors are found, evaluate the associated LCS and method blank results. If the blank is acceptable and the LCS results for spike and surrogate recoveries are within acceptance limits, review the sample values. A technical judgement based on the available information should include one of the following choices:

- Report as matrix effect. The sample(s) have a history or by analysis dictate a matrix effect (everything in control but the sample).
- Flag the results as estimated concentrations.
- Reanalyze the sample.

5.10.7 MS/MSD recoveries shall be evaluated against the laboratory acceptance limits determined by control charting. If MS/MSD recoveries exceed the acceptance limits, evaluate the associated LCS. If the LCS recoveries are within the laboratory acceptance limits, flag the MS/MSD results as due to matrix interferences. If the LCS is out of control, look for calculation errors; if none are found, reanalyze the LCS. If results are still noncompliant, reprepare and reanalyze the LCS, MS/MSD and all samples associated with the LCS.

5.10.8 Calibration curves must meet the criteria given in 5.7.1, otherwise all samples must be re-analyzed.

5.10.9 Daily calibration checks must meet the criteria given in 5.7.2, otherwise all samples must be re-analyzed.

5.10.10 Continuing calibration checks must meet the criteria of 5.7.3, otherwise all samples analyzed after the failed check

must be re-analyzed.

5.10.11 Data shall be reviewed in accordance with ITAS Division SOP, ITAS-IT-QC-0003, "ITAS Data and Report Review Responsibilities".

6. Nonconformance and Corrective Action

6.1 One time procedural variations are allowed only if deemed necessary in the professional judgement of supervision to accommodate variation in sample matrix, radioactivity, chemistry, sample size or other parameters. Any variation in procedure shall be completely documented using a Nonconformance Memo and approved by the Technical Director and QA/QC Coordinator. If contractually required, the client will be notified. The Nonconformance Memo will be filed in the project file.

6.2 Any unauthorized deviations from this procedure must be documented as a nonconformance, with a cause and corrective action described. An ITAS Nonconformance Memo shall be used for this documentation. The original Nonconformance Memo will be filed in the project file.

7. Records Management and Documentation

7.1 Documentation that an individual is authorized to perform this procedure independently will be retained in the individual's training file. Authorization will be designated by the supervisor's or instructor's signature and the date on the training documentation.

7.2 Taping the computer system for backup and storage must be done on a regular basis. Records are kept in sufficient quality and detail to facilitate the restoration of archived data to the data system for retrieval.

7.3 Each instrument is assigned an analysis logbook where the analyst records all analysis information.

7.3.1 At a minimum the logbook should include:

- Computer Data File Name
- Lab ID
- Standard ID Number
- Injection Date
- Analyst's Initials
- Comments, as appropriate

7.3.2 Each analysis logbook is checked and verified by the group supervisor routinely.

7.3.3 All analysis logbooks are controlled by the laboratory QA/QC Coordinator.

7.4 All sample preparation and analysis activities shall be documented using the appropriate worksheets and report forms. Results shall be reported in accordance with client requirements.

Table 1 Target List and Estimated Quantitation Limits[1]

Compound	Abbrev.	Water (µg/L)	Soil (mg/Kg)
Octahydro-1,3,5,7-tetranitro-1,3,4,7-tetrazocine	HMX	26	0.50
Hexahydro-1,3,5-trinitro-1,3,5-triazine	RDX	24	0.50
1,3,5-Trinitrobenzene	135TNB	24	0.25
1,3-Dinitrobenzene	13DNB	24	0.25
Methyl-2,4,6-trinitrophenylnitramine	Tetryl	26	0.65
Nitrobenzene	NB	28	0.26
2,4,6-Trinitrotoluene	246TNT	24	0.25
2,6-Dinitrotoluene	26DNT	24	0.26
2,4-Dinitrotoluene	24DNT	26	0.25
2-Nitrotoluene	2NT	26	0.25
4-Nitrotoluene[2]	4NT	24	0.25
3-Nitrotoluene	3NT	28	0.25

[1] Estimated quantitation limits are highly matrix dependent and may not always be achievable.
[2] 4-Nitrotoluene is generally used as a surrogate standard.

Table 2 Calibration Standards (suggested)[1]

Compound	Level 1 (µg/ml)[2]	Level 2 (µg/ml)[2]	Level 3 (µg/ml)[2]	Level 4 (µg/ml)[2]	Level 5 (µg/ml)[2]	Level 6 (µg/ml)[2]
HMX	0.013	0.026	0.065	0.13	0.195	0.26
RDX	0.012	0.024	0.060	0.12	0.18	0.24
135TNB	0.012	0.024	0.060	0.12	0.18	0.24
13DNB	0.012	0.024	0.060	0.12	0.18	0.24
Tetryl	0.013	0.026	0.065	0.13	0.195	0.26
NB	0.014	0.028	0.070	0.14	0.21	0.28
246TNT	0.012	0.024	0.060	0.12	0.18	0.24
26DNT	0.012	0.024	0.060	0.12	0.18	0.24
24DNT	0.013	0.026	0.065	0.13	0.195	0.26
2NT	0.013	0.026	0.065	0.13	0.195	0.26
4NT	0.012	0.024	0.060	0.12	0.18	0.24
3NT	0.014	0.028	0.070	0.14	0.21	0.28

[1] Concentrations are suggested levels only, and a minimum of 5 levels are required.
[2] 50/50 Acetonitrile/Reagent Water

Table 3 Spiking Mix Concentrations (suggested)[1]

Surrogate Mix[2]	
Compound	**Concentration (µg/ml)**
4NT	120

LCS, MS/MSD Spike Mix[3]	
Compound	**Concentration (µg/ml)**
HMX	13
RDX	12
135TNB	12
13DNB	12
Tetryl	13
NB	14
246TNT	12
26DNT	12
24DNT	13
2NT	13
4NT	12
3NT	14

[1] Concentrations are suggested only.
[2] Suggested spike level is 12 µl for soil and 10 µl for aqueous samples. Do not spike the LCS or MS/MSD with surrogate spike; the LCS, MS/MSD spike mix includes the surrogate.
[3] Suggested spike level is 120 µl for soil and 100 µl for aqueous samples.

Table 4 QC Requirements Summary

QC	Frequency	Criteria	Corrective Action
Method Blank	1 per extraction batch	Analytes < EQL	See Section 5.10.4
LCS	1 per extraction batch	Current laboratory control limits	See Section 5.10.5
MS/MSD	1 per QC batch	Current laboratory control limits	See Section 5.10.7
Surrogates	Every sample	Current laboratory control limits	See Section 5.10.6
Initial Calibration	Initially and as required	RSD ≤ 25% or regression analysis	See Section 5.7.1
Daily Calibration Check	Every 24-hours	≤ 25%D	See Section 5.7.2
Continuing Calibration Check	Every 10 samples	≤ 25%D	See Section 5.7.3

INTERNATIONAL TECHNOLOGY CORPORATION

IT Analytical Services

DIVISION

Standard Operating Procedure

TITLE: ACID DIGESTION OF SOILS AND SLUDGES (METHOD 3050A)

Prepared by: _____ **Date:** 1/7/94

Reviewed by: _____ Technical Specialist **Date:** 12/1/93

_____ Director QA/QC, ITAS **Date:** 12/2/93

_____ Director Health and Safety, ITAS **Date:** 12/22/93

Approved by: _____ Standardization Committee Member **Date:** 1/4/94

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Controlled Copy No: _____ 12/21/98

Key Words: Acid Digestion, Metals, Method 3050A

Revision #	0				
Date	09/16/93				

Regional Office
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IT Corporation is a wholly owned subsidiary of International Technology Corporation

Title: Acid Digestion of Soil and Sludge (Method 3050A)

1. Purpose and Application

1.1 Method 3050A is an acid digestion procedure used to prepare soil and sludge samples for analysis by flame (FLAA) or graphite furnace atomic absorption spectroscopy (GFAA) or by inductively coupled plasma emission spectroscopy (ICP). The samples may be analyzed by SW846 7000 series methods or EPA 200 series methods.

1.2 Sample prepared by this method may be analyzed by ICP for the metals listed below or by FLAA or GFAA as listed below.

FLAA/ICP		GFAA
Aluminum	Manganese	Arsenic
Barium	Molybdenum	Beryllium
Beryllium	Nickel	Cadmium
Cadmium	Osmium	Chromium
Calcium	Potassium	Cobalt
Chromium	Silver	Iron
Cobalt	Sodium	Lead
Copper	Thallium	Molybdenum
Iron	Vanadium	Selenium
Lead	Zinc	Thallium
Magnesium		Vanadium

1.3 The scope of this procedure has been extended to include the following metals.

ICP or FLAA		GFAA
Arsenic	Tin	Nickel
Boron	Titanium	Copper
Selenium	Silicon	Silver
Antimony	Strontium	Antimony

1.4 Method 3050A may also be applicable to the analysis of other metals in soil/sludge type samples provided a minimum of four LCS's are prepared, digested and analyzed showing no appreciable analyte loss.

1.5 Responsibility

Position	Responsibility
Analyst	Perform routine analyses associated with this procedure.
	Maintain equipment and inventory of needed supplies.
	Perform 100% data review.
Group Leader	Schedule samples for analysis.
	Perform 20% data review.

2. References

2.1 "Test Methods for Evaluating Solid Waste; Physical/Chemical Methods", SW-846, Third Edition, USEPA, Method 3050A, Revision 1, July 1992.

2.2 USEPA "Methods for Chemical Analysis of Water and Wastes", EPA-600/4-79-020, March 1983.

3. Associated SOPs

3.1 See Appendix A.

3.2 SOP No.:ITAS-IT-QC-0003, ITAS Data and Report Review Responsibilities.

4. Definitions

4.1 DI Water: Water which is the equivalent of ASTM Type II water.

4.2 LCS (Laboratory Control Sample): A reference material with known concentrations that is carried through the preparation and analysis procedure. The LCS is used to monitor loss/recovery values.

4.3 QC Batch: A group of samples, a maximum of 20, analyzed together with the same method, reagents and manipulation within a maximum of one week.

5. Procedure

5.1 Summary

5.1.1 A representative 1 to 1.2 g (wet weight) sample is digested in nitric acid and hydrogen peroxide. The digestate is then refluxed in nitric acid or hydrochloric acid depending on the instrumental method of analysis. The diluted samples have an approximate acid concentration of 5.0% (V/V).

5.1.2 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. The analyst must be given two blind performance samples to be analyzed or to be processed for analysis. Upon successful completion of these performance evaluation (PE) samples, these analyses must be documented as initial qualification. Requalification must be performed annually thereafter for this procedure. The group/team leader must document the training and PE performance and submit the results to the QA/QC Coordinator for inclusion in associate training files.

5.2 Safety

5.2.1 All applicable safety and compliance guidelines set forth by IT Corporation, and by federal, state and local regulations must be followed during performance of this procedure. All work must be stopped in the event of a known or potential compromise to the health or safety of any ITAS associate, and must be reported immediately to a laboratory supervisor.

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

5.2.3 A fume hood must be used during the procedure due to the corrosive nature of the samples and reagents.

5.2.4 The health and safety hazards of many of the chemicals used in this procedure have not been fully defined. Additional health and safety information can be obtained from the MSDS files maintained in the laboratory. The following specific hazards are known:

5.2.4.1 The following materials are known to be corrosive:

Nitric Acid
Hydrochloric Acid.

5.2.4.2 The following material is known to be an oxidizer:

Hydrogen Peroxide

5.2.5 Waste generated in the procedure will be segregated, and disposed of according to the facility hazardous waste procedures. The Health and Safety Coordinator shall be contacted if additional information is required.

5.3 Interferences

5.3.1 Sludge samples can contain diverse matrix types, each of which may present its own analytical challenge. Spiked samples and any relevant standard reference material shall be processed to aid in determining whether Method 3050 is applicable to a given waste.

5.4 Preservation and Holding Time

5.4.1 All samples must have been collected using a sampling plan that addresses the considerations discussed in Chapter Nine of "Test Methods for Evaluating Solid Waste", Third Edition.

5.4.2 All sample containers must be prewashed with detergents, acids, and DI water. Plastic and glass containers are both suitable.

5.4.3 Nonaqueous samples must be refrigerated upon receipt and analyzed within the holding time. Holding time is 180 days.

5.5 Required Equipment

5.5.1 Griffin Beaker: 250 mL, or equivalent.

5.5.2 75 mm Watch Glass: Ribbed or Plain.

5.5.3 Whatman No. 41 Filter Paper or Equivalent.

5.5.4 Hot Plate: Capable of maintaining 95°C.

5.5.5 Filter Funnel: Polyethylene or glass, another filtration system may be used, provided that it has been shown to not cause loss of the desired elements.

5.5.6 Graduated Cylinder: 100 mL Capacity

5.5.7 Laboratory Balance: Capable of reading to 0.01 g.

5.5.8 120 mL Plastic Sample Bottles

5.5.9 Repipettor(s): 500 or 1000 mL Capacity. For dispensing reagents.

5.5.10 Auto-Pipettors: 500 and 1000 uL. Eppendorf or equivalent.

5.6 Reagents / Standards

5.6.1 DI Water: ASTM Type II or equivalent.

5.6.2 Concentrated Nitric Acid, reagent grade or higher (HNO_3): Acid must be analyzed to determine levels of impurities or come with a certificate of analysis from the manufacturer. If method blank is < reporting limit, the acid may be used.

5.6.3 Nitric Acid (1:1): Prepared by adding concentrated nitric acid to DI water. Acid must be analyzed to determine levels of impurities or come with a certificate of analysis from the manufacturer. If method blank is < reporting limit, the acid may be used.

5.6.4 Concentrated Hydrochloric Acid, reagent grade or higher (HCl): Acid must be analyzed to determine levels of impurities or come with a certificate of analysis from the manufacturer. If method blank is < reporting limit, the acid may be used.

5.6.5 Hydrogen Peroxide (30 %): Oxidant must be analyzed to determine levels of impurities or come with a certificate of analysis from the manufacturer. If method blank is < reporting limit, the H_2O_2 may be used.

5.6.6 Spiking Solutions. See Appendix A on how to prepare, levels, and use.

5.7 Calibration

5.7.1 Laboratory Balance: Check accuracy of laboratory balance daily with balance check weight. If it is outside tolerance, have balance calibration checked before use.

5.7.2 Aqueous Pipettors shall be calibrated quarterly. If a pipettor fails calibration, it shall be taken out of service until calibration criteria can be met.

5.8 Analysis / Operation

5.8.1 Mix the sample thoroughly to achieve homogeneity.

5.8.2 Into a 250 mL beaker, weigh between 1.00 g and 1.20 g (To the nearest 0.01 g) of sample. Record weight in preparation logbook.

5.8.3 Add 10 mL 1:1 HNO_3, mix the slurry, and cover with a watch glass.

5.8.4 Heat sample at 95°C and reflux for 10 to 15 minutes without boiling.

5.8.5 Allow beaker to cool for 2 to 3 minutes.

5.8.6 Add 5 mL HNO_3 (conc) and replace watch glass.

5.8.7 Return to heat and reflux without boiling for 30 minutes.

5.8.8 Repeat steps 5.8.5, 5.8.6, and 5.8.7 to ensure complete oxidation.

5.8.9 Allow the sample to evaporate to approximately 15-10 mLs without boiling, while

maintaining a covering of solution over the bottom of the beaker.

5.8.10 Allow beaker to cool for 2 to 3 minutes.

5.8.11 Add 2 mL DI water, 3 mL 30 % H_2O_2 and cover the beaker with watch glass.

5.8.12 Return to heat until effervescence subsides. (Care must be taken to ensure that losses do not occur due to excessively vigorous effervescence.)

5.8.13 Allow beaker to cool for 2 to 3 minutes.

5.8.14 Add 3 mL of 30 % H_2O_2 and repeat step 5.8.12.

5.8.15 If the digestate is being prepared for analysis by ICP or FLAA, proceed to step 5.8.17.

5.8.16 If the digestate is being prepared for analysis by GFAA, proceed to step 5.8.18.

5.8.17 Final Reflux For Samples Prepared for ICP Analyses.

5.8.17.1 Allow beaker to cool for 2 to 3 minutes.

5.8.17.2 Add 5.0 mL HCl (conc) and 10 mL DI water and cover with watch glass.

5.8.17.3 Return to hot plate and heat for 15 minutes without boiling.

5.8.17.4 Allow beaker to cool.

5.8.17.5 Gravity filter, or vacuum filter, the digestate using Whatman No. 41 filter paper or equivalent and a filter funnel into a 100 mL graduated cylinder. The beaker and filter apparatus should be quantitatively rinsed with DI water to ensure a complete transfer.

5.8.17.6 Dilute to 100 mL final volume with DI water and mix.

5.8.17.7 Transfer digestate to a clean 120 mL prelabeled sample bottle.

5.8.17.8 The sample is now ready for ICP or FLAA analysis.

5.8.18 Final reflux for samples prepared for GFAA analyses.

5.8.18.1 Continue heating until volume is reduced to between 10 to 20 mL.

5.8.18.2 Allow beaker to cool.

5.8.18.3 Gravity filter, or vacuum filter, the digestate using Whatman No. 41 filter paper or equivalent and a filter funnel into a 100 mL graduated cylinder. The beaker and filter apparatus should be quantitatively rinsed with DI water to ensure a complete transfer.

5.8.18.4 Dilute to 100 mL final volume with DI water and mix.

5.8.18.5 Transfer digestate to a clean 120 mL prelabeled sample bottle.

5.8.18.6 The sample is now ready for analysis.

5.9 Calculations

5.9.1 The concentrations determined are to reported on the basis of the actual weight of the sample. If a dry weight analysis is desired, then the percent solids of the sample must be determined. Refer to Appendix A for the Percent Solids Determination SOP.

5.10 Quality Control

5.10.1 For each preparation batch of samples processed, a preparation blank (PBS), which is 1.00 g of DI water) must be carried through the entire sample preparation and analytical process. The blank will be useful in determining if samples are being contaminated.

5.10.2 A laboratory control sample (LCS) must be digested with each preparation batch. The LCS is from a reference material purchased from the EPA or other vendor with certified reference values, or a spiked 100 mL DI water sample.

5.10.3 Spiked samples must be employed to determine accuracy. A matrix spike (MS) and matrix spike duplicate (MSD) shall be digested with each QC batch of samples processed. The analyst must make sure that all metals to be

analyzed are included in the spikes. Refer to the following table for normal spiking procedures.

Method Of Analysis	Normal Spike Added
ICP	As per individual laboratory policies.
GFAA	1.0 ml XITA-1 or other appropriate standards

5.10.4 Refer to Appendix A for the SOP to prepare spiking solutions.

5.10.5 Additional spiking may be necessary to spike for analytes not present in the normal spiking solutions. It is the responsibility of the analyst to prepare spike solutions and to ensure that all requested analytes are spiked.

5.10.6 Additional client specific QC may be requested for a batch of samples. Any QC requested but not explained in sections 5.10.1 through 5.10.4 shall be performed in addition to the normal quality control.

6. Nonconformance and Corrective Action

6.1 Any deviation outside this SOP is considered a nonconformance and will be documented by use of a nonconformance memo.

6.2 Any problems that may arise during the procedure shall be reported to the supervisor for appropriate corrective action.

7. Records Management and Documentation

7.1 All digestion documentation will be kept in a numbered sample preparation logbook containing sample ID's, sample weight or volume, date and analyst. See Appendix B for individual laboratory logsheets.

7.2 All records will be managed and kept in accordance to SOPs listed in Appendix A.

APPENDIX A
Laboratory Specific SOP's

APPENDIX B
INORGANIC PREPARATION LOGSHEET

QUANTERRA STANDARD OPERATING PROCEDURE

TITLE: INDUCTIVELY COUPLED PLASMA-ATOMIC EMISSION SPECTROSCOPY, SPECTROMETRIC METHOD FOR TRACE ELEMENT ANALYSIS, METHOD 6010A AND METHOD 200.7

(SUPERSEDES: REVISION 0)

Prepared by: _____
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TABLE OF CONTENTS

1. SCOPE AND APPLICATION 3
2. SUMMARY OF METHOD 3
3. DEFINITIONS 4
4. INTERFERENCES 4
5. SAFETY 5
6. EQUIPMENT AND SUPPLIES 6
7. REAGENTS AND STANDARDS 7
8. SAMPLE COLLECTION, PRESERVATION AND STORAGE 7
9. QUALITY CONTROL 8
10. CALIBRATION AND STANDARDIZATION 14
11. PROCEDURE 15
12. DATA ANALYSIS AND CALCULATIONS 21
13. METHOD PERFORMANCE 23
14. POLLUTION PREVENTION 23
15. WASTE MANAGEMENT 23
16. REFERENCES 24
17. MISCELLANEOUS (TABLES, APPENDICES, ETC.) 24

LIST OF APPENDICES:

APPENDIX A - TABLES 30
APPENDIX B - QUANTERRA ICP DATA REVIEW CHECKLIST 40
APPENDIX C - TJA CALIBRATION RECOMMENDATION 42
APPENDIX D - MSA GUIDANCE 44
APPENDIX E - TROUBLESHOOTING GUIDE 46
APPENDIX F - CONTAMINATION CONTROL GUIDELINES 48
APPENDIX G - PREVENTATIVE MAINTENANCE 50

1. SCOPE AND APPLICATION

1.1. This procedure describes the analysis of certain metals by Inductively Coupled Plasma Atomic Emission Spectroscopy (ICP) using SW-846 protocol Method 6010A and Methods for Chemical Analysis of Waters and Wastes protocol, Method 200.7 as published in 40 CFR Part 136, Appendix C. Table I of Appendix A lists the elements approved for analysis by Methods 6010A and 200.7. Additional elements may be analyzed under Methods 6010A and 200.7 provided the method performance criteria presented in Section 13.0 are met.

1.2. ICP analysis provides for the determination of metal concentrations over several orders of magnitude. Detection limits, sensitivity and optimum concentration ranges of the metals will vary with the matrices and instrumentation used.

1.3. Method 6010A is applicable to the determination of dissolved, suspended, total recoverable and total elements in ground water, aqueous samples, soils, sludges, wastes, sediments, and TCLP, EP and other leachates/extracts. All matrices require digestion prior to analysis with the exception of analyses for dissolved metals in filtered and acidified aqueous samples. Although digestion is not specifically required by the method, some clients and regulators do require digestion of dissolved samples and this must be clarified before project initiation.

1.4. Method 200.7 is applicable to the determination of dissolved, suspended, total recoverable and total elements in surface water, domestic and industrial waste waters. All matrices require digestion prior to analysis with the exception of analyses for dissolved metals in filtered and acidified aqueous samples if the criteria in Section 11.1 are met.

1.5. This SOP is not applicable to the analysis of drinking water samples due to the wide array of state specific requirements which must be accommodated. Refer to facility specific SOPs for guidance on performing drinking water analyses.

2. SUMMARY OF METHOD

2.1. This method describes a technique for the determination of elements in solution. The basis of the method is the measurement of atomic emission by an optical spectroscopic technique. Samples are nebulized and the aerosol that is produced is transported to the plasma torch where excitation occurs. Characteristic atomic-line emission spectra are produced by a radio frequency inductively coupled plasma (ICP). The spectra are dispersed by a grating spectrometer and the intensities of the lines are monitored by photomultiplier tubes. The photocurrents from the photomultiplier tubes are processed and controlled by a computer system. A background correction technique is required to compensate for variable background contribution to the determination of

trace elements. Background must be measured adjacent to analyte lines during analysis. The position selected for the background intensity measurement, on either or both sides of the analytical line, will be determined by the complexity of the spectrum adjacent to the analyte line. The position used must be free of spectral interferences and reflect the same change in background intensity as occurs at the analyte wavelength measured. Background correction is not required in cases where a background corrective measurement would actually degrade the analytical result. The possibility of additional interferences should also be recognized and appropriate actions taken.

2.2. Consult the appropriate SOP's for details on sample preparation methods.

3. DEFINITIONS

3.1. Dissolved Metals: Those elements which pass through a 0.45 um membrane. (Sample is acidified after filtration).

3.2. Suspended Metals: Those elements which are retained by a 0.45 um membrane.

3.3. Total Metals: The concentration determined on an unfiltered sample following vigorous digestion.

3.4. Total Recoverable Metals: The concentration determined on an unfiltered sample following treatment with hot, dilute mineral acid.

4. INTERFERENCES

4.1. Spectral, physical and chemical interference effects may contribute to inaccuracies in the determinations of trace elements by ICP. Spectral interferences are caused by:

- Overlap of a spectral line from another element.
- Unresolved overlap of molecular band spectra.
- Background contribution from continuous or recombination phenomena.
- Stray light from the line emission of high concentration elements.

4.1.1. A background correction technique is required to compensate for variable background contribution to the determination of trace elements. Background correction is not required in cases where a background corrective measurement would actually degrade the analytical result.

4.1.2. Interelement correction factors (IEC's) are necessary to compensate for spectral overlap. Interelement interferences occur when elements in the sample

emit radiation at wavelengths so close to that of the analyte that they contribute significant intensity to the analyte channel. If such conditions exist, the intensity contributed by the matrix elements will cause an excessively high (or sometimes low) concentration to be reported for the analyte. Interelement corrections (IEC's) must be applied to the analyte to remove the effects of these unwanted emissions. To calculate an IEC, divide the observed concentration of the analyte by the observed concentration of the "interfering element."

4.1.3. Physical interferences are generally considered to be effects associated with sample transport, nebulization and conversion within the plasma. These interferences may result in differences between instrument responses for the sample and the calibration standards. Physical interferences may occur in the transfer of solution to the nebulizer (e.g., viscosity effects), at the point of aerosol formation and transport to the plasma (e.g., surface tension) or during excitation and ionization processes within the plasma itself. Changes in viscosity and surface tension can cause significant inaccuracies, especially in samples containing high dissolved solids or high acid concentrations. If physical interferences are present, dilution of the sample, use of a peristaltic pump, use of an internal standard and/or use of a high solids nebulizer can reduce the effect.

4.1.4. Chemical interferences are characterized by molecular compound formation, ionization effects and solute vaporization effects. Normally these effects are not significant with the ICP technique but if observed can be minimized by buffering the sample, matrix matching or standard addition procedures.

5. SAFETY

5.1. Procedures shall be carried out in a manner that protects the health and safety of all Quanterra associates.

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

5.3. The health and safety hazards of many of the chemicals used in this procedure have not been fully defined. Additional health and safety information can be obtained from the Material Safety Data Sheets (MSDS) maintained in the laboratory.

5.3.1. The following materials are known to be **corrosive**:

sulfuric acid, hydrochloric acid, nitric acid and hydrofluoric acid. (NOTE: sulfuric and hydrofluoric acids are used in cleaning the ICP torch and hydrofluoric acid is also commonly used in air toxics preparations.)

5.3.2. The following materials are known to be **oxidizing agents**:

nitric acid and hydrogen peroxide.

5.3.3. The plasma emits strong UV light and is harmful to vision. AVOID looking directly at the plasma.

5.3.4. The RF generator produces strong radio frequency waves, most of which are unshielded. People with pacemakers should not go near the instrument while in operation.

5.4. Exposure to chemicals must be maintained **as low as reasonably achievable**, therefore, unless they are known to be non-hazardous, all samples should be opened, transferred and prepared in a fume hood, or under other means of mechanical ventilation. Metals digestates can be processed outside of a fume hood. Solvent and waste containers will be kept closed unless transfers are being made.

5.5. The preparation of standards and reagents will be conducted in a fume hood with the sash closed as far as the operation will permit.

5.6. All work must be stopped in the event of a known or potential compromise to the health and safety of a Quanterra associate. The situation must be reported **immediately** to a laboratory supervisor.

5.7. The use of hydrofluoric acid requires special safety precautions. Consult the facility EH&S Manager and laboratory supervisor for guidance.

6. EQUIPMENT AND SUPPLIES

6.1. Inductively Coupled Plasma Atomic Emission Spectrometer equipped with autosampler and background correction.

6.2. Radio Frequency Generator.

6.3. Nitrogen or argon gas supply, welding grade or equivalent.

6.4. Coolflow or appropriate water cooling device.

6.5. Peristaltic Pump.

6.6. Calibrated automatic pipettes or Class A glass volumetric pipettes.

6.7. Class A volumetric flasks.

6.8. Autosampler tubes.

7. REAGENTS AND STANDARDS

7.1. Intermediate standards are purchased as custom Quanterra multielement mixes or as single element solutions. All standards must be stored in FEP fluorocarbon or previously unused polyethylene or polypropylene bottles. Intermediate standard solutions must be replaced prior to the expiration date provided by the manufacturer. If no expiration date is provided, the intermediate solutions may be used for up to one year and must be replaced sooner if verification from an independent source indicates a problem.

7.2. Working calibration and calibration verification solutions may be used for up to 3 months and must be replaced sooner if verification from an independent source indicates a problem. Standards should be prepared in a matrix of 5% hydrochloric and 5% nitric acids. An exception to this is in the event the Trace ICP is utilized without the internal standard. In this case, the standard acid matrix must be matched to the final preparation matrix as listed in Section 11.10.

7.3. Refer to Tables III, IV, IVA, V and VI (Appendix A) for details regarding the working standard concentrations for calibration, calibration verification, interference correction and spiking solutions.

7.4. Concentrated nitric acid (HNO_3), trace metal grade or better.

7.5. Concentrated hydrochloric acid (HCl), trace metal grade or better.

7.6. Reagent water must be produced by a Millipore DI system or equivalent. Reagent water must be free of the analytes of interest as demonstrated through the analysis of method blanks.

8. SAMPLE COLLECTION, PRESERVATION AND STORAGE

8.1. Sample holding times for metals are six months from time of collection to the time of analysis.

8.2. Aqueous samples are preserved with nitric acid to a pH of <2 and may be stored in either plastic or glass. If boron or silica are to be determined, plastic containers are

preferred. Refrigeration is not required. Preservation must be verified prior to analysis.

8.3. Soil samples do not require preservation but must be stored at 4°C ± 2° until the time of analysis.

9. QUALITY CONTROL

Table VII (Appendix A) provides a summary of quality control requirements including type, frequency, acceptance criteria and corrective action.

9.1. Initial Demonstration of Capability

Prior to analysis of any analyte using either Method 200.7 or Method 6010A, the following requirements must be met.

9.1.1. Instrument Detection Limit (IDL) - The IDL for each analyte must be determined for each analyte wavelength used on each instrument. The IDL must be determined annually. If the instrument is adjusted in any way that may affect the IDL, the IDL for that instrument must be redetermined. The IDL shall be determined by multiplying by 3, the standard deviation obtained from the analysis of a standard solution (each analyte in reagent water) at a concentration 3x - 5x the previously determined IDL, with seven consecutive measurements. Each measurement must be performed as though it were a separate analytical sample (i.e., each measurement must be followed by a rinse and/or any other procedure performed between the analysis of separate samples). The result of the IDL determination must be below the Quanterra reporting limit. The CLP IDL procedure can be used for this method.

9.1.2. Method Detection Limit (MDL) - An MDL must be determined for each analyte/matrix prior to the analysis of any samples. The MDL is determined using seven replicates of reagent water, spiked with all the analytes of interest, that have been carried through the entire analytical procedure. MDL's must be redetermined on an annual basis in accordance with 40 CFR Part 136 Appendix B requirements as detailed in Quanterra QA Policy QA-005. The spike level must be between the calculated MDL and 10X the MDL to be valid. The result of the MDL determination must be below the Quanterra reporting limit.

9.1.3. Linear Range Verification (LR) - The linear range must be determined on an annual basis for each analyte wavelength used on each instrument. The standards used to define the linear range limit must be analyzed during a routine analytical run. The determined concentration of the linear range standard must be within 5% of the true value. The linear range is the

concentration above which results cannot be reported without dilution of the sample. If the instrument is adjusted in any way that may affect the LR's, the LR's must be redetermined.

9.1.4. Background Correction Points - To determine the appropriate location for off-line background correction when establishing methods, the user must scan the area on either side adjacent to the wavelength and record the apparent emission intensity from all other method analytes. This spectral information must be documented and kept on file. Background correction points must be set prior to determining IEC's. Refer to the facility specific instrument operation SOP and ICP instrument manual for specific procedures to be used in setting background correction points.

9.1.5. Interelement Corrections (IEC) - ICP interelement correction factors must be determined prior to the analysis of samples and annually thereafter. If the instrument is adjusted in any way that may affect the IEC's, the IEC's must be redetermined. When initially determining IEC's for an instrument, wavelength scans must be performed to ensure that solutions in use are free from contaminants. If an IEC varies significantly from the previously determined IEC then the possibility of contamination should be investigated. The purity of the IEC check solution can be verified by using a standard from a second source or an alternate method (i.e., GFAA or ICP-MS). Published wavelength tables (e.g. MIT tables, Inductively Coupled Plasma-Atomic Spectroscopy: Prominent Lines) can also be consulted to evaluate the validity of the IEC's. Refer to the facility specific instrument operation SOP and instrument manufacturer's recommendations for specific procedures to be used in setting IECs. An IEC must be established to compensate for any interelement interference which results in a false analyte signal greater than ± the RL as defined in Tables I, IA or II.

Note: Trace ICP IEC's are more sensitive to small changes in the plasma and instrument setup conditions. Adjustments in the IEC's will be required on a more frequent basis for the Trace as reflected by the ICSA response.

9.1.6. Rinse Time Determination - Rinse times must be determined annually. To determine the appropriate rinse time for a particular ICP system, the linear range verification standard (see 9.1.3) should be aspirated as a regular sample followed by the analysis of a series of rinse blanks. The length of time required to reduce the analyte signals to < RL will define the rinse time for a particular ICP system. For some analytes it may be impractical to set the rinse time based on the linear range standard result (i.e., analyte not typically detected in environmental samples at that level and an excessive rinse time would be required at the linear range level). Rinse time studies can be conducted at additional concentration levels. These additional studies must be documented

and kept on file if a concentration other than the linear range level is used to set the rinse time. The concentration levels used to establish the rinse time must be taken into consideration when reviewing the data.

9.2. Method Blank (MB) - One method blank must be processed with each preparation batch. The method blank consists of reagent water containing all reagents specific to the method that is carried through the entire analytical procedure, including preparation and analysis. The method blank is used to identify any system and process interferences or contamination of the analytical system that may lead to the reporting of elevated analyte concentrations or false positive data. The method blank should not contain any analyte of interest at or above the reporting limit (exception: common laboratory contaminants, see below) or at or above 5% of the measured concentration of that analyte in associated samples, whichever is higher (sample result must be a minimum of 20x higher than the blank contamination level).

- If the analyte is a common laboratory contaminant (copper, iron, lead (Trace only) or zinc) the data may be reported with qualifiers if the concentration of the analyte in the method blank is less than two times the RL. **Such action must be taken in consultation with the client and must be addressed in the project narrative.**

- Repreparation and reanalysis of all samples associated with an unacceptable method blank is required when reportable concentrations are determined in the samples (see exception noted above).

- If there is no analyte greater than the RL in the samples associated with an unacceptable method blank, the data may be reported with qualifiers. **Such action must be taken in consultation with the client and must be addressed in the project narrative.**

- If the above criteria are not met and reanalysis is not possible, then the sample data must be qualified. **This anomaly must be addressed in the project narrative and the client must be notified.**

- For dissolved metals samples which have not been digested, a CCB result is reported as the method blank. The CCB run immediately prior to the start of the dissolved sample analyses must be used for this purpose. No more than 20 samples can be associated with one CCB.

9.3. Laboratory Control Sample (LCS) - One aqueous LCS must be processed with each preparation batch. The LCS must contain all analytes of interest and must be carried through the entire analytical procedure. Aqueous LCS spike levels are provided in Table III (Appendix A). The LCS is used to monitor the accuracy of the analytical process. On-going monitoring of the LCS results provides evidence that the laboratory is performing the method within acceptable accuracy and precision guidelines.

- If any analyte is outside established control limits the system is out of control and corrective action must occur. Until in-house control limits are established, a control limit of 80 - 120% recovery must be applied.

