APPENDIX A
Accutest Laboratories
Certifications and Quality Assurance Manual

APPENDIX A-1
Accutest Laboratories Certifications

Accutest Laboratories

Statement of Qualifications

and Experience

2012

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Our Mission

To **SAFELY** produce **quality** data that is accurate, timely and of the highest **integrity**.

To provide **service** that consistently exceeds our clients' expectations.

To continuously improve our **performance** by developing and implementing the latest **technology**.

To achieve steady **growth** benefiting our clients, our employees and our company.

ACCUTEST LABORATORIES

Total Performance You Can Count On

Section 1

Introduction

Overview

Accutest Laboratories is a nationwide environmental testing laboratory that has successfully delivered defensible data for over 50 years. Founded in 1956, Accutest is the **nation's 3rd largest environmental testing laboratory combining advanced technology and experienced personnel to deliver "Total Performance You Can Count On".** Our commitment is to provide our clients with appropriate test methods that meet the most stringent quality standards in the environmental testing industry.

Headquartered in Dayton, New Jersey, Accutest operates from state-of-the-art, integrated laboratories in New Jersey, Massachusetts, Florida, Texas, Louisiana, Colorado, and California. **The Company operates over 220,000 square feet of total laboratory space.** Accutest maintains ten fully-staffed Service Centers conveniently located in New York, Pennsylvania, North Carolina, Louisiana, Illinois, Colorado, California, and Arizona. Accutest has a fleet of courier services for sample pickup and container delivery within a 100-mile radius of its laboratories. We also provide contract courier services nationwide.

Accutest provides reliable and comprehensive testing services including organic and inorganic analysis of air, water, soil, waste characterization, petroleum forensics, gas fractioning, energetics and explosives, and emerging contaminants to industrial, consulting/engineering and government clients in support of Federal and State Environmental Programs. **Accutest's extensive Federal and State regulatory experience** provides clients with the required expertise to assist in the development of analytical protocols and sampling and analysis plans.

Our staff consists of over **629 professionals** including chemists, biologists, chemical engineers, computer scientists, technicians, and support personnel. The Accutest senior management team averages over 16 years experience with the company. This translates into the environmental expertise that clients have come to rely on.

The National Environmental Laboratory Accreditation Program (NELAP) forms the foundation of our stringent **Corporate Quality Assurance Program**. This enables Accutest to hold multi-state accreditations and certifications that conform to a National standard. Accutest has also received Department of Defense Environmental Laboratory Accreditation (DoD ELAP) and ISO/IEC 17025:2005 Certificate of Accreditation from the Laboratory Accreditation Bureau (L-A-B) to perform environmental testing in support of environmental restoration programs.

LabLink^{SM}, the most comprehensive data retrieval and information management system in the industry, provides real-time data resources that enables clients to generate electronic data deliveries on demand 24/7/365 days a year.

Overview (continued)

Through LabLink[SM], **Accutest manages an extensive historical database of more than 915 million archived test results** which can be easily searched and retrieved.

Accutest's quality of service consistently exceeds our client's expectations. This is exemplified by the fact that **85% of our business is from repeat clients**. Accutest participates in National Corporate Analytical Programs which are routinely audited by independent third parties. Through steady, carefully managed growth, leading technology, and outstanding service, Accutest provides quality data of the highest integrity that is delivered reliably and uniformly to clients nationwide.

Client Services and Project Management

Accutest provides services that consistently exceed our clients' expectations. Our primary objective is to create and maintain long-term relationships with a dedication to quality, client services, technology and strong, consistent project management. We take great pride in our customer focus and the ability of our highly qualified staff to provide consistent, accurate information and support to the clients we serve.

Serving clients' needs and striving to find better ways to fulfill those needs has been the heart of Accutest since 1956. Our philosophy "Total Performance You Can Count On" is our promise to deliver the best customer service in the industry. Our clients can count on us to be there to address and resolve the challenges which they face.

We strive to enhance communications and teamwork through a work environment dedicated to continuous process evaluation, redesign and improvement. Our project managers have a sense of ownership which fosters their commitment to total customer satisfaction.

Clients are assigned to project managers who become their advocates and their dedicated representatives and contacts for all analytical inquiries. The assigned project manager ensures that client expectations and program objectives are met by obtaining a thorough understanding of all relevant technical and contractual requirements.

Effective and timely communication develops trusted client partnerships, which in turn leads to successful project outcomes. Our project managers are committed to delivering the full measure of our expertise to ensure complete client satisfaction with each and every Accutest service. Project managers understand through experience that successful completion of every project requires up-front planning. Accutest's Life Cycle of Project Management Services is exhibited by the following flow diagram.

Life Cycle of Project Management Services

Project Initiation	The client's decision to employ Accutest is the first step in the project lifecycle. Upon contacting Accutest, a project manager is assigned to the client who coordinates all project phases.
Specifications Defined	Project specifications originate from QAPPs, RFPs, work plans and other related documents. Accutest representatives provide the client with technical assistance, translating their specifications into clear laboratory instructions.
Project Setup & Bottle Configuration	Once the specifications are defined, the setup process begins. The representative develops a schedule with the client and generates the bottle order configuration to match the analytical specifications.
Bottle Order & Sample Pickup	Bottle kits are delivered to the client's site in advance of sampling. Accutest employs local courier services for most clients or commercial carriers when the site is beyond the courier's service range.
Chain-of-Custody & Login Review	Upon arrival at the laboratory, all samples are thoroughly checked to assure regulatory integrity. Chain-of-Custody information is translated to electronic login information and compared to the project specifications.
Status Monitoring & Update	The project manager verifies the login accuracy and resolves specification discrepancies prior to analysis. Representatives monitor project status during analysis, which is communicated to the client using Accutest's electronic tools.
Data Delivery	When the analysis is completed electronic reports are generated to the client's specifications, which can be delivered by using Accutest's sophisticated electronic tools or by conventional means.
Interpretation Assistance	Representatives are available to provide assistance after data delivery. Responses to client queries are prompt and accurate, frequently providing them with key information to aid their decision process.
Invoicing	Invoices are generated from the same electronic information used to communicate analytical specifications to the laboratory. This guarantees that it accurately reflects the specification requested by the client.

The efforts of our client service group; project managers, and technical sales representatives, field and analytical staff; combine to make Accutest an extension of your capability.

Laboratory Information Management System (LIMS)

The Accutest Laboratory Information Management System (LIMS) is the most critical and central tool in the operation of the laboratory. **Accutest's LIMS is one of the most powerful and sophisticated tools in the industry today**. The LIMS at each location resides on an independent server sized to ensure highly efficient performance for internal users and on-line customers simultaneously. The LIMS is based on a state-of-the-art Oracle relational database that has been specifically designed to handle the complex issues faced by environmental laboratories. The LIMS automates virtually every phase of laboratory operations, including sample receipt/login, sample scheduling and tracking, data acquisition, calculations, quality control (QC), final reports, electronic deliverables, and invoicing. As a result, it allows Accutest to deliver the most accurate and consistent product in the industry.

Electronic Data Deliverables

One of the most significant trends in this industry is the increased dependence on Electronic Data Deliverables (EDDs). EDDs that are produced or modified manually cannot be relied upon for accuracy. Accutest consistently provides the most accurate EDDs through totally automated data transfer. At the front end, virtually all laboratory data is transferred automatically to the LIMS without manual transcription, followed by multiple levels of technical review. From this point, the LIMS becomes the single source for all deliverables including data reports, QC reports, and EDDs. Simply put, this means that the raw data matches the paper reports, and the reports match the EDDs.

LabLink^{SM}

LabLink^{SM} is the live, on-line client interface to the Accutest LIMS, the most comprehensive on-line data service in the industry. Available via the internet to all Accutest clients, LabLink^{SM} provides real-time access to project status, current and historical data, on-line reports, EDDs, and billing information. LabLink^{SM} can be configured to send reports via email automatically upon data completion.

We provide clients with a personal, secure internet account that encrypts all communications to maintain data confidentiality. LabLink^{SM} has established a new standard for electronic data management. From the minute samples are received, LabLink^{SM} provides up-to-the-minute access to project information from a PC via a secure website.

The Most Comprehensive On-Line Service in the Industry, LabLink^{SM} service saves time, effort and money. Through LabLink^{SM}, Accutest has made a long term commitment to provide the most comprehensive on-line service in the industry that includes the following features:

- **Automated Sample Receipt Confirmation:** Allows client verification that samples have arrived at the laboratory safely and have been logged in properly. This ensures the correct tests, sample identification (IDs) and Turn-Around-Time (TAT) have been accurately communicated from the client to Accutest. An electronic copy of the chain-of-custody is delivered with the log in report for review.

LabLink[SM] (continued)

- **Compare Results to Multiple Regulatory Limits**: Summary tables compare project results to multiple regulatory limits in real time. The Tables include highlights for hits and exceedances.

- **Complete Project Status Information:** From the minute samples are received, there is complete access to job, sample, and test information. In addition, status information is available in **real time** through LabLink[SM], which allows tracking sample progress through the laboratory.

- **On-line Chain-of-Custody Documents:** As part of sample login, chain-of-custody documents are scanned into PDF files that are available on-line to LabLink[SM] users with a single click.

- **Immediate Access to Test Results:** LabLink[SM] provides complete access to test results the **minute** they are approved by the laboratory. The LabLink[SM] data query provides powerful options (**e.g. hits only**) to get data feedback as quickly as possible. The results can also be compared to a variety of Federal and State regulatory limits.

- **Access to Quality Control Data:** Method blank, MS/MSD, blank spike and surrogates are available on-line. Chromatograms, spectra and other raw data can also be reviewed.

- **Historical Data Query:** With LabLink[SM], it is just as easy to view historical data. Powerful query options and sort criteria can be specified and executed in seconds to evaluate trends.

- **e-Hardcopy Reports available via Auto-email or on-line:** Finished data may be generated in ***e-Hardcopy*** format complete with a signed cover page, chain-of-custody and comprehensive QC data. LabLink[SM] allows the user to activate Auto-email on projects of interest, which automatically initiates generation of an ***e-Hardcopy*** report that will be sent automatically upon completion of a job.

- **Billing/Invoice Information:** LabLink[SM] also allows access to preliminary billing information. This enables the user to check quote prices **before** the invoice is delivered. In addition, historical billing information is maintained, allowing up-to-the-minute project financial summaries. Final invoices can be downloaded on demand in PDF format.

- **Electronic Deliverables on Demand:** EDDs may be generated on demand through LabLink[SM]. This capability is unprecedented in the industry.

- **How to Get On-Line?** Getting started with LabLink[SM] couldn't be easier. All that is needed is access to the Internet. Accutest will then set up a secure LabLink[SM] account and user to be on-line. Best of all, access to LabLink[SM] is provided at no additional charge - simply for doing business with Accutest.

Section 2.0
Analytical Methodology & Regulatory Programs

Analytical Methodology

The Accutest staff has extensive knowledge and experience applying analytical chemistry methods employed in environmental monitoring. This includes methods approved for Federal and State environmental regulatory programs and in-house methods developed for client's unique analytical needs. This expertise combined with a quality assurance system that meets national and international standards enables the Company to consistently produce data of known and documented quality. **In 2011, Accutest logged in more than 107,532 jobs, processed over 576,768 samples and reported well over 1,809,276 tests.** All tests were performed within the framework of the Accutest Quality Assurance System in compliance with method specifications, regulatory requirements and client specific data quality objectives. Accutest routinely employs methods from the following compendiums:

- Test Methods for Evaluating Solid Waste, SW-846 (USEPA);
- Methods for Chemical Analysis of Water and Wastes (USEPA);
- Methods for Organic Chemical Analysis of Municipal and Industrial Wastewater (USEPA);
- Methods for the Determination of Metals in Environmental Samples (USEPA);
- Methods for the Determination of Inorganic Substances in Environmental Samples (USEPA);
- Methods for the Determination of Organic Compounds in Drinking Water (USEPA);
- Compendium of Methods for the Determination of Toxic Organic Compounds in Ambient Air (USEPA);
- Standard Methods for the Examination of Water and Wastewater (APHA, AWWA.WEF); and
- Technical Standards – American Society for Testing and Materials (ASTM).

Regulatory Programs

Analysis of environmental samples using methods from these compendiums is performed in accordance with Federal and State regulatory programs including:

- RCRA – Resource Conservation and Recovery Act (USEPA);
- CWA – Clean Water Act (USEPA);
- NPDES – National Pollution Discharge Elimination System (USEPA);
- SDWA – Safe Drinking Water Act (USEPA);
- CERCLA – Comprehensive Environmental Response Compensation Liability Act (USEPA);
- CAA – Clean Air Act (USEPA);
- TSCA – Toxic Substances Control Act (USEPA);
- OSHA – Occupational Safety and Health Act;
- Brownfields Recovery Act (USEPA); and
- Numerous State Specific Programs Supporting Waste Management Activities and Natural Resource Protection.

Analytical Methodology & Regulatory Programs (continued)

Analytical Support Activities

Accutest routinely produces analytical data in support of projects that require testing in conformance with Federal and State remediation and regulatory compliance programs including:

- Site Characterization Investigations;
- Remedial Investigations and Feasibility Studies;
- Remedial Action Activities (Cleanup and/or Removal);
- Delineation Monitoring;
- Groundwater Monitoring (Natural Resource Protection & Drinking Water);
- Underground Storage Tank Monitoring and Cleanup;
- Ambient & Indoor Air Monitoring;
- NPDES Compliance Monitoring;
- Hazardous Waste Identification/Classification;
- Hydraulic Fracturing Groundwater Contaminants; and
- Hydraulic Fracturing Fluids Analysis

Gas Exploration/Hydraulic Fractioning

Accutest has broad expertise providing analytical chemistry services in support of Hydraulic Fracturing activities. Hydraulic Fracturing support analysis are conducted in all Accutest facilities and include the analysis of fracturing fluids, flow-back waters and pre- and post- drilling groundwater.

Fracturing Fluid Analysis includes Metals, Acrylamides, Cyanides, Phenolics, Residual Chlorine, Ions, Volatile Organics and Semi-Volatile Organics using a combination of USEPA methodology. Method selection is customized to reflect the constituents of each drilling fluid.

Groundwater Analysis includes pre- and post-drilling monitoring including Metals Acrylamides, Cyanides, Phenolics, Herbicides, Ions, Volatile Organics, Semi-Volatile Organics, Pesticides and PCBs, Tetrachlordibenzodioxin (TCDD) using a combination of USEPA SW-846 methodology.

Standard pre-drilling parameters; Inorganic include Alkalinity, Chloride, Conductivity, Hardness, Oil and Grease, pH, Sulfate, TDS, TSS, Turbidity, MBAS (surfactants); Metals: Barium, Calcium, Iron, Magnesium, Manganese, Potassium, Sodium, Strontium, Lithium, Cadmium, Mercury, Silver, Lead, Arsenic, Chromium, Selenium, Sulfur. Organics include BTEX, dissolved gases (methane, ethane, butane, propane), TPH (GRO, DRO).

Drinking Water Analysis includes Metals, Cyanides, Coliforms, Phenolics, Ions, Volatile Organics, Semi-Volatile Organics, Haloacetic Acids and Dibromoethane (EDB) using USEPA drinking water methods.

Air Analysis includes USEPA TO-3, TO-14 and TO-15 for volatile organics.

Reports and Deliverables

Accutest's user friendly data reports can be produced in several formats varying in complexity from basic results to fully documented deliverables depending on client needs. Regardless of the report format, all analysis is performed to meet the quality control specifications of the analytical method and the specified regulatory program. The delivered report can therefore be configured with full confidence that the data is supported by the required quality control practices. These reports are designed for easy interpretation and efficient data validation.