- In the event that an MS/MSD analysis is not possible a Laboratory Control Sample Duplicate (LCSD) must be analyzed. The RPD of the LCS and LCSD must be compared to the matrix spike RPD limits.

- In the instance where the LCS recovery is greater than 120% and the sample results are < RL, the data may be reported with qualifiers. **Such action must be taken in consultation with the client and must be addressed in the case narrative.**

- Corrective action will be repreparation and reanalysis of the batch unless the client agrees that other corrective action is acceptable.

- For dissolved metals samples which have not been digested, a CCV result is reported as the LCS. The CCV run immediately prior to the start of the dissolved sample analyses must be used for this purpose. No more than 20 samples can be associated with one CCV.

9.4. Matrix Spike/Matrix Spike Duplicate (MS/MSD) - One MS/MSD pair must be processed for each preparation batch. A matrix spike (MS) is a field sample to which known concentrations of target analytes have been added. A matrix spike duplicate (MSD) is a second aliquot of the same sample (spiked identically as the MS) prepared and analyzed along with the sample and matrix spike. Some client specific data quality objectives (DQO's) may require the use of sample duplicates in place of or in addition to MS/MSD's. The MS/MSD results are used to determine the effect of a matrix on the precision and accuracy of the analytical process. Due to the potential variability of the matrix of each sample, these results may have immediate bearing only on the specific sample spiked. Samples identified as field blanks cannot be used for MS/MSD analysis. Spiking levels are provided in Tables III and VI (Appendix A).

- If any analyte recovery or RPD falls outside the acceptance range, the recovery of that analyte must be in control for the LCS. Control limits of 80 - 120 % recovery and 20% RPD must be applied to the MS/MSD. If the LCS recovery is within limits, then the laboratory operation is in control and the results may be accepted. If the recovery of the LCS is outside limits corrective action must be taken. Corrective action will include repreparation and reanalysis of the batch. MS/MSD results which fall outside the control limits must be addressed in the narrative.

- If the native analyte concentration in the MS/MSD exceeds 4x the spike level for that analyte, the recovery data are reported as NC (i.e., not calculated). If the reporting software does not have the ability to report NC then the actual recovery must be reported and narrated as follows: "Results outside of limits do not

necessarily reflect poor method performance in the matrix due to high analyte concentrations in the sample relative to the spike level."

- If an MS/MSD is not possible due to limited sample volume then a laboratory control sample duplicate (LCSD) should be analyzed. The RPD of the LCS and LCSD must be compared to the matrix spike RPD limits.

- For dissolved metals samples which have not been digested, a MS/MSD must be performed per batch of up to 20 samples by spiking two aliquots of the sample at the levels specified in Table III (Appendix A).

9.5. Serial Dilution (L) - Serial dilution analysis is performed to determine whether significant physical or chemical interferences exist due to the sample matrix. One sample per preparation batch must be processed as a serial dilution sample. The serial dilution is performed by running a sample at a 4x dilution. Samples identified as field blanks cannot be used for serial dilution analyses. The results of the diluted sample, after correction for dilution, should agree within 10% of the original sample determination when the original sample concentration is greater than 40x the IDL. If the results are not within 10%, narrate the possibility of chemical or physical interference.

9.6. High Calibration Standard (HCAL) - At the beginning of the run, prior to the analysis of samples, the high standard must be rerun and recovered within 95 - 105%. If any analyte of interest falls outside the acceptance criteria corrective action must occur. The analysis should be terminated, the problem resolved and the instrument recalibrated. (See Section 11.11 or 11.12 for required run sequence.)

9.7. Initial Calibration Verification (ICV/ICB) - Calibration accuracy is verified by analyzing a second source standard (ICV). For analyses conducted under Method 200.7, the ICV result must fall within 5% of the true value for that solution. For Method 6010A, the ICV must fall within 10% of the true value for that solution. An ICB is analyzed immediately following the ICV to monitor low level accuracy and system cleanliness. The ICB result must fall within +/- the reporting limit (RL) from zero. If either the ICV or ICB fail to meet criteria, the analysis should be terminated, the problem corrected, the instrument recalibrated and the calibration reverified. (See Section 11.11 or 11.12 for required run sequence).

9.8. Continuing Calibration Verification (CCV/CCB) - Calibration accuracy is monitored throughout the analytical run through the analysis of a known standard after every 10 samples. The CCV must be a mid-range standard made from a 2x dilution of the calibration standard. For analyses that require Method 200.7, the CCV result must fall within 5% of the true value for that solution. For Method 6010A, the CCV must fall within 10% of the true value for that solution. A CCB is analyzed immediately following each CCV. (See Section 11.11 or 11.12 for required run sequence.) The CCB result must fall within +/- RL from zero. Sample results may only be reported

when bracketed by valid ICV/CCV and ICB/CCB pairs. If a mid-run CCV or CCB fails, the analysis for the affected element must be terminated, the problem corrected, the instrument recalibrated, the calibration verified and the affected samples reanalyzed. (Refer to Section 11.13 for an illustration of the appropriate rerun sequence).

9.9. Interference Check Analysis (ICSA/ICSAB) - The validity of the interelement correction factors is demonstrated through the successful analysis of interference check solutions. The ICSA contains only interfering elements, the ICSAB contains analytes and interferents. Refer to Table V (Appendix A) for the details of ICSA and ICSAB composition. Custom Quanterra multielement ICS solutions must be used. All analytes must be spiked into the ICSAB solution, therefore, if a non-routine analyte is required then it must be manually spiked into the ICSAB using a certified ultra high purity single element solution or custom lab-specific mix. Elements known to be interferents on a required analyte must be included in the ICP run when that analyte is determined. Aluminum, iron, calcium and magnesium must always be included in all ICP runs.

9.9.1. The ICSA and ICSAB solutions must be run at the beginning and end of the run or every 8 hours, whichever is more frequent. (See Section 11.11 or 11.12 for required run sequence.)

9.9.2. The ICSAB results for the interferents must fall within 80 - 120% of the true value. If any ICSAB interferent result fails criteria, the analysis should be terminated, the problem corrected, the instrument recalibrated and the samples rerun.

9.9.3. The ICSAB analytes must be recovered within 80 - 120% of the true value. If the ICSAB analytes do not meet criteria the analysis must be terminated, the problem corrected, the instrument recalibrated and the samples rerun for the affected analytes.

9.9.4. ICSA results for the non-interfering elements with reporting limits ≤ 10 ug/L must fall within the Quanterra guidelines of +/- 2x RL from zero. If the ICSA results for the non-interfering elements do not fall within +/- 2x RL from zero the field sample data must be evaluated as follows:

- If the non-interfering element concentration in the ICSA is the result of contamination versus a spectral interference, and this reason is documented, the field sample data can be accepted.

- If the affected element was not required then the sample data can be accepted.

- If the interfering elements are not present in the field sample at a concentration which would result in a false positive or negative result greater than +/- 2x RL from zero then the field sample data can be accepted.

- If the interfering element is present in the field sample at a level which would result in a false analyte signal greater than ± 2x RL from zero, the data can be accepted only if the concentration of the affected analyte in the field sample is more than 10X the analyte signal in the ICSA.

- If the data does not meet the above conditions then the IEC's must be re-evaluated and corrected if necessary and the affected samples reanalyzed or the sample results manually corrected through application of the new IEC to the raw results. If the results are recalculated manually the calculations must be clearly documented on the raw data.

9.10. CRI - To verify linearity near the RL for ICP analysis, a CRI standard is run at the beginning of each sample analysis run following the ICB. The CRI standard must contain all analytes at two times the RL. The CRI results must fall within 50% of the true value or the run must be terminated, the problem corrected and the instrument recalibrated.

9.11. Method of Standard Addition (MSA) -This technique involves adding known amounts of standard to one or more aliquots of the processed sample solution. This technique compensates for a sample interferent that may enhance or depress the analyte signal, thus producing a different slope from that of the calibration standards. It will not correct for additive interferences which cause a baseline shift. Refer to Section 11.17 for additional information on when MSA is required as well as Appendix D for specific MSA requirements.

9.12. Quality Assurance Summaries - Certain clients may require specific project or program QC which may supersede the SOP requirements. Quality Assurance Summaries (QAS) should be developed to address these requirements.

10. CALIBRATION AND STANDARDIZATION

10.1. Set up the instrument with the operating parameters recommended by the manufacturer. Allow the instrument to become thermally stable before beginning calibration (approximately 30 minutes of warm-up is required).

10.2. Calibrate the instrument according to the instrument manufacturer's recommendations. Refer to the facility specific instrument SOP and ICP instrument manual for detailed set up and operation protocols.

10.3. Calibration must be performed daily and each time the instrument is set up. Instrument runs may be continued over periods exceeding 24 hours as long as all calibration verification and interference check QC criteria are met. The instrument standardization date and time must be included in the raw data.

10.4. Refer to Section 9.0 for calibration verification procedures, acceptance criteria and corresponding corrective actions.

11. PROCEDURE

11.1. For 200.7 analyses, dissolved samples must be digested unless it can be documented that the sample meets all of the following criteria:

A. COD is < 20 ppm.
B. Visibly transparent with a turbidity measurement of 1 NTU or less.
C. Colorless with no perceptible order.
D. Is of one liquid phase and free of particulate or suspended matter following acidification.

11.2. A minimum of two exposures for each standard, field sample and QC sample is required. The average of the exposures is reported. For Trace ICP analyses, the results of the sum channel must be used for reporting.

11.3. Prior to calibration and between each sample/standard the system is rinsed with the calibration blank solution. The minimum rinse time between analytical samples is 60 seconds unless following the protocol outlined in 9.1.6 it can be demonstrated that a shorter rinse time may be used. Triton-X can be added to the rinse solution to facilitate the rinse process.

11.4. The use of an autosampler for all runs is strongly recommended.

11.5. The use of automated QC checks through the instrument software is highly recommended for all calibration verification samples (ICV,CCV), blanks (ICB,CCB,PB), interference checks (ICSA,ICSAB) and field samples (linear range) to improve the data review process.

11.6. To facilitate the early identification of QC failures and samples requiring rerun it is strongly recommended that sample data be reviewed periodically throughout the run.

11.7. To facilitate the data review and reporting processes it is strongly recommended that all necessary dilutions and post digestion spikes be performed before closing out the instrument run.

11.8. For unattended overnight auto-runs it is strongly recommended that the frequency of ICSA/ICSAB analysis be increased to every 4 hours.

11.9. The use of an internal standard is recommended on the non-Trace ICP's.

11.10. The use of an internal standard is required on the Trace ICP unless the calibration and QC standards are matrix matched to each digestion procedure used as follows:

Preparation Method	% HNO_3	% HCl
CLP Aqueous	1	5
CLP Soil	5	2.5
SW846 3050	5	5
SW846 3005	2	5
SW846 3010	3	5

The following procedural guidelines must be followed when using an internal standard:

11.10.1. Recommended internal standards are yttrium or scandium. (Note: Any element can be used that is not typically found in environmental samples at a high rate of occurrence.)

11.10.2. The internal standard (IS) must be added to every sample and standard at the same concentration. It is recommended that the IS be added to each analytical sample automatically through use of a third pump channel and mixing coil. Internal standards should be added to blanks, samples and standards in a like manner, so that dilution effects resulting from the addition may be disregarded.

11.10.3. The concentration of the internal standard should be sufficiently high to obtain good precision in the measurement of the IS analyte used for data correction and to minimize the possibility of correction errors if the IS analyte is naturally present in the sample.

11.10.4. The internal standard raw intensity counts must be printed on the raw data.

11.10.5. The analyst must monitor the response of the internal standard throughout the sample analysis run. This information is used to detect potential

problems and identify possible background contributions from the sample (i.e., natural occurrence of IS analyte).

11.10.5.1. If the internal standard counts fall within 30% of the counts observed in the ICB then the data is acceptable.

11.10.5.2. If the internal standard counts in the field samples are more than 30% higher than the expected level, the field samples must then be screened without the addition of the internal standard.

11.10.5.3. If the internal standard element is not identified in the unspiked field sample at a level exceeding 10% of the level spiked, the data may be accepted.

11.10.5.4. If the IS analyte is detected in the unspiked field sample at a concentration greater than 10% of the spiked level then either:

- A different internal standard must be used.
- The IS concentration must be raised.
- The sample must be diluted and rerun.
- The analysis must be run without an internal standard (matrix matching must be substituted.)

11.11. The following analytical sequence must be used for Methods 6010A and 200.7:

Instrument Calibration
ICV
ICB
CRI
HCAL
ICSA
ICSAB
6 samples
CCV
CCB
10 samples
CCV
CCB
Repeat sequence of up to 10 samples between CCV/CCB pairs as required to complete run
CCV
CCB

ICSA
ICSAB
CCV
CCB

Refer to Quality Control Section 9.0 and Table VII (Appendix A) for Method 6010A and 200.7 quality control criteria.

11.12. The following run sequence is consistent with 200.7, 6010A and CLP requirements and may be used as an alternate to the sequence specified in 11.11 if multiple methods must be accommodated in the same analytical run:

Instrument Calibration
ICV
ICB
CRI
HCAL
ICSA
ICSAB
CCV
CCB
10 samples
CCV
CCB
10 samples
CCV
CCB
Repeat sequence of up to 10 samples between CCV/CCB pairs as required to complete run
CCV
CCB
CRI
ICSA
ICSAB
CCV
CCB

Refer to the CLP SOP (CORP-MT-0002) for additional quality control requirements.

11.13. The following run sequence provides an illustration of a mid-run CCV or CCB failure and the appropriate corrective action run sequence as described in Section 9.8:

Original Run: Instrument Calibration
ICV
ICB
CRI
HCAL
ICSA
ICSAB
6 samples
CCV1
CCB1
10 samples
CCV2
CCB2
10 samples **
CCV3 *
CCB3 *
10 samples **
CCV4
CCB4
10 samples
CCV5
CCB5
ICSA
ICSAB
CCV6
CCB6

* Failure occurs at CCV3/CCB3
**Samples requiring rerun for affected analytes

Reanalysis: Recalibrate
ICV
ICB
CRI
HCAL
CCV2
CCB2
10 samples
CCV3
CCB3
10 samples
CCV4
CCB4
ICSA
ICSAB
CCV6
CCB6

Notes: If the reanalysis is conducted under the same instrument setup conditions then it is not necessary to rerun the ICSA/ICSAB at the start of the reanalysis sequence as long as the 8 hour criteria are met. If reanalysis can't be initiated immediately or under the same run conditions then reanalysis must be conducted using the full analysis sequence as detailed in Section 11.11.

Samples between CCV4 and CCV5 do not require reanalysis as they were bracketed by compliant QC samples.

See CORP-MT-0002 for the appropriate reanalysis sequence if CLP requirements must also be met.

11.14. Full method required QC must be available for each wavelength used in determining reported analyte results.

11.15. Guidelines are provided in the appendices on procedures to minimize contamination of samples and standards, preventive maintenance and troubleshooting.

11.16. All measurements must fall within the defined linear range where spectral interference correction factors are valid. Dilute and reanalyze all samples for required analytes that exceed the linear range or use an alternate wavelength for which QC data is established. If an interelement correction exists for an analyte which exceeds the linear range, the IEC may be inaccurately applied. Therefore, even if an overrange analyte may not be required to be reported for a sample, if that analyte is a interferent for any requested analyte in that sample, the sample must be diluted. Acid strength must be maintained in the dilution of samples.

11.17. For TCLP samples, full four point MSA will be required if all of the following conditions are met:

1) recovery of the analyte in the matrix spike is not at least 50%,

2) the concentration of the analyte does not exceed the regulatory level, and,

3) the concentration of the analyte is within 20% of the regulatory level.

The reporting and regulatory limits for TCLP analyses as well as matrix spike levels are detailed in Table VI (Appendix A). Appendix E provides guidance on performing MSA analyses.

11.18. One time procedural variations are allowed only if deemed necessary in the professional judgment of supervision to accommodate variation in sample matrix, radioactivity, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using a Nonconformance Memo and is approved by a

Technical Specialist and QA Manager. If contractually required, the client shall be notified. The Nonconformance Memo shall be filed in the project file.

11.19. Any unauthorized deviations from this procedure must also be documented as a nonconformance, with a cause and corrective action described.

12. DATA ANALYSIS AND CALCULATIONS

12.1. ICV percent recoveries are calculated according to the equation:

$$\%R = 100\left(\frac{Found(ICV)}{True(ICV)}\right)$$

12.2. CCV percent recoveries are calculated according to the equation:

$$\%R = 100\left(\frac{Found(CCV)}{True(CCV)}\right)$$

12.3. Matrix Spike Recoveries are calculated according to the following equation:

$$\%R = 100\left(\frac{SSR - SR}{SA}\right)$$

Where:
SSR = Spike Sample Result
SR = Sample Result
SA = Spike Added

12.4. The relative percent difference (RPD) of matrix spike/matrix spike duplicates or sample duplicates are calculated according to the following equations:

$$RPD = 100\left[\frac{|MSD - MS|}{\left(\frac{MSD + MS}{2}\right)}\right]$$

Where:
MS = determined spiked sample concentration
MSD = determined matrix spike duplicate concentration

$$RPD = 100\left[\frac{|DU1 - DU2|}{\left(\frac{DU1 + DU2}{2}\right)}\right]$$

Where:
DU1 = Sample result
DU2 = Sample duplicate result

12.5. The final concentration for a digested aqueous sample is calculated as follows:

$$mg/L = \frac{C \times V1 \times D}{V2}$$

Where:
C = Concentration (mg/L) from instrument readout
D = Instrument dilution factor
V1 = Final volume in liters after sample preparation
V2 = Initial volume of sample digested in liters

12.6. The final concentration determined in digested solid samples when reported on a dry weight basis is calculated as follows:

$$mg/Kg, dry\ weight = \frac{C \times V \times D}{W \times S}$$

Where:
C = Concentration (mg/L) from instrument readout
D = Instrument dilution factor
V = Final volume in liters after sample preparation
W = Weight in Kg of wet sample digested
S = Percent solids/100

Note: A Percent Solids determination must be performed on a separate aliquot when dry weight concentrations are to be reported. If the results are to be reported on wet weight basis the "S" factor should be omitted from the above equation.

12.7. The LCS percent recovery is calculated according to the following equation:

$$\%R = 100\left(\frac{Found(LCS)}{True(LCS)}\right)$$

12.8. The serial dilution percent difference for each component is calculated as follows:

$$\%Difference = \frac{|I - S|}{I} \times 100$$

Where:
I = Sample result (Instrument reading)
S = Serial dilution result (Instrument reading × 4)

12.9. Appropriate factors must be applied to sample values if dilutions are performed.

12.10. Sample results should be reported with up to three significant figures in accordance with the Quanterra significant figure policy.

13. METHOD PERFORMANCE

13.1. Each laboratory must have initial demonstration of performance data on file for each analyte of interest as described in Section 9.0.

13.2. Refer to Tables I, IA & II in Appendix A for the list of Method 6010A and 200.7 analytes as well as additional analytes that may be analyzed using this SOP.

13.3. Method performance is determined by the analysis of matrix spike and matrix spike duplicate samples as well as method blanks and laboratory control samples. The matrix spike recovery should fall within +/- 20 % and the matrix spike duplicates should compare within 20% RPD. Method blanks must meet the criteria specified in Section 9.2. The laboratory control samples should recover within 20% of the true value until in house control limits are established.

13.4. Training Qualification:

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

14. POLLUTION PREVENTION

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

15. WASTE MANAGEMENT

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

15.2. Standards should be purchased and prepared in volumes consistent with laboratory use to minimize the volume of expired standards to be disposed.

16. REFERENCES

16.1. 40 CFR Part 136, Appendix C, 7-1-92, Method 200.7.

16.2. 40 CFR Part 136, Table IB, 7-1-92.

16.3. Test Methods for Evaluating Solid Waste , Physical/Chemical Methods, SW-846, 3rd Edition, Final Update I, July 1992. Method 6010A.

16.4. CORP-MT-0002, Inductively Coupled Plasma-Atomic Emission Spectroscopy, Method 200.7 CLP-M, SOW ILMO3.0.

16.5. QA-003, Quanterra QC Program.

16.6. QA-004, Rounding and Significant Figures.

16.7. QA-005, Method Detection Limits.

17. MISCELLANEOUS (TABLES, APPENDICES, ETC.)

17.1. Modifications/Interpretations from reference method

17.1.1. Modifications from both Method 6010A and 200.7.

17.1.1.1. Method 200.7 and Chapter 1 of SW846 specify the use of reagent water with a purity equivalent to ASTM Type II water. This SOP requires that reagent water must be free of the analytes of interest as demonstrated through the analysis of method blanks.

17.1.1.2. The alternate run sequence presented in Section 11.12 is consistent with method requirements. Additional QC analyses were added to accommodate the CLP protocol requirements.

17.1.2. Modifications from Method 200.7.

17.1.2.1. Blank subtraction is not performed as per Quanterra QC policy. Method blank results are provided in the analytical report.

17.1.2.2. Method 200.7 defines the IDL as the concentration equivalent to a signal, due to the analyte, which is equal to three times the standard

deviation of a series of ten replicate measurements of a reagent blank signal. Quanterra labs utilize the CLP IDL definition as defined in Section 9.1.1 of this SOP.

17.1.2.3. The calibration blank is prepared in an acid matrix of 5% HNO_3/5% HCl instead of the specified 2% HNO_3/10% HCl matrix as the former matrix provides for improved performance relative to the wide variety of digestate acid matrices which result from the various EPA preparation protocols applied.

17.1.2.4. Section 7.6.3 of 200.7 indicates that the QCS (ICV) should be prepared at a concentration near 1 ppm. The ICV specified in this SOP accommodates the 1 ppm criteria for the majority of analytes. For the remaining analytes, this SOP specifies ICV concentrations which are appropriate to the range of calibration. The intent of the ICV, verification of calibration standard accuracy, is independent of the ICV concentration used.

17.1.2.5. The ICS criteria applied by this SOP differ from those stated in the method. 200.7 states that results should fall within the established control limits of 1.5 times the standard deviation of the mean value. These control criteria were based on a specific solution made available by EMSL-Cincinnati to labs several years ago. Since this solution is no longer available, Quanterra has modeled their ICSA/ICSAB solutions on the design of the ICSA/ICSAB solution provided by EPA directly to EPA contract laboratories. The control limits listed in this SOP are those which EPA states applicable to the EPA designed solution.

17.1.2.6. Method 200.7 states the CCB should be within 2x the standard deviation of the average blank reading. The intent of this requirement is to ensure that the calibration is not drifting at the low end. In the absence of guidance on how to determine the mean blank level from EPA, Quanterra has adopted an absolute control limit of +/- RL from zero for calibration blank criteria.

17.1.3. Modifications from Method 6010A.

17.1.3.1. Chapter 1 of SW-846 states that the method blank should not contain any analyte of interest at or above the MDL. This SOP states that the method blank must not contain any analyte of interest at or above the reporting limit. Common lab contaminants are allowed up to two times the reporting limit in the blank following consultation with the client.

17.1.3.2. Calibration is performed according to instrument manufacturer's recommendations. Method 6010A provides contradictory instructions for instrument calibration by stating to calibrate both according to instrument manufacturer's recommendations as well as to use a blank and three standards. TJA has stated that the use of multiple standards may be detrimental to determinations near the detection limit due to the inability of the linear equation to force-fit through the origin (see Appendix C) and recommends the calibration be performed using a blank and one standard. Leeman recommends a blank and three standards. EPA has stated that manufacturer's recommendations should take precedence and that the next version of the method, 6010B, will clear up the issue by stating "The calibration curve should consist of a minimum of a blank and a standard." This SOP requires verification of the initial instrument calibration using a CRI at two times the RL, an ICV at 5 - 25% of the calibration level, a CCV at 50% of the calibration level and by rerunning the high calibration standard (HCAL) post calibration to demonstrate linearity (See Tables I, IA and II).

17.1.3.3. Section 5.6 of 6010A states that the instrument check standard (CCV) should be prepared from a second source standard. This SOP states that the CCV will be from the same source as the calibration standards. The purpose of the second source standard is to verify the accuracy of the calibration standards. The intent of this requirement is met through the analysis of a second source ICV standard prior to the analysis of samples. The use of a same source CCV provides for a more accurate and consistent measure of instrument drift from initial calibration.

17.1.3.4. Section 5.7 states that spiking of the ICS solution with analytes is not required if the ICP will display overcorrection as a negative number. All Quanterra instrumentation has this capability and therefore the spike analysis is not required. Quanterra does run a spiked ICSAB but the analytes are not spiked at the 10x IDL level referenced in 6010A. The ICSAB solution run by Quanterra is based on the design of the ICSA/ICSAB solution provided by EPA directly to contract environmental labs.

17.1.3.5. Method 6010A uses a Quality Control Standard (QCS) on a weekly basis to verify calibration standard accuracy. Quanterra refers to the QCS as an ICV and the accuracy verification is performed on a daily basis. The QCS described in Method 6010A is made to contain analytes at 10x the IDL. The Quanterra ICV solution is not made at 10x IDL for all elements as this concentration is not appropriate relative to the standard reporting limits. Quanterra designed the ICV to be a reliable indicator of calibration standard accuracy by raising the analyte concentrations to a level where the analytical determination is not impacted by low level curve bias.

17.1.3.6. Method 6010A states the CCB should be within 3x the standard deviations of the average blank reading. The intent of this requirement is to ensure that the calibration is not drifting at the low end. In the absence of guidance on how to determine the mean blank level from EPA, Quanterra has adopted an absolute control limit of +/- RL from zero for calibration blank criteria.

17.2. Modifications from previous SOP

None.

17.3. Facility Specific SOP's

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

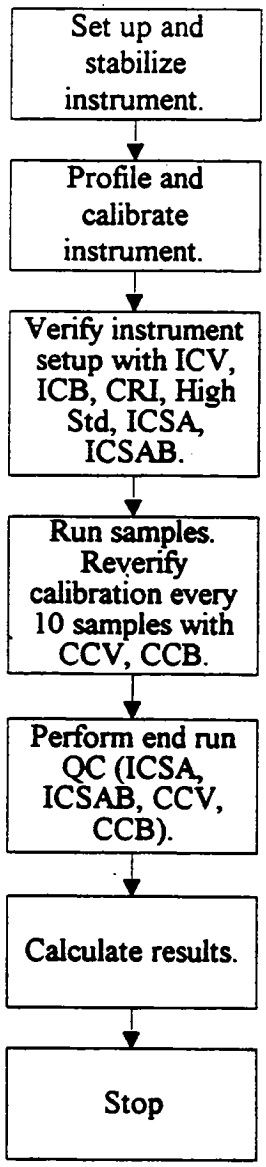
17.4. Documentation and Record Management

The following documentation comprises a complete ICP raw data package:

- Raw data (direct instrument printout signed by analyst).
- Relevant sample preparation benchsheets.
- Run log printout from instrument software where this option is available (TJA) or manually generated run log (i.e., Ward WSL printout).
- Data review checklist - See Appendix B.
- Standards documentation (including prep date, source and lot #).

- Non-conformance summary (if applicable).

17.5. Flow Diagram



APPENDIX A

TABLES

TABLE I. Method 200.7 and 6010A Analyte List

ELEMENT	Symbol	CAS #	6010A analyte	200.7 analyte	Reporting Limit (ug/L) Water	Reporting Limit (mg/kg) Soil
Aluminum	Al	7429-90-5	X	X	200	20
Antimony	Sb	7440-36-0	X	X	60	6
Arsenic	As	7440-38-2	X	X	300	30
Barium	Ba	7440-39-3	X	X	200	20
Beryllium	Be	7440-41-7	X	X	5.0	0.5
Boron	B	7440-42-8		X	200	20
Cadmium	Cd	7440-43-9	X	X	5.0	0.5
Calcium	Ca	7440-70-2	X	X	5000	500
Chromium	Cr	7440-47-3	X	X	10	1
Cobalt	Co	7440-48-4	X	X	50	5
Copper	Cu	7440-50-8	X	X	25	2.5
Iron	Fe	7439-89-6	X	X	100	10
Lead	Pb	7439-92-1	X	X	100	10
Lithium	Li	7439-93-1	X		50	5
Magnesium	Mg	7439-95-4	X	X	5000	500
Manganese	Mn	7439-96-5	X	X	15	1.5
Molybdenum	Mo	7439-98-7	X	X	40	4
Nickel	Ni	7440-02-0	X	X	40	4
Phosphorus	P	7723-14-0	X		300	30
Potassium	K	7440-09-7	X	X	5000	500
Selenium	Se	7782-49-2	X	X	250	25
Silicon	Si	7631-86-9		X	500	50
Silver	Ag	7440-22-4	X	X	10	1
Sodium	Na	7440-23-5	X	X	5000	500
Strontium	Sr	7440-28-0	X		50	5
Thallium	Tl	7440-28-0	X	X	2000	200
Vanadium	V	7440-62-2	X	X	50	5
Zinc	Zn	7440-66-6	X	X	20	2

TABLE IA. Method 200.7 and 6010A Trace ICP Analyte List

ELEMENT	Symbol	CAS #	Reporting Limit (ug/L) Water	Reporting Limit (mg/kg) Soil
Arsenic	As	7440-38-2	10	1.0
Lead	Pb	7439-92-1	3.0	0.3
Selenium	Se	7782-49-2	5.0	0.5
Thallium	Tl	7440-28-0	10	1.0
Antimony	Sb	7440-36-0	10	1.0
Cadmium	Cd	7440-43-9	2.0	0.2
Silver	Ag	7440-22-4	5.0	0.5
Chromium	Cr	7440-47-3	5.0	0.5

TABLE II. Non-Routine Analyte List

ELEMENT	Symbol	CAS #	Reporting Limit (ug/L) Water	Reporting Limit (mg/kg) Soil
Tin	Sn	7440-31-5	100	10
Titanium	Ti	7440-03-26	50	5
Bismuth	Bi	7440-06-99	200	20
Zirconium	Zr	7440-06-77	100	10
Tungsten	W	7440-03-37	500	50
Tellurium	Te	1349-48-09	500	50
Thorium	Th	7440-02-91	500	50
Uranium	U	7440-06-11	500	50
Palladium	Pd	7440-00-53	100	10

NOTE: Analysis of all elements listed may not be available at all Quanterra facilities.

TABLE III. Matrix Spike and Aqueous Laboratory Control Sample Levels

ELEMENT	LCS Level (ug/l)	Matrix Spike Level (ug/l)
Aluminum	2000	2000
Antimony	500	500
Arsenic	2000	2000
Barium	2000	2000
Beryllium	50	50
Cadmium	50	50
Calcium	50000	50000
Chromium	200	200
Cobalt	500	500
Copper	250	250
Iron	1000	1000
Lead	500	500
Lithium	1000	1000
Magnesium	50000	50000
Manganese	500	500
Molybdenum	1000	1000
Nickel	500	500
Phosphorous	10000	10000
Potassium	50000	50000
Selenium	2000	2000
Silver	50	50
Sodium	50000	50000
Strontium	1000	1000
Thallium	2000	2000
Vanadium	500	500
Zinc	500	500
Boron	1000	1000
Silicon	10000	10000
Tin	2000	2000
Titanium	1000	1000
Bismuth	1000	1000
Zirconium	1000	1000
Tellurium	1000	1000
Thorium	1000	1000
Uranium	1000	1000
Tungsten	1000	1000
Palladium	1000	1000

TABLE IV. ICP Calibration and Calibration Verification Standards

Element	Calibration Level	RL (ug/L)	CRI (ug/L)	ICV (ug/L)	CCV (ug/L)
Aluminum	100000	200	400	25000	50000
Antimony	10000	60	120	1000	5000
Arsenic	10000	300	600	1000	5000
Barium	10000	10	20	1000	5000
Beryllium	10000	5	10	1000	5000
Cadmium	10000	5	10	1000	5000
Calcium	100000	5000	10000	25000	50000
Chromium	10000	10	20	1000	5000
Cobalt	10000	50	100	1000	5000
Copper	10000	20	40	1000	5000
Iron	100000	100	200	25000	50000
Lead	10000	100	200	1000	5000
Lithium	10000	50	100	1000	5000
Magnesium	100000	5000	10000	25000	50000
Manganese	10000	15	30	1000	5000
Molybdenum	10000	40	80	1000	5000
Nickel	10000	40	80	1000	5000
Phosphorous	10000	300	600	1000	5000
Potassium	100000	5000	10000	25000	50000
Selenium	10000	250	500	1000	5000
Silver	2000	10	20	500	1000
Sodium	100000	5000	10000	25000	50000
Strontium	10000	50	100	1000	5000
Thallium	20000	2000	4000	5000	10000
Vanadium	10000	50	100	1000	5000
Zinc	10000	20	40	1000	5000
Boron	10000	200	400	1000	5000
Silicon	10000	500	1000	1000	5000
Tin	10000	100	200	1000	5000
Titanium	10000	50	100	1000	5000
Bismuth	10000	200	400	1000	5000
Zirconium	10000	100	200	1000	5000
Tellurium	10000	500	1000	1000	5000
Thorium	10000	500	1000	1000	5000
Uranium	10000	500	1000	1000	5000
Tungsten	10000	500	1000	1000	5000
Palladium	10000	100	200	1000	5000

TABLE IVA. Trace Calibration and Calibration Verification Standards

Element	Calibration Level	RL (ug/L)	CRI (ug/L)	ICV (ug/L)	CCV (ug/L)
Aluminum	50000	200	400	12500	25000
Antimony	1000	10	20	250	500
Arsenic	1000	10	20	250	500
Barium	4000	10	20	1000	2000
Beryllium	4000	5	10	1000	2000
Cadmium	1000	2	4	250	500
Calcium	100000	5000	10000	25000	50000
Chromium	4000	5	10	1000	2000
Cobalt	4000	50	100	1000	2000
Copper	4000	25	40	1000	2000
Iron	50000	100	200	12500	25000
Lead	1000	3	6	250	500
Magnesium	100000	5000	10000	25000	50000
Manganese	4000	15	30	1000	2000
Molybdenum	4000	40	80	1000	2000
Nickel	4000	40	80	1000	2000
Potassium	100000	5000	10000	25000	50000
Selenium	1000	5	10	250	500
Silver	2000	5	10	500	1000
Sodium	100000	5000	10000	25000	50000
Thallium	2000	10	20	500	1000
Vanadium	4000	50	100	1000	2000
Zinc	4000	20	40	1000	2000

TABLE IV. ICP Calibration and Calibration Verification Standards

Element	Calibration Level	RL (ug/L)	CRI (ug/L)	ICV (ug/L)	CCV (ug/L)
Aluminum	100000	200	400	25000	50000
Antimony	10000	60	120	1000	5000
Arsenic	10000	300	600	1000	5000
Barium	10000	10	20	1000	5000
Beryllium	10000	5	10	1000	5000
Cadmium	10000	5	10	1000	5000
Calcium	100000	5000	10000	25000	50000
Chromium	10000	10	20	1000	5000
Cobalt	10000	50	100	1000	5000
Copper	10000	20	40	1000	5000
Iron	100000	100	200	25000	50000
Lead	10000	100	200	1000	5000
Lithium	10000	50	100	1000	5000
Magnesium	100000	5000	10000	25000	50000
Manganese	10000	15	30	1000	5000
Molybdenum	10000	40	80	1000	5000
Nickel	10000	40	80	1000	5000
Phosphorous	10000	300	600	1000	5000
Potassium	100000	5000	10000	25000	50000
Selenium	10000	250	500	1000	5000
Silver	2000	10	20	500	1000
Sodium	100000	5000	10000	25000	50000
Strontium	10000	50	100	1000	5000
Thallium	20000	2000	4000	5000	10000
Vanadium	10000	50	100	1000	5000
Zinc	10000	20	40	1000	5000
Boron	10000	200	400	1000	5000
Silicon	10000	500	1000	1000	5000
Tin	10000	100	200	1000	5000
Titanium	10000	50	100	1000	5000
Bismuth	10000	200	400	1000	5000
Zirconium	10000	100	200	1000	5000
Tellurium	10000	500	1000	1000	5000
Thorium	10000	500	1000	1000	5000
Uranium	10000	500	1000	1000	5000
Tungsten	10000	500	1000	1000	5000
Palladium	10000	100	200	1000	5000

TABLE IVA. Trace Calibration and Calibration Verification Standards

Element	Calibration Level	RL (ug/L)	CRI (ug/L)	ICV (ug/L)	CCV (ug/L)
Aluminum	50000	200	400	12500	25000
Antimony	1000	10	20	250	500
Arsenic	1000	10	20	250	500
Barium	4000	10	20	1000	2000
Beryllium	4000	5	10	1000	2000
Cadmium	1000	2	4	250	500
Calcium	100000	5000	10000	25000	50000
Chromium	4000	5	10	1000	2000
Cobalt	4000	50	100	1000	2000
Copper	4000	20	40	1000	2000
Iron	50000	100	200	12500	25000
Lead	1000	3	6	250	500
Magnesium	100000	5000	10000	25000	50000
Manganese	4000	15	30	1000	2000
Molybdenum	4000	40	80	1000	2000
Nickel	4000	40	80	1000	2000
Potassium	100000	5000	10000	25000	50000
Selenium	1000	5	10	250	500
Silver	2000	5	10	500	1000
Sodium	100000	5000	10000	25000	50000
Thallium	2000	10	20	500	1000
Vanadium	4000	50	100	1000	2000
Zinc	4000	20	40	1000	2000

TABLE V. Interference Check Sample Concentrations*

Element	ICSA (ug/L)	ICSAB (ug/L)
Aluminum	500000	500000
Antimony	-	1000
Arsenic	-	1000
Barium	-	500
Beryllium	-	500
Cadmium	-	1000
Calcium	500000	500000
Chromium	-	500
Cobalt	-	500
Copper	-	500
Iron	200000	200000
Lead	-	1000
Magnesium	500000	500000
Manganese	-	500
Molybdenum	-	1000
Nickel	-	1000
Potassium	-	10000
Selenium	-	1000
Silver	-	1000
Sodium	-	10000
Thallium	-	10000
Vanadium	-	500
Zinc	-	1000
Tin	-	1000

* Custom Quanterra solutions contain analytes common to all Quanterra facilities. Non-routine elements not listed above must be spiked into the ICSAB at 1000 ug/L.

TABLE VI. TCLP Reporting Limits, Regulatory Limits and Matrix Spike Levels

ELEMENT	RL (ug/L)	Regulatory Limit (ug/L)	Spike Level (ug/L)
Arsenic	500	5000	5000
Barium	10000	100000	50000
Cadmium	100	1000	1000
Chromium	500	5000	5000
Lead	500	5000	5000
Selenium	250	1000	1000
Silver	500	5000	1000

TABLE VII. Summary Of Quality Control Requirements

QC PARAMETER	FREQUENCY *	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
Reanalysis of High Standard (HCAL)	Beginning of every analytical run, after CRI.	95 - 105 % recovery.	Terminate analysis; Correct the problem; Recalibrate.
ICV	Beginning of every analytical run.	Method 200.7: 95 - 105 % recovery. Method 6010A: 90 - 110 % recovery.	Terminate analysis; Correct the problem; Recalibrate.
ICB	Beginning of every analytical run, immediately following the ICV.	The result must be within +/- RL from zero.	Terminate analysis; Correct the problem; Recalibrate.
CCV	Every 10 samples and at the end of the run.	Method 200.7: 95 - 105 % recovery. Method 6010A: 90 - 110 % recovery.	Terminate analysis; Correct the problem; Recalibrate and rerun all samples not bracketed by acceptable CCV.
CCB	Immediately following each CCV.	The result must be within +/- RL from zero.	Terminate analysis; Correct the problem; Recalibrate and rerun all samples not bracketed by acceptable CCB.
ICSA	Beginning and end of every run and every 8 hours.	Analyte results must be within +/- 2x RL from zero for analytes with RL ≤ 10 ug/L.	See Section 9.9.
ICSAB	Immediately following each ICSA.	Results must be within 80 - 120% recovery.	See Section 9.9.
CRI	Immediately following ICB.	Results must be within 50 - 150% recovery.	Terminate analysis; Correct the problem; Recalibrate.

* See Sections 11.11 and 11.12 for exact run sequence to be followed.

TABLE VII. Summary of Quality Control Requirements (Continued)

QC PARAMETER	FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
Serial Dilution	One per prep batch.	For samples > 40x IDL, dilutions must agree within 10%.	Narrate the possibility of physical or chemical interference.
Method Blank	One per sample preparation batch of up to 20 samples.	The result must be less than or equal to the RL. Common lab contaminants may be accepted up to 2x the RL after consultation with the client (See 9.2). Sample results greater than 20x the blank concentration are acceptable. Samples for which the contaminant is < RL may not require redigestion or reanalysis (see Section 9.2).	Redigest and reanalyze samples. Note exceptions under criteria section. See Section 9.2 for additional requirements.
Laboratory Control Sample (LCS)	One per sample preparation batch of up to 20 samples.	Aqueous LCS must be within 80 - 120% recovery or in-house control limits. Samples for which the contaminant is < RL and the LCS results are > 120% may not require redigestion or reanalysis (see Section 9.3)	Terminate analysis; Correct the problem; Redigest and reanalyze all samples associated with the LCS.
Matrix Spike	One per sample preparation batch of up to 20 samples.	80 - 120 % recovery. If the MS/MSD is out for an analyte, it must be in control in the LCS. For TCLP See Section 11.17.	In the absence of client specific requirements, flag the data; no flag required if the sample level is > 4x the spike added. For TCLP see Section 11.17.
Matrix Spike Duplicate	See Matrix Spike	80 - 120 % recovery; RPD ≤ 20% (See MS).	See Corrective Action for Matrix Spike.

APPENDIX B

QUANTERRA ICP DATA REVIEW CHECKLIST

Quanterra ICP Data Review Checklist

Run/Project Information:

Run Date: _____ **Analyst:** _____ **Instrument:** _____
Prep Batches Run: _____

Circle Methods used: **6010A / 200.7: CORP-MT-0001 Rev 0**
CLP : CORP-MT-0002 Rev 0

Review Items

A. Calibration/Instrument Run QC	Yes	No	N/A	2nd Level
1. Instrument calibrated per manufacturer's instructions and at SOP specified levels ?				
2. ICV/CCV analyzed at appropriate frequency and within control limits ? (6010A, CLP = 90 - 110%, 200.7 = 95 -105%)				
3. ICB/CCB analyzed at appropriate frequency and within +/- RL or +/- CRDL (CLP) ?				
4. High Std. (HCAL) reanalyzed before samples and recovered within QC limits ? (6010A/200.7 95-105%, CLP- N/A)				
5. CRI run and recovered within QC limits ? (+/- 50% for non-CLP)				
6. ICSA/ICSAB run at required frequency and within SOP limits ?				
B. Sample Results				
1. Were samples with concentrations > the linear range for any parameter diluted and reanalyzed ?				
2. All reported results bracketed by in control QC ?				
3. Sample analyses done within holding time ?				
C. Preparation/Matrix QC				
1. LCS done per prep batch and within QC limits ?				
2. Method blank done per prep batch and < RL or CRDL (CLP) ?				
3. MS run at required frequency and within limits ?				
4. MSD or DU run at required frequency and RPD within SOP limits ?				
5. Serial dilution done per prep batch (or per SDG for CLP) ?				
6. Post digest spike analyzed if required (CLP only) ?				
D. Other				
1. Are all nonconformances documented appropriately ?				
2. Current IDL/LR/IEC data on file ?				
3. Calculations checked for error ?				
4. Transcriptions checked for error ?				
5. All client/project specific requirements met ?				
6. Date/time of analysis verified as correct ?				

Analyst: _____ **Date:** _____
Comments: _____

2nd Level Reviewer : _____ **Date:** _____
Comments: _____

APPENDIX C

TJA CALIBRATION RECOMMENDATION

Thermo Jarrell Ash Corporation

85 Forge Parkway
Franklin, MA 02038-3148

(508) 520-1880
Fax: (508) 520-1732
Telex: 174230 TJA UT

Ms. Ruth Wolfe
Enseco, RMAL
4955 Yarrow St.
Arvada, CO 8002

Dear Ruth,

I would like to respond to your question concerning the recommended standardization procedure for the ICP instrument you own. As the manufacturer, we recommend a 2-point standardization verified by an independent standard which is normally done with every analytical run when using EPA protocols. Since the instrument is known to be linear for at least five orders of magnitude of concentration, using multiple standards is unnecessary and may be detrimental to determinations near the detection limit. This is due to the inability of the linear equation to force-fit through the origin. Therefore, if the multiple standards are not made to exact specifications, there may be a bias near the origin.
Most EPA methods recognize this and follow the procedure I've recommended above, verifying the upper linear range on a quarterly basis.
If you need additional information concerning the standardization procedure please let me know.

Regards,

Bob Foster

Bob Foster

APPENDIX D

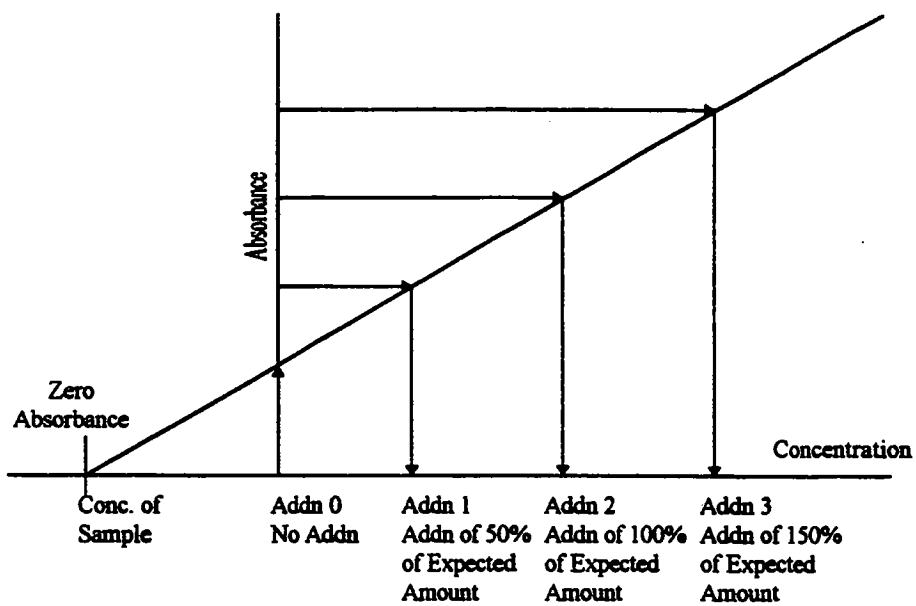
MSA GUIDANCE

Appendix D. MSA Guidance

Method of Standard Addition

Four equal volume aliquots of sample are measured and known amounts of standards are added to three aliquots. The fourth aliquot is the unknown and no standard is added to it. The concentration of standard added to the first aliquot should be 50% of the expected concentration. The concentration of standard added to the second aliquot should be 100% of the expected concentration and the concentration of standard added to the third aliquot should be 150% of the expected concentration. The volume of the unspiked and spiked standard should be the same.

In order to determine the concentration of analyte in the sample, the analytical value of each solution is determined and a plot or linear regression performed. On the vertical axis the analytical value is plotted versus the concentrations of the standards on the horizontal axis. An example plot is shown in Figure 1. When the resulting line is extrapolated back to zero absorbance, the point of interception of the horizontal axis is the concentration of the unknown.



For the method of standard additions to be correctly applied, the following limitations must be taken into consideration:

- The plot of the sample and standards must be linear over the concentration range of concern. For best results, the slope of the curve should be similar to that of a plot of the aqueous standard curve.

- The effect of the interference should not vary as the ratio of the standard added to the sample matrix changes.

APPENDIX E

TROUBLESHOOTING GUIDE

APPENDIX E. TROUBLESHOOTING GUIDE

Problem	Possible Cause/ Solution
High Blanks	Increase rinse time Clean or replace tip Clean or replace torch Clean or replace sample tubing Clean or replace nebulizer Clean or replace mixing chamber Lower Torch
Instrument Drift	RF not cooling properly Vacuum level is too low Replace torch (Crack) Clean or replace nebulizer (blockage) Check room temperature (changing) Replace pump tubing Room humidity too high Clean torch tip (salt buildup) Check for argon leaks Adjust sample carrier gas Reprofile Horizontal Mirror Replace PA tube
Erratic Readings, Flickering Torch or High RSD	Check for argon leaks Adjust sample carrier gas Replace tubing (clogged) Check drainage(back pressure changing) Increase uptake time (too short) Increase flush time (too short) Clean nebulizer, torch or spray chamber Increase sample volume introduced Check that autosampler tubes are full Sample or dilution of sample not mixed Increase integration time (too short) Realign torch Reduce amount of tubing connectors
Cu/Mn Ratio Outside Limits or Low Sensitivity	Plasma conditions changed Clean nebulizer, torch or spray chamber Replace tubing (clogged) Realign torch Check IEC's
Standards reading twice normal absorbance or concentration	Incorrect standard used Incorrect dilution performed

APPENDIX F

CONTAMINATION CONTROL GUIDELINES

APPENDIX F. CONTAMINATION CONTROL GUIDELINES

The following procedures are strongly recommended to prevent contamination:

All work areas used to prepare standards and spikes should be cleaned before and after each use.

All glassware should be washed with detergent and tap water and rinsed with 1:1 nitric acid followed by deionized water.

Proper laboratory housekeeping is essential in the reduction of contamination in the metals laboratory. All work areas must be kept scrupulously clean.

Powdered or Latex Gloves must not be used in the metals laboratory since the powder contains silica and zinc as well as other metallic analytes. Only vinyl or nitrile gloves should be used in the metals laboratory.

Glassware should be periodically checked for cracks and etches and discarded if found. Etched glassware can cause cross contamination of any metallic analytes.

Autosampler trays should be covered to reduce the possibility of contamination. Trace levels of elements being analyzed in the samples can be easily contaminated by dust particles in the laboratory.

The following are helpful hints in the identification of the source of contaminants:

Yellow pipet tips and volumetric caps can sometimes contain cadmium.

Some sample cups have been found to contain lead.

The markings on glass beakers have been found to contain lead. If acid baths are in use for glassware cleaning, they should be periodically checked for contaminants since contaminant concentrations will increase over time.

New glassware especially beakers can be a source of silica and boron.

Reagents or standards can contain contaminants or be contaminated with the improper use of a pipette.

Improper cleaning of glassware can cause contamination.

Latex gloves contain over 500 ppb of zinc.

APPENDIX G

PREVENTIVE MAINTENANCE

APPENDIX G. PREVENTIVE MAINTENANCE

A maintenance log is used to record when maintenance is performed on instruments. When an instrument problem occurs indicate the date, time and instrument number, then identify the problem and corrective action in the maintenance log.

The following procedures are required to ensure that that the instrument is fully operational.

Daily
Change sample pump tubing and pump windings
Check argon gas supply level
Check rinse solution and fill if needed
Check waste containers and empty if needed
Check sample capillary tubing is clean and in good condition
Check droplet size to verify nebulizer is not clogged.
Check sample flow for cross flow nebulizer
Check Cu/Mn ratio-should be 30% of value at date that IECs were performed
Check pressure for vacuum systems

As Needed
Clean plasma torch assembly to remove accumulated deposits
Clean nebulizer and drain chamber; keep free-flowing to maintain optimum performance
Replace peristaltic pump tubing, sample capillary tubing and autosamper sipper probe

Weekly
Apply silicon spray on autosampler tracks
Check water level in coolflow

Monthly
Clean air filters on back of power unit to remove dust
Check D mirror for air instruments

Bi-yearly
Change oil for vacuum systems
Replace coolant water filter (may require more or less frequently depending on quality of cooling water)

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SOP No. CORP-MT-0007
Revision No. 0
Revision Date: 08-01-95
Page: 1 of 40

QUANTERRA STANDARD OPERATING PROCEDURE

TITLE: PREPARATION AND ANALYSIS OF MERCURY IN SOLID SAMPLES BY COLD VAPOR ATOMIC ABSORPTION SPECTROSCOPY, SW846 7471A AND MCAWW 245.5

(SUPERSEDES: NONE)

Prepared by: _____
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TABLE OF CONTENTS

1. SCOPE AND APPLICATION 3

2. SUMMARY OF METHOD 3

3. DEFINITIONS 3

4. INTERFERENCES 3

5. SAFETY 5

6. EQUIPMENT AND SUPPLIES 6

7. REAGENTS AND STANDARDS 7

8. SAMPLE COLLECTION, PRESERVATION AND STORAGE 9

9. QUALITY CONTROL 9

10. CALIBRATION AND STANDARDIZATION 12

11. PROCEDURE 13

12. DATA ANALYSIS AND CALCULATIONS 18

13. METHOD PERFORMANCE 20

14. POLLUTION PREVENTION 20

15. WASTE MANAGEMENT 20

16. REFERENCES 20

17. MISCELLANEOUS (TABLES, APPENDICES, ETC. . .) 21

LIST OF APPENDICES:

APPENDIX A - TABLES 26

APPENDIX B - QUANTERRA Hg DATA REVIEW CHECKLIST 30

APPENDIX C - MSA GUIDANCE 32

APPENDIX D - TROUBLESHOOTING GUIDE 35

APPENDIX E- CONTAMINATION CONTROL GUIDELINES 37

APPENDIX F - PREVENTATIVE MAINTENANCE 39

1. SCOPE AND APPLICATION

1.1. This procedure describes the preparation and analysis of mercury (Hg, CAS # 7439-97-6) by Cold Vapor Atomic Absorption Spectroscopy (CVAA) using SW-846 Method 7471A and MCAWW Method 245.5.

1.2. CVAA analysis provides for the determination of total mercury (organic and inorganic). The combination of the oxidants, potassium permanganate and potassium persulfate, has been found to give 100% recovery with both types of compounds. Detection limits, sensitivity and optimum concentration ranges for mercury analysis will vary with the matrices, instrumentation and volume of sample used.

1.3. Methods 7471A and 245.5 are applicable to the preparation and analysis of mercury in soils, sediments, bottom deposits and sludge-type materials. All matrices require sample preparation prior to analysis.

1.4. The Quanterra reporting limit for mercury in solid matrices is 0.1 mg/kg based a 0.2 g sample aliquot (wet weight).

2. SUMMARY OF METHOD

2.1. This SOP describes a technique for the determination of mercury in solution. The procedure is a physical method based on the absorption of radiation at 253.7 nm by mercury vapor. A representative portion of the sample is digested in sulfuric and nitric acids. Organic mercury compounds are oxidized with potassium permanganate and potassium persulfate and the mercury reduced to its elemental state with stannous chloride and aerated from solution in a closed system. The mercury vapor passes through a cell positioned in the light path of an atomic absorption spectrophotometer. Absorbance is measured as a function of mercury concentration. Concentration of the analyte in the sample is determined by comparison of the sample absorbance to the calibration curve (absorbance vs. concentration).

3. DEFINITIONS

3.1. Total Metals: The concentration determined on an unfiltered sample following digestion.

4. INTERFERENCES

Chemical and physical interferences may be encountered when analyzing samples using this method.

4.1. Potassium permanganate which is used to breakdown organic mercury compounds also eliminates possible interferences from sulfide. Concentrations as high as 20 mg/L of sulfide as sodium sulfide do not interfere with the recovery of inorganic mercury from reagent water.

4.2. Copper has also been reported to interfere; however, copper concentrations as high as 10 mg/L had no effect on the recovery of mercury from spiked samples.

4.3. Chlorides can cause a positive interference. Samples high in chlorides require additional permanganate (as much as 25 mL) because, during the oxidation step, chlorides are converted to free chlorine, which also absorbs radiation at 253.7 nm. Care must be taken to ensure that free chlorine is absent before the mercury is reduced and swept into the cell. This is accomplished by adding excess hydroxylamine reagent (25 mL) and purging the sample headspace before stannous chloride is added. Both inorganic and organic mercury spikes have been quantitatively recovered from seawater using this technique.

Note: Sufficient addition of permanganate is apparent when the purple color persists at least 15 minutes. Some samples may require dilution prior to digestion due to extremely high concentrations of chloride.

4.4. Interference from certain volatile organic materials that absorb at this wavelength may also occur. If suspected, a preliminary run without stannous chloride can determine if this type of interference is present. While the possibility of absorption from certain organic substances present in the sample does exist, this problem is not routinely encountered. This is mentioned only to caution the analyst of the possibility. If this condition is found to exist, the mercury concentration in the sample can be determined by subtracting the result of the sample run without the reducing reagent (stannous chloride) from that obtained with the reducing reagent.

4.5. Samples containing high concentrations of oxidizable organic materials, as evidenced by high COD levels, may not be completely oxidized by this procedure. When this occurs the recovery of mercury will be low. The problem can be eliminated by reducing the volume of original sample used.

4.6. The most common interference is laboratory contamination which may arise from impure reagents, dirty glassware, improper sample transfers, dirty work areas, etc. Be aware of potential sources of contamination and take appropriate measures to minimize or avoid them.

5. SAFETY

5.1. Procedures shall be carried out in a manner that protects the health and safety of all Quanterra associates.

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

5.3. The health and safety hazards of many of the chemicals used in this procedure have not been fully defined. Additional health and safety information can be obtained from the Material Safety Data Sheets (MSDS) maintained in the laboratory. The following specific hazards are known:

5.3.1. The following materials are known to be **corrosive:**

hydrochloric acid, nitric acid and sulfuric acid.

5.3.2. The following materials are known to be **oxidizing agents**:

nitric acid, potassium permanganate, potassium persulfate and magnesium perchlorate.

5.3.3. Mercury is a highly toxic element that must be handled with care. The analyst must be aware of the handling and clean-up techniques before working with mercury. Since mercury vapor is toxic, precaution must be taken to avoid its inhalation, ingestion or absorption through skin. All lines should be checked for leakage and the mercury vapor must be vented into a hood or passed through a mercury absorbing media such as:

5.3.3.1. Equal volumes of 0.1 M $KMnO_4$ and 10% H_2SO_4, or

5.3.3.2. Iodine, 0.25%, in a 3% KI solution.

5.3.4. Magnesium sulfate is known to be a reproductive toxin (mutagen).

5.4. Exposure to chemicals must be maintained **as low as reasonably achievable.** Therefore, unless they are known to be non-hazardous, all samples should be opened, transferred and prepared in a fume hood, or under other means of mechanical ventilation. Solvent and waste containers will be kept closed unless transfers are being made.

5.5. The preparation of standards and reagents will be conducted in a fume hood with the sash closed as far as the operation will permit.

5.6. All work must be stopped in the event of a known or potential compromise to the health and safety of a Quanterra associate. The situation must be reported **immediately** to a laboratory supervisor.

5.7. Do not look directly into the beam of the Hg lamp. The UV light that these lamps radiate is harmful to the eyes.

5.8. Cylinders of compressed gas must be handled with caution, in accordance with local regulations. It is recommended that, wherever possible, cylinders be located outside the laboratory and the gas led to the instrument through approved lines.

5.9. The CVAA apparatus must be properly vented to remove potentially harmful fumes generated during sample analysis.

6. EQUIPMENT AND SUPPLIES

6.1. Temperature controlled water bath (capable of maintaining temperature of 90- 95 °C) or autoclave capable of obtaining 15 lbs., 120 °C.

6.2. Atomic Absorption Spectrophotometer equipped with:

6.2.1. Absorption Cell with quartz end windows perpendicular to the longitudinal axis. Dimensions of the cell must result in sufficient sensitivity to meet the SOP defined reporting limit. The quartz windows must be maintained to provide accurate measurements. Any scratches or fingerprints can alter the absorption of UV radiation.

6.2.2. Mercury specific hollow cathode lamp (HCL) or electrodeless discharge lamp (EDL).

6.2.3. Peristaltic pump which can deliver 1 L/min air.

6.2.4. Flowmeter capable of measuring an airflow of 1 L/min.

6.2.5. Recorder or Printer.

6.2.6. Aeration Tubing: A straight glass frit having a course porosity and Tygon tubing is used for the transfer of mercury vapor from the sample bottle to the absorption cell and return.

6.2.7. Drying device (a drying tube containing magnesium perchlorate or magnesium sulfate and/or a lamp with a 60 W bulb) to prevent condensation in cell. The lamp is positioned to shine on the absorption cell maintaining the air temperature in the cell about 10 °C above room temperature. Other drying devices that acheive the same purpose are also acceptable (i.e., Gortex filter).

Note: Instruments designed specifically for the measurement of mercury using the cold vapor technique may be substituted for the atomic absorption spectrophotometer.

6.3. BOD bottles or equivalent.

6.4. Nitrogen or argon gas supply, welding grade or equivalent.

6.5. Calibrated automatic pipettes or Class A glass volumetric pipettes.

6.6. Class A volumetric flasks.

6.7. Top-loading balance, capable of reading up to two decimal places.

6.8. Thermometer (capable of accurate readings at 95 °C).

6.9. Disposable cups or tubes.

7. REAGENTS AND STANDARDS

7.1. Reagent water must be produced by a Millipore DI system or equivalent. Reagent water must be free of the analytes of interest as demonstrated through the analysis of method blanks.

7.2. Stock (1000 ppm) mercury standards (in 10% HNO_3) are purchased as custom Quanterra solutions. All standards must be stored in FEP fluorocarbon or previously unused polyethylene or polypropylene bottles. Stock standard solutions must be replaced prior to the expiration date provided by the manufacturer. If no expiration date is provided, the stock solutions may be used for up to one year and must be replaced sooner if verification from an independent source indicates a problem.

7.3. Intermediate mercury standard (10 ppm): Take 1 mL of the stock mercury standard (7.2) and dilute to 100 mL with reagent water. The intermediate standard must be made monthly and must be prepared in a matrix of 2% HNO_3. This acid (2 mL of concentrated HNO_3) must be added to the flask/bottle before the addition of the stock standard aliquot.

7.4. Working mercury standard (0.1 ppm): Take 1 mL of the intermediate mercury standard (7.3) and dilute to 100 mL with reagent water. The working mercury standard must be made daily and must be prepared in a matrix of 0.15% HNO_3. This acid (150 uL of concentrated HNO_3) must be added to the flask/bottle before the addition of the stock standard aliquot.

7.5. The calibration standards must be prepared fresh daily from the working standard (7.4) by transferring 0, 0.2, 0.5, 1.0, 2.0, 5.0 and 10.0 mL aliquots of the working mercury standard into sample prep bottles and proceeding as specified in Section 11.1

Note: Alternate volumes of standard may be prepared as long as the accuracy and final standard concentrations as detailed in Table I are maintained.

7.6. The initial calibration verification standard must be made from a different stock solution than that of the calibration standards.

7.7. Refer to Table I (Appendix A) for details regarding the working standard concentrations for calibration, calibration verification and spiking solutions. All standards must be processed through the entire analytical procedure including sample preparation.

7.8. Nitric acid (HNO_3), concentrated, trace metal grade or better.

Note: If a high reagent blank is obtained, it may be necessary to distill the nitric acid.

7.9. Sulfuric acid (H_2SO_4), concentrated, trace metal grade or better.

7.9.1. Sulfuric acid, 0.5 N: Dilute 14.0 mL of concentrated H_2SO_4 to 1 liter with reagent water.

7.10. Hydrochloric acid (HCl), concentrated, trace metal grade or better.

7.11. Aqua Regia: Prepare immediately before use by carefully adding three volumes of concentrated HCl to one volume of concentrated HNO_3.

7.12. Stannous sulfate solution: Add 25 g of stannous sulfate to 250 mL of 0.5 N sulfuric acid. This mixture is a suspension and should appear cloudy. This solution should be made daily and should be stirred continuously during use.

Note: Stannous chloride may be used in place of stannous sulfate. Prepare the stannous chloride solution according to the recommendations provided by the instrument manufacturer.

7.13. Sodium chloride-hydroxylamine hydrochloride solution: Add 12 g of sodium chloride and 12 g of hydroxylamine hydrochloride to every 100 mL of reagent water.

Note: Hydroxylamine sulfate may be used in place of hydroxylamine hydrochloride.

7.14. Potassium permanganate, 5% solution (w/v): Dissolve 5 g of potassium permanganate for every 100 mL of reagent water.

7.15. Potassium persulfate, 5% solution (w/v): Dissolve 5 g of potassium persulfate for every 100 mL of reagent water.

8. SAMPLE COLLECTION, PRESERVATION AND STORAGE

8.1. Sample holding time for mercury is 28 days from time of collection to the time of sample analysis.

8.2. Soil samples do not require preservation but must be stored at 4° C ± 2° C until the time of analysis.

9. QUALITY CONTROL

Table II (Appendix A) provides a summary of quality control requirements including type, frequency, acceptance criteria and corrective action.

9.1. Initial Demonstration of Capability

Prior to the analysis of any analyte using 7471A or the 245.5, the following requirements must be met.

9.1.1. Method Detection Limit (MDL) - An MDL must be determined for each analyte/matrix prior to the analysis of any samples. The MDL is determined using seven replicates of reagent water, spiked with all the analytes of interest, that have been carried through the entire analytical procedure. MDLs must be redetermined on an annual basis in accordance with 40 CFR Part 136 Appendix B requirements. The spike level must be between the calculated MDL and 10X the MDL to be valid. The result of the MDL determination must be below the Quanterra reporting limit.

9.2. Preparation Batch - A group of up to 20 field samples that are of the same matrix and are processed together using the same procedures and reagents.

9.3. Method Blank (MB) - One method blank must be processed with each preparation batch. The method blank consists of reagent water containing all reagents specific to the method that is carried through the entire analytical procedure, including preparation and analysis. The method blank is used to identify any system and process interferences or contamination of the analytical system that may lead to the reporting of elevated analyte concentrations or false positive data. The method blank should not contain any analyte of interest at or above the reporting or at or above 5% of the measured concentration of that analyte in associated samples, whichever is higher (sample result must be a minimum of 20 times higher than the blank contamination level).

- Repreparation and reanalysis of all samples associated with an unacceptable method blank is required when reportable concentrations are determined in the samples (see exception noted above).

- If there is no analyte greater than the RL in the samples associated with an unacceptable method blank, the data may be reported with qualifiers. **Such action must be taken in consultation with the client and must be addressed in the project narrative.**

- If the above criteria are not met and reanalysis is not possible, then the sample data must be qualified. **This anomaly must be addressed in the project narrative and the client must be notified.**

9.4. Laboratory Control Sample (LCS) - One aqueous LCS must be processed with each preparation batch. The LCS is used to monitor the accuracy of the analytical process. On-going monitoring of the LCS results provides evidence that the laboratory is performing the method within acceptable accuracy and precision guidelines. The LCS must be carried through the entire analytical procedure. The CCV results can be reported as the LCS results since all CCVs (as well as all other standards) are processed through the sample preparation step with the field samples. No more than 20 samples can be associated with one CCV used for the purpose of reporting LCS data.

- If the LCS is outside established control limits the system is out of control and corrective action must occur. Until in-house control limits are established, a control limit of 80 - 120% recovery must be applied.

- In the instance where the LCS recovery is > 120% and the sample results are < RL, the data may be reported with qualifiers. Such action must be taken in consultation with the client and must be addressed in the case narrative."

- In the event that an MS/MSD analysis is not possible, a Laboratory Control Sample Duplicate (LCSD) must be analyzed. The RPD of the LCS and LCSD must be compared to the matrix spike RPD limits.

- Corrective action will be repreparation and reanalysis of the batch unless the client agrees that other corrective action is acceptable.

9.5. Matrix Spike/Matrix Spike Duplicate (MS/MSD) - One MS/MSD pair must be processed for each preparation batch. A matrix spike (MS) is a field sample to which known concentrations of target analytes have been added. A matrix spike duplicate (MSD) is a second aliquot of the same sample (spiked identically as the MS) prepared and analyzed along with the sample and matrix spike. Some client specific data quality objectives (DQO's) may require the use of sample duplicates in place of or in addition to MS/MSD's. The MS/MSD results are used to determine the effect of a matrix on the precision and accuracy of the analytical process. Due to the potential variability of the matrix of each sample, these results may have immediate bearing only on the specific sample spiked. Samples identified as field blanks cannot be used for MS/MSD analysis. Spiking levels are provided in Table I (Appendix A).

- If analyte recovery or RPD falls outside the acceptance range, the recovery of that analyte must be in control for the LCS. Until in-house control limits are established, a control limit of 75 - 125 % recovery and 20% RPD must be applied to the MS/MSD. If the LCS recovery is within limits, then the laboratory operation is in control and the results may be accepted. If the recovery of the LCS is outside limits, corrective action must be taken. Corrective action will include repreparation and reanalysis of the batch. MS/MSD results which fall outside the control limits must be addressed in the narrative.

- If the native analyte concentration in the MS/MSD exceeds 4 times the spike level for that analyte, the recovery data are reported as NC (i.e., not calculated). If the reporting software does not have the ability to report NC then the actual recovery must be reported and narrated as follows: "Results outside of limits do not necessarily reflect poor method performance in the matrix due to high analyte concentrations in the sample relative to the spike level."

- If an MS/MSD is not possible due to limited sample volume, then a laboratory control sample duplicate (LCSD) should be analyzed. The RPD of the LCS and LCSD must be compared to the matrix spike RPD limits.

9.6. Initial Calibration Verification (ICV/ICB) - Calibration accuracy is verified by analyzing a second source standard (ICV). The ICV result must fall within 20% of the

true value for that solution. An ICB is analyzed immediately following the ICV to monitor low level accuracy and system cleanliness. The ICB result must fall within +/- the reporting limit (RL) from zero. If either the ICV or ICB fail to meet criteria, the analysis should be terminated, the problem corrected and the instrument recalibrated. (See Section 11.2.10 and Section 11.2.11 for required run sequence). If the cause of the ICV or ICB failure was not directly instrument related the corrective action will include repreparation of the associated samples.

9.7. Continuing Calibration Verification (CCV/CCB) - Calibration accuracy is monitored throughout the analytical run through the analysis of a known standard after every 10 samples. The CCV must be a mid-range standard at a concentration other than that of the ICV. The CCV result must fall within 20% of the true value for that solution. A CCB is analyzed immediately following each CCV. (See Section 11.2.10 and 11.2.11 for required run sequence.) The CCB result must fall within +/- RL from zero. Each CCV and CCB analyzed must reflect the conditions of analysis of all associated samples. Sample results may only be reported when bracketed by valid ICV/CCV and ICB/CCB pairs. If a mid-run CCV or CCB fails, the analysis must be terminated, the problem corrected, the instrument recalibrated, the calibration verified and the affected samples reanalyzed. If the cause of the CCV or CCB failure was not directly instrument related the corrective action will include repreparation of the associated samples.

9.8. Method of Standard Addition (MSA) -This technique involves adding known amounts of standard to one or more aliquots of the sample prior to preparation. This technique compensates for a sample interferent that may enhance or depress the analyte signal, thus producing a different slope from that of the calibration standards. It will not correct for additive interferences which cause a baseline shift. Refer to Section 11.2.12 for additional information on when full 4 point MSA is required as well as Appendix D for specific MSA requirements.

10. CALIBRATION AND STANDARDIZATION

10.1. Calibration standards must be processed through the preparation procedure as described in Section 11.1.

10.2. Due to the differences in preparation protocols separate calibration and calibration verification standards must be prepared for aqueous and solid matrices.

10.3. Calibration must be performed daily (every 24 hours) and each time the instrument is set up. The instrument calibration date and time must be included in the raw data.

10.4. Set up the instrument with the operating parameters recommended by the manufacturer. Allow the instrument to become thermally stable before beginning calibration (approximately 30 minutes of warm-up is required). Refer to the facility specific instrument SOP and CVAA instrument manual for detailed setup and operation protocols.

10.5. Calibrate the instrument according to instrument manufacturer's instructions, using a minimum of five standards and a blank. One standard must be at the Quanterra reporting limit. Analyze standards in ascending order beginning with the blank. Refer to Section 7.5 and Table I for additional information on preparing calibration standards and calibration levels.

10.6. The calibration curve must have a correlation coefficient of ≥0.995 or the instrument shall be stopped and recalibrated prior to running samples. Sample results can not be reported from a curve with an unacceptable correlation coefficient.