Reports can be produced in hard copy format or as a fully indexed electronic document. Accutest specializes in electronic data products, which not only complement the data report, but also provide the client with numerous electronic products and delivery options that simplify data management and review. These options include EDDs in over 1093 data formats, including commercial and custom client formats, which are delivered in over 95% of projects. Access to leading edge, interactive online features enables the user to configure data reports that meet data reporting needs, including the ability to automatically receive data upon completion which has been formatted to client specifications. Hardcopy and electronic deliverable options are as follows:

Hard Copy

- Full Deliverables – Comprehensive, Validation Ready (Level 4)
- Reduced Deliverables – Summary Data with Quality Control (Level 2 & 3)
- Standard Commercial Format – Results only (Level 1)
- State – Specific Formats

Electronic

- GIS/Key, Locus & more)
- Customized Client Specific Database Import Formats
- Custom Spreadsheet Reports
- State Regulatory Required EDD Formats
- Department of Defense (ADR, SEDD, IRPIMS & others)

Interactive Online Reporting

- **LabLink**^{SM} – Accutest Proprietary On-Line Data Management Service
- **e-Hard Copy** – Fully Navigable, Indexed, Interactive PDF Report

Section 3.0

Quality Assurance Program

At Accutest, we continually build quality into the product delivered to clients as a design specification. This is accomplished by incorporating the elements of our Quality System into every laboratory process as an intrinsic component of day-to-day operations. This approach reflects our dedication to a quality system that meets national and international standards. This is achieved through a Corporate Quality Assurance Program that establishes the framework for the quality systems operated and maintained at each Accutest facility. This Program reflects knowledge of the regulatory analytical process and the role our product plays in safeguarding human health and the environment.

Quality Assurance Program (continued)

Policy Statement

The management and staff of Accutest share the responsibility for product quality. The Quality System is designed to ensure that all processes and procedures, which are components of environmental data production, meet established industry requirements. These processes and procedures must be adequately documented from a procedural and data traceability perspective as executed by the staff. It also assures that analytical data of known quality, meeting the quality objectives of the analytical method in use and the data user's requirements, are consistently produced in the laboratory. This enables the ultimate data user to make rational, confident, cost-effective decisions on the assessment and resolution of environmental issues.

The Quality System provides staff with data quality and operational feedback data. This enables a determination whether the laboratory has achieved the established quality and operational standards, which are dictated by the client or established in regulation. The information obtained from the Quality

System is used to assess operational performance from a quality perspective and to perform corrective action as necessary.

The Quality Assurance Standard

Accutest operates a quality program which meets the requirements for laboratory operations established by the international community. Accutest has implemented a Quality System that follows ISO Guide 17025, General Requirements for the Competence of Calibration and Testing Laboratories. The structure of ISO Guide 17025 has been adopted by the National Environmental Laboratory Accreditation Program (NELAP), the voluntary national accreditation program originally established by the USEPA. NELAP has established a uniform national standard for environmental laboratories that places a strong emphasis on quality systems.

The Quality System at Accutest has been designed to meet NELAP Standards. Accutest was among the first laboratories to submit applications to the approved state accrediting authorities for recognition as a NELAP Accredited laboratory. All laboratories in the Accutest network are NELAP Accredited.

Corporate Quality Assurance Program

The Corporate Quality Assurance Program establishes the quality framework for each individual Accutest facility. The Corporate Quality Assurance Director determines corporate policies and defines the quality responsibilities at the facility level. He is responsible for monitoring the quality system at each facility and providing feedback to the management staff.

The reporting relationship between Corporate Quality Assurance and the Quality Assurance Officers at each laboratory reflects a dotted line responsibility. This type of relationship enables the Corporate Program to be implemented locally while enhancing day-to-day operational efficiency at each facility.

Quality Assurance at Each Laboratory

The Accutest philosophy enables each facility to implement a quality policy using their unique operating style. This approach provides the autonomy needed to meet the requirements of the local regulatory jurisdictions using procedures that efficiently meet their operational objectives.

The Quality Assurance Program at each facility incorporates the elements of NELAP and ISO Guide 17025. The operations management staff is responsible for implementing the program elements and operating the quality system.

The Quality Assurance Officer monitors the program, provides feedback to local and corporate management, and assists with corrective action and training if needed.

In order to measure the success of the Corporate Quality Assurance Program, Accutest participates in the following regulatory proficiency test programs:

- Water Pollution Study (NELAP fields of testing);
- Water Supply Study (NELAP fields of testing);
- RCRA Water PT Samples;
- RCRA Soil PT Samples;
- Independent Single Blind PT Samples; and
- Independent Double Blind PT Samples.

Accutest also participates in numerous national corporate proficiency test programs/audits sponsored by our clients, utilizing independent, third party consulting firms.

Accutest State Certifications & Accreditations

Accutest maintains accreditation for the majority of the state regulatory analytical programs offered in the United States. The program specific accreditations maintained in each state are essential for submitting analytical data to meet data reporting requirements.

Many states maintain accreditation programs for drinking water only. In these states, accreditation is not required to conduct analysis for other regulatory programs administered by them. The Accutest State Certification & Accreditation Map is available at www.accutest.com. Or, you may contact your Accutest Representative for a current list of our certifications.

Accutest Laboratories' Certifications, Accreditations & Permits
November 1, 2013

New Jersey Facility (Mid-Atlantic) - USEPA Federal Identification Number NJ00141
Florida Facility (Southeast) - USEPA Federal Identification Number FL00946
Texas Facility (Gulf Coast) - USEPA Federal Identification Number TX01484
Massachusetts Facility (New England) - USEPA Federal Identification Number MA00136
California Facility (West) - USEPA Federal Identification Number CA00150
Colorado Facility (Mountain States) - USEPA Federal Identification Number CO00049
Louisiana Facility (ALGC-Louisiana) - USEPA Federal Identification Number LA00013

Certifying Authority	Accutest Facility	Certification Program	Registration No.	Expiration Date
Alaska	Mid Atlantic	Contaminated Sites	UST-103	May-14
Alaska	Mid Atlantic	Potable Water	NJ00141	June-14
Alaska	Southeast	Contaminated Sites	UST-088	August-13
Alaska	West	Contaminated Sites	UST-092	September-13
Arizona	Gulf Coast	Non-Potable Water, Solid/Hazardous Waste, Air Toxics	AZ0769	July-14
Arizona	Mid Atlantic	Air Toxics	AZ0786	September-14
Arizona	West	Non-Potable Water, Solid/Hazardous Waste	AZ0762	November-13
Arkansas	ALGC-Louisiana	Solid/Hazardous Wastes, Non-Potable Water	88-0734	August-13
Arkansas	Gulf Coast	Non-Potable Water, Solid/Hazardous Waste	88-0756	March-14
Arkansas	Southeast	Solid/Hazardous Wastes, Non-Potable Water	88-0620	September-13
California (NELAP)	Mid Atlantic	Potable/Non-Potable Water, Solid/Hazardous Waste	01152CA	July-14
California (NELAP)	Southeast	Potable Water, Solid/Hazardous Waste	04226CA	June-14
California (NELAP)	West	Non-Potable Water, Solid/Hazardous Waste	08258CA	July-14
Colorado	Mountain States	Potable Water	None	May-14
Colorado	New England	Potable Water	None	January-14
Connecticut	Mid Atlantic	Potable/Non-Potable Water, Solid/Hazardous Waste	PH-0585	June-15
Connecticut	New England	Potable/Non-Potable Water, Solid/Hazardous Waste, ETPH	PH-0109	June-15
Delaware	Mid Atlantic	DNREC HSCA Program	Not Applicable	Recognized
DoD ELAP	Mid Atlantic	Non-Potable Water, Solid/Chemical Waste, Air Toxics	L2248	April-16
DoD ELAP	New England	Non-Potable Water, Solid/Chemical Waste, Air Toxics	L2235	January-16
DoD ELAP	Southeast	Non-Potable Water, Solid/Chemical Waste	L2229	December-15
DoD ELAP	West	Non-Potable Water, Solid/Chemical Waste	L2242	May-16
Florida (NELAP)	ALGC-Louisiana	Potable, Non-Potable, Solid Waste	E87657	June-14
Florida (NELAP)	Gulf Coast	Non-Potable Water, Solid/Hazardous Waste, Air & Emissions	E87628	June-14
Florida (NELAP)	Mid Atlantic	Potable, Non-Potable, Solid Waste, UST, Air Toxics	E87482	June-14
Florida (NELAP)	New England	Potable, Non-Potable Water/Solid/Hazardous Waste, Air Toxics	E87579	June-14
Florida (NELAP)	Southeast	Potable, Non-Potable, Solid Waste, Air Toxics	E83510	June-14
Georgia	ALGC-Louisiana	Non-Potable Water, Solid/Chemical Materials	None Provided	June-14
Georgia	New England	Non-Potable Water, Solid/Chemical Materials, Air and Emissions	None Provided	June-14

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Texas Facility (Gulf Coast) - USEPA Federal Identification Number TX01484
Massachusetts Facility (New England) - USEPA Federal Identification Number MA00136
California Facility (West) - USEPA Federal Identification Number CA00150
Colorado Facility (Mountain States) - USEPA Federal Identification Number CO00049
Louisiana Facility (ALGC-Louisiana) - USEPA Federal Identification Number LA00013

Certifying Authority	Accutest Facility	Certification Program	Registration No.	Expiration Date
Georgia	Southeast	Non-Potable Water, Solid/Chemical Materials, Air and Emissions	None Provided	June-14
Idaho	Mountain States	Potable Water	CO00049	May-14
Illinois (NELAP)	Mid Atlantic	Potable & Non-Potable Water; Haz Waste	002515	May-14
Illinois (NELAP)	New England	Potable & Non-Potable Water; Haz Waste	20018-003245	July-14
Illinois (NELAP)	Southeast	Non-Potable Water; Haz Waste	200063-003253	August-14
Indiana	Mid Atlantic	Potable Water	C-NJ-03	June-14
Iowa	Southeast	UST, Solid/Hazardous Wastes, Non-Potable Water	IA366	February-14
ISO/IEC 17025:2005	Mid Atlantic	Non-Potable Water, Solid/Chemical Waste, Air Toxics	L2248	April-16
ISO/IEC 17025:2005	New England	Non-Potable Water, Solid/Chemical Waste, Air Toxics	L2235	January-16
ISO/IEC 17025:2005	Southeast	Non-Potable Water, Solid/Chemical Waste	L2229	December-15
ISO/IEC 17025:2005	West	Non-Potable Water, Solid/Chemical Waste	L2242	May-16
Kansas (NELAP)	Gulf Coast	Solid/Hazardous Wastes, Non-Potable Water	E-10366	July-14
Kansas (NELAP)	Mid Atlantic	Potable/Non-Potable Water, Solid/Hazardous Wastes	E-10356	October-14
Kansas (NELAP)	Southeast	Solid/Hazardous Wastes, Non-Potable Water	E-10327	October-13
Kentucky	ALGC-Louisiana	Underground Storage Tank Program	31	June-14
Kentucky	Gulf Coast	Underground Storage Tank Program	96	March-14
Kentucky	Mid Atlantic	Potable Water	90131	December-13
Kentucky	New England	Underground Storage Tank Program	97	June-14
Kentucky	Southeast	Underground Storage Tank Program	0065	June-14
Louisiana (NELAP)	ALGC-Louisiana	Non-Potable Water, Solid/Hazardous Waste	02048	June-14
Louisiana (NELAP)	Gulf Coast	Non-Potable Water, Solid/Hazardous Waste, Air Emissions	04004	June-14
Louisiana (NELAP)	Mid Atlantic	Non-Potable Water, Solid/Hazardous Waste, Air Toxics	04106	June-14
Louisiana (NELAP)	Southeast	Non-Potable Water, Solid Chemical Materials	03051	June-14
Louisiana DHH (NELAP)	ALGC-Louisiana	Potable Water	LA110022	December-13
Maine	New England	Potable & Non-Potable Water, Maine DRO/GRO, RCRA, Air Toxics	MA00136-2012004	April-14
Maryland	Mid Atlantic	Potable Water	167	March-14
Massachusetts	Mid Atlantic	Potable/Non-Potable Water	NJ141	June-14
Massachusetts	New England	Potable & Non-Potable Water	M-MA136	June-14
Massachusetts	Southeast	Non-Potable Water	M-FL946	June-14

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Colorado Facility (Mountain States) - USEPA Federal Identification Number CO00049
Louisiana Facility (ALGC-Louisiana) - USEPA Federal Identification Number LA00013

Certifying Authority	Accutest Facility	Certification Program	Registration No.	Expiration Date
Minnesota	New England	Potable/Non-Potable Water, Solid/Chemical Waste, Air Toxics	025-999-441-508250	December-13
Mississippi	Southeast	Potable Water	Not Applicable	N/A
Montana	ALGC-Louisiana	Petroleum Release Section	Not Applicable	Recognized
Montana	Mid Atlantic	Petroleum Release Section	Not Applicable	Recognized
Montana	New England	Petroleum Release Section	Not Applicable	Recognized
Nebraska	Mountain States	Private/Non-Compliance Potable Water	None	May-14
Nevada	Southeast	Non-Potable Water, Solid/Hazardous Wastes	FL009462013-1	July-14
Nevada	West	Potable/Non-Potable Water, Solid/Hazardous Wastes	CA001502010A	July-14
New Hampshire (NELAP)	New England	Potable/Non-Potable Water, Solid & Chemical Materials	250212+D26	January-14
New Hampshire (NELAP)	New England	Potable/Non-Potable Water (1,4-Dioxane SIM)	206113	July-14
New Jersey (NELAP)	Mid Atlantic	Potable/Non-Potable Water, Solid Waste, Air Toxics	12129	June-14
New Jersey (NELAP)	Mountain States	Potable/Non-Potable Water, Solid/Hazardous Waste	CO007	June-14
New Jersey (NELAP)	New England	Non-Potable Water/Solid/Hazardous Waste	MA926	June-14
New Jersey (NELAP)	Southeast	Solid/Hazardous Wastes, Non-Potable Water	FL002	June-14
New York (NELAP)	Mid Atlantic	Potable/Non-Potable Water, Solid/Hazardous Waste, Air	10983	April-14
New York (NELAP)	New England	Potable/Non-Potable Water/Solid/Hazardous Waste, Air	11791	April-14
North Carolina	ALGC-Louisiana	Solid/Hazardous Wastes, Non-Potable Water	487	December-13
North Carolina	Mid Atlantic	Solid/Hazardous Wastes, Non-Potable Water	346	December-13
North Carolina	New England	Solid/Hazardous Wastes, Non-Potable Water	653	December-13
North Carolina	Southeast	Solid/Hazardous Wastes, Non-Potable Water	573	December-13
North Dakota	Mountain States	Potable Water/Non-Potable, Solid/Hazardous Waste	R-027	January-14
North Dakota	New England	Potable/Non-Potable Water, Solid/Hazardous Waste	R-188	June-14
Ohio Voluntary Action Pr.	Mid Atlantic	Solid/Hazardous Wastes, Non-Potable Water	CL0056	January-15
Oklahoma	Gulf Coast	Non-Potable Water, Solid/Hazardous Waste	9103-2013-031	August-14
Oklahoma	Southeast	Non-Potable Water, Solid/Hazardous Waste	9959-2013-089	August-14
Oklahoma	Mountain States	Potable Water	D9942-2013-089	August-14
Oregon (NELAP)	West	Potable & Non-Potable Water; Haz Waste	CA200011-007	October-14
Oregon (NELAP)	West	NWTPH-Dx/Gx in Non-Potable Water, Haz Waste	CA300006-002	July-14
Pennsylvania (NELAP)	Mid Atlantic	Potable & Non-Potable Water; Haz Waste	68-00408-010	May-14

Accutest Laboratories' Certifications, Accreditations & Permits
November 1, 2013

New Jersey Facility (Mid-Atlantic) - USEPA Federal Identification Number NJ00141
Florida Facility (Southeast) - USEPA Federal Identification Number FL00946
Texas Facility (Gulf Coast) - USEPA Federal Identification Number TX01484
Massachusetts Facility (New England) - USEPA Federal Identification Number MA00136
California Facility (West) - USEPA Federal Identification Number CA00150
Colorado Facility (Mountain States) - USEPA Federal Identification Number CO00049
Louisiana Facility (ALGC-Louisiana) - USEPA Federal Identification Number LA00013