10.7. Refer to Section 9.0 for calibration verification procedures, acceptance criteria and corrective actions.

11. PROCEDURE

11.1. Standard and Sample Preparation:

11.1.1. All calibration and calibration verification standards (ICV, ICB, CCV, CCB) are processed through the digestion procedure as well as the field samples.

11.1.2. Transfer 0, 0.2, 0.5, 1.0, 2.0, 5.0 and 10.0 mL aliquots of the working standard (7.5) into a series of sample digestion bottles.

Note: Alternate volumes of standard may be prepared as long as the accuracy and final standard concentrations as detailed in Table I are maintained.

11.1.3. Add reagent water to each standard bottle to make a total volume of 10 mL. Continue preparation as described under 11.1.5 or 11.1.6 below.

11.1.4. Transfer triplicate 0.2 g portions of a well mixed sample into a clean sample digestion bottle. Continue preparation as described under 11.2.2 or 11.2.3 below.

11.1.5. Water Bath protocol:

11.1.5.1. To each **standard** bottle: Add 5 mL of aqua regia.
To each **sample** bottle: Add 5 mL of reagent water and 5 mL of aqua regia.

11.1.5.2. Heat for 2 minutes in a water bath at 95 ° C.

11.1.5.3. Cool.

11.1.5.4. Add 50 mL of distilled water.

11.1.5.5. Add 15 mL of potassium permanganate solution.

11.1.5.6. Add 8 mL of potassium persulfate solution, mix thoroughly

11.1.5.7. Heat for 30 minutes in the water bath at 95 °C.

11.1.5.8. Cool.

11.1.5.9. Add 6 mL of sodium chloride-hydroxylamine sulfate solution to reduce the excess permanganate.

11.1.5.10. To each **standard** bottle: Add 50 mL of reagent water.
To each **sample** bottle: Add 55 mL of reagent water.

11.1.5.11. Continue as described under Section 11.2.

11.1.6. Autoclave protocol:

11.1.6.1. Add 5 mL concentrated of H_2SO_4 and 2 mL of concentrated HNO_3.

11.1.6.2. Add 5 mL of saturated potassium permanganate solution.

11.1.6.3. Add 8 mL of potassium persulfate solution.

11.1.6.4. Cover digestion bottle with aluminum foil or screw cap loosely applied.

11.1.6.5. Heat samples at 121 °C and 15 lbs. pressure for 15 minutes.

11.1.6.6. Cool.

11.1.6.7. Make up to volume of 100 mL with reagent water.

Note: Alternate final volumes may be used as long as the standards and sample are treated the same way.

11.1.6.8. Add 6 mL of sodium chloride-hydroxylamine sulfate solution to reduce excess permanganate.

11.1.6.9. Continue as described under Section 11.2 .

11.2. Sample Analysis:

11.2.1. Because of differences between various makes and models of CVAA instrumentation, no detailed operating instructions can be provided. Refer to the facility specific instrument operating SOP and the CVAA instrument manual for detailed setup and operation protocols.

11.2.2. All labs are required to detail the conditions/programs utilized for each instrument within the facility specific instrument operation SOP.

11.2.3. Manual determination:

11.2.3.1. Treating each sample individually, purge the head space of the sample bottle for at least one minute.

11.2.3.2. Add 5 mL of stannous chloride solution and immediately attach the bottle to the aeration apparatus.

11.2.3.3. Allow the sample to stand quietly without manual agitation while the sample is aerated (1 L/min flow). Monitor the sample absorbance during aeration. When the absorbance reaches a maximum and the signal levels off, open the bypass valve and continue aeration until the absorbance returns to its baseline level. Close the bypass valve and remove the aeration device.

11.2.3.4. Place the aeration device into 100 mLs of 1% HNO_3 and allow to bubble rinse until the next sample is analyzed.

11.2.4. Automated determination: Follow instructions provided by instrument manufacturer.

11.2.5. Perform a linear regression analysis of the calibration standards by plotting maximum response of the standards vs. ug of mercury. Determine the mercury concentration in the samples from the linear regression fit of the calibration curve. Calibration using computer or calculation based regression curve fitting techniques on concentration/response data is acceptable.

11.2.6. All measurements must fall within the defined calibration range to be valid. Dilute and reanalyze all samples for analytes that exceed the highest calibration standard.

11.2.7. If the sample results are negative and the absolute value of the negative result is greater than the reporting limit, the sample must be diluted and reanalyzed.

11.2.8. The samples must be allowed to cool to room temperature prior to analysis or a decrease in the response signal can occur.

11.2.9. Baseline correction is acceptable as long as it is performed after every sample or after the CCV and CCB; resloping is acceptable as long as it is immediately preceded and followed by a compliant CCV and CCB.

11.2.10. The following analytical sequence must be used with 7471A and 245.5:

Instrument Calibration
ICV
ICB
Maximum 10 samples
CCV
CCB
Repeat sequence of 10 samples between CCV/CCB pairs as required to complete run
CCV
CCB

Refer to Quality Control Section 9.0 and Table II (Appendix A) for quality control criteria to apply to Methods 7471A and 245.5.

Note: Samples include the method blank, LCS, MS, MSD, duplicate, field samples and sample dilutions.

11.2.11. The following run sequence is consistent with 7471A, CLP and 245.5 and may be used as an alternate to the sequence in 11.2.10. This run sequence is recommended if multiple method requirements must be accommodated in

one analytical run:

Instrument Calibration
ICV
ICB
CRA*
CCV
CCB
10 samples
CCV
CCB
Repeat sequence of 10 samples between CCV/CCB pairs as required to complete run.
CCV
CCB

Refer to the appropriate CLP SOP (CORP-MT-0008) for quality control requirements for QC samples.

* Refer to the CLP SOP for information on the CRA.

11.2.12. For TCLP samples, full four point MSA will be required if all of the following conditions are met:

1) recovery of the analyte in the matrix spike is not at least 50%,

2) the concentration of the analyte does not exceed the regulatory level, and,

3) the concentration of the analyte is within 20% of the regulatory level.

Appendix E provides guidance on performing MSA analyses. For TCLP mercury determinations, MSA spikes must be added prior to sampe preparation.

11.3. To facilitate the early identification of QC failures and samples requiring rerun it is strongly recommended that sample data be reviewed periodically throughout the run.

11.4. Guidelines are provided in the appendices on procedures to minimize contamination of samples and standards, preventive maintenance and troubleshooting.

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

radioactivity, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using a Nonconformance Memo and is approved by a Technical Specialist and QA Manager. If contractually required, the client shall be notified. The Nonconformance Memo shall be filed in the project file.

11.6. Any unauthorized deviations from this procedure must also be documented as a nonconformance, with a cause and corrective action described.

12. DATA ANALYSIS AND CALCULATIONS

12.1. ICV percent recoveries are calculated according to the equation:

$$\%R = 100\left(\frac{Found(ICV)}{True(ICV)}\right)$$

12.2. CCV percent recoveries are calculated according to the equation:

$$\%R = 100\left(\frac{Found(CCV)}{True(CCV)}\right)$$

12.3. Matrix spike recoveries are calculated according to the following equation:

$$\%R = 100\left(\frac{SSR - SR}{SA}\right)$$

Where:
SSR = Spike Sample Result
SR = Sample Result
SA = Spike Added

12.4. The relative percent difference (RPD) of matrix spike/matrix spike duplicates or sample duplicates are calculated according to the following equations:

$$RPD = 100\left[\frac{|MSD - MS|}{\left(\frac{MSD + MS}{2}\right)}\right]$$

Where:
MS = determined spiked sample concentration

MSD = determined matrix spike duplicate concentration

$$RPD = 100\left[\frac{|DU1 - DU2|}{\left(\frac{DU1 + DU2}{2}\right)}\right]$$

Where:
DU1 = Sample result
DU2 = Sample duplicate result

12.5. For automated determinations, the final concentration determined in solid samples when reported on a dry weight basis is calculated as follows:

$$mg/kg,\ dry\ weight = (C \times V \times D)/(W \times S)$$

Where:
C = Concentration (ug/L) from instrument readout
V = Volume of digestate (L)
D = Instrument dilution factor
W = Weight in g of wet sample digested
S = Percent solids/100

Note: A Percent Solids determination must be performed on a separate aliquot when dry weight concentrations are to be reported. If the results are to be reported on a wet weight basis, the "S" factor should be omitted from the above equation.

12.6. For manual (total) determinations, the final concentration determined in solid samples when reported on a dry weight basis is calculated as follows:

$$mg/kg,\ dry\ weight = (C)/(W \times S)$$

Where:
C = Concentration (ug) from instrument readout
W = Weight in g of wet sample digested
S = Percent solids/100

Note: A Percent Solids determination must be performed on a separate aliquot when dry weight concentrations are to be reported. If the results are to be reported on a wet weight basis, the "S" factor should be omitted from the above equation.

12.7. The LCS percent recovery is calculated according to the following equation:

$$\%R = 100\left(\frac{Found(LCS)}{True(LCS)}\right)$$

12.8. Sample results should be reported with up to three significant figures in accordance with the Quanterra significant figure policy.

13. METHOD PERFORMANCE

13.1. Each laboratory must have initial demonstration of performance data on file for each analyte of interest as described in Section 9.0.

13.2. Method performance is determined by the analysis of method blank, laboratory control sample, matrix spike and matrix spike duplicate samples. The matrix spike recovery should fall within +/- 25 % and the matrix spike duplicates should compare within 20% RPD. The method blanks must meet the criteria in Section 9.3. The laboratory control sample should recover within 20% of the true value until in house limits are established.

13.3. Training Qualification:

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

14. POLLUTION PREVENTION

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

15. WASTE MANAGEMENT

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

16. REFERENCES

16.1. Test Methods for Evaluating Solid Waste , Physical/Chemical Methods, SW-846, 3rd Edition, Final Update II, Revision I, September 1994, Method 7471A (Mercury).

16.2. "Methods for the Chemical Analysis of Water and Wastes", EPA-600/4-79-020, U.S.EPA, August 1983, Method 245.5.

16.3. U.S.EPA Statement of Work for Inorganics Analysis, ILMO3.0.

16.4. QA-003, Quanterra QC Program.

16.5. QA-004, Rounding and Significant Figures.

16.6. QA-005, Method Detection Limits.

17. MISCELLANEOUS (TABLES, APPENDICES, ETC. . .)

17.1. Modifications/Interpretations from reference method.

17.1.1. Modifications from both 7471A and 245.5.

17.1.1.1. A potassium persulfate oxidation step has been included to facilitate the breakdown of organic mercurials which are not completely oxidized by potassium permanganate. Use of potassium persulfate in combination with the permanganate improves the recovery of mercury from organo-mercury compounds. The use of persulfate has been incorporated in several recent EPA mercury protocols.

17.1.1.2. The alternate run sequence presented in Section 11.2.11 is consistent with method requirements. An additional QC analysis (CRA) was added to accommodate the CLP protocol requirements.

17.1.2. Modifications from Method 7471A

17.1.2.1. Chapter 1 of SW846 specify the use of reagent water with a purity equivalent to ASTM Type II water. This SOP specifies the use of a Millipore DI system or equivalent to produce reagent water. This SOP requires that reagent water must be free of the analytes of interest as demonstrated through the analysis of method blanks.

17.1.2.2. Chapter 1 of SW-846 states that the method blank should not contain any analyte of interest at or above the MDL. This SOP states that the method blank must not contain any analyte of interest at or above the reporting limit.

17.1.3. Modifications from 245.5

17.1.3.1. Method 245.5 Section 9.3 states concentrations should be reported as follows: Between 0.1 and 1 ug/g, to the nearest 0.01 ug; between 1 and 10 ug/g, to the nearest 0.1ug; above 10 ug/g, to the nearest ug. Quanterra reports all Hg results under this SOP to two significant figures.

17.2. Modifications from previous SOP

None.

17.3. Facility Specific SOPs

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

17.4. Documentation and Record Management

The following documentation comprises a complete CVAA raw data package:

- Raw data (direct instrument printout)
- Run log printout from instrument software where this option is available or manually generated run log. (A bench sheet may be substituted for the run log as long as it contains an accurate representation of the analytical sequence).
- Data review checklist - See Appendix B
- Standards Documentation (source, lot, date).
- Copy of digestion log.
- Non-conformance summary (if applicable).

Figure 1. Solid Sample Preparation for Mercury - Autoclave Procedure

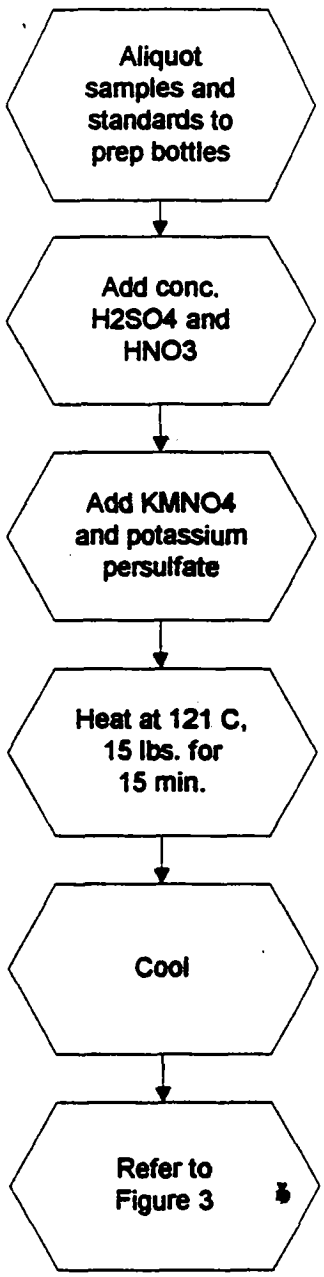


Figure 2. Solid Sample Preparation for Mercury - Water Bath Procedure

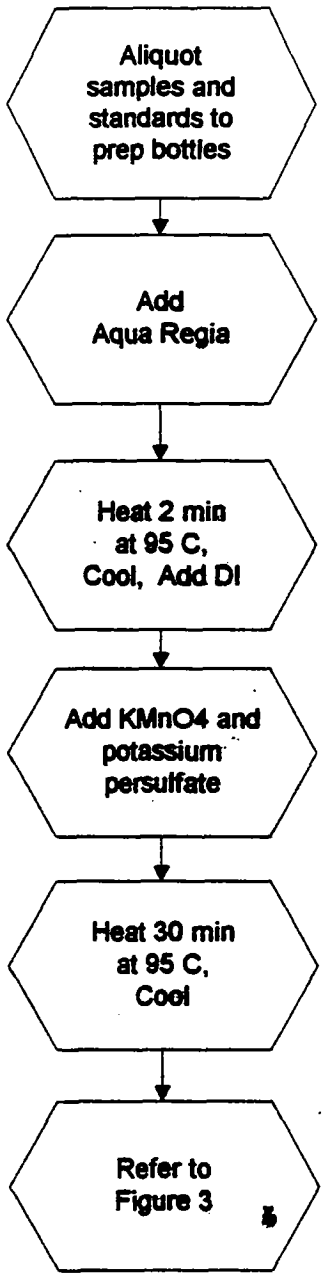
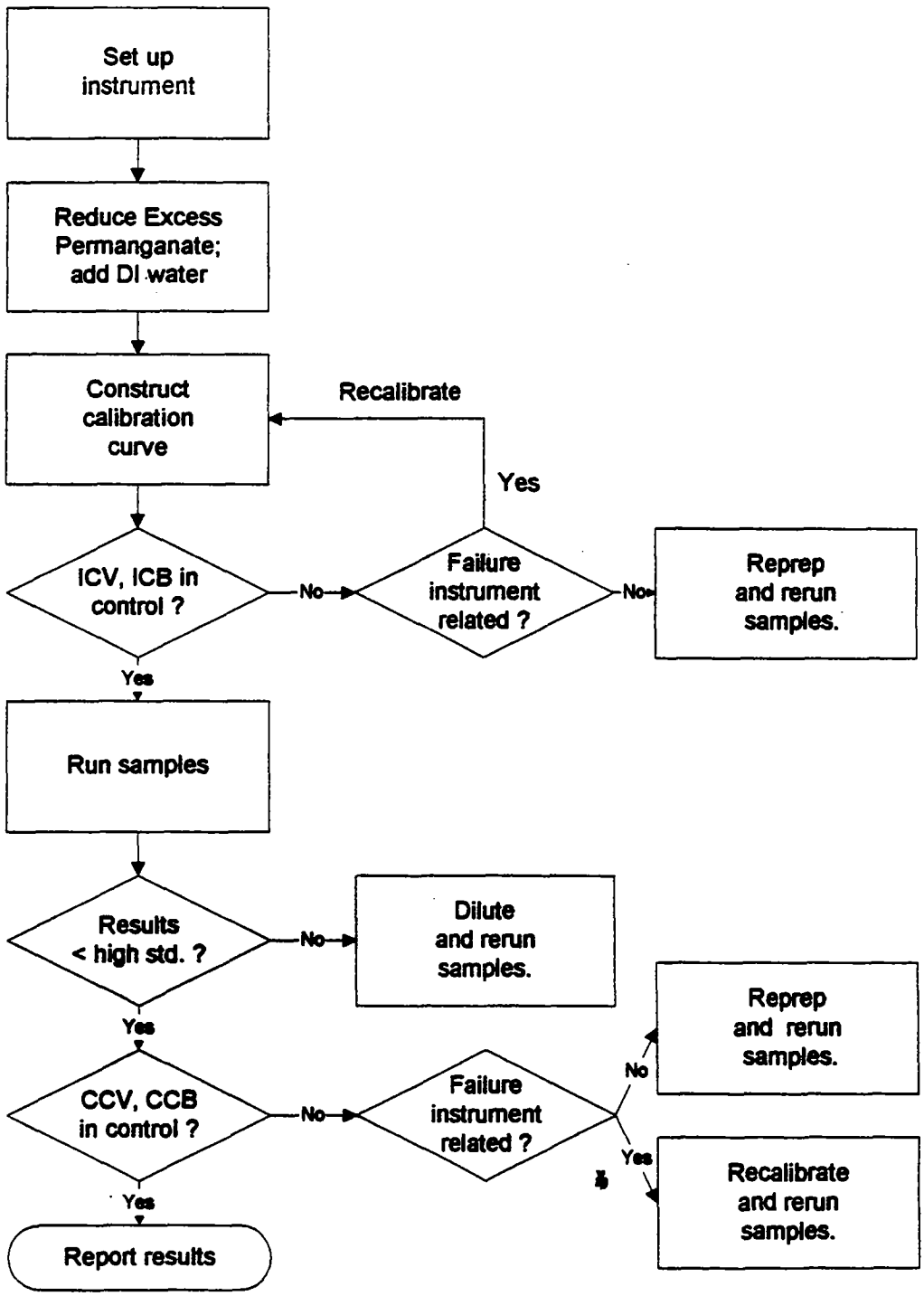


Figure 3. CVAA Mercury Analysis



APPENDIX A

TABLES

TABLE I. MERCURY REPORTING LIMITS, CALIBRATION STANDARD*, QC STANDARD AND SPIKING LEVELS

Soil RL (mg/kg)	0.1
Std 0 (mg/L)	0
Std 1 (mg/L)	0.0002
Std 2 (mg/L)	0.0005
Std 3 (mg/L)	0.001
Std 4 (mg/L)	0.002
Std 5 (mg/L)	0.005
Std 6 (mg/L) **	0.010
ICV (mg/L)	0.001 or 0.0025 ***
CCV/LCS (mg/L)	0.0025 or 0.005 ***
MS (mg/L)	0.001

* **SOP specified calibration levels must be used unless prevented by the instrument configuration or client specific requirements. Deviations from specified calibration levels must be documented in the facility specific instrument operation SOP and must be approved by the facility technical manager and Quality Assurance Manager.**

** **Optional standard which may be used to extend the calibration range as allowed by the instrument configuration. If the instrument configuration prevents the use of 6 standards, the 2 ppb standard may be eliminated in favor of the 10 ppb standard.**

*** **Concentration level dependent on high calibration standard used. CCV must be 50% of the high standard concentration and the ICV must be 20-25% of the high standard concentration.**

TABLE II. Summary Of Quality Control Requirements

QC PARAMETER	FREQUENCY *	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
ICV	Beginning of every analytical run.	80 - 120 % recovery.	Terminate analysis; Correct the problem; Recalibrate or reprep batch (see Section 9.6).
ICB	Beginning of every analytical run, immediately following the ICV.	The result must be within +/- RL from zero.	Terminate analysis; Correct the problem; Recalibrate or reprep batch (see Section 9.6).
CCV	Every 10 samples and at the end of the run.	80 - 120 % recovery.	Terminate analysis; Correct the problem; Recalibrate and rerun all samples not bracketed by acceptable CCV or reprep batch (see Section 9.7).
CCB	Immediately following each CCV.	The result must be within +/- RL from zero.	Terminate analysis; Correct the problem; Recalibrate and rerun all samples not bracketed by acceptable CCB or reprep batch (see Section 9.7).
Method Blank	One per sample preparation batch of up to 20 samples.	The result must be less than or equal to the RL. Sample results greater than 20x the blank concentration are acceptable. Samples for which the contaminant is < RL do not require redigestion (See Section 9.3).	Redigest and reanalyze samples. Note exceptions under criteria section. See Section 9.3 for additional requirements.

*See Sections 11.2.10 and 11.2.11 for exact run sequence to be followed.

TABLE II. Summary of Quality Control Requirements (Continued)

QC PARAMETER	FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
Laboratory Control Sample (LCS)	One per sample preparation batch of up to 20 samples.	Aqueous LCS must be within 80 - 120% recovery or in-house control limits.	Terminate analysis; Correct the problem; Redigest and reanalyze all samples associated with the LCS (see Section 9.4).
Matrix Spike	One per sample preparation batch of up to 20 samples.	75 - 125 % recovery or in-house control limits. If the MS/MSD is out for an analyte, it must be in control in the LCS.	In the absence of client specific requirements, flag the data; no flag required if the sample level is > 4x the spike added. (see Section 9.5) For TCLP see Section 11.3.12
Matrix Spike Duplicate	See Matrix Spike	75 - 125 % recovery or in-house control limits; RPD ≤ 20%. (See MS)	See Corrective Action for Matrix Spike.

APPENDIX B
QUANTERRA Hg DATA REVIEW CHECKLIST

Quanterra Hg Data Review Checklist

Run/Project Information

Run Date: _____ **Analyst:** _____ **Instrument:** _____
Prep Batches Run: _____

Circle Methods used: **7470A / 245.1 : CORP-MT-0005 Rev 0** **7471 / 245.5 : CORP-MT-0007 Rev 0**
CLP - AQ : CORP-MT-0006 Rev 0 **CLP - SOL : CORP-MT-0008 Rev 0**

Review Items

A. Calibration/Instrument Run QC	Yes	No	N/A	2ndLevel
1. Instrument calibrated per manufacturer's instructions and at SOP specified levels ?				
2. ICV/CCV analyzed at appropriate frequency and within control limits?				
3. ICB/CCB analyzed at appropriate frequency and within +/- RL or +/- CRDL (CLP)?				
4. CRA run (CLP only)?				
B. Sample Results				
1. Were samples with concentrations > the high calibration standard diluted and reanalyzed?				
2. All reported results bracketed by in control QC ?				
3. Sample analyses done within holding time?				
C. Preparation/Matrix QC				
1. LCS done per prep batch and within QC limits ?				
2. Method blank done per prep batch and < RL or CRDL (CLP) ?				
3. MS run at required frequency and within limits ?				
4. MSD or DU run at required frequency and RPD within SOP limits?				
D. Other				
1. Are all nonconformances documented appropriately ?				
2. Current IDL/MDL data on file?				
3. Calculations and Transcriptions checked for error ?				
4. All client/ project specific requirements met?				
5. Date of analysis verified as correct ?				

Analyst: _____ **Date:** _____
Comments:

_____

_____

2nd Level Reviewer : _____ **Date:** _____

_____

_____

APPENDIX C

MSA GUIDANCE

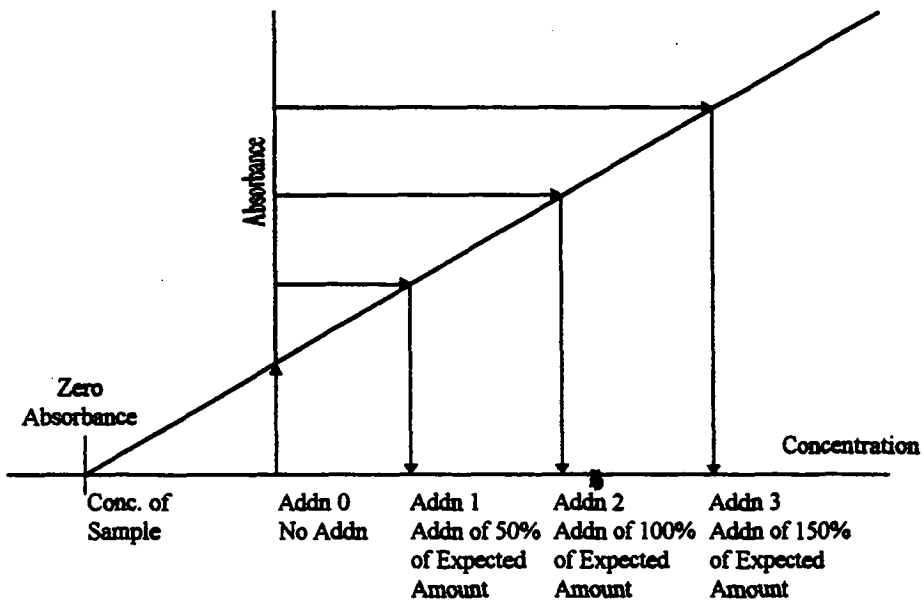
APPENDIX C. MSA GUIDANCE

Method of Standard Addition

Four equal volume aliquots of sample are measured and known amounts of standards are added to three aliquots. The fourth aliquot is the unknown and no standard is added to it. The concentration of standard added to the first aliquot should be 50% of the expected concentration. The concentration of standard added to the second aliquot should be 100% of the expected concentration and the concentration of standard added to the third aliquot should be 150% of the expected concentration. The volume of the unspiked and spiked aliquots should be the same (i.e., the volume of the spike added should be negligible in relation to the volume of sample).

To determine the concentration of analyte in the sample, the absorbance (or response) of each solution is determined and a linear regression performed. On the vertical axis the absorbance (or response) is plotted versus the concentrations of the standards on the horizontal axis using 0 as the concentration of the unspiked aliquot. An example plot is shown in Figure 1. When the resulting line is extrapolated back to zero absorbance, the point of interception of the horizontal axis is the concentration of the unknown. Calculate the correlation coefficient (r) and the x-intercept (where y=0) of the curve. The concentration in the digestate is equal to the negative x-intercept.

Figure 1



- For the method of standard additions to be correctly applied, the following limitations must be taken into consideration.
- The plot of the sample and standards must be linear over the concentration range of concern. For best results, the slope of the curve should be similar to that of a plot of the aqueous standard curve.
- The effect of the interference should not vary as the ratio of the standard added to the sample matrix changes.

APPENDIX D

TROUBLESHOOTING GUIDE

APPENDIX D. TROUBLESHOOTING GUIDE

Problem	Possible Cause
Poor or No Absorbance or Sensitivity Check failed	Incorrect wavelength Dirty windows Window loose Etched or dirty optics Wrong lamp Bad lamp Not enough or no sample introduced Empty sample cup Incorrectly made standards Gas leak EDL power supply set on "Continuous"
Erratic Readings	Source lamp not aligned properly Lamp not prewarmed Injection tip partially clogged Contaminated reagents Contaminated glassware Drying tube saturated Bad lamp Injection tip hitting outside of tube Injection tip coated or not set properly Leak in sample tubing Power fluctuations Air bubbles in tubing
EDL Won't Light	Lamp cable not plugged in Lamp power set at 0 Lamp is dead Power supply fuse is blown Short in cord
Standards reading twice or half normal absorbance or concentration	Incorrect standard used Incorrect dilution performed Dirty cell
Background Correction Light Blinking	Background screen or attenuator faulty

APPENDIX E

CONTAMINATION CONTROL GUIDELINES

APPENDIX E. CONTAMINATION CONTROL GUIDELINES

The following procedures are strongly recommended to prevent contamination:

All work areas used to prepare standards and spikes should be cleaned before and after each use.

All glassware should be washed with detergent and tap water and rinsed with 1:1 nitric acid followed by deionized water.

Proper laboratory housekeeping is essential in the reduction of contamination in the metals laboratory. All work areas must be kept scrupulously clean.

Powdered or Latex Gloves must not be used in the metals laboratory since the powder contains silica and zinc, as well as other metallic analytes. Only vinyl or nitrile gloves should be used in the metals laboratory.

Glassware should be periodically checked for cracks and etches and discarded if found. Etched glassware can cause cross contamination of any metallic analytes.

Autosampler trays should be covered to reduce the possibility of contamination. Trace levels of elements being analyzed in the samples can be easily contaminated by dust particles in the laboratory.

The following are helpful hints in the identification of the source of contaminants:

Reagents or standards can contain contaminants or be contaminated with the improper use of a pipette.

Improper cleaning of glassware can cause contamination.

Separate glassware if an unusually high sample is analyzed and soak with sulfuric acid prior to routine cleaning.

APPENDIX F

PREVENTIVE MAINTENANCE

APPENDIX F. PREVENTIVE MAINTENANCE

A maintenance log is used to record when maintenance is performed on instruments. When an instrument problem occurs indicate the date, time and instrument number, then identify the problem and corrective action in the maintenance log.

The following procedures are required to ensure that that the instrument is fully operational.

Cold Vapor Atomic Absorption (Leeman PS 200) [1]

Daily	Semi-annually	Annually
Clean lens.	Check Hg lamp intensity.	Change Hg lamp.
Check aperture.		Check liquid/gas separator.
Check argon flow.		
Check tubing.		
Check drain.		
Replace drying tube.		

Cold Vapor Atomic Absorption (PE 5000) [1]

Daily	Monthly
Clean aspirator by flushing with DI water.	Clean cell in aqua regia.
Check tubing and replace if needed.	Clean aspirator in aqua regia.
Clean windows with methanol.	
Change silica gel in drying tube.	
Check argon gas supply.	
Adjust lamp.	

INTERNATIONAL TECHNOLOGY CORPORATION

IT Analytical Services

Knoxville Laboratory Standard Operating Procedure

Title: **Receipt and Log-in of Commercial Samples**

Prepared by: Rebecca L. Charles Date: 12/1/93

Reviewed by: Robyn M. Wagner Date: 11/30/93
Technical Specialist

Date: 12/01/93
Quality Control Coordinator

Date: 12/1/93
Director, Quality and Compliance, ITAS

Date: 12-1-93
Director, Health and Safety, ITAS

Approved by Date: 12-1-93
Laboratory Director

UNCONTROLLED COPY

Controlled Copy No: _____

Key Words: **Sample Receipt, Log-in, Commercial Samples**

Revision #	0					
Date:	11/01/93					

Title: Receipt and Log-in of Commercial Samples

1. **Purpose, Application, and Responsibility**

1.1 The purpose of this procedure is to provide instructions for the receipt and log-in of commercial samples.

1.2 This procedure is applicable to samples received from all clients except the USEPA CLP.

1.3 It is the responsibility of the receiving and log-in group leader or designee to act as sample custodian.

1.4 It is the responsibility of the group leader, or designee, to train receiving personnel in the performance of this procedure and delegate the performance of this procedure to personnel who are qualified in the conduct of this procedure.

1.5 It is the responsibility of the qualified analyst to follow this procedure, to report deviations from this procedure, and to perform this procedure independently only when properly qualified.

1.6 It is the responsibility of the project manager, or designee, to provide a Quality Assurance Summary for all samples received.

2. **References**

2.1 ITAS Quality Assurance Management Plan, current revision.

2.2 ITAS Operations Specific Quality Assurance Management Plan, current revision.

3. **Associated SOPs**

3.1 ITAS-KN-QC-0005, "Nonconformance and Corrective Action".

3.2 ITAS-KN-AD-0002, "Internal Chain-of-Custody and Sample Security for ITAS-Knoxville".

3.3 ITAS-OR-2307, "Sample Receipt and Radioactive Material Receipt/Shipment Surveys".

3.4 ITAS-OR-5006, "Sample Preservation Verification".

3.5 ITAS-KN-QC-0007, "QC Sample Initiation and Documentation".

3.6 ITAS-KN-QC-0010, "Temperature Monitoring".

4. **Definitions**

4.1 Sample Delivery Group (SDG): A sample delivery group is defined by one of the following, whichever occurs first:

- A case; or
- 20 field samples within a case; or
- Each 14-day calendar period (30 days for radiological analysis) during which field samples in a case are received, beginning with receipt of the first sample in the case or SDG.

4.2 RFA/COC: Request for Analysis/Chain of Custody form.

4.3 Client Sample Number: A unique identification number designated by a client for each sample. The sample number appears on the sample RFA/COC.

4.4 Time - When required to record time on any deliverable item, time shall be expressed as Military Time, i.e., a 24-hour clock.

4.5 Master Sample Receipt Log: A document for tracking the receipt of samples and assigning sequential work order numbers and laboratory sample numbers.

4.6 Sample: Physical evidence collected from a facility or from the environment and may consist of one or more containers.

4.7 CUR: Condition Upon Receipt Variance Report. (See Figure 1)

4.8 WOC: Work order Checklist (See Figure 2)

4.9 Project Codes: Alphanumeric code which consists of a 3 to 4 alphabetic client code followed by a sequential work order number.

4.10 Lab sample number - sequentially assigned unique number identifying client samples

4.11 Commercial - a client other than the USEPA CLP.

5. Procedure

5.1 Summary

5.1.1 Upon receipt, coolers are checked for custody seals. Coolers are opened under a hood. Conditions are checked and documented. SDG groups and QC samples are assigned. Samples are assigned to a work order and laboratory sample numbers are affixed to the containers. Samples are placed in storage locations. All pertinent information is logged into a laboratory computer data base. A summary of the sample log-in along with original paperwork is submitted to the project manager for review.

5.1.2 Qualifications

5.1.2.1 The Associate must have at a minimum an Associate's degree in a scientific discipline or two years work experience in an analytical laboratory in a related area.

5.1.3 Training

5.1.3.1 Initial training on this procedure will consist, at a minimum, of an oral review followed by at least one manual demonstration. Training will be given by personnel already qualified to perform this procedure.

5.1.3.2 The trainee will perform this procedure under the direct supervision of the instructor until acceptable performance is achieved.

5.1.3.3 The instructor will document acceptable performance by completing, signing and dating a checklist consisting of applicable elements of this procedure.

5.1.3.4 The training documentation is retained in the individual's training file.

5.2 Safety

5.2.1 All procedures shall be carried out in a manner that protects the health and safety of all IT associates.

5.2.2 Eye protection that satisfies ANSI Z87.1, laboratory coat, and appropriate gloves must be worn while samples are being handled. Disposable gloves that have become contaminated will be removed and discarded; other gloves will be cleaned immediately.

5.2.3 Exposure to chemicals will be maintained **as low as reasonably achievable**; therefore, unless they are known to be non-hazardous, all samples will be opened, transferred and prepared in a fume hood, or under other means of mechanical ventilation.

5.2.4 All work must be stopped in the event of a known, or potential, compromise to the health or safety of any ITAS associate. This situation must be reported **immediately** to a laboratory supervisor.

5.3 Interferences

5.3.1 None.

5.4 Preservation and Holding Times

5.4.1 Use the preservation and holding times listed in the "Operation Specific Quality Assurance Management Plan" unless otherwise specified by the client.

5.5 Required Equipment

5.5.1 Black ink pen.

5.5.2 Calibrated thermometer.

5.5.3 Computer with log-in software applications.

5.5.4 Sealable storage bags; quart and gallon size.

5.5.5 Labels.

5.5.6 Refrigerators capable of maintaining 4°C $\pm$ 2°C.

5.5.7 Freezer capable of maintaining -10°C to -20°C.

5.6 Reagents and Standards

5.6.1 None.

5.7 Calibration

5.7.1 Thermometers shall be calibrated yearly.

5.8 Operation

5.8.1 Notify the project manager immediately of any Condition upon Receipt Variance Reports (CUR) generated during the log-in process. (See Figure 1)

5.8.2 Upon receipt, verify that the number of packages received agrees with the airbill, waybill or carrier's records.

5.8.3 Verify the presence and condition of custody seals on the cooler or shipping container. Record verification on the Work Order Checklist (WOC). Refer to Figure 2. Document missing or broken custody seals on a CUR.

5.8.4 Open the shipping container under a vented fume hood. If there are noxious fumes, stop work and notify the Health and Safety coordinator. If there are broken or leaking samples that are known or suspected to contain radioactive material, stop work and notify the Radiation Safety Officer.

5.8.5 Check the shipping containers for the Request for Analysis/Chain-of-Custody (RFA/COC) forms and record their presence or absence on the WOC. Document missing, improperly completed or unrelinquished RFA/COC forms on a CUR.

5.8.6 Sign and enter the date and time of sample receipt on the RFA/COC forms and airbills.

5.8.7 Take the temperature of the coolers containing samples that require refrigeration. Insert a calibrated thermometer into the middle of the cooler. After the thermometer has equilibrated, read the temperature.

5.8.8 The acceptable temperature range for refrigerated samples is 2°C to 6°C. Note the temperature on the RFA/COC. Note whether the temperature was within specifications on the WOC. Document samples received out of required temperature specifications on a CUR.

5.8.9 If shipment is received on the weekend, the shipping containers may be moved to a walk-in sample refrigerator until the next business day after the RFA/COC is signed and the cooler temperature taken.

5.8.10 Unpack the cooler; document any broken or leaking containers or volatile samples with headspace on a CUR.

5.8.11 Verify that all information on the sample containers matches the information on the RFA/COC form(s). Record verification on WOC. Document discrepancies in information, missing samples, or additional samples on a CUR.

5.8.12 Document any samples received out of holding time on a CUR.

5.8.13 If samples are known or suspected to contain radioactive material, survey the shipping containers and samples. Refer to SOP ITAS-OR-2307, "Sample Receipt and Radioactive Material Receipt/Shipment Surveys".

5.8.14 Verify the pH of preserved samples (other than samples for volatile organic analyses). Refer to SOP ITAS-OR-5006, "Sample Preservation Verification". Record preservation verification on WOC. Document any samples preserved incorrectly on a CUR.

NOTE: Do not open samples for volatile organic analyses.

5.8.15 Relinquish samples that have been designated for screening to the Health Physics department and enter the required information in the screening log book.

5.8.16 Samples should be grouped in work orders by client, project, day of receipt, SDG group, matrix and/or client requirements. Work order numbers are sequentially assigned. To manually assign work order numbers, enter the work order number on the Master Sample Receipt Log using the next available number. For computer systems that automatically generate work order numbers, work order numbers will be generated at log-in.

5.8.17 Assign project codes if required.

5.8.18 Create a project file to contain all information related to the work order.

5.8.19 Label sample containers with a sequentially assigned lab sample numbers. Label before computer log-in if numbers are manually assigned. Record number range on the

Master Sample Receipt Log. Label after computer log-in if numbers are automatically assigned by the computer data base.

5.8.20 Assign QC and SDG according to SOP ITAS-KN-QC-0007. Include information in project file.

5.8.21 Determine available storage space. Assign samples requiring refrigeration to monitored sample refrigerators. Assign frozen samples to monitored sample freezers. Assign other samples to ambient shelf locations. Samples for volatile organic analysis should be stored in separate refrigerators from other samples.

5.8.22 Enter the work order information into the computer data base according to software documentation. The information entered should include, but is not limited to:

- Project code or Client Name
- Work order number
- Date and Time of receipt
- Due date
- Disposal method and days to disposal
- Contract number and/or Project Name
- The SDG number or QC reference number
- Lab sample number
- Client number
- Analysis requested
- Collection date
- Type and number of containers
- Storage location
- Radiological Category
- Project Manager
- QAS number

5.8.23 Record the project code and work order number on all documents relevant to the project.

5.8.24 Initiate an Internal Chain of Custody form and move samples to assigned storage locations. Shelby tubes for volatile organic analysis should be sealed in an outer container such as a sealable plastic bag.

5.8.25 Generate a printout of the completed sample log-in and include in the project folder.

5.8.26 Complete the WOC and include in project folder.

5.8.27 Include any required screening information, original paperwork and CURs in project folder.

5.8.28 Submit project file for peer review.

5.8.29 Submit project file for project manager review.

5.8.30 Projects requiring corrections are submitted back to the Receiving and Log-in group along with the Project Review Checklist with any required corrections indicated in the "action needed" section

5.8.31 Correct the log-in and complete the Corrected Project Coversheet.

5.8.32 Resubmit the project file to the project manager.

5.9 Calculations

5.9.1 None.

5.10 Quality Control

5.10.1 The sample log-in group peer reviews all sample delivery documents and signs and dates the project folder.

5.10.2 The project manager reviews all the documents in the project file and verifies that the client's samples have been properly logged in.

6. Nonconformance and Corrective Action

6.1 One time procedural variations are allowed only if deemed necessary in the professional judgement of supervision to accommodate variation in sample matrix, radioactivity, chemistry, sample size or other parameters. Any variation in procedure shall be completely documented using a Nonconformance Memo and approved by the Technical Director and QA/QC Coordinator. If contractually required, the client will be notified. The Nonconformance Memo will be filed in the project file.

6.2 Any unauthorized deviations from this procedure must be documented as a nonconformance, with a cause and corrective action described. An ITAS Nonconformance Memo shall be used for this documentation. The original Nonconformance Memo will be filed in the

project file.

6.3 The following is a list of some possible nonconformances:

6.3.1 Cooler temperatures not taken or recorded.

6.3.2 RFA/COC not signed or dated by receiving personnel.

6.3.3 Sample variances not documented on a CUR form.

6.3.4 Samples dropped or broken during receiving.

6.3.5 Samples stored improperly.

6.3.6 Airbills not signed.

7. Records Management and Documentation

7.1 Documentation that an individual is authorized to perform this procedure independently will be retained in the individual's training file. Authorization will be designated by the supervisor's or instructor's signature and the date on the training documentation.

7.2 All receiving documents, Sample Receipt Log, and all other relevant documents including CURs and WOCs must be filed in the project file.

7.3 Each logbook is checked and verified by the group leader or designee routinely.

7.4 All logbooks are controlled by the laboratory QA/QC Coordinator.

FIGURE 1.0

INTERNATIONAL TECHNOLOGY CORPORATION

Work Order No.:_____

Condition Upon Receipt Variance Report
ITAS - Knoxville Laboratory/Middlebrook Facility

Client:_____ Date:_____

Project No:_____ Initiated by:_____

Analysis Requested:_____ RFA/COC Numbers:_____

Client Sample Numbers Affected:_____

Condition/Variance (Check all that apply):

1. ☐ Not enough sample received for proper analysis.
   Received approximately:_____
2. ☐ Sample received broken/leaking.
3. ☐ Sample received without proper preservative.
   - ☐ Cooler temperature not within 4°C ± 2°C
     Record temperature:_____
   - ☐ pH_____
   - ☐ other:_____
4. ☐ Sample received in improper container.
5. ☐ Sample received without proper paperwork. Explain:
   _____
6. ☐ Paperwork received without sample.
7. ☐ No sample ID on sample container.
8. ☐ Custody tape disturbed/broken/missing.
9. ☐ Sample splits performed by lab.
10. ☐ Volatile sample received with approximately _____ mm headspace.
11. ☐ Sample ID on container does not match sample ID on paperwork. Explain: _____
    _____
    _____
12. ☐ All coolers on airbill not received with shipment.
13. ☐ Other (explain below):
    _____
    _____
    _____

Notes:
_____
_____

Corrective Action:

☐ Client's Name: _____ Informed verbally on: _____ By: _____

☐ Client's Name: _____ Informed in writing on: _____ By: _____

☐ Sample(s) processed "as is". Comments: _____

☐ Sample(s) on hold until: _____ If released, notify: _____

Sample Control Supervisor Review:_____ Date: _____

Project Management Review: _____ Date: _____

SIGNED ORIGINAL MUST BE RETAINED IN THE PROJECT FILE

FIGURE 2.0
ITAS KNOXVILLE LABORATORY

WORK ORDER CHECKLIST FOR SAMPLE RECEIVING AND REPORTING

CLIENT:_____ WORK ORDER NO.:_____

SAMPLE RECEIPT					
DATE RECEIVED:			TIME:		
CONDITION	Y	N	CONDITION	Y	N
RELINQUISHED COC & RFA W/SHIPMENT?			ALL SAMPLES LISTED ON RFA/COC RECEIVED?		
QAS AVAILABLE AT RECEIPT?			SAMPLES PROPERLY PRESERVED?		
DO SAMPLE LABELS & COC MATCH?			CUSTODY SEALS PRESENT & INTACT?		
TEMPERATURE WITHIN SPECIFICATION?					
IF NO WAS CHECKED FOR ANY OF THE ABOVE, A "CONDITION UPON RECEIPT" VARIANCE FORM MUST BE INITIATED. CUR NUMBER (WHERE APPLICABLE): _____					
SUBMITTED TO PM: Signature:			Date & Time:		
RELEASED BY PM: Signature:			Date & Time:		
COMMENTS:					

WORK ORDER INDEX CHECKLIST			
SECTION	RECORD DESCRIPTION	CHECK Y, N, OR N/A	IF NO: COMMENT
A	CORRESPONDENCE		
B	REQUEST FOR ANALYSIS		
	CHAIN-OF-CUSTODY		
	SCREENING RECORDS		
	WORK ORDER PRICED AT LOG-IN		
	CONTRACTOR RECORDS		
C	SAMPLE DATA		
	QC DATA		
	SUBCONTRACTOR DATA		
D	DATA REPORT		
	EDT		
E	NONCONFORMANCE MEMOS		
	CONDITION UPON RECEIPT VARIANCES		

OPERATION SPECIFIC STANDARD OPERATING PROCEDURE

TITLE: DOCUMENT STORAGE

(SUPERSEDES: KNOX-AD-0001, Revision 0)

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1.0 Purpose

1.1 The purpose of this procedure is to describe the requirements for record transmittal, on-site record storage prior to off-site transfer, off-site storage and retrieval of documents and records at the Quanterra, Knoxville Laboratory.

1.2 This procedure applies to all quality and project documents and records either received or produced at the Quanterra, Knoxville Laboratory.

2.0 Responsibilities

Position	Responsibility
Records Management Facilitator (RMF)	• To train laboratory personnel on the requirements of the Records Management Policy • To maintain records for those not assigned to or controlled by other individuals in the laboratory, as specified by the Quanterra records retention schedule. • To periodically assess the status of the document transmittal, storage and retrieval process. • To receive, verify receipt of, store and retrieve records and documents.
Records Management Team (RMT), composed of the laboratory's Group Leaders, Team Leaders, Project Managers, Management and Administrative Supervisors	• To perform a record survey of all records utilized by the laboratory • To ensure that all quality, operations, and project records that are received or produced in their respective departments are properly identified and maintained while in use. • To ensure that all quality, operations, and project records are properly transmitted to Records Management when the records are no longer in use.
Laboratory Personnel	• To safeguard all records and documents from damage and loss. • To properly identify and maintain all records and documents while in use. • To retain records in compliance with the most current Quanterra Record Retention Schedule and for certifying their compliance annually.
Regional Records Management Team, Regional Contract Compliance Officer (RCCO), or designee	• To conduct annual compliance audits to verify compliance to the Quanterra Records Management Policy.

3.0 Safety

3.1 Normal office dependent safety precautions must be taken in performing this SOP. If personnel are required to perform any portion of the procedure in laboratory areas, appropriate personal protective equipment and precautions must be utilized.

3.2 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.

3.3 Bankers boxes (greater than 12 1/2" x 15 1/2" x 10 1/2") shall not be utilized for the storage and archival of inactive records. Larger boxes filled with paper exceed the weight limit requirement of 60 pounds.

4.0 Procedure

4.1 Quanterra Records Management Policy

4.1.1 Each Quanterra associate is required to attend a records awareness session that will describe the Quanterra Records Management Policy and its implementation.

4.1.2 A Records Management Team will be established utilizing management personnel from each department at the laboratory.

4.1.3 A Records Management Facilitator (RMF) shall be selected from within the laboratory to oversee the implementation and maintenance of the Quanterra Records Management Policy for the laboratory.

4.1.4 A Records Survey (see example in Appendix I) shall be completed by each department to determine the type of records that are utilized by the department and to establish the retention period that is applied to each individual type of record.

4.1.5 The retention period for each record shall be obtained from the Quanterra Record Retention Schedule (Appendix II). Retention periods may be superseded by corporate requirements.

NOTE: Any discrepancies in retention periods that are identified will not be changed at the regional level without approval by the Quanterra Contract Compliance Officer.

4.1.6 Records shall be destroyed as specified in the retention schedule (or as superseded by a contract requirement) when their retention period ends. The anniversary date used for record retention shall be the end of the calendar year based on the year of generation.

4.1.7 An annual Clean Up Day shall be scheduled for the destruction of records.

4.1.8 Destruction of records shall be documented on a Certificate of Record Destruction (see example in Appendix III) when destroyed by any person other than the record originator. The person supervising the destruction of records is responsible for completing this certificate.

4.1.9 No records shall be destroyed when litigation, government investigation or audit is underway (pending or imminent). Whenever a subpoena or demand for discovery is in effect, the RCCO and RMF shall receive notice from the Legal Department and shall in turn notify any appropriate regional personnel of any records to be retained for potential production or disclosure. Records held for litigation or audit may be released only by the organization initiating the hold, by the use of a Request for Destruction of Records, acknowledged in writing. Refer to Suspension of Destruction (Appendix VIII).

4.1.10 The Certificate of Compliance (see example in Appendix IV) will serve as evidence of proper maintenance of records, including the proper destruction of records, for the originator of the record. Each Quanterra associate is required to complete a Certificate of Compliance.

4.1.11 For additional guidance on record disposition, refer to General Guidance on Records Disposition (Appendix IX).

4..2 On-site Document Storage

NOTE Electronic records shall be stored in a manner to protect them from fire, moisture and proximity to strong magnetic fields. Examples include fire-proof file cabinets or a fire rated storage facility. Refer to KNOX-IS-0002 Revision 0 "Electronic Data and Software Backup" for filing system to be utilized.

4.2.1 On-site document storage is maintained in the Quanterra, Knoxville laboratory safe, which is a concrete, fire-proof enclosure with approximately 226 square feet of storage space.

4.2.2 Each laboratory associate shall review their records and determine the identity of records ready to be transferred to the safe.

4.2.3 Records that are ready to be transferred from a laboratory area to the safe are placed into a records storage box and labelled with a unique identification number by the group or person submitting the box.

4.2.4 A Record Transfer Form (see example in Appendix V) must be completed for each box.

4.2.5 The Record Transfer Form shall be reviewed by the group leader or designee prior to the records being moved and copies of the form(s) being made. This review shall be documented by the reviewer signing (or initialing) and dating the Records Transfer Form.

4.2.6 Two copies of the Record Transfer Form are to be made.

4.2.7 The original and copies of the Record Transfer Form are distributed as follows:

- Original - Records Management Facilitator
- Copy - placed inside appropriate box
- Copy - retained by originating department

4.2.8 The RMF, Reporting Group Supervisor or designee is notified that boxes are ready for transfer to the safe.

4.2.9 The RMF, Reporting Group Supervisor or designee determines the capacity of the safe prior to the boxes being moved.

4.2.10 If the safe is filled to capacity, then boxes currently in the safe are assigned an offsite storage box number and prepared for shipment to offsite storage.

4.2.11 If the safe is not filled to capacity, then the boxes from the laboratory will be moved to the safe.

4.2.12 When records are to be checked out of the safe, the associate shall obtain the key from the RMF, the Reporting Group Supervisor or designee. The associate shall document this key check out and the reason for records access on a key sign out form. The RMF, the Reporting Group Supervisor or designee have the responsibility and authority to disallow access to associates who do not provide sufficient reason for records access.

NOTE: Records that have been removed from the safe **must** be returned within **30 days.**

4.2.13 The associate is responsible for ensuring the the signout form is completed and that the key is returned upon completion.

4.2.14 The Archived Data Retrieval Log (see example in Appendix VI) shall be completed for each record removed from the safe.

4.2.15 A signout card shall be completed and placed in the space from which the data was removed.

4.2.16 Upon return of each record, the Archived Data Retrieval Log shall be completed and the signout card removed.

4.2.17 The associate that signed the Archived Data Retrieval Log is responsible for ensuring that the record is returned to the appropriate box.

4.2.18 If it becomes necessary to add additional records to a box that has already been placed in the safe, the following actions shall be utilized:

4.2.18.1 For the addition of an individual record (i.e. - revised case narrative, revised electronic data transfer, etc.) to a work order folder, refer to KNOX-AD-0002 "Project Records" for the necessary requirements.

4.2.18.2 For the addition of a data folder, a modified Record Transfer Form signed by the associate making the addition shall be submitted to the RMF.

4.2.18.3 Distribution of the modified form will follow the same procedure as the original distribution. The RMF or designee will file the modified Record Transfer Form in

the Record Transfer Form Log along with the original Record Transfer Form.

4.3 Offsite Document Storage

4.3.1 When the safe is filled to capacity, boxes currently in the safe are assigned an offsite storage box number and prepared for shipment to offsite storage.

4.3.2 The RMF or designee will ensure that the Record Transfer Form (and modified Record Transfer Form, if applicable) is in the box and all records listed on the Record Transfer Form are in the box.

4.3.2.1 If any records are missing from the box, the RMF or designee will review the Archived Data Retrieval Log and contact the responsible associate to determine the location of the record in question.

4.3.3 The RMF or designee will sign and date as Records Information Management (RIM) Authorization on the original Records Transfer Form (or modified Record Transfer Form, if applicable). This form will be filed by the RMF or designee by the date the form was originated.

4.3.4 Records are then shipped to offsite storage.

4.3.4.1 The offsite storage location in use is a document storage facility located at International Technology Corporation, Environmental Technology Development Center, 1570 Bear Creek Road, Kingston, Tennessee 37763.

4.3.4.2 This secure facility was designed and built to meet NQA-1 construction and fire protection requirements for record storage facilities. The facility is equipped with a halon fire protection system. Keys to this facility are controlled by the RMF and Facilities Manager or designee.

4.3.4.3 With the approval of the RMF and Quanterra's Manager

of Records Information Management Services, a commercial offsite records center may be used.

4.3.5 The RMF or designee shall be contacted for retrieval of records from offsite storage. A Data Retrieval From Document Storage Form (see example Appendix X) shall be provided to the RMF. A time will be established for retrieval of requested records from offsite storage.

NOTE To retrieve records from offsite storage, a **one week** time period is required.

4.3.6 When offsite storage records are scheduled for destruction, a Notice of Impending Records Destruction form (see example in Appendix VII) will be completed.

4.3.7 A copy of the Notice of Impending Records Destruction form will be forwarded to the appropriate supervisor for review.

4.3.8 Records will be destroyed on the scheduled date unless the appropriate supervisor responds within **30 days**. If no response is received, the records will be destroyed per the Quanterra Record Retention Policy.

4.3.9 No records shall be destroyed when litigation, government investigation or audit is underway (pending or imminent). Whenever a subpoena or demand for discovery is in effect, the RCCO and RMF shall receive notice from the Legal Department and shall in turn notify any appropriate regional personnel of any records to be retained for potential production or disclosure. Records held for litigation or audit may be released only by the organization initiating the hold, by the use of a Request for Destruction of Records, acknowledged in writing. Refer to Suspension of Destruction (Appendix VIII).

5.0 Definitions

5.1 Active Records - Those records that are referenced frequently in the daily operations of the business.

5.2 Administrative Value - The period of time a record may be needed within the company for administrative or operational purposes.

5.3 Archive - The permanent retention of a record for historical reasons.

5.4 Centralized Records Storage - Storage system in which records for an entire business are gathered and stored in one location for the company.

5.5 Charge-out Form - A form documenting an authorized employee to borrow records from the files.

5.6 Commercial Records Center - A records center operated by a private service company that takes responsibility for housing and maintaining records of various companies.

5.7 Cross-reference - An aid to finding a record stored by a filing segment other than the one selected for coding. Coding a record with a second filing caption and creating a special cross-reference sheet under the special caption.

5.8 Data - Raw material (information) used in the process of collecting information.

5.9 Decentralized Files - Records located and maintained in or near the department that is immediately responsible for the function they relate to.

5.10 Degauss - To reformat or de-magnetize media. (Example: floppy disc, hard drive, magnetic media.)

5.11 Disaster Recovery Plan - A program of activities that should be followed in the event of a disaster. The plan should cover all activities and all vital records should be identified with copies stored off-site or in fireproof file cabinets..

5.12 Disposition - The process of destroying obsolete records.

5.13 Document - The smallest unit of filing. Sometimes called a page.

5.14 Electronic Filing - Storage and retrieval of information in digital form.

5.15 Electronic Mail - The electronic transfer of correspondence and memos from one computer to another.

5.16 Evidential Value - The value of a record that documents the history of an organization.

5.17 File - A group of related documents.

5.18 Fiscal/Tax Value - The period of time a record must be retained for financial reasons or for tax requirements.

5.19 Guide - A divider used in files to identify sections of the file and to provide physical support for records.

5.20 Hardcopy - Records that are printed on paper.

5.21 Inactive Record - Records that are referred to less than once a month.

5.22 Indexing - Each part of a name or number that is considered in determining the proper filing location of a record.

5.23 In-house Records Center - An area within the company used to store inactive records.

5.24 Instrument Streamer Tapes - Magnetic tapes with portions of raw data that is used to produce data from laboratory instruments.

5.25 Key - The code by which records have been filed by sequential order on magnetic tape.

5.26 Legal Value - Value inherent in records that provide legal proof of business transactions. The period of time a record must be retained to meet statutory requirements or for other legal reasons.

5.27 Library - A collection of files.

5.28 Life cycle of Records - The period from a records' origination or receipt to its final disposition.

5.29 Local Area Network - A group of interconnected processing devices used to distribute data or information throughout a given geographic area.

5.30 Magnetic Media - Various types of records (tapes, disks, diskettes) on which the computer may electronically store its output.

5.31 Media - The floppy disks, paper tapes, magnetic tapes, cassettes, magnetic disks or cartridges on which information is stored.

5.32 Micrographics - The filming, storage, and retrieval of information, data, or records in miniaturized form.

5.33 Nonessential Records - Records that are not essential to the restoration of business and should be destroyed once their purpose has been fulfilled. Documents or copies of documents made for convenience are disposed of after use.

5.34 Numeric Filing - A filing system that relies on the use of code numbers that are assigned to businesses, clients, individual names, subjects, or geographic locations.

5.35 Off-site Records Center - An off-site location used to house inactive and vital records.

5.36 Outfolders/Outsheets - A guide indicating what material has been taken from the file and by whom.

5.37 Periodic Audit - A regular examination of records management to determine how well the program is functioning and to find ways to improve its performance.

5.38 Permanent Record - Records that are legally required to be kept indefinitely and considered to be so valuable or unique in documenting the history of an organization that they are preserved in the archives.

5.39 Purge - Removal from a file of information that is of no further value and has met the retention period.

5.40 Record - Any form of recorded information, regardless of medium.

5.41 Records Center - A low-cost storage facility for housing the inactive and semi-active records of an organization; the center may be inhouse, either on-premises or off-premises, or in commercial storage.

5.42 Records Center Box - A cardboard box designed to hold approximately one cubic foot of records, either legal-size or letter-size.

5.43 Records Disposition - The final stage of a record's life cycle, concluding with either disposition or permanent storage.

5.44 Records Management - The systematic control of an organization's records from their creation or receipt to their ultimate preservation or destruction.

5.45 Records Retention Schedule - A document that outlines specific periods set by government regulations and company policies as to how long records should be retained and disposed of.

5.46 Record Series - A group of similar or related records, used or filed as a unit and permit evaluation as a unit for retention scheduling purposes.

5.47 Records Survey - An identification and quantification of all of the records possessed by an organization.

5.48 Retention Period - A time frame that has been determined following a review of government regulations and company policies on the period of time that a document is required to be retained.

5.49 Semi-active Records - Records that are used about twice a month and stored in less accessible areas.

5.50 Shredder - A machine used for the destruction of records by reducing documents to fine strips, shreds, or particles. The Quanterra standards for shredding are:

- Non-government related documents = pieces not wider than 1/16 inch
- Government related documents or contracts = pieces not wider than 1/16 inch (+/- 1/64 inch) with length shredded no more than 1/2 inch

5.51 Suspension of Destruction - The process by which destruction of **all** records (any form of destruction) must be stopped immediately until written notification is received from legal counsel.

5.52 Tickler File - A file organized by date. Documents or reminder notices are filed under the date on which they should by reviewed or acted upon.

5.53 Useful Records - Records that are convenient to have on hand to prevent a delay in resuming normal operations after a disaster, but not considered essential because the data can be easily replaced or obtained from other sources.

5.54 Vital Records - Those records that are essential to

(1) resume or continue operations in the event of a disaster,
(2) preserve the rights of the organization's owners, employees, and customers, and/or
(3) protect the organization legally and financially.

6.0 Appendices

6.1 Appendix I-Records Survey Form

6.2 Appendix II-Quanterra Record Retention Schedule

6.3 Appendix III-Certificate of Record Retention

6.4 Appendix IV-Certificate of Compliance

6.5 Appendix V-Record Transfer Form

6.6 Appendix VI-Archived Data Retrieval Log

6.7 Appendix VII-Notice of Impending Records Destruction Form

6.8 Appendix VIII-Suspension of Destruction

6.9 Appendix IX-General Guidance on Records Disposition

6.10 Appendix X-Data Retrieval From Document Storage Form

6.11 Appendix XI-References

6.12 Appendix XII-History of Revision

APPENDIX I

QUANTERRA ENVIRONMENTAL SERVICES

KNOXVILLE LABORATORY

RECORDS SURVEY

Page 1 of 2

Department _____ Date _____
Authorization Access _____

Fill in blanks with appropriate information. Direct any questions to Records Management.

Record Title - The name to which the record is referred.
Definition - Define what the record is and its function if this is not obvious.
How Long Kept - Length of time the record is kept in your department.
How is it Indexed - List method used (i.e.- name, title, alphabetically, numerically, etc.)
Matrix - Paper, film, fiche, magnetic tape, etc.
Copy/Original - Specify whether record is the copy or the original of the organization.
Active/Inactive - Specify whether record is active, semi-active, or inactive.
Quantity - Cubic inches/Cubic feet; amount of records generated per year.

RECORD TITLE _____
DEFINITION _____
HOW LONG KEPT _____
HOW IS IT INDEXED _____
MATRIX _____
COPY/ORIGINAL _____
ACTIVE/INACTIVE _____
QUANTITY _____

RECORD TITLE _____
DEFINITION _____
HOW LONG KEPT _____
HOW IS IT INDEXED _____
MATRIX _____
COPY/ORIGINAL _____
ACTIVE/INACTIVE _____
QUANTITY _____

APPENDIX I

Page 2 of 2

RECORD TITLE _____
DEFINITION _____
HOW LONG KEPT _____
HOW IS IT INDEXED _____
MATRIX _____
COPY/ORIGINAL _____
ACTIVE/INACTIVE _____
QUANTITY _____

RECORD TITLE _____
DEFINITION _____
HOW LONG KEPT _____
HOW IS IT INDEXED _____
MATRIX _____
COPY/ORIGINAL _____
ACTIVE/INACTIVE _____
QUANTITY _____

RECORD TITLE _____
DEFINITION _____
HOW LONG KEPT _____
HOW IS IT INDEXED _____
MATRIX _____
COPY/ORIGINAL _____
ACTIVE/INACTIVE _____
QUANTITY _____

RECORD TITLE _____
DEFINITION _____
HOW LONG KEPT _____
HOW IS IT INDEXED _____
MATRIX _____
COPY/ORIGINAL _____
ACTIVE/INACTIVE _____
QUANTITY _____

APPENDIX II

QUANTERRA ENVIRONMENTAL SERVICES - KNOXVILLE LABORATORY

QUANTERRA RECORD RETENTION SCHEDULE
(Revised 11/93)

RECORD SERIES TITLE	VITAL RECORD	RETENTION BY YEAR	DISPOSITION
Office of Record: **ACCOUNTING**			
General Ledgers	Vital	50	Archive
■Financial Statements/Trial Balances	Vital	10	Shred
■Journals/Journal Entries		10	Shred
■Financial Plans		5	Shred
■Worksheets, Budgets		2	Shred
■Forecasts		ACT + 2	Shred
Accounts Payable Records		6	Shred
■Check Registers	Vital	6	Shred
■Vendor Files (Req's, Packing Slips, Brochures)		4	Shred
■Reconciliations (Bank Statements)		REC + 2	Shred
■Petty Cash Receipts		4	Trash
■Check Files (Cancelled)		6	Shred
Accounts Receivable Records		6	Shred
■Invoice Register (include Credit Memos)	Vital	3	Shred
■Client Collection History & Correspondence	Vital	7	Shred
■Credit Applications		1	Shred
Fixed Asset Ledgers & Equipment Records	Vital	7	Shred
Capital Expenditure Requests	Vital	6	Shred

APPENDIX II

RECORD SERIES TITLE	VITAL RECORD	RETENTION BY YEAR	DISPOSITION
Office of Record: **ACCOUNTING** (continued)			
■Depreciation Schedules		5	Shred
■Vehicle Records		ACT	Shred
■Maintenance & Repair Records (exclude Lab Equipment)		ACT	Shred
■Titles, Property	Vital	ACT + 20	Shred
Purchase Orders (Originals)		5	Shred
Purchase Requisitions (Originals)		5	Shred
Payroll Registers	Vital	AUD + 3	Shred
Vacation Registers (Accrued)		2	Trash
W-2 Forms (Copies)		5	Shred
W-4 Forms (Copies)		5	Shred
Timesheets		5	Shred
Commissions		6	Shred
Expense Reports (Originals)		6	Shred
Sales/Use Tax Reports & Filings		6	Shred
Tax Returns (Income & Payroll) & Supporting Schedules		6	Shred
Property Tax Records		5	Shred
Audits, Accounting		AUD + 7	Shred
Audits, Sales Tax		5	Shred
Accounting Details		4	Shred
Accruals/Reserves & Supporting Papers		6	Shred

APPENDIX II

RECORD SERIES TITLE	VITAL RECORD	RETENTION BY YEAR.	DISPOSITION
Office of Record: **ACCOUNTING** (continued)			
Insurance Payments & Vouchers (Unemployment)		3	Shred
Insurance Payments (Workers Compensation)		3	Shred
Permits, Building	Vital	EXP + 2	Shred
Permits, Business		ACT + 1	Shred
Permits, Zoning		ACT + 1	Shred
Accounting Procedures and Systems Updates		S + 7	Shred
Reports, Securities & Exchange Commission (SEC)		50	Shred
Annual Reports (Corning Incorporated)		D - 1	Trash
Office of Record: **ADVERTISING/MARKETING**			
Advertisements, Art Work, Newspaper Clips, Photos		IND/5	Shred
Mailing Lists		S	Shred
Media Kits, Newsletters, and Releases		D - 2	Archive
Customer Satisfaction Surveys/Feedback		2	Shred
Office of Record: **ENVIRONMENT, HEALTH, & SAFETY**			
Accident Logs (OSHA Form 200)	Vital	7	Shred
Accident Reports, Injury/Incident Reports	Vital	7	Shred
Air Emissions Monitoring		5	Shred
Noise Exposure Measurements		2	Shred
Audits, Health & Safety		7	Shred
Chemical Hygiene Plan		S	Shred

APPENDIX II

RECORD SERIES TITLE	VITAL RECORD	RETENTION BY YEAR	DISPOSITION
Office of Record: **ENVIRONMENT, HEALTH, & SAFETY** (continued)			
Community Right to Know - Chemical Inventories		7	Shred
Emergency Action and Contingency Plans	Vital	S	Shred
Employee Exposure/IH Monitoring Records		30	Shred
Fire Alarm System Inspections & Reports		3	Shred
Fire Extinguisher Records (Tags)		1	Shred
Fire Prevention Programs		S	Shred
Hazard Communications Training Records		7	Shred
Hazardous Waste Manifests & Related Reports	Vital	7	Shred
Health and Safety Inspection Records and Logs		3	Shred
Logs, OSHA Inspection	Vital	7	Shred
Material Safety Data Sheets (MSDS)		S	Trash
OSHA Form 100		5	Shred
Permits, Environmental & Related Reports		7	Shred
Radioactive Materials Records	Vital	7	Shred
Office of Record: **HUMAN RESOURCES**			
Affirmative Action Plans	Vital	IND	HR
Disability Claims		30	Shred
Equal Employment Opportunity Records (EEO)		IND	HR
Job Descriptions/Position Summaries		S	Shred
Medical Records	Vital	50	Shred

APPENDIX II

RECORD SERIES TITLE	VITAL RECORD	RETENTION BY YEAR	DISPOSITION
Office of Record: **HUMAN RESOURCES** (continued)			
Personnel Files & Related Records (incl Perf. Rev.)	Vital	IND	HR
Benefits & Goal Sharing Plans		7	Shred
Resumes/Applications		2	Shred
Climate Surveys		5	Shred
Training Course Materials		RA	Shred
Workers Compensation Files		18	Shred
Office of Record: **INFORMATION SERVICES**			
Data Processing Procedures		S	Shred/Delete
Daily (Incremental) Backups	Vital	14 days	Delete/Degauss
Weekly (Image) Backups	Vital	6 weeks	Delete/Degauss
Monthly (Image) Backups	Vital	1	Delete/Degauss
Quarterly (Image) Backups	Vital	7	Delete/Degauss
Database Archives	Vital	7	Delete/Degauss
Computer Software Distribution Media License		IND/3	Delete/Degauss
Programming Source Codes		IND/1	Delete/Degauss
VAX License Keys		EXP + 1	Shred
PC System Backups - Monthly		S	Delete
Instrument Streamer Tapes		1	Degauss

APPENDIX II

RECORD SERIES TITLE	VITAL RECORD	RETENTION BY YEAR	DISPOSITION
Office of Record: **LABORATORY**			
Contract Lab Program (CLP) - Private/Federal		7/2	Shred
Instrument Validation Package (Quarterly)		7	Shred
Laboratory Logs	Vital	7	Shred
■Analysis, Calibration, Field	Vital	7	Shred
■Canister Cleaning, Gas Tank Supply Pressure	Vital	7	Shred
■Injection, Job Numbers, Instrument Maintenance	Vital	7	Shred
■Raw Data, Temperature (Refrigerator, Oven, etc.)	Vital	7	Shred
■Samples, Shipping, Standards, Testing	Vital	7	Shred
Laboratory Data	Vital	7	Shred
■Raw Data, Test Data	Vital	7	Shred
■pH Strip Charts	Vital	7	Shred
■Field Data	Vital	7	Shred
Method Development/Method Validation Data		IND/3	Shred
Office of Record: **LABORATORY MANAGEMENT**			
Annual Reports (Locations)			Archive
Authorizations		ACT	Shred
Budgets		3	Shred
Business Plans		3	Shred
Charters	Vital	IND/5	Archive
Correspondence, Customer (inc. Telephone Logs)		7	Shred/Delete

APPENDIX II

RECORD SERIES TITLE	VITAL RECORD	RETENTION BY YEAR	DISPOSITION
Office of Record: **LABORATORY MANAGEMENT** (continued)			
Courier Schedules		S	Trash
Directives		S	Shred
Equipment Records (Shipments, Airbills,etc.)		4	Shred
Equipment Maintenance Records		ACT	Shred
Feasibility Studies		2	Shred
Insurance Certificates		S	Shred
Insurance Policies	Vital	EXP + 7	Shred
Licenses		ACT + 1	Shred
Logs, Copy Room Activity		1	Trash
Manuals, Office Equipment Operations		S	Trash
Manuals, Training		S	Shred
Office Layouts and Improvements		ACT	Shred
Organizational Charts		S	Shred
Production Summaries		3	Shred
Profit and Loss Statements		3	Archive
Regulatory Information	Vital	S + 7	Trash
Training Records (Hour, Course Types, etc.)		3	Trash
World Class Quality Assessments		3	Shred
Total Quality Management (QITs)		3	Shred
Manuals, Computer		S	Trash
Project Files/Data	Vital	7	Shred