Certifying Authority	Accutest Facility	Certification Program	Registration No.	Expiration Date
Pennsylvania (NELAP)	New England	Non-Potable, Solid/Hazardous Waste	68-01121	September-14
Pennsylvania (NELAP)	Southeast	Non-Potable, Solid/Hazardous Waste	68-03573-001	August-14
Rhode Island	Mid Atlantic	Potable/Non-Potable Water, Air	LAO00176	December-13
Rhode Island	New England	Potable/Non-Potable Water	LAO00071	December-13
South Carolina	ALGC-Louisiana	Potable/Non-Potable Water, Solid/Hazardous Waste	73004001	June-13
South Carolina	Mid Atlantic	Solid/Hazardous Wastes, Non-Potable Water	94009001	June-14
South Carolina	Southeast	Solid/Hazardous Wastes, Non-Potable Water	96038001	June-14
Texas (NELAP)	ALGC-Louisiana	Potable/Non-Potable Water, Solid/Hazardous Waste	T104704186-12-5	December-13
Texas (NELAP)	Gulf Coast	Non-Potable Water, Solid/Hazardous Waste	T104704220-13-10	March-14
Texas (NELAP)	Mid Atlantic	Non-Potable Water, Solid/Hazardous Waste, Air Toxics	T104704234-13-7	July-14
Texas (NELAP)	Mountain States	Potable Water (Non-Compliance)	T104704511-13-2	February-14
Texas (NELAP)	Southeast	Non-Potable Water, Solid & Chemical Materials	T-104704404-13-5	May-14
US Army Corps of Eng.	Gulf Coast	HTRW, Solid/Hazardous Waste	Not Applicable	Self Declared
US Dept. of Agriculture	ALGC-Louisiana	Foreign Soils Permit	P330-11-00394	November-14
US Dept. of Agriculture	Gulf Coast	Foreign Soils Permit	P330-13-00012	January-16
US Dept. of Agriculture	Mid Atlantic	Foreign Soils Permit	P330-12-00102	April-15
US Dept. of Agriculture	New England	Foreign Soils Permit	P330-13-00053	February-16
US Dept. of Agriculture	Southeast	Foreign Soils Permit	P330-13-00090	March-16
US Dept. of Agriculture	West	Foreign Soils Permit	P330-12-00338	December-15
Utah (NELAP)	Mountain States	Potable, Non-Potable Water, Solid/Hazardous Wastes	CO000492012-2	January-14
Utah (NELAP)	Southeast	Potable, Non-Potable, Solid/Chemical Materials	FL009462013-3	July-14
Virginia	Mid Atlantic	Potable Water	00004-1783	June-14
Virginia (NELAP)	Gulf Coast	Air Toxics	460226-2085	March-14
Virginia (NELAP)	Mid Atlantic	Potable, Non-Potable, Solid/Chemical Materials, Air Toxics	460174-2425	September-14
Virginia (NELAP)	Southeast	Potable, Non-Potable, Solid/Chemical Materials	460177-2426	September-14
Washington	Southeast	Non-Potable, Solid/Chemical Materials	C918	March-14
Washington	West	Potable, Non-Potable, Solid/Chemical Materials	C925-13	October-14
West Virginia	ALGC-Louisiana	Non-Potable Water, Solid/Hazardous Wastes	257	June-14
West Virginia	Mid Atlantic	Non-Potable Water, Solid/Hazardous Wastes	329	October-13

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Colorado Facility (Mountain States) - USEPA Federal Identification Number CO00049
Louisiana Facility (ALGC-Louisiana) - USEPA Federal Identification Number LA00013

Certifying Authority	Accutest Facility	Certification Program	Registration No.	Expiration Date
Wisconsin	New England	Potable, Non-Potable Water, Solid/Hazardous Wastes	399080220	August-14
Wyoming	Mountain States	Potable Water	Not Applicable	May-14

Audits & Proficiency Testing

Accutest participates in national proficiency test programs to maintain the accreditations required by the NELAP states and those required by non-NELAP states. Accutest traditionally obtains acceptable performance on over 98% of the parameters evaluated. In 2011 Accutest reported results for over 14,000 proficiency test parameters performed under the following programs:

- Water Pollution Study (NELAP fields of testing);
- Water Supply Study (NELAP fields of testing);
- RCRA Water PT Samples; and
- RCRA Soil PT Samples
- Clean Air Act Emissions PT Samples

Accutest also participates in numerous single and double blind proficiency test programs sponsored by national, corporate clients, utilizing independent, third party consulting firms.

Audits are an essential component of Accutest's Quality Assurance Program. Accutest conducts extensive, internal audits of every aspect of the analytical system annually. This includes an assessment of numerous analytical methods and an assessment of the laboratory quality system. Audit findings are the basis of corrective actions that continually elevate the Company's performance.

Audits are also conducted by state accrediting bodies for initial and continued accreditation. Findings from these audits are also used to improve process performance.

Accutest continuously monitors key indicators to measure operational efficiency which is essential to project execution. The indicators used to evaluate performance include analytical report turn-around-time, reissued reports, holding times and rejected data.

Statistical evaluations are used to measure and refine overall performance, promoting continuous improvement.

Section 4.0

Facilities and Resources

Laboratories

Accutest's state-of-the-art facilities have been designed specifically for environmental testing. Each facility is functionally designed to provide efficient processing of a large volume of samples and provide a comfortable, safe working environment for laboratory staff. The facilities are equipped with centralized process gas distribution, water purification centers and separate, dedicated HVAC systems. These systems maintain critical positive/negative pressure relationships between internal laboratories, ensuring adequate ventilation and preventing atmospheric cross-contamination. A site location map is provided on the next page.

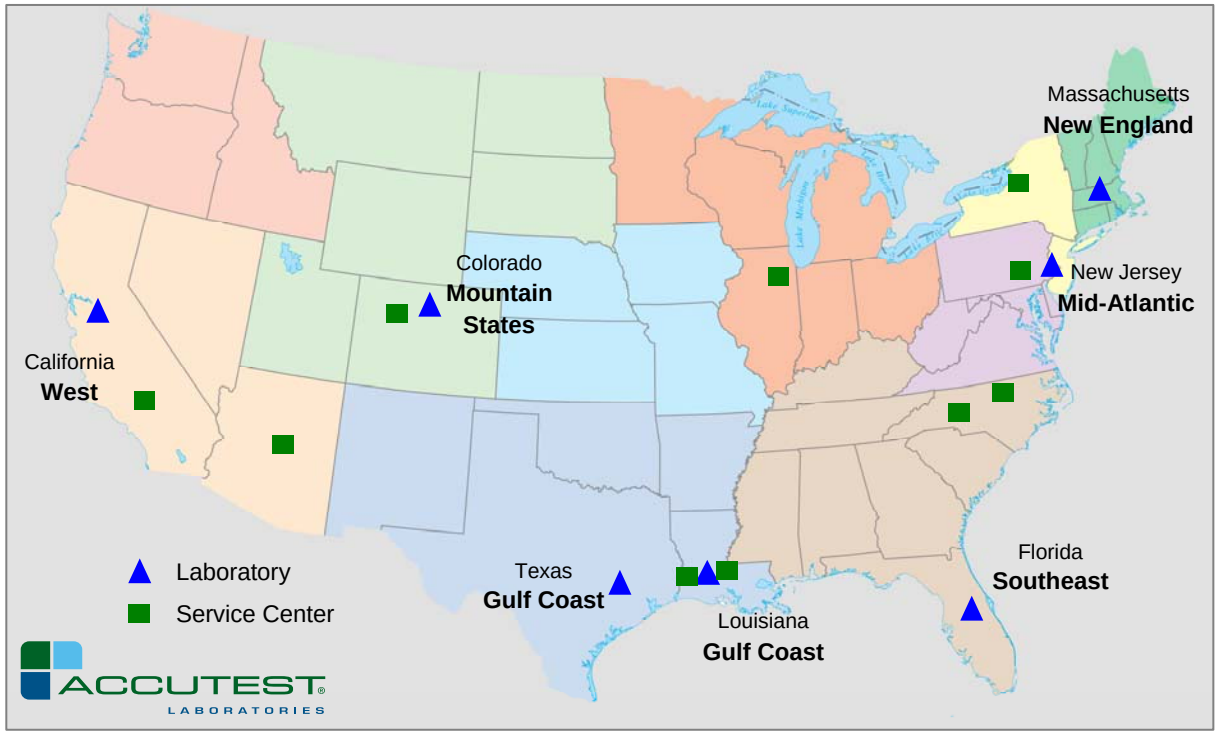
Service Centers

Accutest operates ten (10) Service Centers conveniently located in the States of New York, Pennsylvania, North Carolina, Louisiana, Illinois, Colorado, California, and Arizona to ensure that samples can be shipped to each of the laboratories quickly and to accommodate project specific locations in that region.

Courier Services

Accutest maintains a fleet of couriers that handle sample pickup and container delivery within a 100-mile radius of our laboratories. All employees are trained and have experience in handling environmental samples and sample documentation. Accutest has established contract courier services in a number of other states to provide local delivery and pickup services.

Accutest Locations



Laboratories

Mid-Atlantic
2235 US HWY 130
Dayton, NJ 08810
Tel: 732-329-0200

New England
495 Tech Center West
Marlborough, MA 01752
Tel: 508-481-6200

Southeast
4405 Vineland Road
Orlando, FL 32811
Tel: 407-425-6700

Gulf Coast (TX)
10165 Harwin Drive
Houston, TX 77036
Tel: 713-271-4700

Gulf Coast (LA)
500 Ambassador Caffery Pkwy
Lafayette, LA 70583
Tel: 337-237-4775

Mountain States
4036 Youngfield Street
Wheat Ridge, CO 80033
Tel: 303-425-6021

West
2105 Lundy Avenue
San Jose, CA 95131
Tel: 408-588-0200

Sales Centers: Southbury, CT Nashville, TN Tampa, FL St. Louis, MO

Service Centers

Syracuse Service Center
6780 Northern Blvd. Ste 202
East Syracuse, NY 13057
Tel: 315-329-4763

Exton Service Center
924 Springdale Drive
Exton, PA 19341
Tel: 610-363-7400

Raleigh Service Center
6308 Angus Drive, Ste C
Raleigh, NC 27617
Tel: 919-208-7171

Charlotte Service Center
2828-C Queen City Drive
Charlotte, NC 28208
Tel: 704-929-1533

Baton Rouge Service Center
17485 Opportunity Ave. Ste 1B
Baton Rouge, LA 70817
Tel: 225-752-8929

Lake Charles Service Center
2818 S. Beglis Pkwy
Sulphur, LA 70665
Tel: 337-287-4879

Chicago Service Center
1449 Tonne Road
Elk Grove Village, Illinois 60007
Tel: 847-258-3057

Rifle Service Center
3161 Baron Lane, Unit F
Rifle, CO 81650
Tel: 970-309-5460

Irvine Service Center
17165 Von Karman Ave. Ste 112
Irvine, CA 92614
Tel: 949-250-9900

Phoenix Service Center
1741 W. University Drive, #149
Tempe, Arizona 85281
Tel: 602-501-5673

Equipment and Instrumentation

Accutest maintains automated, computerized analytical instrumentation to support large complex projects and routine analyses of standard analytical parameters. Our commitment to clients is reflected by the capital investment in facilities, equipment and technology. Our production capability, capacity and redundancy of instrumentation assure the reliability and performance needed to deliver major analytical projects successfully. Instruments are dedicated to specific matrices and analyses to accelerate productivity and prevent cross contamination. A major factor when evaluating a laboratory is the age, model and condition of its equipment. Accutest maintains a significant proportion of advanced late model instrumentation.

Major Instrumentation Summary

Analytical Instrument	Accutest Network Total
GC/MS VOA	93
GC/MS SVOA	38
GC VOA	46
GC SVOA	83
HPLC	15
ICP/ICPMS	18/7

Health and Safety Program

Accutest operates a formal Health and Safety Program that complies with the requirements of the Occupational Safety and Health Administration (OSHA). Our goal is to provide a safe and healthy working environment for our employees and clients while protecting the public and preserving the Company's assets and property. Accutest complies with all applicable government regulations pertaining to the safety and health in the laboratory and the workplace.

The objective of our Health and Safety Program is to promote safe work practices that minimize the occurrence of injuries and illness to the staff through proper health and safety training, correct laboratory technique application and the use of engineering controls. The program consists of key policies and practices that are essential to safe laboratory operation. All employees receive training on the program's elements. Job specific training is conducted to ensure safe practices for specified tasks. All employees are required to participate in the program, receive initial and annual training, and comply with the program requirements.

Key elements of the program include Safety Education and Training, Hazard Communication, Chemical Protection (with an emphasis on Personal Protective Equipment and Engineering Controls), Chemical Hygiene, Waste Handing and Emergency procedures. The program is administered at the corporate level by the Health & Safety Director, and each facility has a designated Health & Safety Officer and Safety Committee. Regular facility audits are conducted to ensure compliance to the program.

All field personnel receive appropriate OSHA training which is updated on a regular basis. In addition, they receive training and approval to cover client and site specific requirements.

Field Services

Accutest's Field Services Department offers Occupational Safety and Health Administration (OSHA) certified sampling technicians to support environmental projects. Accutest has the capacity to fully equip and mobilize teams utilizing the appropriate level of protection to accommodate project safety requirements.

Our field technicians have extensive experience in a wide range of field sampling situations. They have been successfully audited by State regulatory agencies and are accredited to perform analysis for the short holding time field parameters in the "analyze immediately" category (pH, specific conductance, temperature, residual chlorine and dissolved oxygen).

The field staff is also well versed in sample collection using conventional grab techniques, time weighted compositing using automated or manual procedures using sequential-discrete or single jar composite and soil core collection. Customized sampling approaches based on unique client needs can also be developed upon request.

Accutest Sampling Programs & Capabilities:

- National Pollution Discharge Elimination System (NPDES)
- RCRA (Resource Conservation & Recovery) Wastes
- Groundwater Wells
- Wastewater
- Ambient Air
- Drinking Water – Municipal & Residential
- Low Flow Groundwater Wells
- Subsurface Soil
- Sediment
- Statistical Sampling Design

Section 5.0

Key Staff Profiles

Reza Tand
Vice President, Operations

Years with Firm: 23
Total Years Experience: 26
Degree: BS, Chemistry

Experience: Mr. Tand is Corporate Vice President of Operations, and provides technical, operational, strategic growth and administrative leadership and management of personnel. Previously, Mr. Tand was the Director of the New England division and brings over 29 years of analytical expertise in all facets of environmental testing. He is directly responsible for lab operations in Mid-Atlantic (Corporate), New England, Louisiana, and Mountain States divisions which specialize in analysis of organics and inorganics in water, soil and air matrices.

Harry Behzadi
Vice President, Operations

Years with Firm: 17
Total Years Experience: 28
Degree: Ph.D., Analytical Chemistry

Experience: Dr. Behzadi is currently Vice President of Operations. Previously, Dr. Behzadi was the Director of the Southeast and Gulf Coast Divisions. He has extensive experience in trace organics and inorganic analyses of environmental samples. He has been responsible for laboratory management, analytical method development, professional training and QA/QC in both the environmental and pharmaceutical industries. Dr. Behzadi has extensive experience in the R&D, operation, maintenance and trouble shooting of the following instrumentation: GC/MS, GC, HPLC, ICP, AA, IR, GPC and UV/VIS. Previously, Dr. Behzadi has served as an environmental laboratory organics manager and a pharmaceutical laboratory manager.

Dr. Kesavalu Bagawandoss
Technical Director

Years with Firm: 3
Total Years Experience: 31
Degree: BS, Chemistry; MS, Chemistry; Ph.D. Engineering (Environmental); Jurist Doctor (Law); Licensed to Practice Law in Louisiana

Experience: Dr. Doss served as Chief Operations Officer for Integrate, Inc., providing Data Validation Services, Laboratory Audits and Litigation Support Services for 6 years. He served as a Laboratory Director for 15 years providing analytical services to the Gulf Coast Region. Additionally, he served as the Laboratory Director for Industrial Hygiene services accredited by AIHA. Provided Superfund Analytical Services for USEPA for 20 years continuously for various contracts, including Dioxins/Dibenzofurans, Organics and Inorganics. Expertise also includes Laboratory setups, Clean room setups, Methods development, Fingerprinting, Alkylated PAH's, Biomarkers, Frac Fluid Analyses, Air Toxics, Biota Analyses by MSPD and Land Treatment of Oil Refinery Wastes.