APPENDIX II

RECORD SERIES TITLE	VITAL RECORD	RETENTION BY YEAR	DISPOSITION
Office of Record: **LEGAL/COMPLIANCE**			
Deeds	Vital	Deed + 2	Shred
Lease Files and Abstracts	Vital	Lease + 2	Shred
Merger Documents	Vital	IND/5	Archive
Contracts and Agreements (Customers)	Vital	EXP + 7	Shred
Contracts and Agreements (Suppliers)	Vital	IND/5	Shred
Office of Record: **ORIGINATOR (ALL EMPLOYEES)**			
Address Lists/Contacts		S	Shred
Backups, Personal Computer		S	Delete
Calendars (Appt. Books, etc.) - Paper & Elec.		CY + 1	Shred
Contact/Address Lists		S	Shred
Correspondence, Routine (Paper & E-Mail)		CY + 1	Trash/Delete
Expense Reports (Employee Copies)		2	Shred
Forms (Blank)/Form Letters		S	Trash
Interview Notes		1	Shred
Meeting Agenda/Notices		D - 1	Trash
Meeting Minutes/Notes (including CATs & QITs)		D - 2	Shred
Objectives (MBOs)		2	Shred
Period Reports/Monthly Reports		D - 5	Shred
Presentation Materials		RA	Shred
Purchase Orders (Copies)		3	Shred
Purchase Requisitions (Copies)		1	Shred

APPENDIX II

RECORD SERIES TITLE	VITAL RECORD	RETENTION BY YEAR.	DISPOSITION
Office of Originator: **ORIGINATOR (ALL EMPLOYEES)** (continued)			
Status Lists		S	Shred
Subject/Reference Files (Records Assembled by Subject)		RA	Shred
Office of Record: **PROJECT MANAGEMENT/ADMINISTRATION/SALES**			
Bids & Proposals		EXP + 2	Shred
Project Management Files		EXP + 7	Shred
■Contract Copies, Work Orders, Task Orders		EXP + 7	Shred
■Bids & Proposals Copies, if any		EXP + 7	Shred
■Invoice Copies/Summaries, if any		EXP + 7	Shred
Client Information Files		7	Shred
Client Lists		RA	Shred
Market Research		S	Shred
Marketing Plans		5	Shred
Price Lists		S + 7	Shred
Price Quotes		2	Shred
Sales Act. Reports, Sales Summaries, Call Reports		2	Shred
Telephone Logs		7	Shred
Office of Record: **QUALITY ASSURANCE**			
Audits, Quality Assurance (include all related docs)		7	Shred
■Internal Facility Audits		7	Shred
■External Facility Audits (Agency-State/Fed, Client)		7	Shred

APPENDIX II

RECORD SERIES TITLE	VITAL RECORD	RETENTION BY YEAR	DISPOSITION
Office of Record: **QUALITY ASSURANCE** (continued)			
■Data Audits		7	Shred
Certification/Approval Docs (Fed, State, Client)	Vital	IND/3	Shred
Employee Signature Lists		7	Trash
Method Detection Limit Studies		7	Shred
Performance Evaluation Studies		7	Shred
■Results/Raw Data		7	Shred
■Reports		7	Shred
■Corrective Action Reports		7	Shred
Forms		5	Trash
Policy Development/Background Files		5	Shred
Published Methods		IND	Trash
Quality Assurance Monthly Reports		7	Shred
Quality Assurance Plan Documents		7	Shred
■Quality Assurance Program Plans		7	Shred
■Quality Assurance Project Plans		7	Shred
■QA Policy Manual		7	Shred
Quality Control Charts		7	Shred
Quality Program Records		7	Shred
■KRI Data/Reports		7	Shred
■QIT Information		7	Shred
Regulatory Compliance Records		7	Shred

APPENDIX II

RECORD SERIES TITLE	VITAL RECORD	RETENTION BY YEAR	DISPOSITION
Office of Record: **QUALITY ASSURANCE** (continued)			
Standard Operating Procedures	Vital	IND/3	Shred
■Laboratory	Vital	IND/3	Shred
■Management	Vital	IND/3	Shred
■Administrative	Vital	IND/3	Shred
Office of Record: **RECIPIENT (ALL EMPLOYEES)**			
Articles, Studies, Periodicals, Trade Journals, etc.		RA	Trash
Catalogues, Vendor (Equipment & Supplies)		S	Trash
Conference/Seminar Information		RA	Trash
Handouts (Internal Programs)		RA	Shred
Itineraries		D -1	Shred
Manuals (Training, Procedures, Equip Operation)		S	Shred
Professional Association Info/Memberships		RA	Trash
Publications - Government		S	Trash
Reference and Training Materials		RA	Shred
Telephone Messages		D -1	Trash
Travel Information/Itineraries		D -1	Shred
OFFICE OF RECORD: **SECURITY/FACILITIES**			
Engineering Drawings - Facilities	Vital	IND/3	Shred
Keypad Information		ACT	Shred
Visitor Registration		1	Shred

APPENDIX II

QUANTERRA RECORD RETENTION SCHEDULE LEGEND

CODE	LEGEND DESCRIPTION
ACT	Retain While Active
ACT + #	Retain While Active + # of Year (s)
Archive	Corning Corporate Archives [Call (303)223-8457 for Instructions]
AUD + #	Retain # of Year (s) Following Audit
CI	Corning Incorporated
CLP	Contract Laboratory Program
CY + #	Retain Current Year + # of Previous Year (s)
D - #	Discretionary Retention Not to Exceed # of Year (s)
DARM	Dept of Archives & Records Mgmt Corning Inc, Corning, NY
Deed + #	Retain Life of Deed + # of Year (s)
DOD + #	Retain # of Year (s) After Date of Delivery
EH&S	Environmental Health and Safety
EPA	Environmental Protection Agency
EXP + #	Retain # Year (s) After Expiration
HR	Human Resources
IND/#	Indefinite Retention; Review Status Every # of Year (s)
ISD	Information Services
LAB	Lab Group Associated with Generation of Project Data
Lease + #	Retain # Year (s) Following Term of Lease
OSHA	Occupational Safety and Health Administration
PA	Project Administration
QA	Quality Assurance

APPENDIX II

CODE	LEGEND DESCRIPTION
RA	Review Annually
REC + #	Reconciliation + # Year (s)
S	Retain Until Superseded
S + #	Retain Until Superseded + # of Year (s)
SET + #	Retain Settlement + # of Year (s)
Shred	Shred of Incinerate (Confidential Information)
Term + #	Retain # of Year (s) Following Term of Investment
Trash	Trash of Recycle (Not Confidential Records)
Vital	Record Series is Vital; Protect by Storing a Duplicate Copy Off-site or in Fire Rated Storage

Research Sources:

- Guide to Retention Requirements in the Code of Federal Regulations
- Legal Requirements for Business Records by D.S. Skupsky
- National & International Retention Standards by F.E. Guymon
- Retention! Automated Database, Zasio Enterprises, Inc.

APPENDIX III

CERTIFICATE OF RECORD DESTRUCTION

Name _____

Location _____ Department _____

Description of Records:

_____

_____

_____

_____

_____

I certify that the records described above meet the identification and description above. These records have been destroyed in accordance with the Quanterra Record Retention Policy (latest revision).

Authorized Signature _____

Title _____

Date _____

APPENDIX IV

RECORDS MANAGEMENT PROGRAM

CERTIFICATE OF COMPLIANCE

Name _____

Location _____

Department _____

I hereby certify that during the past year, I have maintained my records in compliance with the Quanterra Records Retention Policy (latest revision) and in accordance with my region's and facility's Record Management Program.

Signature _____

Title _____

Date _____

APPENDIX V

QUANTERRA ENVIRONMENTAL SERVICES
KNOXVILLE LABORATORY

RECORD TRANSFER FORM

Complete this form for each box of records being archived. (Use an additional form if necessary.)
Transfer of documents shall be made to the Records Management Facilitator, or designee.

Released By/Date _____ Reviewed By/Date _____
Dept. Name _____ Dept. Number _____

LAB BOX IDENTIFICATION _____

☐ Original ☐ Modified PAGE _____ of _____

RECORD TITLE _____
RECORD SERIES _____
TIME PERIOD COVERED _____ RETENTION PERIOD _____

BOX CONTENTS

(FOR RIM USE ONLY)
RIM Authorization _____ Date _____ Box Number _____ Bar Code _____
DATE RECORDS TO BE REVIEWED _____

APPENDIX VI

QUANTERRA ENVIRONMENTAL SERVICES - KNOXVILLE LABORATORY

ARCHIVED DATA RETRIEVAL LOG

SIGN OUT	DATE	BOX ID	DOCUMENT ID	SIGN IN	DATE

APPENDIX VII

NOTICE OF IMPENDING RECORDS DESTRUCTION

TO: _____ DATE: _____
LOCATION: _____

FROM: _____

THE RECORDS THAT YOU HAVE SENT TO OFF-SITE STORAGE HAVE EXCEEDED THEIR RETENTION PERIOD!
(Attached - See acknowledgement copy of specific records)

In order to comply with Record Retention Policy, they will be destroyed by the date marked on your acknowledgement copy.

If for some reason these records cannot by destroyed, you have **30 days** to respond. Please contact your Record Liaison located at your facility.

RESPONSE DATE: _____

YOU WILL NOT RECEIVE ANOTHER NOTICE OF DESTRUCTION!

IF WE DO NOT HEAR FROM YOU BY THE ABOVE DATE, YOUR RECORDS WILL BE DESTROYED PER THE QUANTERRA RECORD RETENTION POLICY.

APPENDIX VIII

SUSPENSION OF DESTRUCTION

In the event of litigation, government investigation or when an audit is pending or imminent, all destruction of records must stop, even when permitted by the records retention schedule. These records should be segregated from the files and marked to be on "hold" with the reason indicated.

Any staff member receiving information regarding potential litigation, government investigation or audit should immediately inform our legal counsel. Notices will be sent to managers, records manager or records liaisons and other appropriate staff. Immediately stop the destruction of **ALL** records, since it is sometimes unclear at first which records are involved.

Legal counsel will advise all staff in writing when record destruction may resume. They also will advise what areas are not affected as information becomes available and grant permission to continue to destroy records which are not required.

Records will be systemically destroyed in accordance with the records retention program and in the regular course of business. Quanterra will destroy their records once a year during their annual "Clean Up" day. A "Notice of Destruction" must be signed prior to destruction prior to destruction of records by the record liaison for each facility.

APPENDIX IX

GENERAL GUIDANCE ON RECORDS DISPOSITION

1. Office of Record of a Document - The office of record is responsible for retaining the record for its entire life cycle. If you are not the office record for a particular record, retain record as Informational Only, and discard it when it is no longer needed for reference.

2. Informational Copy - Document owner is not the office of record. The copy of the record is retained for operational needs only, not to exceed the retention period for the office of record.

3. Original Document - A person who receives an original document and edits the document in any way, creates a new record or original, and is responsible for its retention. All other copies are considered informational copies.

4. Computer Generated Reports - Sales, usage, revenue and pricing reports, etc., must be retained by the office of record, not necessarily the generating source.

5. Non-Quanterra Created Documents - Only the person who receives the original document directly from the outside source is responsible for its retention, all other copies are informational.

6. Junk Memos - Read, then pitch when the deed is done.

7. Reference Files - If you know you won't need it, don't keep it. Be sure to examine your reference files annually and eliminate those with no value.

8. Correspondence - It is very important that **any** project file related information must be filed with the project file. All other general correspondence, company generated memos, etc. have a 2 year retention period versus the 7 year retention period for project file related records.

9. Purchase Orders - The original needs to be kept for 5 years in Accounting or Purchasing, all copies can be destroyed when their usefulness is finished.

10. For disagreement on retention period or a new record series, bring up during Record Survey process. After discussion, the policy may be revised.

11. Older records can be destroyed at the end of the maximum period for the majority of the records involved (example: 7 years for project files). The assumption is made that most records relate to that category, and rather than review old records for proper categorization, we will retain for the longer period in the policy.

APPENDIX X

QUANTERRA ENVIRONMENTAL SERVICES
KNOXVILLE LABORATORY

DATA RETRIEVAL FROM DOCUMENT STORAGE FORM

Allow one week for retrieval from offsite storage.

Requested By _____ Date _____

DATE NEEDED _____

PROVIDE AS MUCH INFORMATION AS POSSIBLE.

Record Title _____
Lab Box Id _____
Date (s) Covered _____
Other Information _____

Record Title _____
Lab Box Id _____
Date (s) Covered _____
Other Information _____

Record Title _____
Lab Box Id _____
Date (s) Covered _____
Other Information _____

Record Title _____
Lab Box Id _____
Date (s) Covered _____
Other Information _____

Record Title _____
Lab Box Id _____
Date (s) Covered _____
Other Information _____

APPENDIX XI

1.0 References

1.1 ENSECO Records Management Policy, latest revision.

1.2 Quanterra Quality Management Plan, latest revision.

1.3 IT Analytical Services - Knoxville/Middlebrook Laboratory Standard Operating Procedure - AD910306R0 "Transfer of Document Files to Off-site Storage", Revision 0.

APPENDIX XII

HISTORY OF REVISION

REV NO.	DATE	PAGES AFFECTED	REASON FOR REVISION
0	09/20/94	All	To supersede ITAS SOP AD910306R0. To describe procedure utilized by the laboratory for storage prior to off-site storage and off-site storage. To incorporate Quanterra Records Management Policy.
1	12/05/94	4, 5, 6, 7,8,13, 31,36,37,38	To change check out period of documents from safe from one week to 30 days to conform to Quanterra policy. To clarify access controls to the safe, and receipt inspection of records prior to placing in the safe. Revised Records Transfer Form (Appendix V). Added Data Retrieval From Document Storage Form (Appendix X).

OPERATION SPECIFIC STANDARD OPERATING PROCEDURE

TITLE: FACILITY SECURITY

(SUPERSEDES: ITAS-KN-QC-0003, Revision 0)

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Approved By: [illegible] 05/19/95
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Environmental, Health and Safety Coordinator

Approved By: Brian F. Wagner 5/19/95
Laboratory Director

Proprietary Information Statement:

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1.0 Purpose

1.1 This procedure describes the security systems in place at the Quanterra-Knoxville Laboratory. The security systems are adequate to limit access to the laboratory to authorized personnel and to control access to samples and confidential client information.

2.0 Responsibilities

2.1 The Quality Assurance Manager is responsible for maintaining the security of the Quality and Operation records located in the QA office.

2.2 The Laboratory Systems Manager is responsible for supervising the security of project records and controlled access areas for sample storage.

2.3 The Laboratory Director or designee is responsible for controlling keys, the arming and disarming of the alarm system and distribution of access codes to Associates.

2.4 Each laboratory associate is responsible for securing and maintaining the custody of samples during the analysis process and for following the elements of this procedure which include, but are not limited to: logging off the network system after use, signing in and out documents and keys, using their access code to gain entrance to the building and accompanying all visitors while they are on the premises.

3.0 Safety

3.1 Normal office-dependent safety precautions must be taken in performing this SOP. If personnel are required to perform any portion of the procedure in laboratory areas, appropriate personal protective equipment and precautions must be utilized.

3.2 All work must be stopped in the event of a known, or potential compromise to the health or safety of a Quanterra associate. The situation must be reported **immediately** to a laboratory supervisor.

4.0 Procedure

4.1 Physical Security

4.1.1 The building is equipped with an access code security system which permits access to the building by use of authorized codes only. All Quanterra-

Knoxville associates are issued pass codes which can be used on the employee entrances.

4.1.2 The main entrance is unlocked during normal working hours and is overseen at all times by a receptionist to prevent unauthorized entry. All other doors are locked to prevent unauthorized entry.

4.1.3 All visitors must use the main entrance, where they must sign-in and receive a visitor's badge. All visitors must be accompanied by a Quanterra associate while they are on the premises. Visitors must sign-out before leaving the premises.

4.2 Sample Security

4.2.1 The sample storage coolers and non-refrigerated storage are located in the laboratory area which is restricted to laboratory personnel and escorted visitors.

4.2.2 The security of the samples is documented in the chain of custody record that is created for each sample. All samples must be signed out and back in to sample receiving personnel through the use of internal chain of custody record.

4.2.3 The exterior door to the Sample Receipt/Coding department is shut and locked at all times. This door cannot be accessed by either a key or a code.

4.3 Administrative Controls

4.3.1 Keys are issued to primary users of an area. These people must sign that they have received a key. The controlled distribution signature form is kept by the Laboratory Director Administrative Assistant.

4.3.2 Access to the network computer systems is gained by password where each associate is given a password to enter the network system and a different password to gain access to the LIMS, which is a part of the network that contains information regarding sample status.

4.3.2.1 Root access to the system and system files will be limited to the LAN analyst and the LIMS Administrator.

4.3.2.2 Different users will have different levels of access to the LIMS. Supervisors will have data editing and review privileges. Lab technicians and data entry persons will have data entry, query, and viewing privileges.

4.3.3 On-site document storage is maintained in the Quanterra, Knoxville laboratory safe, which is a concrete, fire-proof enclosure with approximately 226 square feet of storage space. The safe remains locked unless documents are being transferred into and out of the area. Documents are signed out and signed back in the room by the person retrieving the document. See associated SOP KNOX-AD-0001, current revision, "Document Storage".

4.4 Any deviations from this procedure must be documented as a nonconformance, with a cause and corrective action described.

5.0 Definitions

5.1 Laboratory Information Management System - (LIMS) a computer system used by Quanterra-Knoxville to track the information regarding environmental samples being analyzed by the different departments.

6.0 Appendices

6.1 Appendix I: References

Appendix I References

1.1 Quality Assurance Program Requirements for Nuclear Facilities, NQA-1, American Society of Mechanical Engineers, July, 1989.

1.2 Quanterra Quality Assurance Management Plan for Environmental Services, current revision.

1.3 Quanterra Quality Management Plan, current revision.

Sampling and Analysis Plan
Site Investigation at the
G-8 Burning Ground
Former Plum Brook Ordnance Works
Sandusky, Ohio

Revision 1

Prepared For:
Department of the Army
Nashville District, Corps of Engineers
Nashville, Tennessee

Prepared By:

IT Corporation
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Knoxville, Tennessee 37923

IT Project Number 764678

May 1996

Table of Contents

Page

List of Tables iii
List of Figures iv
List of Acronyms v
1.0 Project Description 1-1
1.1 Site History and Contaminants 1-1
1.2 Summary of Existing Site Data 1-2
2.0 Project Organization and Responsibilities 2-1
3.0 Scope and Objectives 3-1
3.1 Scope 3-1
3.2 Objectives 3-1
3.3 Site-Specific Data Quality Objectives 3-1
3.3.1 Overview 3-1
3.3.2 Data Users and Available Data 3-2
3.3.3 Conceptual Model 3-2
3.3.4 Decision-Making Process, Data Uses, and Needs 3-3
3.3.5 Risk Evaluation Needs 3-3
3.3.6 Data Quality, Types, and Quantity 3-3
3.3.7 Precision, Accuracy, and Completeness 3-3
4.0 Field Activities 4-1
4.1 Geophysics 4-1
4.2 Soil Gas Survey 4-1
4.3 Groundwater 4-1
4.4 Surface and Subsurface Soil 4-1
4.4.1 Rationales 4-1
4.4.1.1 Trenching and Soil Sampling Locations 4-2
4.4.1.2 Sample Collection and Field and Laboratory Analysis 4-3
4.4.1.3 QA/QC Samples and Frequency 4-3
4.4.2 Procedures 4-3
4.4.2.1 Sampling Methods 4-3
4.4.2.2 Trenching 4-3
4.4.2.3 Boring Logs 4-3

Table of Contents (Continued)

Page

4.4.2.4 Sampling for Geotechnical Analyses 4-4
4.4.2.5 Sampling for Chemical Analyses 4-4
4.4.2.6 Trench/Soil Sample Location Survey 4-4
4.4.3 Decontamination Procedures 4-4
4.5 Sediment 4-5
4.5.1 Rationale 4-5
4.5.1.1 Sediment Sampling Locations 4-5
4.5.1.2 Sample Collection and Field and Laboratory Analyses 4-5
4.5.1.3 QA/QC Samples and Frequency 4-5
4.5.2 Procedures 4-6
4.5.2.1 Sampling Methods 4-6
4.5.2.2 Sample Collection Logs 4-6
4.5.2.3 Sampling for Chemical Analyses 4-6
4.5.2.4 Sediment Sampling Location Survey 4-6
4.5.3 Decontamination Procedures 4-7
4.6 Surface Water 4-7
4.7 Other Matrices 4-7
5.0 Sample Chain of Custody/Documentation 5-1
5.1 Field Activity Daily Log 5-1
5.2 Photographs 5-2
5.3 Sample Numbering System 5-2
5.4 Sample Documentation 5-3
5.4.1 Sample Labels 5-3
5.4.2 Sample Logbook 5-3
5.4.3 Field Custody Procedures 5-4
5.4.4 Transfer of Custody and Shipment 5-4
5.4.5 Laboratory Receipt and Entry of Samples 5-5
6.0 Sample Preservation, Packing, and Shipping 6-1
7.0 Investigation-Derived Waste 7-1
8.0 Nonconformances and Corrective Action Procedures 8-1
9.0 Project Schedule 9-1

List of Tables

Table	Title	Follows Page
3-1	Summary of Data Quality Objectives for the G-8 Burning Ground Site Investigation	3-2
4-1	Summary of Pilot and Additional Trenches	4-2
4-2	Soil and Sediment Sample Designations and QA/QC Sample Quantities	4-3
4-3	Summary of Chemical Analyses and Methodologies for Soil and Sediment Samples	4-3
6-1	Laboratory Sample Containers, Preservatives, and Holding Times	6-1

List of Figures

Figure	Title	Follows Page
1-1	Vicinity Map	1-1
1-2	Aerial Photograph of the former Plum Brook Ordnance Works	1-2
2-1	Project Organization for G-8 Burning Ground Site Investigation, PBOW	2-1
3-1	Site Investigation Decision Diagram	3-2
4-1	Locations of Trenching, Soil Sampling, and Sediment Sampling	4-2
4-2	Soil Sampling Log	4-3
4-3	Sample Collection Log	4-6
5-1	Field Activity Daily Log	5-1
5-2	Sample Label	5-3
5-3	Analysis Request and Chain of Custody Record	5-4
8-1	Nonconformance Report	8-1
8-2	Variance Log	8-2

List of Acronyms

AR/COC	Analysis Request and Chain of Custody
ASTM	American Society of Testing and Materials
bgs	below ground surface
DERP	Defense Environmental Restoration Program
DNT	dinitrotoluene
DQO	data quality objective
EPA	U.S. Environmental Protection Agency
GSA	General Services Administration
IDW	investigation-derived waste
IT	IT Corporation
LeRC	Lewis Research Center
MS	matrix spike
NASA	National Aeronautics and Space Administration
NGVD	National Geodetic Vertical Datum
PA	preliminary assessment
PAH	polynuclear aromatic hydrocarbon
PBOW	Plum Brook Ordnance Works
PBS	Plum Brook Station
PCB	polychlorinated biphenyl
QA/QC	quality assurance/quality control
QAPP	quality assurance project plan
SAP	sampling and analysis plan
SSHP	site safety and health plan
SVOC	semivolatile organic compound
TAL	target analyte list
TCL	target compound list
TNT	trinitrotoluene
USACE	U.S. Army Corps of Engineers
VOC	volatile organic compound

List of Acronyms

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QA/QC	quality assurance/quality control
QAPP	quality assurance project plan
SAP	sampling and analysis plan
SOW	statement of work
SSHP	site safety and health plan
SVOC	semivolatile organic compound
TAL	target analyte list
TCL	target compound list
TNT	trinitrotoluene
USACE	U.S. Army Corps of Engineers
VOC	volatile organic compound

1.0 Project Description

The U.S. Army is conducting studies of the environmental impact of suspected hazardous waste sites at properties owned by the U.S Department of Defense. This work is being pursued by the U.S. Army Corps of Engineers (USACE) under the Defense Environmental Restoration Program (DERP). The former Plum Brook Ordnance Works (PBOW) in Sandusky, Ohio is an Army DERP project and is currently being managed and technically overseen by the Nashville, Tennessee USACE District Office. IT Corporation (IT) is contracted to provide engineering services for the site investigation of the suspected G-8 Burning Ground at the former PBOW, under Delivery Order 0015 to Contract No. DACA62-94-D-0030. This sampling and analysis plan (SAP) has been prepared to provide a technical guidance for soil and sediment sample collection and analysis and will be used in conjunction with the quality assurance project plan (QAPP) and the site safety and health plan (SSHP) during the site investigation at the G-8 Burning Ground.

The PBOW site is currently owned by the National Aeronautics and Space Administration (NASA) and is operated as the Plum Brook Station (PBS) of the Lewis Research Center (LeRC). Figure 1-1 shows the geographical location of the former PBOW site.

1.1 Site History and Contaminants

The 9,009-acre PBOW site was built in early 1941 as a manufacturing plant for 2,4,6-trinitrotoluene (TNT), dinitrotoluene (DNT), and pentolite. Production of explosives began in December 1941 and continued until 1945. It is estimated that more than one billion pounds of explosives were manufactured during the 4-year operating period.

After the plant was shut down, decontamination of TNT, acid, pentolite, and DNT processing lines began and was completed during the last quarter of 1945. The property was initially transferred to the Ordnance Department, then to the War Assets Administration after it was certified by the U.S.Army to be decontaminated. In 1949, PBOW was transferred to the General Services Administration (GSA).

NASA acquired the PBOW in 1963 and is presently utilizing the site. On April 18, 1978, NASA declared approximately 2,152 acres of land as excess. The Perkins Township Board of Education acquired 46 acres of the excess and uses its area as a bus transportation center. GSA retains the remaining acreage and currently has a use agreement with the Ohio National Guard for 604 acres of the land. NASA presently controls about 6,400 acres and is using its

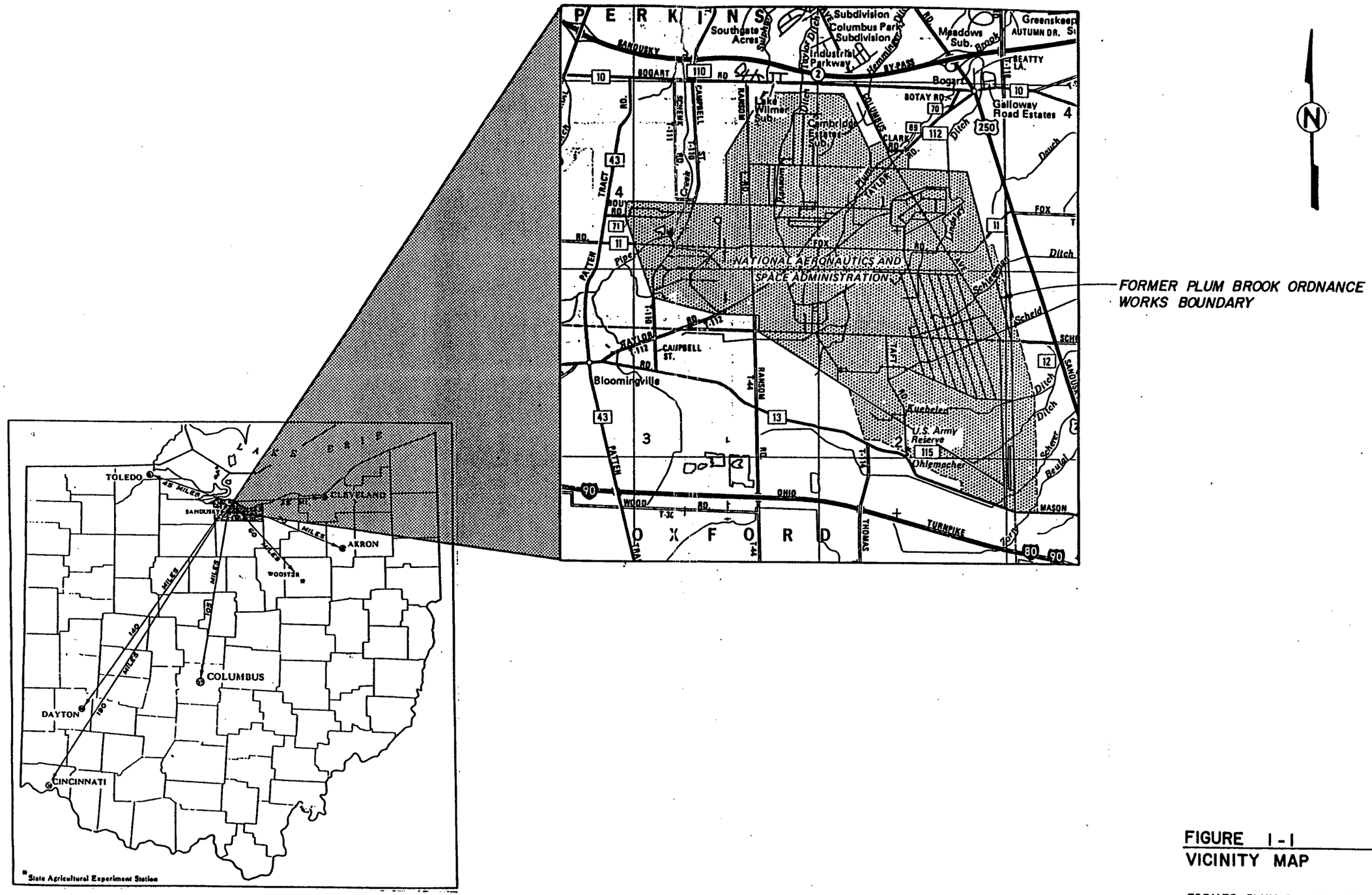


FIGURE 1-1
VICINITY MAP

FORMER PLUM BROOK ORDNANCE WORKS
SANDUSKY, OHIO

INTERNATIONAL TECHNOLOGY CORPORATION

STARTING DATE: 4-26-89	DATE LAST REV.: 2-27-90	INITIATOR: D. BURTON	DRAWING NO.: 409658-B-WP08
DRAWN BY: S. MOORE	DRAWN BY: P.L. SUAREZ	PROJ. MGR.: D. BURTON	PROJECT NO.: 409658

BRUNING 72425

KD00834

site to conduct space research as a satellite operation of NASA's LeRC in Cleveland, Ohio. The details of these land transactions are listed in the site management plan and can be found at the NASA PBS.

The G-8 Burning Ground is located in the southwestern corner of the former PBOW site, east of Guard House G-8. The Guard House G-8 was formerly located at the corner of Campbell Street and Patrol Road, as shown in the aerial photograph (Figure 1-2). The area of the burning pit was estimated to be approximately 2 acres. Environmental contaminants in this burning ground, according to previous investigations at the PBOW site and review of the site history, may include nitroaromatic compounds (explosive residual), volatile organic compounds (VOC), semivolatile organic compounds (SVOC), and metals.

1.2 Summary of Existing Site Data

Previous investigations of this burning ground include a preliminary assessment (PA), a site inspection, and a limited soil, surface water, and sediment sampling. The PA conducted by Science Application International Corporation in 1990 identified this site as an area of concern. The site inspection conducted by Morrison Knudsen Corporation described this site as a clearing area with a small ditch that runs east to west bordering the north side.

In March 1994 and March 1995, representatives from the USACE-Nashville District and from USACE's subcontractor conducted a site reconnaissance at the burning ground. No adverse surface conditions were readily apparent. The area was found to consist of an open area surrounded by hardwood forest, with no readily apparent adverse surface conditions.

Two shallow soil samples (0 to 2 feet in depth), one surface water sample, and one sediment sample were collected by Morrison Knudsen in January 1994, under contract to NASA. Two soil samples were collected from east and west sides of the center of the site and analyzed for VOCs, SVOCs, pesticides/polychorinated biphenyls (PCB), and nitroaromatics. One of the two samples was detected to contain polynuclear aromatic hydrocarbons (PAH) at concentrations ranging from 61 to 140 micrograms per kilogram. These concentrations were below the method quantitation limit. A pair of surface water and sediment samples was collected on the east side of the concrete culvert between Patrol Road and the perimeter fence at the head of a small stream that feeds Pipe Creek. 2-Butanone was detected in the surface water sample at 7 micrograms per liter. Several PAHs were reported to have been detected in the sediment sample, but the concentrations were all below the quantitation limit.

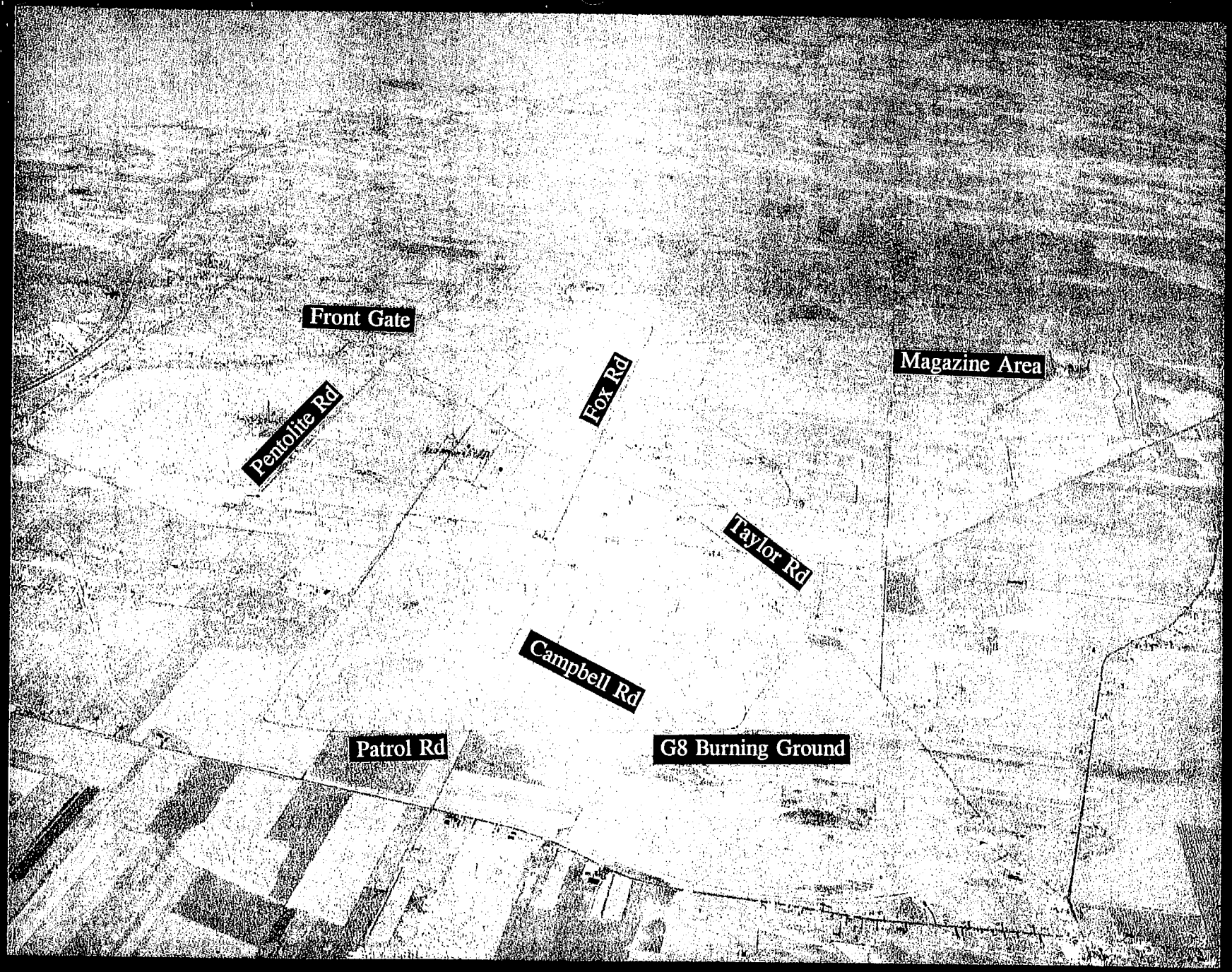


FIGURE 1-2 AERIAL PHOTOGRAPH OF THE FORMER PLUM BROOK ORDNANCE WORKS

The USACE-Nashville District and IT conducted a site visit of the G-8 Burning Ground area from December 5 through 7, 1995. As part of the site visit, a screening geophysical survey using a Geonics EM-31 terrain conductivity meter and a Geometrics G-858G magnetic gradiometer was conducted in the open area thought to be the location of the burning ground. The surveyed area was bounded to the north by a creek, to the west by Campbell Road, and to the east and west by the tree lines. The purpose of the survey was to identify geophysical anomalies that may assist in delineating the boundaries of the former burning pit. However, no apparent anomalies representing burn debris/residue or buried metallic objects within the surveyed area were found.