Key Staff Profiles (continued)

Andrew Dexter Years with Firm: 17
Vice President, Chief Information Officer Total Years Experience: 30

Experience: Mr. Dexter has developed an overall IT strategy for Accutest and has taken the lead role in implementing a new state-of-the-art LIMS system at all Accutest Laboratories. He has over 30 years of experience in systems and laboratory automation as well as 24 years of environmental laboratory experience. Before joining Accutest, Mr. Dexter was one of seven founding members of Automated Compliance Systems, where he played a key roll in developing the LIMS software now implemented at Accutest. He was the sole designer/developer of Seedpak2, an Oracle-based instrument interfacing package that was later licensed to Perkin-Elmer for use in its SQL-LIMS product. Mr. Dexter was the sole designer/developer of AQUARIUS, a software package for automated GC/MS data acquisition.

Brian Davis Years with Firm: 25
Systems Manager Total Years Experience: 29
Degree: BA, English

Experience: Mr. Davis is currently the Corporate Systems Manager for Accutest Laboratories and reports directly to the Vice President/Chief Information Officer located in Dayton, NJ. His responsibilities include System Administration, Database Administration, Network Administration, Software design and development with a specialty in electronic data deliverables (EDD's) and support. His industry experience includes 10 years in Quality Assurance, 2 years auditing external laboratories and 26 years in information technology. Mr. Davis was instrumental in the design and implementation of the state-of-the-art LIMS System at all Accutest Laboratories.

Paul Ioannidis Years with Firm: 4
Laboratory Director, Mid-Atlantic Total Years Experience: 29
Degree: B.S. Biology, M.S. Environmental Science, Ph.D. Environmental Sciences, M.S. International Relations

Experience: Mr. Ioannidis is responsible for managing the daily operations and all facets of activities at the NJ laboratory. He has over 25 years management experience in commercial environmental laboratories including technical operations, quality assurance, business development, international programs and facility general management. Mr. Ioannidis has extensive laboratory management experience, and has been very active in supporting numerous DoD environmental programs, as well as numerous superfund activities at the state and the Federal EPA level. He has extensive experience in international programs, and has supported field, testing, and consulting activities in West Africa, Central America, Europe and South/Central Asia.

Key Staff Profiles (continued)

Phillip M. Worby Years with Firm: 5
Director, Corporate Quality Assurance Total Years of Experience: 34
Degree: B.S., Environmental Studies/Water and Wastewater Treatment

Experience: Mr. Worby is Accutest's Director of Corporate Quality Assurance. He is responsible for the corporate quality assurance program in each of Accutest's laboratory facilities. Phil brings over 34 years of environmental chemistry experience to Accutest and has previously held management positions in commercial environmental laboratories as a Technical Director and as a Quality Assurance Director. Mr. Worby also has extensive environmental regulatory experience throughout the country and is currently serving as the Past President of the Pennsylvania Association of Accredited Environmental Laboratories and is past Chairman of the New Jersey DEP Environmental Laboratory Advisory Committee.

Wen-Wen Chi Years with Firm: 21
Corporate Technical Director/Organics Total Years Experience: 32
Degree: BS, Chemical Engineering

Experience: Ms. Chi has strong knowledge in the end-to-end Organics Operations from sample preparation/extraction, through sample analysis, data interpretation/review, to report generation. She has extensive experience in Priority Pollutant Analysis using GC-GCMS & HPLC, covering EPA 500/600 Series, SW846, MAEPH/NJEPH, CLP work for EPA, Appendix IX, Dioxin Analysis, and T03/T015/NJT015LL for Ambient Air & Soil Vapor Methodologies. She also has expertise in special projects (EPA, SAS), new equipment/methodology evaluations, instrumentation & Lab start-up planning, technical consultation with clients, and application programs development for laboratory operations standardization and automation.

Nancy Cole Years with Firm: 22
Corporate Technical Director/Inorganics Total Years Experience: 27
Degree: MS, Inorganics Chemistry

Experience: Ms. Cole is the Corporate Technical Director/Inorganics for Accutest Laboratories. The Inorganics division includes metals, wet chemistry, and microbiology. Metals includes a variety of analytical techniques such as ICP, ICP/MS, CV AAS, and Atomic Fluorescence Spectrometry. Wet chemistry includes a broad range of classical and instrumental techniques ranging from ion chromatography to UV/Vis and titrametric analyses. Ms. Cole has extensive experience in EPA, SW846, Standard Methods, and ASTM methodologies as well as NELAC and DOD testing standards. She is involved in daily laboratory operations, including project set-up, data review, and client services and client interactions.

Key Staff Profiles (continued)

Brad Madadian
Regional Laboratory Manager, Northeast

Years with Firm: 22
Total Years Experience: 22
Degree: Masters, Chemistry

Experience: Mr. Madadian is responsible for overseeing the operation, procurement, and personnel in New England, Louisiana, and Mountain States divisions. He is directly involved in design and renovation of facilities and infrastructure to improve work flow and efficiency with utmost emphasis in safety. He works closely with the client services group to better serve client inquiries. Previously, Mr. Madadian was the Laboratory Manager of the New England division and has 23 years of experience in environmental testing. He has been involved in various method developments, studies, and has attended numerous technical training and seminars.

Ronald Benjamin
Laboratory Director, Gulf Coast, LA

Years with Firm: 19
Total Years Experience: 33
Degree: BA, Urban Planning

Experience: Mr. Benjamin is responsible for the day to day operations at Accutest's Gulf Coast, LA Laboratory in Lafayette, LA. He has extensive experience in analytical chemistry applied in the fields of chemical manufacturing, wastewater processing, waste management and environmental monitoring. His experience includes bench expertise in the application of organic and inorganic EPA methodology and methods development associated with environmental monitoring for oil and gas exploration and production under API contract. Mr. Benjamin has over twenty years management experience including independent commercial laboratories and compliance management for public utilities.

Paul Canevaro
Laboratory Director, Gulf Coast, TX

Years with the Firm: 5
Total Years Experience: 33
Degree: BS, Chemistry

Experience: Mr. Canevaro is responsible for day to day laboratory operations at Accutest Gulf Coast laboratory in Houston, Texas. He has extensive experience performing analysis in support of the Safe Drinking Water Act, Clean Water Act, Resource Conservation and Recovery Act and USEPA Contract Laboratory Program. His experience includes bench expertise in metals analysis and inorganic chemistry. He has held senior staff positions including lead technical support functions, laboratory oversight and general management of laboratory operations. In those roles, he implemented laboratory operations and grew the operation from a staff of two to seventy, establishing the largest drinking water laboratory in the State of Florida with accreditation in nineteen states.

Key Staff Profiles (continued)

Norman Farmer
Regional Technical Director, Southeast

Years with Firm: 16
Total Years Experience: 22
Degree: BS, Chemical Oceanography

Experience: Mr. Farmer currently oversees the technical operations for Accutest Laboratories, Southeast, Northern California, and Gulf Coast divisions. This includes project coordination between the facilities; instrument trouble shooting, repair, and installation; method validation guidance; and laboratory design and expansion. Mr. Farmer is familiar with the various QC and reporting criteria for Navy, U.S. Army Corps of Engineers, and AFCEE. He is responsible for implementing the DoD QSM processes throughout Accutest. Mr. Farmer also reviews Quality Assurance Project Plans to ensure that all data quality objectives and reporting requirements are met by laboratory.

James Roush
Technical Manager, New England

Years with Firm: 2
Total Years Experience: 15
Degree: BS, Marine Biology

Experience: Mr. Roush is Technical Manager with Accutest New England. As an accomplished environmental scientist and analytical chemist with knowledge of IT administration, his experience includes laboratory and project management, interpretation of chemical data used in environmental forensic, investigations of manufactured gas plants, petroleum releases, railroads, pipelines, and utility sites, environmental and forensic sample analysis including hydrocarbon fingerprinting, alkylated PAHs, and petroleum biomarkers. Mr. Roush has substantial experience with soil, groundwater, sediment, NAPL and soil vapor sampling techniques and has performed vapor and particulate air monitoring on large remediation sites.

Stephen Grant
Vice President, Sales and Marketing

Years with Firm: 13
Total Years of Experience: 25
Degree: BS, Chemistry

Experience: Mr. Grant is Vice President, Sales and Marketing, and has over 24 years of experience in both operations and sales roles in environmental laboratories. The last 14 years have been in direct sales and sales management. He is responsible for developing and implementing the company's sales and marketing strategy on a national basis. In addition, Mr. Grant manages Accutest's National Accounts Program. Prior to working in the environmental industry, he worked as a chemist in a research laboratory.

Key Staff Profiles (continued)

Kevin Gibbons
National Sales Manager

Years with Firm 3
Total years of experience 15
Degree: BA, History

Experience: Mr. Gibbons is the National Sales Manager, where he is responsible for developing many of Accutest's largest national clients. Mr. Gibbons has over 14 years of environmental industry experience, all in a sales capacity. He also serves as Regional Sales Manager for the New England region and is responsible for managing the sales program in that territory. Prior to joining Accutest Mr. Gibbons worked for Environmental Data Resources as National Account Manager.

Matthew Cordova
Director, Corporate Client Services

Years with Firm: 14
Total Years of Experience: 31
Degree: BS, Marine Biology

Experience: As the Client Services Director, Mr. Cordova is responsible for the implementation of the client services and project management activities within the lab. He also manages the sample log in process, to ensure that project specifications are accurately entered into the LIMS and communicated to the laboratory. Working in conjunction with the production managers, Mr. Cordova ensures that the Client Services Department meets project commitments and data quality objectives. Mr. Cordova's environmental laboratory experience includes, Atomic Spectroscopy, Inorganic Chemistry and management of Client Services, Quality Assurance, Health and Safety and Laboratory Operations.

Section 6.0

Major Project Experience (continued)

Experience (Engineering Firms)			
Project Type		**Project Description**	**Region**
1	**RIFS**	***Site Investigation/Remediation Soil*** and groundwater investigation for Fortune 50 Manufacturing Company. Full TCL/TAL analyses with emphasis on hexavalent chromium analyses. Project included treatability and leaching studies.	**Mid-Atlantic**
2	**RCRA**	***Background Pre-Drill Groundwater*** analysis in support of hydraulic fracturing activities in the Marcellus Shale. Analyses include volatiles, dissolved gases, metals and various wet chemistry parameters.	**Mid-Atlantic**
3	**RI**	***Long-Term RI to Establish Extent/Range of Pollutants*** in lake and stream sediments and water. Additional investigation/remediation of surrounding industrial sites.	**Mid-Atlantic**
4	**RCRA**	***Massachusetts Turnpike Authority, Central Artery*** Concentrated effort to process 3000 post-excavation samples for a full suite of analysis. 48-72 hour TAT. LabLink used to electronically transfer the data for immediate validation and use.	**New England**
5	**RIFS RCRA**	***Site Investigation/Remedial Investigation for Major Aerospace Company***. Large on-going Site Investigation/Remedial Investigation in support of on-going construction activities. Full TCL/TAL analysis, expedited TAT, custom EDD and 3rd party validation.	**Southeast**
6	**RIFS RCRA**	***Multiple Superfund RIFS Activities for Large Utility Company*** Full TCL/TAL analyses for Superfund sites in Puerto Rico, North Carolina and Florida. Full deliverables and extensive electronic deliverables.	**Southeast**
7	**CLP RIFS RCRA**	***EPA Region 6 Laboratory Testing Support Contract*** Performed laboratory analytical services in support of evaluating on shore contamination originating from the Deep Water Horizon Oil Spill in the Gulf of Mexico. Services involved collection and analysis of environmental samples from field teams on a daily basis for over six months.	**Gulf Coast**
8	**RCRA UST**	***Kentucky Natural Resources and Environmental Protection*** Underground Storage Tanks (UST) Program for over 300 sites in Eastern Kentucky. Analyzed groundwater and soil samples for BTEX, PAHs, Metals and RCRA Characteristics. Analyzed over 1000 samples in a 4 month period, all of which required expedited turnaround.	**Central**
9	**RCRA**	***Major Petroleum Company Analysis*** of soil and water in support of oil and gas exploration activities. Analytical suite includes volatiles, semivolatiles, metals and various hydrocarbon analyses.	**Mid-West**
10	**RIFS**	***Brownfields Investigations*** Multiple Brownfields sites throughout Oklahoma through a contract with a national consulting firm.	**Mid-West**
11	**RCRA**	***Groundwater Monitoring and Waste Characterization*** Analysis at several major landfills including Full Appendix IX parameters and site-specific compounds. Modified analytical methods to reach site reporting limits.	**West**
12	**RIFS**	***Site Investigation at Former Aerospace Manufacturing Site*** Full suite of analyses including Perchlorate and hexavalent chromium.	**West**

Major Project Experience (continued)

Experience (Industrial Projects)			
Project Type		**Project Description**	**Region**
1	**RCRA NPDES**	***Electric & Gas Company*** Extensive contract to support the Materials Management Group. Analytical services for MGP sites and facilities. Contract also includes emergency response and field sampling.	**Mid-Atlantic**
2	**RCRA**	***Petroleum Refinery, St. Croix US Virgin Islands*** Large, on-going sampling and analysis program requiring Full Appendix IX and Appendix III analyses as part of a RCRA Facility Investigation (RFI) and RCRA permitted land and wastewater treatment unit monitoring. Customized preprinted chains of custody and sample bottle labels were supplied to the client in order to minimize time spent in the field.	**Mid-Atlantic**
3	**RIFS RCRA**	***Blanket Analytical Contract for large Aerospace Manufacturer*** Contract includes Corporate Remediation and Facility Environmental Management. Full TCL/TAL, Appendix IX and RCRA analysis. Extensive development of electronic deliverable for integration into client database. Field sampling services supplied upon request.	**New England**
4	**RCRA NPDES**	***Regional Petroleum Distributor*** Analytical support throughout New England for groundwater monitoring and site investigations. Major analyses include volatiles, PAH's, metals, soil vapor and ambient air. Provide routine courier service in multiple states.	**New England**
5	**RIFS RCRA**	***Superfund Site in West Palm Beach*** Exclusive laboratory contract to support RIFS activity. Analyzed over 1800 samples for full suite of analysis. Extensive PCB studies.	**Southeast**
6	**RCRA NPDES UST**	***Texas Natural Resource Conservation Commission Analytical Services*** Contract in support of Field Operations Division. Programs include UST, RCRA, and NPDES, utilizing EPA 600 and 8000 Series Methods.	**Gulf Coast**
7	**RCRA**	***Electric/Utility Company, Gulf Coast Region*** Serve as the primary analytical testing laboratory for a major electric utility company in the Gulf Coast region in support of RCRA characterization of waste and transformer oils. Provided rapid TAT and emergency response capabilities.	**Gulf Coast**
8	**RCRA**	***Custom analysis of hydraulic fracturing fluid*** for several oil & gas exploration companies. Also developed specialized analysis for dissolved gases and air.	**Gulf Coast**
9	**RIFS RCRA**	***Multiple RIFS/RCRA sites throughout the mid-west***. Analyses of soil and ground-water for full TCL/TAL. State-specific methods utilized as needed. Custom electronic data deliverables.	**Central**
10	**RCRA**	***Former Steel Manufacturing Site*** Waste Characterization and groundwater monitoring. Analysis included full TCLP on rush turnaround.	**Mid-West**
11	**DOE**	***High Profile Groundwater and Soil Investigation and Remediation Project*** of a two mile linear accelerator. Thousands of samples on an expedited turnaround time during the life of the project.	**West**

Major Project Experience (continued)

Experience (DoD Projects)			
Project Type		**Project Description**	**Region**
1	**DoD**	***US Army Corps of Engineers, Philadelphia District.*** Indefinite delivery contract for several large scale investigation and remediation projects within the district. Provide full data deliverable packages, SEDD EDD and ADR EDD.	**Mid-Atlantic**
2	**DoD**	***Regulatory Compliance Sampling and Analysis*** throughout Naval Air Station. Sampled groundwater monitoring wells, wastewater grab and 24-hour composites, soil and waste drum samples. Analyzed for priority pollutants, waste classification and general chemistry parameters.	**New England**
3	**DoD**	***Department of the Army, Natick R&D Laboratories*** Analytical to support Field Site Investigations, Treatability Studies and Groundwater Monitoring. Analyses included full TCL+ and Mercury.	**New England**
4	**DoD**	***Department of the Airforce, Airforce Center for Environmental Excellence*** Multiple RIFS, SI and Groundwater Monitoring Programs at Cape Canaveral. 600 samples analyzed on an expedited basis for TCL Volatiles and EPA Method 8310. Provided reduced CLP deliverables package and an ERPIMS EDD***.***	**Southeast**
5	**DoD**	***Department of the Navy, Southern Division Navy Clean Contract*** 1600 samples analyzed according to NFESC protocol. Analyses included Full TCL/TAL and EPA Method 8330. Sites include NAS Cecil Field, NAS Jax, NAS Key West, NTC Orlando, NAS Pensacola, and Eglin AFB.	**Southeast**
6	**DoD**	***Department of the Navy, Atlantic Division Navy Clean Contract*** RIFS activities at Camp Lejuene, USN Cherry Point, and Yorktown Naval Weapons Station. Data electronically transferred via LabLink to expedite the validation of 250 samples.	**Southeast**
7	**DoD**	***US Army Corps of Engineers*** Fort Wingate, New Mexico, site investigation: Expedited 150 samples for Full TCL/TAL plus Explosives.	**Gulf Coast**
8	**DoD**	***US Army Corps of Engineers*** quarterly ground-water and soil monitoring at The Plum Brook Ordnance Works Sandusky, Ohio and The West Virginia Ordnance Works remediation, Mason County, WV. 300 samples per quarter analyzed for Nitroaromatics, Volatiles, Semi-Volatiles, TAL Metals and PCB's. Results in ERPIMS deliverables and custom EDDs.	**Central**
9	**DoD**	***Department of the Army, Chicago District*** Treatability Study for the Indiana Harbor Confined Disposal Facility. Analysis included Full TCL/TAL using SW846 Methodology.	**Central**
10	**DoD**	**Department of the Air Force, Air Force Center for Environmental Excellence** Multiple groundwater and soil monitoring programs at McConnell Air Force Base, Kansas. 450 samples analyzed for Volatiles, Semi-Volatiles, PCB's, TPH, Pesticides and Herbicides. Soil and ground-water disposal parameters including a full suite of TCLP parameters.	**Mid-West**
11	**DoD**	***US Army Corps of Engineers*** Quarterly ground-water and soil monitoring at former BRAC installation. 300 samples analyzed for Volatiles and inorganic analysis. Project requires utilization of Passive Diffusion Sampling Bags (PDS) provided prefilled by laboratory. Level 4 deliverables and ADR EDD.	**West**
12	**DoD**	***Department of the Air Force*** RIFS over 400 samples from Air force base located in the Hawaiian Islands. 2-3 day TAT for Pesticides, PAH's and PCB's with level 4 deliverables and ERPIMS EDD.	**West**

Representative Client List

AECOM
AMEC
Amerada Hess
Arcadis
Ashland Chemical
ATC
Atofina Petroleum Chemicals
Beazer
BEM Systems
Brenntag
Brown and Caldwell
Cabot Oil & Gas
Chesapeake Energy
Chrysler
Citgo
Clean Harbors
Conestoga-Rovers & Associates
Conoco Phillips
CSXT
Dow Chemical
EA Engineering
El Paso
Ensafe
ERM
Exxon Mobil
Ford
Gannet Fleming
General Electric
GEI Consultants, Inc
GES
Getty Petroleum
Golder Associates
Granite Construction
HDR Engineering
Hoffman - La Roche Inc.
Honeywell International Inc.
IBM
Kleinfelder
Langan Engineering
MACTEC
Marathon Petroleum
MWH Americas
NiSource
O'Brien & Gere Engineers, Inc.
Occidental
OP-TECH Environmental Services
PPG Industries
PSI
Republic
Roux Associates
Shaw
Shell
SouthWest Water Corp.
Sovereign Consulting
Stantec
Sunoco
Terracon
Tetra Tech
TRC
United Technologies
Warren Equities
Weston Solutions
Williams Energy
WSP

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APPENDIX A-2
Accutest Laboratories
Quality Assurance Manual

Quality Systems Manual

Volume: XIV: March, 2013

Effective Date: 3/6/13

Document Tracking Number:_____

Laboratory Director: Reza Tand

Laboratory Manager/Technical Director - Inorganics: Brad Madadian

Quality Assurance Officer: Robert Treggiari

Technical Director - Organics: James Roush

Organics Manager: Douglas Yargeau

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Marlborough, MA 01752
508.481.6200

Introduction

The Accutest Laboratories Quality Assurance Program, detailed in this plan, has been designed to meet the quality program requirements of the National Environmental Accreditation Conference (NELAC) and ISO Guide 17025, ISO Guide 17011 and other National environmental monitoring programs. The plan establishes the framework for documenting the requirements of the quality processes regularly practiced by the Laboratory. The Quality Assurance Director is responsible for changes to the Quality Assurance Program, which is appended to the QSM during the annual program review. The plan is also reviewed annually for compliance purposes by the Company President and Laboratory Director and edited if necessary. Changes that are incorporated into the plan are itemized in a summary of changes following the introduction. Plan changes are communicated to the general staff in a meeting conducted by the Director of Quality Assurance following the plan's approval.

The Accutest plan is supported by standard operating procedures (SOPs), which provide specific operational instructions on the execution of each quality element and assure that compliance with the requirements of the plan are achieved. Accutest employees are responsible for knowing the requirements of the SOPs and applying them in the daily execution of their duties. These documents are updated as changes occur and the staff is trained to apply the changes.

At Accutest, we believe that satisfying client requirements and providing a product that meets or exceeds the standards of the industry is the key to a good business relationship. However, client satisfaction cannot be guaranteed unless there is a system that assures the product consistently meets its design requirements and is adequately documented to assure that all procedural steps are executed, properly documented and traceable.

This plan has been designed to assure that this goal is consistently achieved and the Accutest product withstands the rigors of scrutiny that are routinely applied to analytical data and the processes that support its generation.

Summary of Changes
Accutest Laboratories Quality System Manual – January 2013

Section	Page	Description
2	11	Accutest New England Organizational Chart

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1.0 QUALITY POLICY

1.1 ***Accutest Mission***:

Accutest Laboratories provides analytical services to commercial and government clients in support of environmental monitoring and remedial activities as requested. The Laboratory's mission is dedicated to providing reliable data that satisfies client's requirements as explained in the following:

"Provide easy access, high quality, analytical support to commercial and government clients which meets or exceeds data quality objectives and provides them with the data needed to satisfy regulatory requirements and/or make confident decisions on the effectiveness of remedial activities."

These services are provided impartially and are not influenced by undue commercial or financial pressures which might impact the staff's technical judgment. Coincidentally, Accutest does not engage in activities that endanger the trust in our independent judgment and integrity in relation to the testing activities performed.

1.2 ***Policy Statement*:**

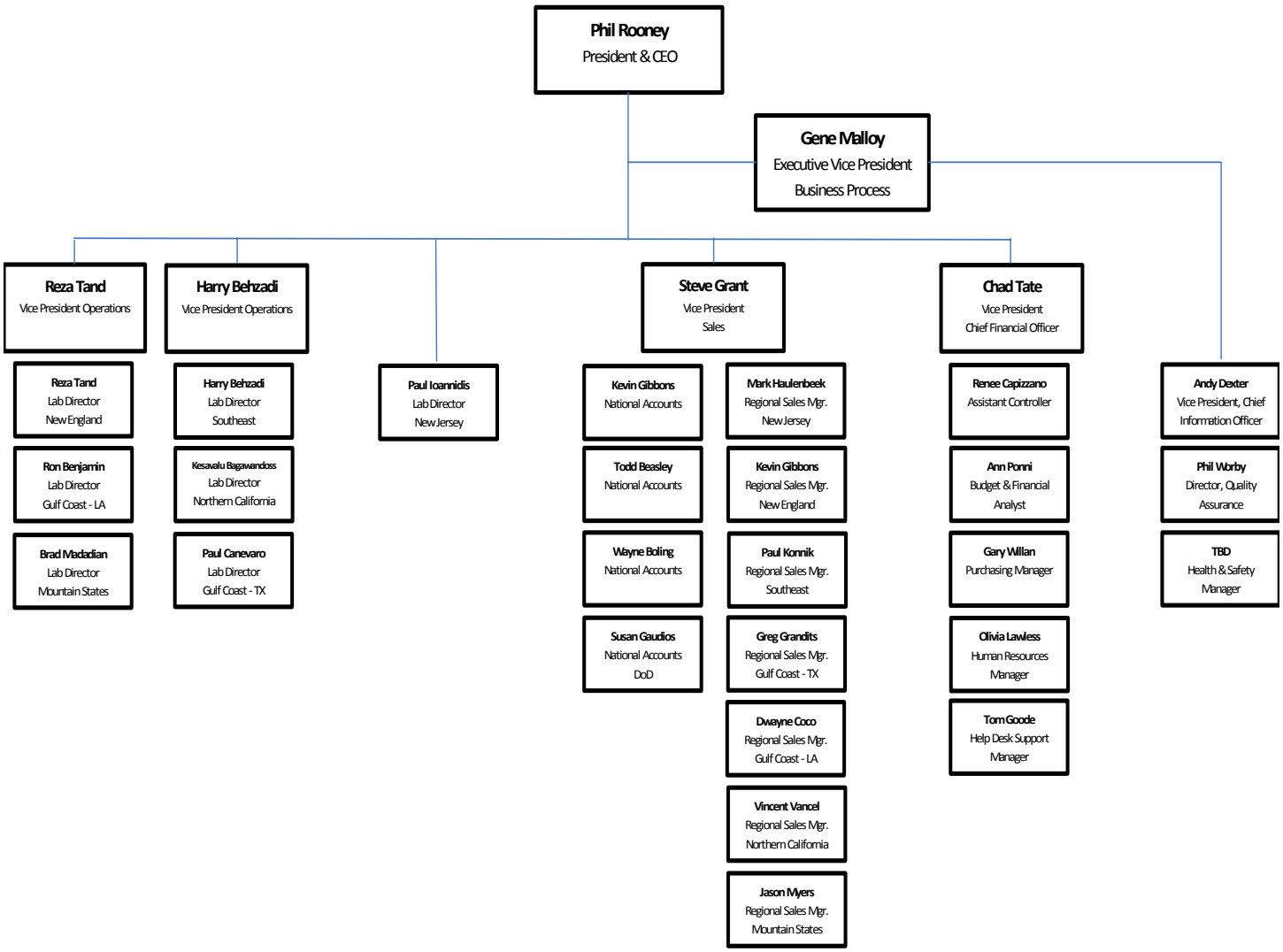
The management and staff of Accutest Laboratories share the responsibility for product quality. Accordingly, Accutest's quality assurance program is designed to assure that all processes and procedures, which are components of environmental data production, meet established industry requirements, are adequately documented from a procedural and data traceability perspective, and are consistently executed by the staff. It also assures that analytical data of known quality, meeting the quality objectives of the analytical method in use and the data user's requirements, is consistently produced in the laboratory. This assurance enables the data user to make rational, confident, cost-effective decisions on the assessment and resolution of environmental issues.

The laboratory Quality System also provides the management staff with data quality and operational feedback information. This enables them to determine if the laboratory is achieving the established quality and operational standards, which are dictated by the client or established by regulation. The information provided to management, through the QA program, is used to assess operational performance from a quality perspective and to perform corrective action as necessary.

All employees of Accutest Laboratories participating in environmental testing receive quality system training and are responsible for knowing and complying with the system requirements. The entire staff shares Accutest's commitment to good professional practice.

Phillip Rooney; President

Accutest Laboratories Organizational Chart



2.0 ORGANIZATION

2.1 ***Organizational Entity***. Accutest Laboratories is a privately held, independent testing laboratory founded in 1956 and registered as a New Jersey Corporation. The headquarters are located in Dayton, New Jersey where it has conducted business since 1987. The New England Laboratory is located in Marlborough, Massachusetts where business has been conducted since 1990. Additional satellite laboratories are maintained in Orlando (Florida), Houston, (Texas), Scott (Louisiana), Denver (Colorado) and San Jose (California).

2.2 ***Management Responsibilities***

Requirement: Each laboratory facility has an established chain of command. The duties and responsibilities of the management staff are linked to the President/CEO of Accutest Laboratories who establishes the agenda for all company activities.

President/CEO. Primary responsibility for all operations and business activities. Delegates authority to laboratory directors, general managers, and the quality assurance director to conduct day to day operations and execute quality assurance duties. Each of the seven operational entities (New Jersey, Massachusetts, Florida, Texas, Louisiana, Denver, and California) report to the President/CEO.

Quality Assurance Director. Design, oversight, and facilitation responsibility for all Quality System elements identified in the Quality Program. Reports directly to the President/CEO.

Laboratory Director. Executes day to day responsibility for laboratory operations including technical aspects of production activities and associated logistical procedures. Reports directly to the President/CEO.

Laboratory Manager. Assists Laboratory Director with operations responsibilities. Reports directly to the Laboratory Director.

Quality Assurance Officer. Design, oversight, and facilitation responsibility for all quality assurance activities established by the Accutest Quality Assurance Program. Reports directly to the Corporate Quality Assurance Director and works closely with the Laboratory Director to ensure appropriate implementation and continued improvement of the quality program.

Technical Directors (Organics/Inorganics). Responsible for day to day operations and activities of the organics and inorganics laboratories including scheduling, production and data quality. Reports directly to the Laboratory Director.

Department Managers. Executes day to day responsibility for specific laboratory areas including technical aspects of production activities and associated logistical procedures. Direct report to the laboratory director.

Section Supervisors. Executes day to day responsibility for specific laboratory units including technical aspects of production activities and associated logistical procedures.

Direct report to the Department Manager.

Accutest management is committed to ensuring that the technical and quality systems specified in the current Department of Defense (DoD) Quality Systems Manual are fully adhered to for applicable DoD projects.