2.0 Project Organization and Responsibilities

The project team will be IT and IT's approved subcontractors.

The IT team has identified management and technical personnel who will participate in this project. The program manager, Don C. Burton, professional engineer will have the authority and responsibility for contract management. The project manager, Mikael Spangberg, will be responsible for overall project execution and allocation of resources necessary to implement the work plan and safety and health plan. The principal investigator, George H. Yu, registered professional geologist, will coordinate multidisciplinary investigation efforts and ensure efficient and effective information transfer among team members and be responsible for the technical quality of the project. Melissa G. Smith, certified industrial hygienist, will serve as the safety and health officer and be responsible for safety and health issues encountered during the execution of the field work. Other key IT associates are identified within the organization and will serve in their respective roles for the completion of this investigation effort.

IT services to be used for this work are primarily located in Knoxville, Tennessee and are:

- Project Management
- Engineer
- Health and Safety
- Quality assurance/quality control (QA/QC)
- Contract Administration
- Data Management
- Hazard Evaluation.

Guidance will be provided to individuals, and their activities will be coordinated by the project manager to ensure that overall project goals and objectives are met. Additional IT technical staff will be available throughout the course of the project. The project manager will determine needs as they arise and obtain the appropriate resources accordingly. Figure 2-1 presents the project organization chart.

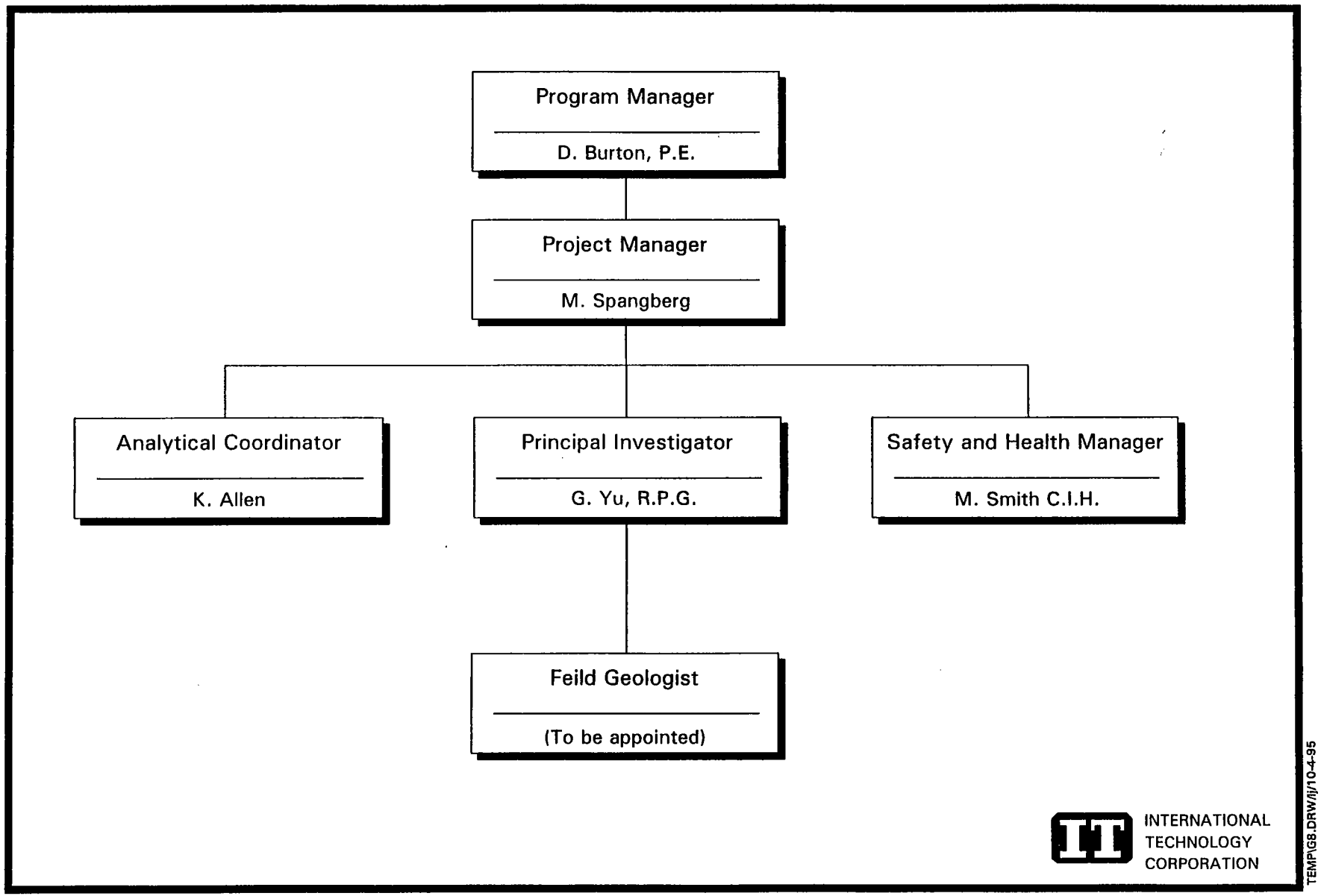


Figure 2-1 Project Organization for G-8 Burning Ground Site Investigation, PBOW

3.0 Scope and Objectives

3.1 Scope

The scope of this site investigation as specified in the statement of work (USACE, 1995) includes a visual site inspection, preparation and completion of project specific work plans, completion of various field investigation activities, evaluation of analytical results from samples collected during the field investigation, preparation and submittal of investigation reports of activities, conclusions, and recommendations for further actions.

The field investigation activities include excavation of 3 pilot trenches and a possible 4 additional trenches, collection of 2 soil samples each from 12 different locations, collection of 2 sediment samples, a land survey, and management of investigation-derived wastes (IDW). Samples collected for chemical analysis include surface soil, subsurface soil, sediment, and field QC samples.

3.2 Objectives

This investigation is intended to provide information concerning possible environmental contamination at the G-8 Burning Ground that can be used in making management decisions for the necessity of further work at the site. This investigation is preliminary in nature and information acquired will provide a foundation upon which other studies of the site, if deemed necessary and appropriate, should be based. Specifically, the objectives of this site investigation are:

- Define the location and the extent of the G-8 Burning Ground.
- Confirm or deny the presence of residual chemical contamination of sediment, surface soils, and subsurface soils.
- Determine whether or not soils are the sample media of importance and if other site media should be further evaluated.

3.3 Site-Specific Data Quality Objectives

3.3.1 Overview

The data quality objectives (DQO) process is followed to evaluate data requirements and to support the decision-making process associated with the action selection for the G-8 Burning Ground at the PBOW site. This section incorporates the components of the DQO process

described in the U.S. Environmental Protection Agency (EPA) publication 9355.9-01 *Data Quality Objectives Process for Superfund*. Table 3-1 summarizes the factors used to determine the sampling quantity and procedures necessary to meet the objectives of the site investigation as stated in Section 3.2 and to establish a basis for future action in this area. A general decision-making process of this site investigation is outlined in Figure 3-1.

3.3.2 Data Users and Available Data

The intended data users and available data related to site investigation at the G-8 Burning Ground in Table 3-1 have been used to formulate a site-specific conceptual model to support the development of the SAP necessary to meet the objectives of the site investigation and to establish the basis for future actions in the investigated area. The data users for information generated during this investigation are primarily the EPA, USACE, NASA, Ohio Environmental Protection Agency, and USACE's supporting contractors. This SAP has been designed to provide the regulatory agencies with sufficient detail to reach a determination as to the adequacy of the sampling and analysis plan. The program has also been designed to provide the USACE with the level of defensible information required to confirm or deny the existence and nature of the residual chemical contamination in sediment, surface soils, and subsurface soils.

3.3.3 Conceptual Model

Four factors are considered in defining the conceptual model for the site:

- Potential contamination sources
- Migration pathway(s)
- Potential receptors
- Contaminants of concern.

Potential sources of contamination in the G-8 Burning Ground include the use of the site for burning TNT product. During previous investigations of limited scale, contaminants of concern, including several organic compounds, have been identified in shallow soil, surface water, and sediment samples. The migration pathways to be considered in the G-8 Burning Ground are the soils, sediments, groundwater, and surface water. Groundwater is not currently considered as a medium of concern because no site-specific groundwater quality data is available. The most likely receptors are wildlife living in the area and humans using the area for recreational purposes. Potential contaminants of concern based on the past use of the site include nitroaromatic compounds; metals; VOCs; SVOCs, especially PAHs from incomplete combustion of TNT products; and PCBs and pesticides.

Table 3-1

Summary of Data Quality Objectives for the G-8 Burning Ground Site Investigation Plum Brook Ordnance Works, Sandusky, Ohio

Potential Data Users	Available Data	Conceptual Model	Media of Concern	Data Uses and Objectives	Data Types	Analytical Level	Data Quantity
EPA OEPA DOD USACE NASA IT Corporation Other Contractors Possible future land users	Former burning pit for TNT product. Detection of several organic compounds in soil, sediment, and surface water.	Contaminated sources: Burning TNT product on site Migration pathways: Soil, sediment, surface water, and groundwater Potential Receptors: Wildlife, human Potential Contaminants of Concern: Nitroaromatics, metals, VOCs, SVOCs, PCBs, and pesticides	Soils, sediments, and surface water	Primarily site investigation to confirm the existence and nature of the contamination and to delineate the lateral extent of the burn pit Suitable for future site characterization and risk assessment	Soils: Nitroaromatics, metals, PCBs, pesticides, VOCs, and SVOCs	Definitive	24 + QA
					Sediment: Nitroaromatics, metals, PCBs, pesticides, VOCs, and SVOCs	Definitive	2 + QA
					Samples for Trenches: Visual inspection of the soil samples from trench	NA[a]	NA

[a]NA - not applicable

Objectives:

- Confirm or deny the presence of contamination in soil and sediment
- Delineate the extent of G-8 Burning Ground

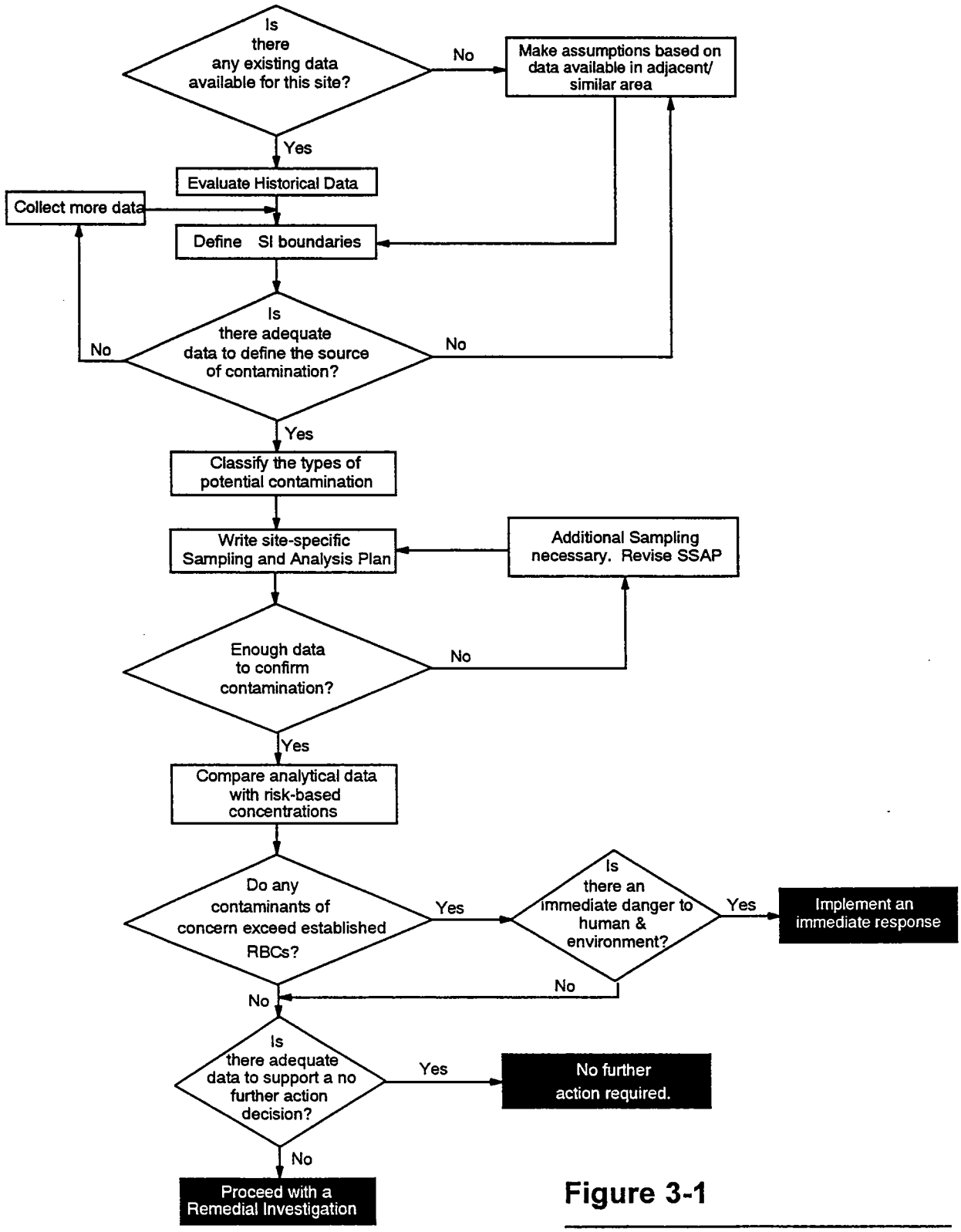


Figure 3-1

Site Investigation Decision Diagram
G-8 Burning Ground, PBOW
Sandusky, Ohio

INTERNATIONAL TECHNOLOGY CORPORATION

RBC - Risk-Based Concentration

3.3.4 Decision-Making Process, Data Uses, and Needs

The site-specific decisions regarding the action selection include:

- Take no further remedial action.
- Proceed with a formal remedial investigation.
- Initiate a remedial action to minimize or eliminate an immediate threat.

The formal decision-making process is presented in Figure 3-1. Data obtained during this investigation will be used to delineate the lateral extent of the burning ground and to confirm or deny the presence of contamination in sediment and soils at the site. Specifically, the data required as described in Sections 4.4 and 4.5 are intended to meet the objectives stated in Section 3.2.

3.3.5 Risk Evaluation Needs

The confirmation of contamination will be based on a comparison of detected VOCs, SVOCs, nitroaromatics, metals, PCBs, and pesticides with the most current list of risk-based concentrations developed by EPA. An EPA definitive data category will be used to achieve detection limits sufficient to determine whether the established risk-based concentrations are exceeded in soils and sediments at the site. Definitive data will be adequate for confirming the presence of site contamination and for supporting a risk assessment, if required, in the future.

3.3.6 Data Quality, Types, and Quantity

Soil and sediment will be sampled and analyzed in order to meet the objectives of the site investigation. QA/QC samples will be collected for all sample types as described in Section 4.4. All samples will be analyzed by EPA-approved methods and comply with EPA DQO/definitive data requirements. In addition to meeting the quality needs of the site investigation, data analyzed with this level of quality are appropriate for all phases of site characterization and risk assessment.

3.3.7 Precision, Accuracy, and Completeness

Laboratory requirements of precision, accuracy, and completeness for this site investigation are provided in detail in Section 3.1 of the QAPP. Briefly, the required analytical precision will be obtained through the analysis and evaluation of duplicate QA samples. Accuracy will be determined through the analysis and evaluation of field blanks and matrix spike (MS) samples. The completeness will be verified by validating a total of 50 percent of the analytical results.

4.0 Field Activities

Field activities during this investigation include excavation of 3 pilot trenches and a possible 4 additional trenches, collection of 2 soil samples each from 12 different locations, collection of 2 sediment samples, a land survey, and management of IDW. Samples collected for chemical analysis include surface soil, subsurface soil, sediment, and field QC samples.

4.1 Geophysics

Geophysical survey for screening purpose using a Geonics EM-31 terrain conductivity meter and a Geometrics G-858G magnetic gradiometer was completed during the site visit between December 5 and 7, 1995. No apparent anomalies representing burn debris/residue or buried metallic objects within the surveyed area were found.

4.2 Soil Gas Survey

Soil gas survey will not be used during this investigation.

4.3 Groundwater

Groundwater sampling and analysis are not currently included in the scope of this investigation.

4.4 Surface and Subsurface Soil

Surficial soil samples refers to samples collected from ground surface to 6 inches below ground surface (bgs). Subsurface soil samples refer to samples collected at depths equal to or greater than 6 inches bgs.

4.4.1 Rationales

Previous limited investigation indicated several organic contaminants present in soil samples collected from this area. The organic contaminants detected in shallow soil samples included PAHs and several nitroaromatic compounds. However, the exact boundary of the burning ground is still elusive and several visual site inspections and the most recent screening geophysical survey did not delineate the size of the burning ground. Therefore, it becomes necessary to adopt a systematic grid sampling strategy in which soil samples will be collected based on predetermined grids with regular intervals. Such sampling strategy will minimize bias and provide a complete site coverage. The possibilities exist that the burning ground may have been covered with fill materials after it was abandoned by the facility. Therefore, identification of the exact location and size of the burning ground is essential to placing soil

sampling locations in a systematic manner. Pilot trench locations will be randomly selected in three central areas of the clearing area thought to be the burning ground. After the location of the burning ground is found, four additional trenches will be placed to determine the size of the site. Soil samples will then be collected in areas of high possibility of contamination indicated by the trenching results. If the initial trenching operation shows no indication of the burning ground, no additional trench will be done and soil samples for chemical analysis will be collected from the entire area where the burning ground was believed to have existed to ensure a controlled data coverage according to a predetermined sampling grid.

4.4.1.1 Trenching and Soil Sample Locations

Trench Locations. Three pilot trenches (BG8-TR01 through BG8-TR03) will be placed initially in the central areas of the suspected burning ground. Depending on the results of the pilot trenches, four additional trenches (BG8-TR04 through BG8-TR07) will be positioned in areas outside of the pilot trenches to determine the size of the burning pit. The locations of the trenches as shown in Figure 4-1 are tentative and will be adjusted according to site conditions by IT field coordinator in consultation with USACE-Nashville District. The dimensions of trenches will be 15 feet long, 3 feet wide, and 3 feet deep. Table 4-1 summarizes the proposed trenching operations. Soil samples for trenches will be visually inspected for color, organic content, odor, and any residues that may indicate previous use of the site. After inspection, the trenches will be backfilled and marked for later land survey.

Surficial Soil Sampling Locations. Twelve surficial soil samples will be placed in twelve different locations (BG8-SO01 through BG8-S012) within the burning ground based on the grids as shown in Figure 4-1. These locations are tentative and will be adjusted by using grid intervals if the results of trenching better define the location and size of the burning ground.

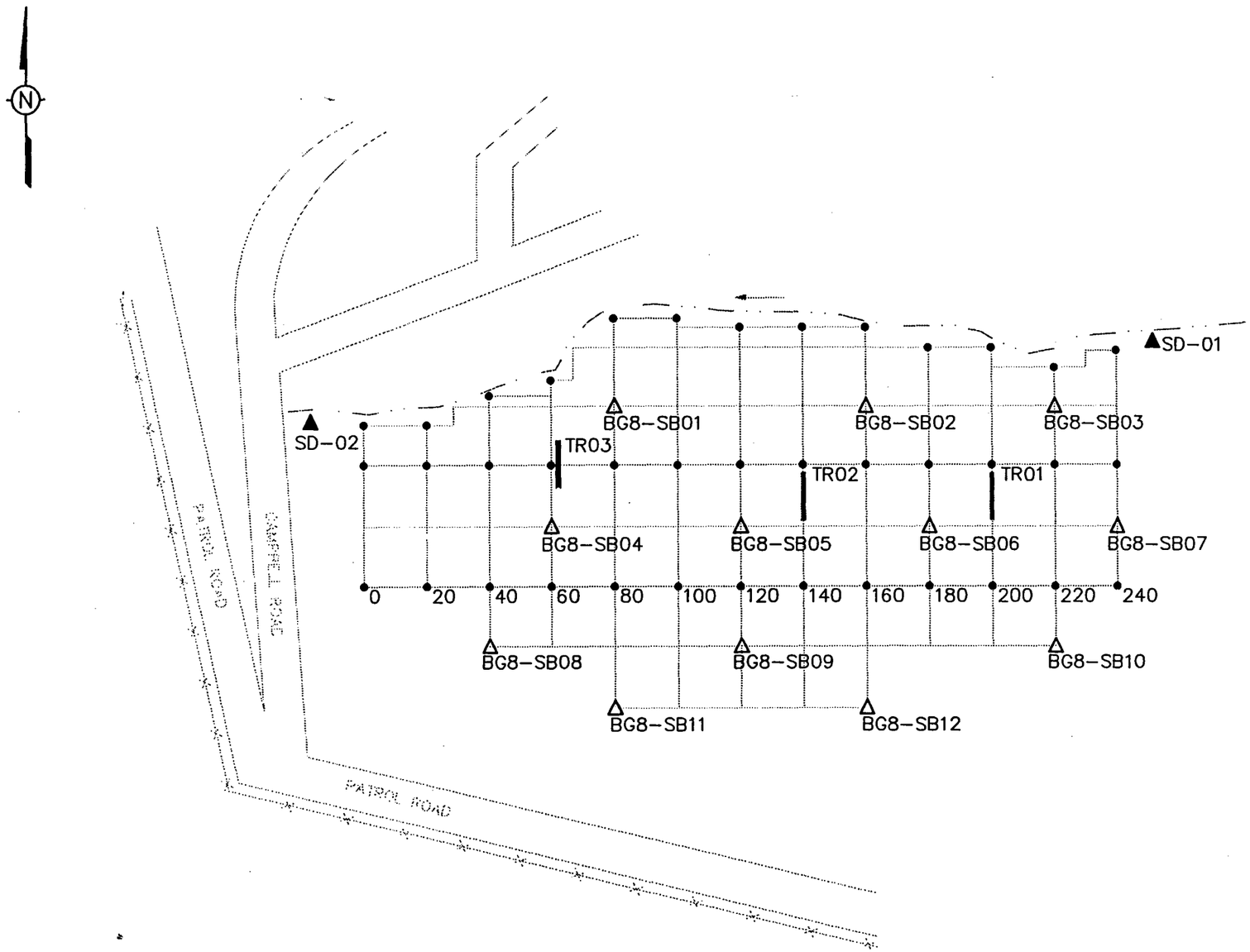
Subsurface Soil Sample Locations. Twelve subsurface soil samples will be collected in the same location as the surface soil sampling location. Subsurface samples will be collected from either a depth of 2 to 3 feet bgs or at soil/bedrock interface, whichever is encountered first.

Table 4-1

Summary of Pilot and Additional Trenches
Site Investigation at the G-8 Burning Ground
Former Plum Brook Ordnance Works, Sandusky, Ohio

Trench Designation	Location[a]	Dimension	Actions
Pilot Trench			
PBOW-96-BG8-TR01	40 feet south of the creek at stake (80N, 200E)	15' (L) x 3' (W) x 3' (D)	Visual inspection and photograph
PBOW-96-BG8-TR02	45 feet south of the creek at stake (85N, 140E)		
PBOW-96-BG8-TR03	20 feet south of the creek at stake (70N, 60E)		
Optional Trench			
PBOW-96-BG8-TR04	Location depends on the results of the pilot trenching	15' (L) x 3' (W) x 3' (D)	Visual inspection and photograph
PBOW-96-BG8-TR05			
PBOW-96-BG8-TR06			
PBOW-96-BG8-TR07			

[a]Refer to Figure 4-1 for the geophysical survey stakes.



LEGEND:

- GEOPHYSICAL SURVEY STAKE
- CREEK
- PERIMETER FENCE
- PILOT TRENCH
- SEDIMENT SAMPLE LOCATION
- SOIL BORING LOCATION

SCALE:

0 40 80 FEET

FIGURE 4-1

LOCATIONS OF TRENCHING, SOIL AND SEDIMENT SAMPLING G-8 BURNING GROUND

PBOW
SANDUSKY, OHIO

INTERNATIONAL TECHNOLOGY CORPORATION

DRAFT. CHCK. BY: C. TUMLIN	INITIATOR: G. YU	DWG. NO.: 764678-B-01	
ENGR. CHCK. BY: G. YU	PROJ. MGR.: D. BURTON	PROJ. NO.: 764678	
STARTING DATE: 12/19/95	DATE LAST REV.:		
DRAWN BY: D. BILLINGSLEY	DRAWN BY:		

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Soil sample locations (BG8-SO01 through BG8-S012) selected based on the stated rationales are also shown on Figure 4-1. Sample designations and sampling depths are summarized in Table 4-2. These locations are tentative and are subject to change if site condition warrants.

4.4.1.2 Sample Collection and Field and Laboratory Analysis

A total of 24 soil samples will be collected for chemical analysis. Twelve of these samples will be surficial samples to be collected from the 12 locations down to 6 inches bgs. The other 12 samples will be subsurface samples to be collected from either a depth of 2 to 3 feet bgs or at the soil/bedrock interface, whichever is shallower. All characterization soil samples will be analyzed for target compound list (TCL) VOCs, TCL SVOCs, target analyte list (TAL) metals, pesticides/PCBs, and nitroaromatics using analytical methods as listed in Table 4-3. Soil samples submitted for VOCs analysis will be collected first as discrete samples taken from the bottom of the sampler to prevent the escape of volatile organics.

4.4.1.3 QA/QC Samples and Frequency

QA/QC samples will be collected to ensure the reliability of field sampling procedures and materials. These samples will be collected and/or handled in the same manner as the characteristic samples. Specifically, these include field duplicate, field split, MS, and matrix spike duplicate samples. QA/QC sample quantities and sample designations are provided in Table 4-2. Detailed description of QA/QC samples is presented in Section 4.9 of the QAPP.

4.4.2 Procedures

The following sections present the procedures and methodologies to be used for soil sampling.

4.4.2.1 Sampling Methods

Soil sampling depth will be advanced using stainless-steel hand bucket auger or a Little Beaver. A drill rig is not required due to the shallow sampling depth.

4.4.2.2 Trenching

A backhole will be used for trench excavation. Soil samples from the trench for visual inspection will be collected using a hand auger.

4.4.2.3 Boring Logs

The IT field geologist will log continuous soil core from the ground surface to the total depth of the boring, in accordance with American Society of Testing and Materials (ASTM)-D2488-

Table 4-2

Soil and Sediment Sample Designations and QA/QC Sample Quantities
Site Investigation at the G-8 Burning Ground
Former Plum Brook Ordnance Works, Sandusky, Ohio

Sample Designation	Sample Location	Sampling Depth[a] (ft)	QA/QC Sample Designation		
			Field Duplicates	Matrix Spike/ Matrix Spike Duplicate	Field Split Sample
Soil					
PBOW-96-SO01-BG8-xxxx-(0-0.5)	BG8-SB01	0-0.5'			
PBOW-96-SO01-BG8-xxxx-(2-3)		2-3'			
PBOW-96-SO02-BG8-xxxx-(0-0.5)	BG8-SB02	0-0.5'	PBOW-96-FD02-BG8-xxxx-(2-3)	PBOW-96-MS/MSD02-BG8-xxxx(2-3)	PBOW-96-FS02-BG8-xxxx-(2-3)
PBOW-96-SO02-BG8-xxxx-(2-3)		2-3'			
PBOW-96-SO03-BG8-xxxx-(0-0.5)	BG8-SB03	0-0.5'			
PBOW-96-S003-BG8-xxxx-(2-3)		2-3'			
PBOW-96-SO04-BG8-xxxx-(0-0.5)	BG8-SB04	0-0.5'			
PBOW-96-SO04-BG8-xxxx-(2-3)		2-3'			
PBOW-96-SO05-BG8-xxxx-(0-0.5)	BG8-SB05	0-0.5'			
PBOW-96-SO05-BG8-xxxx-(2-3)		2-3'			
PBOW-96-SO06-BG8-xxxx-(0-0.5)	BG8-SB06	0-0.5'			
PBOW-96-SO06-BG8-xxxx-(2-3)		2-3			
PBOW-96-SO07-BG8-xxxx-(0-0.5)	BG8-SB07	0-0.5'			
PBOW-96-SO07-BG8-xxxx-(2-3)		2-3'			

Table 4-2

Soil and Sediment Sample Designations and QA/QC Sample Quantities
Site Investigation at the G-8 Burning Ground
Former Plum Brook Ordnance Works, Sandusky, Ohio

Sample Designation	Sample Location	Sampling Depth[a] (ft)	QA/QC Sample Designation		
			Field Duplicates	Matrix Spike/ Matrix Spike Duplicate	Field Split Sample
Soil (continued)					
PBOW-96-SO08-BG8-xxxx-(0.0.5)	BG8-SB08	0-0.5'			
PBOW-96-SO08-BG8-xxxx-(2-3)		2-3'			
PBOW-96-SO09-BG8-xxxx-(0-0.5)	BG8-SB09	0-0.5'			
PBOW-96-SO09-BG8-xxxx-(2-3)		2-3'			
PBOW-96-SO10-BG8-xxxx-(0-0.5)	BG8-SB10	0-0.5'	PBOW-96-FD10-BG8-xxxx-(2-3)		PBOW-96-FS10-BG8-xxxx-(2-3)
PBOW-96-SO10-BG8-xxxx-(2-3)		2-3'			
PBOW-96-SO11-BG8-xxxx-(0-0.5)	BG8-SB11	0-0.5'			
PBOW-96-SO11-BG8-xxxx-(2-3)		2-3'			
PBOW-96-SO12-BG8-xxxx-(0-0.5)	BG8-SB12	0-0.5'			
PBOW-96-SO12-BG8-xxxx-(2-3)		2-3'			
Sediment					
PBOW-96-SD-BG8-xxxx	BG8-SD01	Water/sediment interface	PBOW-96-FD01-BG8-xxxx-(SD)		PBOW-96-FS01-BG8-xxxx-(SD)
PBOW-96-SD-BG8-xxxx	BG8-SD02	Water/sediment interface			

[a]Subsurface soil samples will be collected between depths 2 to 3 feet or at the soil/limestone interface, whichever is shallower.

Table 4-3

Summary of Chemical Analyses and Methodologies for Soil and Sediment Samples Site Investigation at the G-8 Burning Ground Former Plum Brook Ordnance Works, Sandusky, Ohio

Analytical Parameters	Analytical Method
TCL Volatile Organic Compounds	EPA SW-846 8260A[a]
TCL Semivolatile Organic Compounds	EPA SW-846 3540/8270B[a]
TAL Metals	SW-846 3050A/6010A[b] for Ag, Al, As, Ba, Be, Ca, Cd, Co, Cr, Cu, Fe, K, Mg, Mn, Na, Ni, Pb, Sb, Se, Tl, V, Zn 7471A[a] for Hg
Nitroaromatic Compounds	EPA SW-846/8330[c]
TCL PCBs/Pesticides	EPA SW-846 8081[a]

[a]Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, Third Edition, Update II, September 1994.
[b]Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, Third Edition, Update I, July 1992.
[c]Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, Third Edition, Revision 1, December 1990.
TCL - Target compound list.
TAL - Target analyte list.
PCB - Polychlorinated biphenyl.

HTRW DRILLING LOG	DISTRICT	HOLE NUMBER
1 COMPANY NAME	2. DRILL SUBCONTRACTOR	SHEET SHEETS OF

3 PROJECT	4 LOCATION
5 NAME OF DRILLER	6 MANUFACTURER'S DESIGNATION OF DRILL
7 SIZES AND TYPES OF DRILLING AND SAMPLING EQUIPMENT	8 HOLE LOCATION
	9 SURFACE ELEVATION
	10. DATE STARTED / 11 DATE COMPLETED
12. OVERBURDEN THICKNESS	15. DEPTH GROUNDWATER ENCOUNTERED
13. DEPTH DRILLED INTO ROCK	16 DEPTH TO WATER AND ELAPSED TIME AFTER DRILLING COMPLETED
14 TOTAL DEPTH OF HOLE	17 OTHER WATER LEVEL MEASUREMENTS (SPECIFY)

18. GEOTECHNICAL SAMPLES	DISTURBED	UNDISTURBED	19 TOTAL NUMBER OF CORE BOXES

20. SAMPLES FOR CHEMICAL ANALYSIS	VOC	METALS	OTHER (SPECIFY)	OTHER (SPECIFY)	OTHER (SPECIFY)	21 TOTAL CORE RECOVERY %

22. DISPOSITION OF HOLE	BACKFILLED	MONITORING WELL	OTHER (SPECIFY)	23. SIGNATURE OF INSPECTOR

LOCATION SKETCH/COMMENTS SCALE:

PROJECT	HOLE NO.

ENG FORM 5056-R, AUG 94 (Proponent: CECW-EG)

Figure 4-2. HTRW Drilling Log

93 using the United Soil Classification System. The Boring Log as shown in Figure 4-2 will serve as a record of sample collection, sample location and depth, and drilling procedures.

4.4.2.4 Sampling for Geotechnical Analyses

Soil samples for geotechnical analysis will not be included in this investigation.

4.4.2.5 Sampling for Chemical Analyses

All soil samples designated for chemical analysis will be collected as described in Section 4.4.2.1. Soil samples collected from various locations will be placed in appropriate sample containers, sealed, labeled, and shipped. These samples will be analyzed for chemical parameters as listed in Table 4-3, along with analytical methods.

4.4.2.6 Trench/Soil Sample Location Survey

Trench and soil sample locations will be surveyed by a registered professional land surveyor. The horizontal coordinates will be surveyed to the closest 1.0 foot and referenced to the Ohio State Plane Coordinate System. Vertical coordinates (surface elevation) will be to the nearest 0.01 foot and referenced to the 1929 National Geodetic Vertical Datum (NGVD). A wood stake will be placed at each sampling location and at each corner of the trench after completion to facilitate land survey. The survey data will be used to accurately locate each sample location on the site map.

4.4.3 Decontamination Procedures

The following decontamination procedures will be used to decontaminate soil and sediment sampling equipment before site entry, between sampling locations, and before site departure:

- Steam-rinse with potable water (metal soil sampling equipment only).
- Wash and scrub using a brush with nonphosphate detergent.
- Rinse with potable water.
- Rinse with deionized water (ASTM Type II water).
- Rinse with methanol. Follow with a hexane rinse because soil samples and sediment will be analyzed for pesticides/PCBs.
- Final rinse with deionized water. The volume of water used must be at least five times greater than the volume of methanol and hexane used.
- Air dry.

- Wrap in aluminum foil.

4.5 Sediment

Sediment sampling will be conducted during this investigation. Sediment refers to any unconsolidated materials below water surface.