Chain of Command

The responsibility for managing all aspects of the Company's operation is delegated to specific individuals, who have been assigned the authority to act in the absence of the senior staff. These individuals are identified in the following Chain of Command:

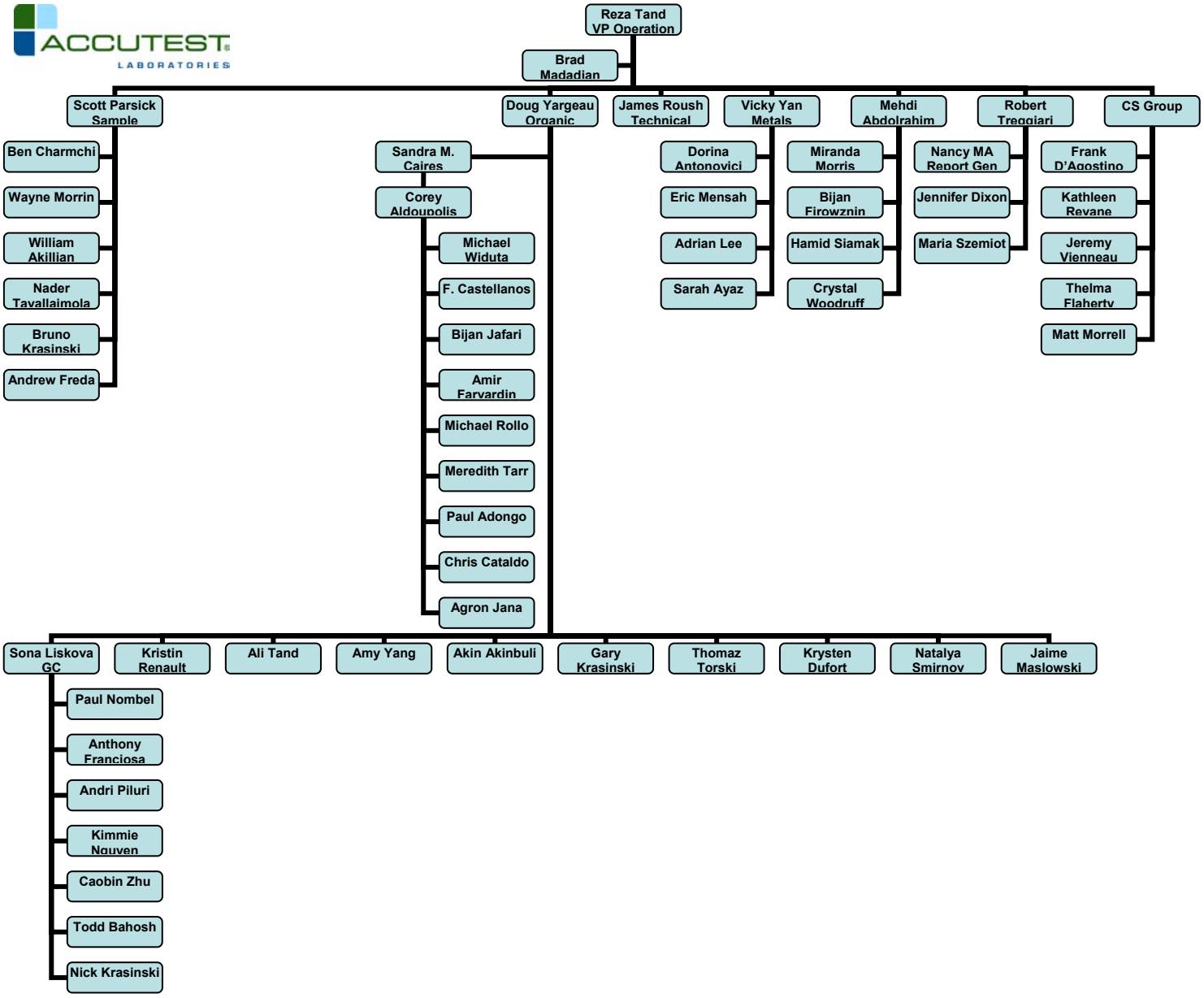
Reza Tand, Vice President, Northeast Operations, Laboratory Director
Brad Madadian, Laboratory Manager, Technical Manager, Inorganics
Douglas Yargeau, Technical Manager, Organics
James Roush, Technical Manager, Organics
Robert Treggiari, Quality Assurance Officer
Scott Parsick, Sample Manager

Key Management Appointed Deputies

In the event that the technical director(s) is absent for a period of time that exceeds 15 consecutive calendar days, the designated appointees shall temporarily perform the technical director(s) job function.

Technical Director – Laboratory Director	Reza Tand
Appointed Deputy	Brad Madadian – Laboratory Manager
Appointed Deputy	Douglas Yargeau – Organics Manager
Technical Director – Inorganics	Brad Madadian
Appointed Deputy	Douglas Yargeau – Organics Manager
Technical Director – Organics	James Roush
Appointed Deputy	Douglas Yargeau – Organics Manager
Technical Director – GCMS	Douglas Yargeau
Appointed Deputy	James Roush – Organics Technical
Quality Assurance Officer	Robert Treggiari
Appointed Deputy	Brad Madadian – Laboratory Manager

Accutest Laboratories of New England Organizational Chart



3.0 QUALITY RESPONSIBILITIES OF THE MANAGEMENT TEAM

3.1 ***Requirement***: Each member of the management team has a defined responsibility for the Quality System. System implementation and operation is designated as an operational management responsibility. System design and implementation is designated as a Quality Assurance Responsibility.

President/CEO. Primary responsibility for all quality activities. Delegates program responsibility to the Quality Assurance Director. Serves as the primary alternate in the absence of the Quality Assurance Director. Has the ultimate responsibility for implementation of the Quality System.

Laboratory Director. Responsible for oversight of implementation and operation of the Quality System in all laboratory areas. Responsible for the oversight, design, and implementation of corrective action for defective processes. Has the authority to delegate Quality System implementation responsibilities.

Quality Assurance Officer. Responsible for design, implementation support, training, and monitoring of the quality system. Identifies product, process, or operational defects using statistical monitoring tools and processes audits for elimination via corrective action. Empowered with the authority to halt production if quality issues warrant immediate action. Monitors implemented corrective actions for compliance. Responsible for selection, approval, and maintenance of statistical Quality Control System (SQC) samples. Responsible for annual QSM and Quality Policy review. Presents Laboratory Director and Corporate Quality Assurance Director with annual Quality System and Management Review..

Technical Directors. Responsible for overseeing the technical aspects of the quality assurance system as they are integrated into method applications and employed to assess analytical control on a daily basis. The Technical directors review and acknowledge the technical feasibility of proposed QA systems involving technical applications of applied methodology.

Department Managers. Responsible for applying the requirements of the Quality System in their section and assuring subordinate supervisors and staff apply all system requirements. Initiates, designs, documents, and implements corrective action for quality deficiencies.

Section Supervisors. Responsible for applying the requirements of the Quality System to their operation and assuring the staff applies all system requirements. Initiates, designs, documents, and implements corrective action for quality deficiencies.

Client Services Manager. Responsible for applying the requirements of the Quality System to their operation and assuring the staff applies all system requirements. Initiates, designs, documents, and implements corrective action for quality deficiencies.

Bench Analysts. Responsible for applying the requirements of the Quality System to the analyses they perform, evaluating QC data and initiating corrective action for quality control deficiencies within their control. Implements global corrective action as directed by superiors.

3.2 ***Program Authority***:

Authority for program implementation originates with the President/CEO who bears ultimate responsibility for system design, implementation, and enforcement of requirements. This authority and responsibility is delegated to the Director of Quality Assurance who performs quality functions independently without the encumbrances or biases created by operational or production responsibilities to ensure an honest, independent assessment of quality issues.

3.3 ***Data Integrity Policy***:

The Accutest Data Integrity Policy reflects a comprehensive, systematic approach for assuring that data produced by the laboratory accurately reflects the outcome of the tests performed on field samples and has been produced in a bias free environment by ethical professionals. The policy includes a commitment to technical ethics, staff training in ethics and data integrity, an individual attestation to data integrity and procedures for evaluating data integrity. Senior management assumes the responsibility for assuring compliance with all technical ethics elements and operation of all data integrity procedures. The staff is responsible for compliance with the ethical code of conduct and for practicing data integrity procedures.

The Accutest Data Integrity Policy is as follows:

"Accutest Laboratories is committed to producing data that meets the data integrity requirements of the environmental regulatory community. This commitment is demonstrated through the application of a comprehensive data integrity program that includes ethics and data integrity training, data integrity evaluation procedures, staff participation and management oversight. Adherence to the specifications of the program assures that data provided to our clients is of the highest possible integrity and can be used for decision making processes with high confidence."

Data Integrity Responsibilities

Management. Senior management retains oversight responsibility for the data integrity program and retains ultimate responsibility for execution of the data integrity program elements. Senior management is responsible for providing the resources required to conduct ethics training and operate data integrity evaluation procedures. They also include responsibility for creating an environment of trust among the staff and being the lead advocate for promoting the data integrity policy and the importance of technical ethics. The Quality Assurance Officer is the designated ethics officer for the New England laboratory.

Staff. The staff is responsible for adhering to the company ethics policy as they perform their duties and responsibilities associated with sample analysis and reporting. By executing this responsibility, data produced by Accutest Laboratories retains its high integrity characteristics and withstands the rigors of all data integrity checks.

The staff is also responsible for adhering to all laboratory requirements pertaining to manual data edits, data transcription and data traceability. These include the application of approved manual peak integration and documentation procedures. It also includes establishing traceability for all manual results calculations and data edits.

Ethics Statement. The Accutest ethics statement reflects the standards that are expected for businesses that provide environmental services to regulated entities and regulatory agencies on a commercial basis. The Ethics Policy is comprised of key elements that are essential to organizations that perform chemical analysis for a fee. As such, it focuses on elements related to personal, technical and business activities.

Accutest Laboratories provides analytical chemistry services on environmental matters to the regulated community. The data the company produces provides the foundation for determining the risk presented by a chemical pollutant to human health and the environment. The environmental industry is dependent upon the accurate portrayal of environmental chemistry data. This process is reliant upon a high level of scientific and personal ethics.

It is essential to the Company that each employee understands the ethical and quality standards required to work in this industry. Accordingly, Accutest has adopted a code of ethics, which each employee is expected to adhere to as follows:

- Perform chemical and microbiological analysis using accepted scientific practices and principles.
- Perform tasks in an honest, principled and incorruptible manner inspiring peers & subordinates.
- Maintain professional integrity as an individual.
- Provide services in a confidential, honest, and forthright manner.
- Produce results that are accurate and defensible.
- Report data without any considerations of self-interest.
- Comply with all pertinent laws and regulations associated with assigned tasks and responsibilities.

Data Integrity Procedures.

Four key elements comprise the Accutest data integrity system. Procedures have been implemented for conducting data integrity training and for documenting that employees conform to the Accutest Data Integrity and Ethics policy.

The data integrity program consists of routine data integrity evaluation and documentation procedures to periodically monitor and document data integrity. These procedures are documented as SOPs. SOPs are approved and reviewed annually following the procedures employed for all Accutest SOPs. Documentation associated with data integrity evaluations is maintained on file and is available for review.

Data Integrity Training. Accutest employees receive technical ethics training during new employee orientation. Employees are also required to attend annual ethics refreshment training and sign an ethical conduct agreement annually, which verifies their understanding of Accutest's ethics policy and their ethical responsibilities. The agreement is refreshed annually and appended to each individual's training file.

The training focuses on the reasons for technical ethic training, explains the impact of data fraud on human health and the environment, and illustrates the consequences of criminal fraud on businesses and individual careers. Accutest's ethics policy and code of ethics are reviewed and explained for each new employee.

Training on data integrity procedures are conducted by individual departments for groups involved in data operations. These include procedures for manual chromatographic peak integration, traceability for manual calculations and data transcription.

Data Integrity Training Documentation. Records of all data integrity training are maintained in individual training folders. Attendance at all training sessions is documented and maintained in the training archive.

Accutest Data Integrity and Ethical Conduct Agreement. All employees are required to sign a Data Integrity and Ethical Conduct Agreement annually. This document is archived in individual training files, which are retained for duration of employment.

The Data Integrity and Ethical Conduct Agreement is as follows:

I. I understand the high ethical standards required of me with regard to the duties I perform and the data I report in connection with my employment at Accutest Laboratories.

II. I have received formal instruction on the code of ethics that has been adapted by Accutest Laboratories and agree to comply with these requirements.

III. I have received formal instruction on the elements of Accutest Laboratories' Data Integrity Policy and have been informed of the following specific procedures:

a. Routine data integrity monitoring is conducted on sample data, which may include an evaluation of the data I produce,

b. Formal procedures for the confidential reporting of data integrity issues are available, which can be used by any employee,

c. A data integrity investigation is conducted when data issues are identified that may negatively impact data integrity.

IV. I am aware that data fraud is a punishable crime that may include fines and/or imprisonment upon conviction.

V. I also agree to the following:

a. I shall not improperly alter analytical conditions from standard analysis to sample analysis;

b. I shall not intentionally report data values (QC Results, dry labbing), which are not the actual values observed or measured;

c. I shall not intentionally modify (improper manual integration, unwarranted software manipulation, QC misrepresentation) data values unless the modification can be technically justified through a measurable analytical process;

d. I shall not manipulate computer software to meet method QC criteria;

e. I shall not intentionally report dates and times of data analysis that are not the true and actual times the data analysis was conducted;

f. I shall not misrepresent laboratory quality control performance;

g. I shall not report results from the analysis of one sample for those of another;

h. I shall not condone any accidental or intentional reporting of inauthentic data by other employees and immediately report it's occurrence to my superiors;

i. I shall immediately report any accidental reporting of inauthentic data by myself to my superiors.

Data Integrity Monitoring. Documented procedures are employed for performing data integrity monitoring. These include regular data review procedures by supervisory and management staff (Section 12.7), supervisory review and approval of manual integrations and periodic reviews of GALP audit trails from the LIMS and all computer controlled analysis.

Data Review. All data produced by the laboratory undergoes several levels of review, which includes two levels of management review. Detected data anomalies that appear to be related to data integrity issues are isolated for further investigation. The investigation is conducted following the procedures described in this section.

Manual Peak Integration Review and Approval. Routine data review procedures for all chromatographic processes includes a review of all manual chromatographic peak integrations. This review is performed by the management staff and consists of a review of the machine integration compared to the manual integration. Manual integrations, which have been performed in accordance with Accutest's manual peak integration procedures are approved for further processing and release. Manual integrations which are not performed to Accutest's specifications are set aside for corrective action, which may include analyst retraining or further investigation as necessary.

GALP Audit Trail Review. Good Automated Laboratory Practice (GALP) audits are comprehensive data package audits that include a review of raw data, process logbooks, processed data reports and GALP audit trails from individual instruments and LIMS. GALP audit trails, which record all electronic data activities, are available for the majority of computerized methodology and the laboratory information management

system (LIMS). These audit trails are periodically reviewed to determine if interventions performed by technical staff constitute an appropriate action. The review is performed on a recently completed job and includes interviews with the staff that performed the analysis. Findings indicative of inappropriate interventions or data integrity issues are investigated to determine the cause and the extent of the anomaly.

Confidential Reporting of Data Integrity Issues: Data integrity concerns may be raised by any individual to their supervisor. Employees with data integrity concerns should always discuss those concerns with their immediate supervisors as a first step unless the employee is concerned with the confidentiality of disclosing data integrity issues or is uncomfortable discussing the issue with their immediate supervisors. The supervisor makes an initial assessment of the situation to determine if the concern is related to a data integrity violation. Those issues that appear to be violations are documented by the supervisor and referred to the Director of Quality Assurance for investigation.

Documented procedures for the confidential reporting of data integrity issues in the laboratory are part of the data integrity policy. These procedures assure that laboratory staff can privately discuss ethical issues or report items of ethical concern without fears of repercussions with senior staff.

Employees with data integrity concerns that they consider to be confidential are directed to the Corporate Human Resources Manager in Dayton, New Jersey. The HR Manager acts as a conduit to arrange a private discussion between the employee and the Corporate QA Director or a local QA Officer.

During the employee - QA discussion, the QA representative evaluates the situation presented by the employee to determine if the issue is a data integrity concern or a legitimate practice. If the practice is legitimate, the QA representative clarifies the process for the employee to assure understanding. If the situation appears to be a data integrity concern, the QA representative initiates a Data Integrity Investigation following the procedures specified in SOP MQA298.

Data Integrity Investigations: Follow-up investigations are conducted for all reported instances of ethical concern related to data integrity. Investigations are performed in a confidential manner by senior management according to a documented procedure. The outcome of the investigation is documented and reported to the company president who has the ultimate responsibility for determining the final course of action in the matter. Investigation documentation includes corrective action records, client notification information and disciplinary action outcomes, which is archived for a period of five years.

The investigations are conducted by the senior staff and supervisory personnel from the affected area. The investigations team includes the Laboratory Director and the Quality Assurance Director. Investigations are conducted in a confidential manner until it is completed and resolved.

The investigation includes a review of the primary information in question by the investigations team. The team performs a review of associated data and similar historical data to determine if patterns exist. Interviews are conducted with key staff to determine the reasons for the observed practices.

Following data compilation, the investigations team reviews all information to formulate a consensus conclusion. The investigation results are documented along with the recommended course of action.

Corrective Action, Client Notification & Discipline: Investigations that reveal systematic data integrity issues will be referred to corrective action for resolution and disposition (Section 13). If the investigation indicates that an impact to data has occurred and the defective data has been released to clients, client notification procedures will be initiated following the steps in Section 13.2.

In all cases of data integrity violations, some level of disciplinary action will be conducted on the responsible individual. The level of discipline will be consistent with the violation and may range from retraining and/or verbal reprimand to termination. A zero tolerance policy is in effect for unethical actions.

4.0 JOB DESCRIPTIONS OF KEY STAFF

4.1 ***Requirement***: Descriptions of key positions within the organization are defined to ensure that clients and staff understand duties and the responsibilities of the management staff and the reporting relationships between positions.

President/Chief Executive Officer. Responsible for all laboratory operations and business activities. Establishes the company mission and objectives in response to business needs. Direct supervision of the Vice President of Operations, each laboratory director, client services, management information systems, quality assurance and health and safety.

Laboratory Director (Technical Director). Reports to the company president. Establishes laboratory operations strategy. Direct supervision of organic chemistry, inorganic chemistry, and sample management. Operational responsibility for Orlando, Florida, Marlborough, Massachusetts and Houston, Texas laboratories. Keeps updated to regulatory guideline and regulation changes that may affect laboratory procedures.

Vice President, Chief Information Officer. Reports to the company president. Develops the IT software and hardware agenda. Provides system strategies to compliment company objectives. Maintains all software and hardware used for data handling.

Quality Assurance Officer. Reports to the Corporate Quality Assurance Director and works closely with the Laboratory Director. Facilitates implementation of the company quality agenda, develops quality procedures, provides assistance to operations on quality procedure implementation, coordinates all quality control activities, monitors the quality system, provides quality system feedback to management to be used for process improvement and oversees health and safety. Keeps updated to regulatory guidelines and regulation changes that may affect laboratory procedures. Performs quality control data review for trend monitoring purposes. Conducts internal audits and prepares reports for management review. Oversees proficiency testing program. Process quality control data for statistical purposes. The Quality Assurance Officer is designated as the calibration coordinator.

Client Service Manager. Reports to the Laboratory Director. Establishes and maintains communications between clients and the laboratory pertaining to client requirements which are related to sample analysis and data deliverables. Initiates client orders and supervises sample login operations. Supervises Client Service Personnel.

Manager, Organics (Organics Technical Director). Reports to the Laboratory Director. Directs the operations of the organics group, consisting of organics preparation and (GC) instrumental analysis. Establishes daily work schedule. Supervises method implementation, application, and data production. Responsible for following Quality Program requirements. Maintains laboratory instrumentation in an operable condition.

Manager, GCMS (Organics Technical Director). Reports to the Laboratory Director. Directs the operations of the organics group, consisting of GCMS instrumental analysis. Establishes daily work schedule. Supervises method implementation, application, and data production. Responsible for following Quality Program requirements. Maintains laboratory instrumentation in an operable condition.

Manager, Inorganics (Inorganics Technical Director). Reports to the Laboratory Director. Directs the operations of the inorganics group, consisting of wet chemistry and the metals laboratories. Establishes daily work schedule. Supervises method implementation, application, and data production. Responsible for following Quality Program requirements. Maintains laboratory instrumentation in an operable condition.

Manager, Sample Management. Reports to the Laboratory Director. Develops, maintains and executes all procedures required for sample pickup, receipt of samples, verification of preservation, and chain of custody documentation. Responsible for maintaining and documenting secure storage, delivery of samples to laboratory units on request, and disposal following completion, and courier services. Responsible for following Quality Program requirements

Supervisor, Wet Chemistry. Reports to the Inorganics Technical Director. Executes daily analysis schedule. Supervises the analysis of samples for wet chemistry parameters using valid, documented methodology. Maintains instrumentation in an operable condition. Reviews data for compliance to quality and methodological requirements.

Supervisor, Metals. Reports to the Inorganics Technical Director. Executes daily analysis schedule. Supervises the analysis of samples for metals using valid, documented methodology. Maintains instrumentation in an operable condition. Reviews data for compliance to quality and methodological requirements.

Supervisor, Organic Preparation. Reports to the Organics Technical Director. Executes the daily sample preparation schedule. Performs the extract of multi-media samples for organic constituents using valid, documented methodology. Prepares documentation for extracted samples. Assumes custody until transfer for analysis. Maintains instrumentation in an operable condition. Responsible for following Quality Program requirements.

Supervisor, GCMS. Reports to the GCMS Technical Director. Coordinates the daily sample analysis schedule. Supervised the analysis for volatile organic compounds using valid, documented methodology. Maintains instrumentation in an operable condition.

Responsible for following Quality Program requirements.

Employee Screening, Orientation, and Training.

All potential laboratory employees are screened and interviewed by human resources and technical staff prior to their hire. The pre-screen process includes a review of their qualifications including education, training and work experience to verify that they have adequate skills to perform the tasks of the job.

Newly hired employees receive orientation training beginning the first day of employment by the Company. Orientation training consists of initial health and safety training including general laboratory safety, personal protection and building evacuation. Orientation also includes quality assurance program training, data integrity training, and an overview of the Company's goals, objectives, mission, and vision.

All technical staff receives training to develop and demonstrate proficiency for the methods they perform. New analysts work under supervision until the supervisory staff is satisfied that a thorough understanding of the method is apparent and method proficiency has been demonstrated, through a precision and accuracy study that has been documented, reviewed and approved by the QA Staff. Data from the study is compared to method acceptance limits. If the data is unacceptable, additional training is required. The analyst may also demonstrate proficiency by producing acceptable data through the analysis of an independently prepared proficiency sample.

Individual proficiency is demonstrated annually for each method performed. Data from initial and continuing proficiency demonstrations are archived in the individual's training folder and training application database.

4.2 ***Training Documentation***. The Quality Assurance Department prepares a training file for every new employee. All information related to qualifications, experience, external training courses, and education are placed into the file. Verification documentation for orientation, health & safety, quality assurance, and ethics training is also included in the file.

Additional training documentation is added to the file as it is developed. This includes documentation of SOP understanding, data for initial and continuing demonstrations of proficiency, performance evaluation study data and notes and attendance lists from group training sessions.

The Quality Assurance Department maintains the employee training database. This database is a comprehensive inventory of training documentation for each individual employee. The database enables supervisors to obtain current status information on training data for individual employees on a job specific basis. It also enables the management staff to identify training documentation in need of completion.

Employee specific database records are created by human resources on the date of hire. Data base fields for job specific requirements such as SOP documentation of understanding and annual demonstration of analytical capability are automatically generated when the supervisor assigns a job responsibility. Employees acknowledge that their SOP responsibilities have been satisfied using a secure electronic process which updates the database record. Reports are produced which summarize the qualifications of

individual employees or departments.

5.0 SIGNATORY APPROVALS

Requirement: Procedures have been developed for establishing the traceability of data and documents. The procedure consists of a signature hierarchy, indicating levels of authorization for signature approvals of data and information within the organization. Signature authority is granted for approval of specific actions based on positional hierarchy within the organization and knowledge of the operation that requires signature approval. A log of signatures and initials of all employees is maintained by QA for cross-referencing purposes.

5.1 *Signature Hierarchy*.

President/Chief Executive Officer. Authorization for contracts and binding agreements with outside parties. Approval of final reports, quality assurance policy, SOPs, project specific QAPs, data review and approval in lieu of technical managers.

Laboratory Director. Approval of final reports and quality assurance policy in the absence of the President. Approval of SOPs, project specific QAPs, data review and approval in lieu of technical managers. Establishes and implements technical policy.

Quality Assurance Officer. Approval of final reports and quality assurance procedures in the absence of the Director. Approval of SOPs, project specific QAPPs, data review and approval in lieu of technical managers.

Client Service Manager. Project specific contracts, pricing, and price modification agreements. Approval and acceptance of incoming work, Client services policy.

Managers, Technical Departments. Methodology and department specific QAPPs. Data review and approval. Technical approval of SOPs . Department specific supplies purchase.

Manager, Sample Management. Initiation of laboratory sample custody and acceptance of all samples. Approval of department policies and procedures. Department specific supplies purchase. Waste manifesting and disposal.

Supervisors, Technical Departments. Data review approval, purchasing of expendable supplies.

5.2 *Signature Requirements*.

All laboratory activities related to sample custody and generation or release of data must be approved using either initials, signatures or electronic, password protected procedures. The individual, who applies his signature initial or password to an activity or document, is authorized to do so within the limits assigned to them by their supervisor. All written signatures and initials must be applied in a readable format that can be cross-referenced to the signatures and initials log if necessary.

5.3 *Signature and Initials Log*.

Quality Assurance maintains a signature and initials log. New employee signatures and initials are appended to the log on the first day of

employment. Signature of individuals no longer employed by the company are retained, but annotated with their date of termination.

6.0 DOCUMENTATION & DOCUMENT CONTROL

Requirement: Document control policies have been established which specify that any document used as an information source or for recording analytical or quality control information must be managed using defined document control procedures. Accordingly, policies and procedures required for the control, protection, and storage of any information related to the production of analytical data and the operation of the quality system to assure its integrity and traceability have been established and implemented in the laboratory. The system contains sufficient controls for managing, archiving and reconstructing all process steps which contributed to the generation of an analytical test result. Using this system, an audit trail for reported data can be produced, establishing complete traceability for the result.

6.1 ***Administrative Records***. Administrative (non-analytical) records are managed by the quality assurance department. These records consist of electronic documents which are retained in a limited access electronic directory or paper documents, which are released to the technical staff upon specific request.

Form Generation, Modification & Control. The quality assurance group approves and manages all forms used as either stand-alone documents or in logbooks to ensure their traceability. Forms are generated as computer files only and are maintained in a limited access master directory. The QA staff also manages and approves modifications to existing forms. Obsolete editions of modified forms are retained for five years.

Approved forms are assigned a 7-character alphanumeric code. The first two alpha characters designate the department that uses the form; the next three digits are sequentially assigned number, and the last two numbers are the version.

New forms must include the name Accutest Laboratories and appropriate spaces for signatures of approval and dates. Further design specifications are the responsibility of the originating department.

The technical staff is required to complete all forms to the maximum extent possible. If information for a specific item is unavailable, the analyst is required to "Z" the information block. The staff is also required to "Z" the uncompleted portions of a logbook or logbook form if the day's analysis does not fill the entire page of the form.

Logbook Control. All laboratory logbooks are controlled documents that are comprised of approved forms used to document specific processes. Quality Assurance maintains logbook generation control. New logs are numbered and issued to the department and is assigned responsibility for the log. Old logs are returned to QA for entry into the document archive system where they are retained for ten (10) years. Laboratory staff may hold a maximum of two consecutively dated logbooks of the same type in the laboratory including the most recently issued book to simplify review of recently completed analysis.

All logbooks are boxed and catalogued according to the document control number.

Logbooks are placed into storage boxes identified with by a alphanumeric number indicating the facility, department and box number (eg. MMS001 is box number one for Massachusetts lab mass spec logs).

Controlled Documents. Key laboratory documents are designated for controlled document status to assure that identities of individuals receiving copies and the number of copies that have been distributed are known. Controlled status simplifies document updates and retrieval of outdated documents. Control is maintained through a document numbering procedure and document control log designating the individual receiving the controlled document and the date of receipt. Document control is also maintained by pre-designating the numbers of official copies of documents that are placed into circulation within the laboratory.

Quality Systems Manual (QSM). All QSMs are assigned a number prior to distribution. The number, date of distribution, and identity of the individual receiving the document are recorded in the document control logbook. Only QSM copies that are for distribution in the laboratory will be controlled. Copies distributed to clients, auditing agencies, or regulatory authorities will be tracked, but not controlled. The numbering system is restarted with each new volume, which corresponds to the annual revision of the QSM. Electronic versions are distributed as read only files that are password protected – or as PDFs. The QSM is reviewed annually.

Standard Operating Procedures (SOPs). SOPs are maintained electronically in department-segregated directories on a write-protected server accessible by all technical staff. Editable SOP documents are in a separate controlled directory with limited access. Any SOP located within these directories has been reviewed and approved by QA and the applicable department manager. Any SOP located within these directories is defined by the lab as approved and active. Unless otherwise specified, SOPs that are printed are not official copies and are marked accordingly.

SOP copies distributed to clients, auditing agencies, or regulatory authorities are tracked, but not controlled.

Electronic versions of outdated SOPs are moved from the active SOP directory to the inactive (archive) directory. SOPs in process of updating or generation are stored and labeled as Draft in the editable directory.

6.2 ***Technical Records***. All records related to the analysis of samples and the production of an analytical result are archived in secure document storage or on electronic media and contain sufficient detail to produce an audit trail which re-creates the analytical result. These records include information related to the original client request, bottle order, sample login and custody, storage, sample preparation, analysis, data review and data reporting.

Each department involved in this process maintains controlled documents which enable them to maintain records of critical information relevant to their department's process.

6.3 ***Quality Control Support Data & Records***. All information and data related to the quality system is stored in a restricted access directory on the network server. Information on this directory is backed-up daily. Users of the quality assurance information and data

have "read-only" access to the files contained in the directory. The QA staff and the Laboratory Director/Manager have write capability in this directory.

This directory contains all current and archived quality system manuals, SOPs, control limits, MDL studies, precision and accuracy data, official forms, internal audit reports, proficiency test scores and metrics calibration information.

The following information (but not limited to) is retained in the directory:

Quality System Manuals
Standard Operating Procedures
Certification Documentation
Change Management Data
External Audit Reports
Internal Audit Reports
Corrective Action Database
Laboratory Forms Directory
Health & Safety Manuals
Inactive Standard Operating Procedures
Method Detection Limit Data
Metrics Inventory & Calibration Data
Performance Limits
Proficiency Test Scores & Statistics
Project Specific Analytical Requirements
QC Report Reviews
Regulatory Agency Quality Documents
Staff Bios And Job Descriptions
Forms

6.4 ***Analytical Records***. All data related to the analysis of field samples are retained as either paper or electronic records that can be retrieved to compile a traceable audit trail for any reported result. All information is linked to the client job and sample number, which serves as a reference for all sample related information tracking.

Critical times in the life of the sample from collection through analysis to disposal are documented. This includes date and time of collection, receipt by the laboratory, preparation times and dates, analysis times and dates and data reporting information. Analysis times are calculated in hours for methods where holding time is specified in hours (≤72 hours).

Sample preparation information is recorded in a separate controlled logbook. It includes sample identification numbers, types of analysis, preparation and cleanup methods, sample weights and volumes, reagent lot numbers and volumes and any other information pertinent to the preparation procedure.

Information related to the identification of the instrument used for analysis is permanently attached to the electronic record. The record includes an electronic data file that indicates all instrument conditions employed for the analysis, including the type of analysis conducted. The analyst's identification is electronically attached to the record. The instrument tuning and calibration data is electronically linked to the sample or linked though paper logs which were used in the documentation of the analysis. Quality control and performance criteria are permanently linked to the paper archive or electronic file.

Paper or electronic records for the identity, receipt, preparation and evaluation of all standards and reagents used in the analysis are documented in prepared records and maintained in controlled documents or files. Lot number information linking these materials to the analysis performed is recorded in the logbooks associated with the samples in which they were used.

Manual calculations or peak integrations that were performed during the data review are retained as paper or electronic (PDF) documents and included as part of the electronic archive. Signatures for data review are retained on paper or as scanned versions of the paper record for the permanent electronic file.

6.5 ***Confidential Business Information (CBI).*** Operational documents including SOPs, Quality Manuals, personnel information, internal operations statistics, and laboratory audit reports are considered confidential business information. Strict controls are placed on the release of this information to outside parties.

Release of CBI to outside parties or organizations may be authorized upon execution of a confidentiality agreement between Accutest and the receiving organization or individual. CBI information release is authorized for third party auditors and commercial clients in electronic mode as Adobe Acrobat .PDF format only.

6.6 ***Software Change Documentation & Control.*** Changes to software are documented as text within the code of the program undergoing change. Documentation includes a description of the change, reason for change and the date the change was placed into effect. Documentation indicating the adequacy of the change is prepared following the evaluation by the user who requested the change.