4.5.1 Rationale

During previous limited investigation, only one sediment sample was collected on the east side of the concrete culvert between Patrol Road and the perimeter fence at the head of a small stream that feeds Pipe Creek. Several PAHs were reported to have been detected in this sediment sample, but the concentrations were below the quantitation limit. This investigation calls for two sediment samples to be collected from the drainage ditch located along the north boundary of the suspected burning pit. During the recent site visit, the ditch contained water flowing west toward Campbell Road.

4.5.1.1 Sediment Sampling Locations

The locations of the two sediment samples along the ditch are shown on Figure 4-1. One sample (BG8-SD01) will be collected from upgradient location while the other (BG8-SD02) will be obtained from the downgradient location near the concrete culvert.

4.5.1.2 Sample Collection and Field and Laboratory Analyses

Two sediment samples will be collected from two locations along the drainage ditch located north of the site. This ditch contained flowing water during a recent site visit. However, it may become dry over a prolonged period of dry weather. If sediment samples cannot be collected because of a dry situation, the USACE-Nashville District will be contacted for alternatives. Sediment samples, if available, will be analyzed for the same parameters as for soil samples. Those will include TCL VOCs, TCL SVOCs, TAL metals, pesticides/PCBs, and nitroaromatics using analytical methods as listed in Table 4-3. Sediment samples submitted for VOCs analysis will be collected first as discrete samples taken from the bottom of the sampler to prevent the escape of volatile organics.

4.5.1.3 QA/QC Samples and Frequency

Sediment QA/QC samples will be obtained following the procedures specified in Section 4.4.1.3. One field duplicate sediment sample and one field split sample will be collected. Quantity of sediment QA/QC samples is summarized in Table 4-2.

4.5.2 Procedures

The following sections present the procedures and methodologies to be used for sediment sampling.

4.5.2.1 Sampling Methods

Sediment samples will be collected with a bucket auger or similar sampling device and logged on the drilling log as shown in Figure 4-2. The distance from the water surface to bottom will be measured and recorded. The sampling device will be lowered slowly approaching the bottom depth to prevent unnecessary bottom disturbance. Any excess liquid will be decanted from the sediment sample before placing it in the appropriate container. After collection, samples will be packed in coolers with ice for delivery to the laboratory. Bedrock (limestone) may be encountered at a very shallow depth along the creek.

4.5.2.2 Sample Collection Logs

Sample collection logs will be used during sediment sample collection activities to document collection information including location, sample depth, sample date, sample number, and sampling method. Physical parameters of the sediment will also be described on this log as shown in Figure 4-3.

4.5.2.3 Sampling for Chemical Analyses

All sediment samples designated for chemical analysis will be collected as described in Section 4.5.2.1. Sediment samples collected will be placed in appropriate sample containers, sealed, labeled, and shipped. These samples will be analyzed for the same chemical parameters as soil samples as listed in Table 4-3.

4.5.2.4 Sediment Sampling Location Survey

Sediment sampling locations will be surveyed by a registered professional land surveyor. The horizontal coordinates will be surveyed to the closest 1.0 foot and referenced to the Ohio State Plane Coordinate System. Vertical coordinates (surface elevation) will be to the nearest 0.01 foot and referenced to the 1929 NGVD. A wood stake will be placed at each sampling location after sediment sample collection to facilitate land survey. The survey data will be used to accurately locate each sediment sampling location on the site map.

SAMPLE COLLECTION LOG

USE THIS FORM FOR ALL SAMPLE COLLECTION EXCEPT GROUNDWATER

PROJECT NO.: _____ PROJECT NAME: _____

REQUEST FOR ANALYSIS NO.: _____ INVESTIGATION SITE: _____

COLLECTION DATE: _____ COLLECTION TIME: _____

SAMPLE NUMBER: _____ - _____ - _____ - _____

WATER SAMPLES (Not Groundwater)

For use with surface water samples, field blanks, equipment rinsates, etc.

Type of Water Body: _____

Sample Filtered (Yes/No): _____

Depth of Take Below Surface: _____ feet

Eh: _____ mV

pH: _____ SU

Conductivity: _____ µmhos/cm

Dissolved Oxygen (D.O.): _____ % or mg/l

Temperature: _____ °C or °F

Turbidity: _____ NTUs

SAMPLE ANALYSIS PARAMETER	ANALYTICAL METHOD NO.

SEDIMENT SAMPLES

Depth of water: _____ feet

Depth of take: _____ feet

SOIL SAMPLES

Depth of take: _____ feet

START	END
inches	inches
inches	inches

Headspace Reading: _____

Purpose (Analytical / Physical): _____

Composite Sample (Yes / No): _____

SAMPLE LOCATION SKETCH

REMARKS / COMMENTS: _____

FORM PREPARED BY: _____

SAMPLE COLLECTED BY: _____

FORM CHECKED BY: _____

FIELD FORM (OCTOBER 1994) SC-LOG

Figure 4-3 Sample Collection Log

4.5.3 Decontamination Procedures

Decontamination of sediment sampling equipment will follow procedures specified in Section 4.4.3.

4.6 Surface Water

Collection of surface water samples will not be conducted during this investigation.

4.7 Other Matrices

No other matrices other than those described in Section 4.4 and 4.5 will be sampled during this investigation.

5.0 Sample Chain of Custody/Documentation

5.1 Field Activity Daily Log

Field data collection activities will be recorded on the Field Activity Daily Log as shown in Figure 5-1. Entries will be described at an appropriate level of detail so that the situation can be reconstructed without relying on memory. All log sheets will be kept in the project files. Project files will be turn over to the USACE at the completion of the project contract.

Entries on the logs will contain a variety of information. Specifically, this information will include:

- Project identification
- Field activity subject
- General work activity
- Unusual events
- Visitors on site
- Subcontractor progress or problems
- Communication with the client or others
- Weather condition
- IT personnel on site
- Sample number and time of day for each sample collected for analysis
- Listing by sample number of samples collected during the day, sorted by chain-of-custody number (compiled at the end of the day)
- Record of telephone call informing the laboratory of sample shipment
- Accomplishment of decontamination of drilling equipment and sampling equipment
- Accomplishment of required calibration checks
- Disposition of decontamination fluids

INTERNATIONAL TECHNOLOGY CORPORATION

DAILY LOG			
DATE			
NO.			
SHEET		OF	

FIELD ACTIVITY DAILY LOG

PROJECT NAME	PROJECT NO.
FIELD ACTIVITY SUBJECT:	
DESCRIPTION OF DAILY ACTIVITIES AND EVENTS:	
VISITORS ON SITE	CHANGES FROM PLANS AND SPECIFICATIONS, AND OTHER SPECIAL ORDERS AND IMPORTANT DECISIONS.
WEATHER CONDITIONS:	IMPORTANT TELEPHONE CALLS:
IT PERSONNEL ON SITE:	
SIGNATURE	DATE:

327A-7-86

Figure 5-1

- Variances from project plans and procedures (details will be recorded in the variance log)
- Accomplishment of tailgate safety meetings
- Review of project procedures with site personnel
- Photographs taken and identification numbers
- Air monitoring readings, as appropriate.

5.2 Photographs

Color photographs (35-millimeter color print) will be taken of trenching and soil sampling activities. The photograph number and roll number will be logged on the Field Activity Daily Log to identify which sampling site is depicted in the photograph. Each film roll will be identified by taking a photograph of an information sign on the first frame. The job and film roll number will be written on each sign to identify the pictures contained on the roll; for example:

Plum Brook Ordnance Works G-8 Burning Ground
Site Location (trench or boring number)
Roll Number 1
Frame Number 1 of 36
February 02, 1996 - (photographer's name)

Color print will be identified with the project number, date taken, and a brief description. Negatives and color prints will be kept in project central files.

5.3 Sample Numbering System

Sample numbering system to be used during this investigation will conform to USACE-Nashville District's numbering convention. Specifically, each sample will be assigned a unique sample identification number that describes where the sample was collected. Each number consists of a group of letters and numbers, separated by hyphens. The sample media and numbering system is described as follows.

Project Code	Year	Sample Type[a]	Site Number	Sample Number	Depth
PBOW	96	XX	BG8	XXXX	(XXXX)

[a]Sample type:

SD - sediment sample
SO - soil sample
TR - trench

"SO03-BG8-0129-(0'-0.5')" signifies that this soil sample is collected at a depth between 0 and 0.5 foot bgs from soil sampling 03 with a laboratory number 0129. The complete sample number will be recorded by the IT field geologist in the Field Activity Daily Log and in the Boring Log or in the Sediment Sample Collection Log.

5.4 Sample Documentation

5.4.1 Sample Labels

All physical samples for laboratory analysis obtained at the sites will be placed in an appropriate sample container for shipment to the laboratory. Each sample container will be identified with a separate identification label (Figure 5-2). The following information will be included on the label:

- Project identification
- Sample identification
- Sample location
- Date of collection
- Time of collection
- Required analytical methods
- Sampler's initial.

5.4.2 Sample Logbook

A sample log book will be used by the IT field geologist or sample technician to keep track of sample collection, designation, and packing and shipping information for all samples. Upon completion of the filed work, the sample log book will be kept in the project files for future reference.

Sample Label

INTERNATIONAL TECHNOLOGY CORPORATION

F
H R

Project Name _____

Project No. _____

Sample No. _____

Collection Date/Time _____

Collector's Name _____

Sample Location _____

Sample Type/Depth/Description _____

Analyze For _____ Preservative _____

Bottle _____ of _____ _____ Filtered _____ Nonfiltered

23-8-85

Figure 5-2

5.4.3 Field Custody Procedures

The Analysis Request and Chain of Custody Record (AR/COC) form (Figure 5-3), along with any instructions for filling the form, will be furnished by the laboratory that will analyze the samples.

Field custody procedures include:

- Before sampling begins, the principal investigator or the field coordinator will instruct site personnel in the AR/COC procedures, as necessary.
- The quantity, types, and identification of samples will be reviewed.
- Sampling locations will be finalized and annotated on a site map.
- The field coordinator has overall responsibility for the care and custody of the samples collected until they are transferred or properly dispatched to the laboratory. Each individual who collects a sample is responsible for sample custody until the sample is transferred to someone else via the AR/COC record form.
- Shipment information is recorded for shipment of samples at the end of the shift, day, or collection period on the Field Activity Daily Log.

5.4.4 Transfer of Custody and Shipment

Transfer of custody and shipping procedures include:

- An AR/COC form will be initiated in the field for each sample. The white copy of this record will accompany each sample. The yellow copy will be retained at the site and will be placed in the project files at the conclusion of field activities.
- If the laboratory sample custodian judges sample custody to be invalid (i.e., sample arrived damaged), a Nonconformance Report form will be initiated. The project manager and QA officer will be notified. The project manager will make a decision as to the fate of the sample(s) in question. The sample(s) will either be processed "as is" with custody failure noted along with the analytical data or rejected with sampling rescheduled if necessary. The project manager and QA officer will sign off the Nonconformance Report, noting the reason for disposition.
- Each time responsibility for custody of the sample changes, the new custodian will sign and date the record.
- The custody of individual sample containers will be documented by recording each container's identification on an AR/COC form.

INTERNATIONAL TECHNOLOGY CORPORATION

ANALYSIS REQUEST AND CHAIN OF CUSTODY RECORD*

Reference Document No.
Page 1 of ____

Project Name/No. 1 _____
Sample Team Members 2 _____
Profit Center No. 3 _____
Project Manager 4 _____
Purchase Order No. 6 _____
Required Report Date 11 _____

Samples Shipment Date 7 _____
Lab Destination 8 _____
Lab Contact 9 _____
Project Contact/Phone 12 _____
Carrier/Waybill No. 13 _____

Bill to: 5 _____
Report to: 10 _____

ONE CONTAINER PER LINE

Sample Number 14	Sample Description/Type 15	Date/Time Collected 16	Container Type 17	Sample Volume 18	Pre-servative 19	Requested Testing Program 20	Condition on Receipt 21	Disposal Record No. 22
							FOR LAB USE ONLY	
							FOR LAB USE ONLY	

Special Instructions: 23

Possible Hazard Identification: 24
Non-hazard ☐ Flammable ☐ Skin Irritant ☐ Poison B ☐ Unknown ☐

Sample Disposal: 25
Return to Client ☐ Disposal by Lab ☐ Archive _____ (mos.)

Turnaround Time Required: 26
Normal ☐ Rush ☐

QC Level: 27
I. ☐ II. ☐ III. ☐ Project Specific (specify): _____

1. Relinquished by 28 (Signature/Affiliation)	Date: Time:	1. Received by 28 (Signature/Affiliation)	Date: Time:
2. Relinquished by (Signature/Affiliation)	Date: Time:	2. Received by (Signature/Affiliation)	Date: Time:
3. Relinquished by (Signature/Affiliation)	Date: Time:	3. Received by (Signature/Affiliation)	Date: Time:

Comments: 29

White: To accompany samples
Yellow: Field copy
*See back of form for special instructions.

MCA 3/15/91

Figure 5-3

- The analyses to be performed for each sample will be recorded on an AR/COC form. The white copy will accompany the samples. The yellow carbon copy will be retained at the site.

- Upon sample destruction or disposal, the custodian responsible for the disposal will complete the AR/COC form, file a copy, and send a copy to the project manager or the designated representative for record keeping.

5.4.5 Laboratory Receipt and Entry of Samples

Upon receipt at the laboratory, a sample is removed from the shipping container. The sample identifications on the sample bottles are then compared to the information contained on the sample packing list or the included AR/COC. If discrepancies exist, appropriate notes (signed and dated) are made on the AR/COC, and the shipping and receiving supervisor and the project manager are notified.

The following items are checked upon receipt of samples with the AR/COC or the accompanying forms:

- The seals and tapes on the cooler are unbroken and uncut.

- The sample containers in the cooler are intact.

- The identification on the sample bottles corresponds to the entries on accompanying forms.

- The number of sample containers received are equal to the number of samples listed on the AR/COC or accompanying forms.

Identification numbers are stamped on stickers and securely wrapped around each sample.

6.0 Sample Preservation, Packing, and Shipping

Sample containers and caps will be new, certified as being precleaned, containers from I-CHEM, made of materials recommended by the EPA in Title 40 Code of Federal Regulations Part 136 and SW-846 (3rd Edition). Sample containers and preservatives/preservation methods are summarized in Table 6-1. Sample containers will be supplied by the laboratory.

Each sample container will be bagged before placement in the cooler. Sample holding times will be calculated from the date the sample is collected, not the date that it is received by the laboratory.

Samples will be placed in coolers as soon as possible after collection and will be packed so as to minimize the possibility of container breakage by using vermiculite or styrofoam peanuts to fill void spaces in the cooler. Sample will be cooled as promptly as feasible to a temperature of approximately 4 degrees Celsius and maintained at that temperature by means of blue ice or packaged ice from the time the shipping carton is sealed using tape and custody tape until it is received at the laboratory. Coolers will be shipped to the laboratory by a next-day delivery service. Notification of shipment, including airbill number, will be telephoned or faxed to the laboratory the day of sample collection. If this is not possible, the laboratory will be notified the following morning.

Complete AR/COC records will be included with each shipment of coolers.

Table 6-1

Laboratory Sample Containers, Preservatives, and Holding Times
Site Investigation at the G-8 Burning Ground
Former Plum Brook Ordnance Works, Sandusky, Ohio

Matrix	Analytical Parameter	Analytical Method	Container Type & Quantity	Preservative	Holding Time
Soils and Sediments	Volatile Organics	8260A	2 x 125-mL Wide-Mouth Glass w/ Teflon-Lined Cap	Cool to 4°C	14 days from collection
	Semivolatile Organics	8270B	1 x 250-mL Wide-Mouth Amber Glass w/Teflon-Lined Cap	Cool to 4°C	Extract 14 days from collection, analyze 40 days from extraction
	Pesticides/PCB	8081			
	Metals	3050A/6010A 7471A	1 x 125-mL Wide-Mouth Glass w/Teflon-Lined Cap	Cool to 4°C	180 days from collection (Hg - 28 days) from collection
	Explosives	8330	1 x 125-mL Wide-Mouth Amber Glass w/Teflon-Lined Cap	Cool to 4°C	Extract 14 days from collection, analyze 40 days from extraction

7.0 Investigation-Derived Waste

IDW during field activities at the former PBOW are anticipated to be minimal in quantity because of the shallow boring depth. IDW includes soils from trench excavation, cuttings from soil sampling, excess soil samples, decon water, decon fluid, and disposable personal protective equipment. Trench soil and cuttings from soil sampling and excess soil samples will be returned immediately after sample collection to the point of origin (backfill). Other IDW will be handled following the guidance document EPA\540\G-\91\009, May 1991, *Management of Investigation-Derived Wastes During Site Inspection.* Specifically, IDW will be managed in the following manner:

- Disposable personal protective equipment will be double-bagged and disposed of in the dumpster.

- Decontamination water will be collected in 55-gallon drums and transported to the designated outside disposal area located in the Pentolite Road Red Water Pond, south of the Pentolite Road, for disposal.

- Decontamination fluid will be kept in a stainless-steel pan and will be allowed to evaporate. Residues will be disposed of in the on-site disposal area as previously described.

8.0 Nonconformances and Corrective Action Procedures

Nonconforming equipment, items, activities, conditions, and unusual incidents that could affect compliance with the project requirements will be identified, controlled, and reported in a timely manner. A nonconformance is defined as a malfunction, failure, deficiency, or deviation that renders the quality of an item unacceptable and indeterminate. The originator (any IT employee) of a Nonconformance Report (Figure 8-1) will describe the finding in this report and will notify the project manager and QA officer. Each nonconformance will be reviewed and a disposition given for the item, activity, or condition. The disposition of a nonconformance will be documented and approved by the IT organization responsible for the issuance of the nonconformance. The QA officer will concur with the disposition of the nonconformance.

The laboratory coordinator is responsible for assessment of QC sample information. If data fall outside accepted limits, the QA/QC coordinator will immediately notify the laboratory project manager and the responsible group leader. If the situation is not corrected and an out-of-control condition occurs or is expected to occur, the QA officer will notify the project manager. The project manager and group leaders are responsible for identifying the source of the nonconformance and initiating corrective action. Completion of corrective action should be evidenced by data returning to prescribed acceptable limits.

The modification, repair, rework, or replacement of nonconforming equipment, items, or activities will require the reverification of acceptability. In certain instances, as determined by the project manager or QA officer, these actions may require that corrective action be completed and verified before site work continues.

The equipment, item, or activity that has the deficiency may be temporarily stopped while the nonconformance is being investigated. If, in the opinion of the project manager and the QA officer, the nonconformance does not significantly affect the technical quality or use of the work, the work may continue pending resolution of the nonconformance. The basis for such decision will be documented on the Nonconformance Report and submitted to the QA officer for review and approval. The documentation will include the statement that the decision was made prior to continuing the work. The records of nonconformances and their dispositions will be kept in the project central files.

IT CORPORATION

NONCONFORMANCE REPORT

PROJECT NO. _____ NR NO _____

PAGE 1 OF

PROJECT NAME _____ DATE: _____

NONCONFORMANCE:

IDENTIFIED BY: _____ DATE: _____

CORRECTIVE ACTION REQUIRED:

TO BE PERFORMED BY: _____ DATE: _____

MUST CORRECTION BE VERIFIED? YES NO

TO BE VERIFIED BY: _____ PREPARED BY: _____ DATE: _____

CORRECTIVE ACTION TAKEN:

PERFORMED BY: _____ DATE: _____

VERIFIED BY: _____ DATE: _____

CC:

Approved By: _____ Date: _____

Approved By: _____ Date: _____

Figure 8-1

Changes (variances) to the approved work plan may be necessary as site conditions require and may be initiated either in the field or in the office. All variances will be noted on the Field Activity Daily Log and will be recorded on a Variance Log shown in Figure 8-2. Variances will be approved by the QA officer and the project manager prior to implementation of the change. Variances that affect the project scope, cost, or schedule must be approved by the USACE before being implemented and the health and safety officer must approve any variances to the SSHP. The project manager and QA officer will document their approval of the change by signing the Variance Log before the change is implemented in the field. The principal investigator or the field coordinator is responsible for initiating and chronologically maintaining all variances that are generated during the field work. The project manager's approval of a variance may be initiated on a verbal basis; such approval will be documented on the Variance Log and the original will be submitted to the project manager while a copy will be maintained in the field. If a variance is requested by the USACE, it will be so noted on the Variance Log.

INTERNATIONAL TECHNOLOGY CORPORATION

VARIANCE NO._____

VARIANCE LOG

PROJECT NO._____ PAGE___ OF___

PROJECT NAME _____ DATE:_____

VARIANCE (INCLUDE JUSTIFICATION)

APPLICABLE DOCUMENT:

CC:

REQUESTED BY: _____ Date: _____

Approved By: _____ Date: _____
Project Manager

_____ Date: _____
Quality Assurance Officer

_____ Date: _____

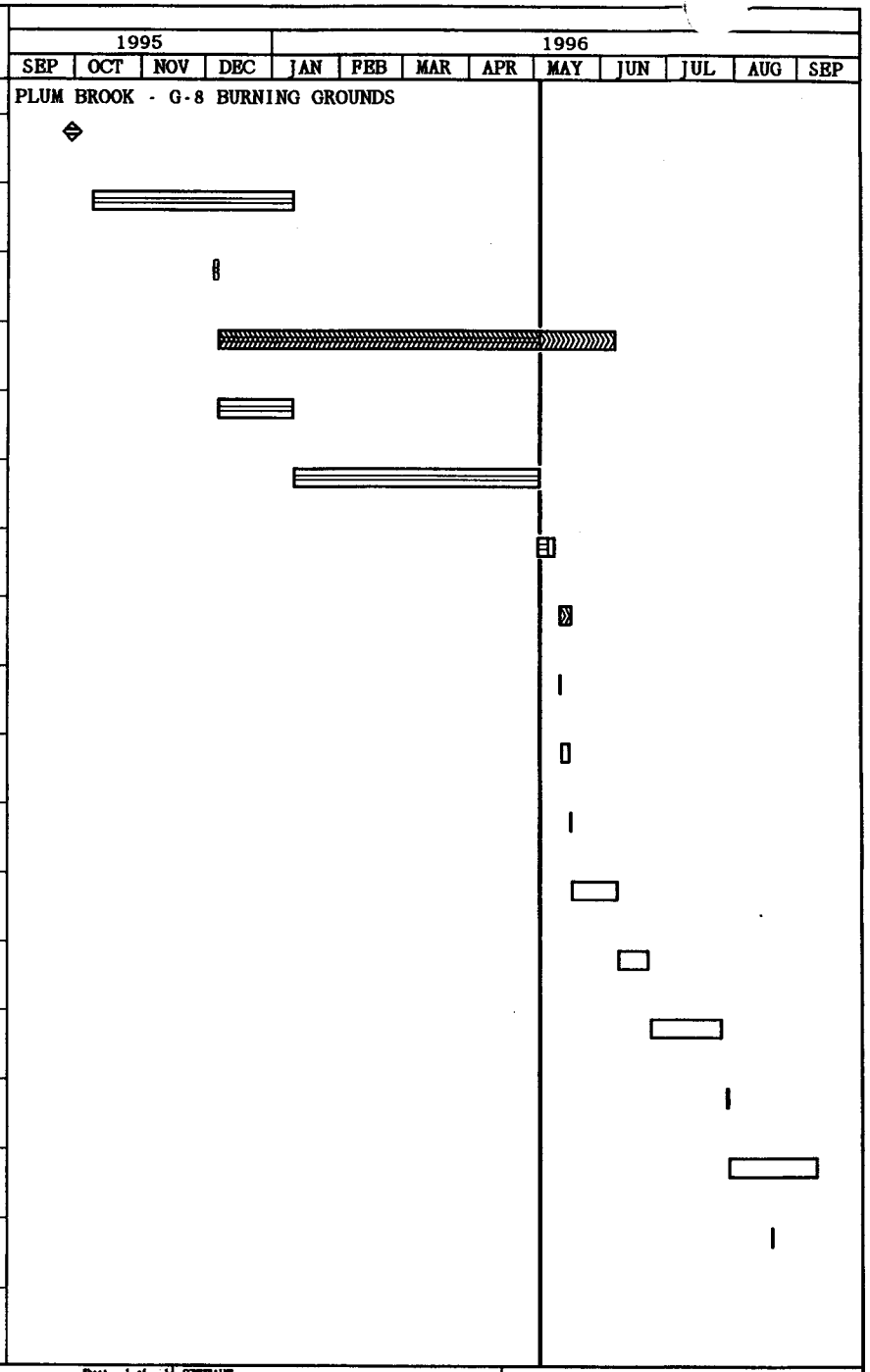
Figure 8-2

Changes (variances) to the approved work plan may be necessary as site conditions require and may be initiated either in the field or in the office. All variances will be noted on the Field Activity Daily Log and will be recorded on a Variance Log shown in Figure 8-2. Variances will be approved by the QA officer and the project manager prior to implementation of the change. Variances that affect the project scope, cost, or schedule must be approved by the USACE before being implemented and the health and safety officer must approve any variances to the SSHP. The project manager and QA officer will document their approval of the change by signing the Variance Log before the change is implemented in the field. The principal investigator or the field coordinator is responsible for initiating and chronologically maintaining all variances that are generated during the field work. The project manager's approval of a variance may be initiated on a verbal basis; such approval will be documented on the Variance Log and the original will be submitted to the project manager while a copy will be maintained in the field. If a variance is requested by the USACE, it will be so noted on the Variance Log.

9.0 Project Schedule

The project schedule is included in this final SAP.

ACTIVITY DESCRIPTION	EARLY START	EARLY FINISH	REM DUR
NOTICE TO PROCEED	30SEP95A		0
RECORDS REVIEW	9OCT95A	10JAN96A	0
SITE VISIT & COORDINATION MEETING	5DEC95A	6DEC95A	0
WORK PLANS	7DEC95A	7JUN96	25
PREPARE & SUBMIT DRAFT WORK PLANS TO USACE/EPA	7DEC95A	10JAN96A	0
REGULATORY REVIEW OF DRAFT WORK PLANS	11JAN96A	3MAY96A	0
REVISE DOCUMENT & SUBMIT FINAL WORK PLANS	3MAY96A	10MAY96	7
FIELD WORK & KICKOFF MEETING	13MAY96	18MAY96	6
MOBILIZATION	13MAY96	13MAY96	1
FIELD SAMPLING/TRENCHING	14MAY96	17MAY96	4
DEMOBILIZATION	18MAY96	18MAY96	1
ANALYTICAL WORK	19MAY96	8JUN96	21
DATA ANALYSIS	9JUN96	22JUN96	14
DRAFT SI REPORT	24JUN96	26JUL96	25
DRAFT SI REPORT MEETING	29JUL96	29JUL96	1
FINAL SI REPORT	30JUL96	9SEP96	30
INVESTIGATION DERIVED WASTE	19AUG96	19AUG96	1



Plot Date 9MAY96
Data Date 4MAY96
Project Start 10OCT95
Project Finish 9SEP96

(c) Primavera Systems, Inc.

Activity Bar/Early Dates
Critical Activity
Progress Bar
Milestone/Ping Activity

NCOE PBOW

Sheet 1 of 1

USACE - NASHVILLE DISTRICT
NCOE PBOW ENVIRONMENTAL RISK ASSESS.
G-8 BURNING GROUNDS

CONSTRAINT
ESE NAMOCK

SCHEDULE REVISIONS

Date	Revision	Checked	Approved

RESPONSE TO ER-H COMMENTS
SITE INVESTIGATION AT THE G-8 BURNING GROUND
FORMER PLUM BROOK ORDNANCE WORKS IN SANDUSKY, OHIO

Reference: *Fax dated 5/3/96 to Don Burton from Doug Webb.*

Comments on QAPP and SAP:

Comment 1: **Section 1.2. Edit third sentence.**

Response 1: The referenced sentence in the QAPP has been reviewed. It is unclear what the reviewer intended by the statement, and no changes have been made to the document.

Comment 2: **Section 7.2. Second paragraph, Add analyzed according to SW-846 methodology or IT Corporations Standard Operating Procedures.**

Response 2: The second paragraph of the referenced section of the QAPP has been edited to include a statement that analyses will be performed in accordance with SW-846 requirements.

Comment 3: **Section 7.3. First sentence. Add documented in the run-log.**

Response 3: The first sentence has been edited to state that instrument set-up, calibration, and operation will be documented in the run-log.

Comment 4: **Section 8.1. First paragraph. Edit last sentence. Add analyst will check computer output.**

Response 4: The referenced sentence has been revised to include the statement that the analyst will check computer output.

Comment 5: **Section 8.3. Add CLP-like format.**

Response 5: Section 8.3 has been revised to include CLP-like format.

Comment 6: **Figure 4-1. The sampling points on the figure appear to be in a pattern, not randomly placed.**

Response 6: As stated in Section 4.4.1, a systematic random sampling strategy utilizing a predetermined grid with regular sampling intervals has been adopted so as to minimize sample bias and maximize site coverage. In addition, Section 4.4.1.1 states that the depicted sample locations are tentative and will be adjusted based on site conditions by IT in consultation with the USACE. Therefore, no changes have been made to the document.

Comment 7: **Page 4-2, para. 4.4.1. This section appears contradictory. Were the sampling points randomly placed over the grid or were they selected systematically? This section seems to imply both. Please explain the methodology used in location selection.**

Response 7: The sampling points were randomly placed on the sampling grid for the purpose of showing tentative locations. These locations are subject to adjustment in the field based upon the results of the geophysical survey and the trenching efforts. As the exact limits of the burning grounds are not known at this time, the use of a grid-system for the placement of sample locations is considered appropriate pending the availability of additional field data.

Comment 8: **Page 4-3, para. 4.4.2.3. All soil sampling locations shall be logged on USACE ENG 5056R and 5056A-R forms. These are considered the only legal record of the boring/sampling event, and are to be legibly complete din the field. Any boring log not legible may be ground for not accepting that boring. Hand auger borings may be logged on the drill log cover sheet by describing soil details in the sketch area if there is room; otherwise the log continuation sheet should be used.**

Response 8: All soil sampling locations will be logged on USACE ENG 5056R and 5056A-R forms. The logs presented in the document have been revised accordingly.

Comment 9: **Page 4-6, para. 4.5.2.2. Log form ENG 5056 R should be completed in the field for each sediment sampling location.**

Response 9: All sediment samples will be logged using USACE ENG 5056R as requested.

Comment 10: **Page 4-4 Sample Identification Table: Please change the sample identification format to something similar to West Virginia Ordnance Works. This is necessary to allow the use of existing GIS table formats.**

Response 10: Sample designations, such as PBOW-96-SO01-BG8-xxxx-(0 - 0.5) were developed using the requirements of Table 2 of the statement of work, dated 1 June 1995. The sample numbering scheme can certainly be changed as requested, but this would not comply with the requirements of the SOW. For example, the sample referenced above would be numbered BG8SO-001-####, and would not include the PBOW, 96, or sample depth identifiers. No changes made to the document pending USACE direction.

Comment 11: **Page 4-8: Need to include a statement that all data sent to the Ohio River Division Laboratory needs to be bound and indexed. This will facilitate the review of the data.**

Response 11: The statement has been added.

Comment 12: **The Draft SSHP is ok for implementation? I don't have any significant comments, although, they should edit their documents for duplications, etc. (e.g., page 4 of 6, two vehicle traffic hazards under heavy equipment).**

Response 12: The SSHP has been edited for duplications.

Comments on Safety and Health Plan

Comment 1: **Section 2.6 Drill Rig Operators. This section is not applicable and should be deleted.**

Response 1: Although use of a drill rig is not anticipated for soil sampling, power augering by a Little-Beaver will be conducted. Section 2.6 has been revised to reflect this change and associated safety requirements.

Comment 2: **Section 3.1 Scope of Work. The phrase "Soil boring" doe snot appear applicable since the soil sampling involves auger samples. Recommend that the time of year when this work is to be performed be noted.**

Response 2: The phrase "soil boring" has been eliminated from the test. Current schedule for the field work has been noted in the test.

Comment 3: **Section 3.3. Physical Hazards. "Drilling hazards" is not applicable. The hazard is in the sue of an auger.**

Response 3: "Drilling hazards" has been revised to "power augering" since a Little-Beaver will be used during soil sampling.

Comment 4: **Section 3.3.1. Drilling Hazards. Delete all items under this section that apply to drilling and add appropriate safety hazards for use of augers. It is noted under "Utility Hazards" that there are no overhead or underground utilities, however, it is stated under Section 4.2.3, pg. 4-7, that a utility survey will be conducted prior to excavating. Please revise these sections to be consistent.**

Response 4: Appropriate safety hazards for operation of a power auger has been added to the text to replace drilling hazards. Section 4.2.3 has been revised as indicated.

Comment 5: **Table 3-1. (a) (1 of 6) Contact with moving equipment/vehicles should also note that backup alarms on vehicles are a recommended control.**

Response 5a: Table 3-1 (1 of 6) has been revised to include a backup alarm on vehicles as recommended.

(b) (2 of 6) Delete the entire table.

Response 5b: This table was revised to replace drilling with power augering and the content of the table has been revised to address safety issues associated with operation of a power auger.

(c) (3 of 6) Delete "drilling." For "Contact with potentially contaminated materials", it appears that use of PPE should be mandatory since presence of contamination is unknown, but likely.

Response 5c: The word "drilling" was deleted from text. Refer to Section 5.2.6. All site workers entering EZ will wear modified Level D PPE. Level C or high level will be required if air monitoring results warrant.

(d) (4 of 6) Vehicle Traffic. Should mention that vehicles shall have, and drivers and passengers shall use, seat belts. Should mention backup alarms. "Vehicle Traffic" is in this table below.

Response 5d: Vehicle here refers to heavy equipment such as dozer, front-end-loader, etc. These vehicles normally do not have seat belts. If a site worker operates a truck, wearing a seat belt is mandatory according to IT's safety policy.

(e) (6 of 6) The items in this table are listed twice.

Response 5e: This table has been edited for duplication.

Comment 6: **Section 3.6.2 Insects and Section 3.6.1 snakes. Since the work is to be performed in the month of February, these sections do not appear to be applicable. If the work is delayed until warmer months, these sections would apply and a section pertaining to Heat Stress would have to be added.**

Response 6: The field work is rescheduled for the month of May. Therefore, Section 3.6.2 and Section 3.6.1 are applicable. Also, Section 4.3, Cold Stress Prevention, was replaced with Heat Stress Prevention.

Comment 7: **Section 7.0 Decontamination. The phrase "drill rig" should be deleted in the third sentence of the first paragraph.**

Response 7: The phrase "drilling" has been replaced with "power auger."

Comment 8: **Table 8-2. Recommend that the work activity "drilling" be deleted since soil samples will be taken with augers and this activity is covered in "Sampling". The frequency of monitoring is incorrect (5 to 10 foot depth).**

Response 8: Table 8-2 has been revised per reviewers recommendation.

Comment 9: **Section 8.3 Noise Monitoring. The terms "drilling" and "drill rig" should be deleted from this section.**

Response 9: These terms have been deleted from Section 8.3.

Comments on Quality Assurance Project Plan

Comment 1: **Section 2.1 USACE Nashville District Project Manager. Recommend adding "Quality Assurance Review of IT's workplans, etc.," to the CEORN PM's responsibility list.**

Response 1: Concur. The recommended phrase has been added to the QAPP.

Comment 2: **Section 4.2.1 Nonsampling Equipment. Delete all equipment that relates to drilling equipment.**

Response 2: Concur. Deletion has been made per reviewer's comment.

Comments on Sampling and Analysis Plan

Comment 1: **General. Since the soil samples are to be taken with an auger, the term "soil boring" appears misleading. Recommend revising all occurrences of "soil boring" to "soil sample."**

Response 1: All occurrences of soil boring have been changed to soil sample.

Comment 2: **Figure 4-1. Recommend revising the legend for "SOIL BORING LOCATION" to "SOIL SAMPLE LOCATION".**

Response 2: The legend on Figure 4-1 has been revised as recommended by the reviewer.

Comment 3: **Section 4.4.2. Drilling Methods. Recommend renaming this section "Soil Sampling Methods" since the term "drilling" implies the use of a drill rig.**

Response 3: Section 4.4.2 has been renamed "Soil Sampling Methods" per reviewer's comments.