6.7 ***Report and Data Archiving***. Data files from sample analysis are downloaded to the intranet server, which is backed up to magnetic tape daily, and archived on-site. The system backup tape clones are archived off-site. Accutest Laboratories maintains the data files or image file copies of original reports in archive for a period of five (5) years. This includes data reports and raw data for field sample analysis, proficiency test analysis and all associated quality control sample analysis. After five years, the files are automatically discarded unless contractual arrangements exist which dictate different requirements. Client or regulatory agency specific data retention practices are employed for several government organizations such as the Department of Defense and the Massachusetts Department of Environmental Protection that require a retention period of ten (10) years.

Accutest archives an electronic (PDF) copy of the original report, and electronic (PDF) copies of the organic and inorganic support data. Organic support data is archived according to instrument batch numbers. Organic and inorganic support data is scanned and saved as a PDF. Closed client files and Chain of Custody documents are maintained in the office area until archiving is necessary. Paper copy data is archived as needed and placed in the on-site long-term storage area (or scanned and archived electronically). The data is placed in storage boxes and marked with the bracketing report numbers. Client report data is filed by the job number.

Method detection limit (MDL) studies are maintained as electronic data files in the Quality Assurance network directory. Raw data for MDL studies and P&A studies are stored in the same manner as described for inorganic and organic raw data.

Laboratory logbooks are archived in labeled boxes in the on-site storage area. Boxes are labeled according to the facility (MA) and department (e.g. MMS for mass spec logbooks, MGC for GC logbooks, etc.), and by sequential numbers (e.g.MMS001 for MA

mass spec data box 001). Retrieval and return of archived data is recorded in an Archive logbook maintained by QA.

6.8 ***Training***. The company maintains a training record for all employees that documents that they have received instruction on administrative and technical tasks that are required for the job they perform. Training records for individuals employed by the company are retained for a period of six months following their termination of employment.

Training File Origination. Quality Assurance initiates training files. QA retains the responsibility for the maintenance and tracking of all training related documentation in the file. The file is begun on the first day of employment. Information required for the file includes a copy of the individual's most current resume, detailing work experience and a copy of any college diplomas or transcript(s). Information added includes documentation of health and safety and Quality Assurance training, data integrity training, and a signed ethics agreement.

Training documentation, training requirements, analyst proficiency information and other training related support documentation is tracked using a customized database application (section 4.2). Database extracts provide an itemized listing of specific training requirements by job function. Training status summaries for individual analysts portray dates of completion for job specific training requirements.

Technical Training. The supervisor of each new employee is responsible for developing a training plan for each new employee. The supervisor evaluates the employees training progress at regular frequencies. Supporting documentation, including SOP documentation of understanding, demonstration of capability and precision and accuracy studies, which demonstrate an analyst's proficiency for a specific test, are added to the training file as completed. Employees and supervisors verify documentation of understanding (DOU) for all assigned standard operating procedures in the training database. Certificates or diplomas for any off-site training are also added to the file.

6.9 ***Master List.*** A master list of all laboratory references (including SOPs, Methods, client QAPPS, regulatory agency and client technical specifications, forms, the QSM, and any other controlled document) is maintained by QA. The master list is updated for each addition, removal, or revision to a controlled document and the date of update is documented on the master list. The master list is maintained on the write-protected QA server.

6.10 ***Controlled Document Procedures.*** Any controlled document that is submitted must be assigned a control number, and this document must be retrieved/replaced when the document is revised or becomes obsolete. If a controlled document is submitted without the intent of retrieval it is not considered controlled, and must clearly be identified with an "UNCONTROLLED DOCUMENT" watermark or similar marking.

7.0 REFERENCE STANDARD TRACEABILITY

***Requirement*:** Documented procedures, which establish traceability between any measured value and a national reference standard, are established by the laboratory as required. All metric measurements are traceable to NIST reference weights or thermometers that are calibrated on a regular schedule. All chemicals used for calibration of a quantitative process are traceable to an NIST reference that is documented by the vendor using a certificate of traceability. The laboratory maintains a documentation system that establishes the traceability links. The procedures for verifying and documenting traceability are documented in standard operating procedures.

7.1 ***Traceability of Metric Measurements - Thermometers***. Accutest uses NIST thermometers to calibrate commercially purchased thermometers prior to their use in the laboratory. If necessary, thermometers are assigned correction factors that are determined during their calibration using an NIST thermometer as the standard. The correction factor is documented in a thermometer log and on a tag attached to the thermometer. The correction factor is applied to temperature measurements before recording the measurement in the temperature log. Calibration of each thermometer is verified and documented on a regular schedule. Glass type thermometers are calibrated against the NIST thermometer on an annual basis. Infrared and probe type or instrument thermometers are calibrated on a quarterly basis. The NIST thermometer is checked for accuracy by a qualified vendor every five (5) years following the specifications for NIST thermometer calibration verification detailed in the United States Environmental Protection Agency's "Manual for the Certification of Laboratories Analyzing Drinking Water", Fifth Edition, January 2005. This calibration verification must be performed by an ISO/IEC 17025 – accredited contractor, and include data points throughout the working range of the thermometer. Refer to the Purchase of Lab Supplies SOP (MQA221) for details.

7.2 ***Traceability of Metric Measurements – Calibration Weights***. Accutest uses calibrated weights, which are traceable to NIST standard weights to calibrate all balances used in the laboratory. Balances are calibrated to specific tolerances within the intended use range of the balance. Calibration checks are required on each day of use. If the tolerance criteria are not achieved, corrective action specified in the balance calibration SOP is applied before the balance can be used for laboratory measurements. Recalibration of all calibration weights is conducted and documented every 5 years. This calibration must be performed by an ISO/IEC 17025 - accredited contractor. Refer to the Purchase of Lab Supplies SOP (MQA221) for details.

7.3 ***Traceability of Metric Measurements – Top Loader and Analytical Balances.*** The laboratory top-loader and analytical balances are calibrated annually by an ISO/IEC 17025 – accredited contractor. The calibration must include the working range of the balance. Refer to the Purchase of Lab Supplies SOP (MQA221) for details.

7.4 ***Traceability of Chemical Standards***. All chemicals, with the exception of bulk dry chemicals and acids, purchased as reference standards for use in method calibration must establish traceability to NIST referenced material through a traceability certificate. Process links are established that enable a calibration standard solution to be traced to its NIST reference certificate.

Chemical standards used for analysis must meet the specifications of the method. These specifications must be stated in the reagents section of the method SOP.

7.5 ***Assignment of Reagent and Standard Expiration Dates.*** Expiration date information for all purchased standards, prepared standard solutions and selected reagents is provided to Accutest by the vendor as a condition of purchase. Neat materials and inorganic reagents are not required to be purchased with expiration dates. Prepared solutions are labeled with the expiration date provided by the manufacturer. In-house prepared solutions are assigned expiration dates that are consistent with the method that employs their use unless documented experience indicates that an alternate date can be applied. If alternate expiration dates are employed, their use is documented in the method SOP. Expiration dates for prepared inorganic reagents, which have not exhibited instability, are established at two years from the date of preparation for tracking purposes. For standards or reagents with a partially defined expiration date (i.e. month and year), the last day of the month will be used as the final expiration date.

7.6 ***Reagents and Supplies Verification.*** Bulk chemicals/reagents and other consumable materials that may affect the quality of environmental tests are inspected or otherwise verified prior to use. An aliquot is removed and analyzed by the applicable test and verified to be free from contamination prior to use on a lot basis. Sample bottles are verified for use on a lot basis by similar means. Other consumable materials are verified for use by various other means.

The earliest expiration date has been established as the limiting date for assigning expiration dates to prepared solutions. The assignment of expiration dates that are later than the expiration date of any derivative solution or material are prohibited.

7.7 ***Documentation of Traceability***. Traceability information is documented in individual logbooks designated for specific measurement processes. The quality assurance group maintains calibration documentation for metric references in separate logbooks.

Balance calibration verification is documented in logbooks that are assigned to each balance. The individual conducting the calibration is required to initial and date all calibration activities. Any defects that occur during calibration are also documented along with the corrective action applied and a demonstration of return to control.

Temperature control is documented in logbooks assigned to the equipment being monitored. A calibrated thermometer is assigned to each individual item. Measurements are recorded along with date and initials of the individual conducting the measurement on a daily or as used basis. Corrective action, if required, is also documented including the demonstration of return to control.

Initial traceability of chemical standards is documented via a vendor-supplied certificate (not available for bulk dry chemicals and acids) that includes lot number and expiration date information. Solutions prepared using the vendor supplied chemical standards are documented in logbooks assigned to specific analytical processes. Alternatively, documentation may be entered into the electronic standards and reagent tracking log. The documentation includes links to the vendor's lot number, an internal lot number, dates of preparation, expiration date, and the preparer's initials.

Supervisors conduct regular reviews of logbooks, which are verified using a signature and date.

8.0 TEST PROCEDURES, METHOD REFERENCES, AND REGULATORY PROGRAMS

Requirements: The laboratory employs client specified or regulatory agency approved methods for the analysis of environmental samples. A list of active methods is maintained, which specifies the type of analyses performed and cross-references the methods to applicable environmental regulations. Routine procedures used by the laboratory for the execution of a method are documented in standard operating procedures. Method performance and sensitivity are demonstrated annually where required. Defined procedures for the use of method sensitivity limits for data reporting purposes are established by the QA Officer and used consistently for all data reporting purposes.

8.1 ***Method Selection & Application***. Accutest employs methods for environmental sample analysis that are consistent with the client's application, which are appropriate and applicable to the project objectives. Accutest informs the client if the method proposed is inappropriate or outdated and suggests alternative approaches.

Accutest employs documented, validated regulatory methods in the absence of a client specification and informs the client of the method selected. These methods are available to the client and other parties as determined by the client. Documented and validated in-house methods may be applied if they are appropriate to the project. The client is informed of the method selection.

8.2 ***Standard Operating Procedures***. Standard operating procedures (SOP) are prepared for routine methods executed by the laboratory, processes related to laboratory operations and sample or data handling. All SOPs are formatted to meet the specifications established by the National Environmental Laboratory Conference, which are detailed in Chapter Five – Quality Systems of the established Standards. The procedures describe the process steps in sufficient detail to enable an individual, who is unfamiliar with the procedure to execute it successfully.

SOPs are evaluated annually and edited if necessary. Reviewed SOPs that do not require modification include an evaluation summary form indicating that an evaluation was conducted and modifications were not needed. SOPs can be edited on a more frequent basis if changes are required for any reason. These may include a change to the methodology, elimination of systematic errors that dictate a need for process changes or modifications to incorporate a new version of the method promulgated by the originating regulatory agency. Procedural modifications are indicted using a revision number. SOPs are available for client review at the Accutest facility upon request.

The complete list of the laboratories SOPs available is maintained in the QA server under the "current SOP" folder and in the training application database.

8.3 ***Method Validation***. Standard methods from regulatory sources are primarily used for all analysis. Standard methods do not require validation by the laboratory. Non-standard, in-house methods are validated prior to use. Validation is also performed for standard

methods applied outside their intended scope of use. Validation is dependent upon the method application and may include analysis of quality control samples to develop precision and accuracy information for the intended use. A final method validation report is generated, which includes all data in the validation study. A statement of adequacy and/or equivalency is included in the report. A copy of the report is archived in the quality assurance directory of the company server.

Non-standard methods are validated prior to use. This includes the validation of modified standard methods to demonstrate comparability with existing methods. Demonstrations and validations are performed and documented prior to incorporating technological enhancements and non standard methods into existing laboratory methods used for general applications. The demonstration includes method specific requirements for assuring that significant performance differences do not occur when the enhancement is incorporated into the method. Validation is dependent upon method application and may include the analysis of quality control samples to develop precision and accuracy information for intended use.

The study procedures and specifications for demonstrating validation include comparable method sensitivity, calibration response, method precision, method accuracy and field sample consistency for several classes of analytical methods are detailed in this document. These procedures and specifications may vary depending upon the method and the modification.

8.4 ***Estimated Uncertainty.*** A statement of the estimated uncertainty of an analytical measurement accompanies the test result when required. Estimated uncertainty is derived from the performance limits established for spiked samples of similar matrices. The degree of uncertainty is derived from the negative or positive bias for spiked samples accompanying a specific parameter. When the uncertainty estimate is applied to a measured value, the possible quantitative range for that specific parameter at that measured concentration is defined. Well recognized regulatory methods that specify values for the major sources of uncertainty and specify the data reporting format do not require a further estimate of uncertainty.

8.5 ***Demonstration of Capability***. Confirmation testing is conducted to demonstrate that the laboratory is capable of performing the method before its application to the analysis of environmental samples. The results of the demonstration tests are compared to the quality control specifications of the method to determine if the performance is acceptable.

Capability demonstrations are conducted initially for each method on every instrument and annually on a method specific basis thereafter. Acceptable demonstrations are documented for individual training files and retained by the QA staff. New analytes, which are added to the list of analytes for an accredited method, are evaluated for applicability through a demonstration of capability similar to those performed for accredited analytes.

The New England facility maintains 2 work cells. These work cells are Organic Prep and Metals. The Organic Prep work cell is defined as the entire department. Extractable test Demonstrations of Capability (DOC) are evaluated for the Organic Prep work cell and for each individual participating in the DOC. The Metals work cell is defined as the prep employee and the analyst.

8.6 ***Method Detection Limit Determination***. Annual method detection limit (MDL) studies are performed as appropriate for routine methods used in the laboratory. The procedure used for determining MDLs is described in 40 CFR, Part 136, Appendix B. Studies are performed for each method on water, soil and air matrices for every instrument that is used to perform the method. MDLs are established at the instrument level. The highest MDL of the pooled instrument data is used to establish a laboratory MDL. MDLs are experimentally verified on every instrument used to perform the analysis through the analysis of standard solutions at 1-4 times the concentration of the experimental MDL. Certain regulatory agencies (such as the Department of Defense - DoD) or clients may have different verification requirements (DoD requires MDLs to be verified by a standard at 1-3 times the MDL). The quality assurance staff manages the annual MDL determination process and is responsible for retaining MDL data on file. Approved MDLs are appended to the LIMS and used for data reporting purposes.

8.7 ***Instrument Detection Limit Determination***. Instrument detection limits (IDLs) are determined for all inductively coupled argon plasma emission spectrophotometers and graphite furnace atomic absorption spectrophotometers. The IDL is determined for each wavelength used for sample analysis. The IDL data is used to estimate instrument sensitivity in the absence of the sample matrix. IDL determinations are conducted at the frequency specified in the appropriate SOPs' for ICP analysis.

8.8 ***Method Reporting Limit.*** The method reporting limit is determined by the concentration of the lowest calibration standard in the calibration curve for most organics methods. This value is adjusted based on several sample preparation factors including sample volume, moisture content (soils), digestion, distillation or dilution. The low calibration standard is selected by department managers as the lowest concentration standard that can be used for calibration while continuing to meet the calibration linearity criteria of the method being used. The validity of the method reporting limits are confirmed through the analysis of a spiked quality control sample at 1-2 times the method reporting limit concentration. By definition, detected analytes at concentrations below the low calibration standard cannot be accurately quantitated and are qualified as estimated values.

8.9 ***Reporting of Quantitative Data.*** Analytical data for all methods is reported without qualification to the reporting limit established for each method. Data, for organic methods may be reported to the established method detection limit depending upon the client's requirements provided that all qualitative identification criteria for the detected parameter have been satisfied. All parameters reported at concentrations between the reporting limit and the method detection limit are qualified as estimated.

Measured concentrations of detected analytes that exceed the upper limit of the calibration range are either diluted into the range and reanalyzed or qualified as an estimated value. The only exception to this applies to ICP and ICP/MS analysis, which can be reported to the upper limit of the experimentally determined linear range without qualification.

8.10 ***Precision and Accuracy Studies***. Annual precision and accuracy (P&A) studies, which demonstrate the laboratories ability to generate acceptable data, are performed for all routine methods used in the laboratory. The procedure used for generating organic P&A

data is referenced in the majority of the regulatory methodology in use. The procedure requires quadruplicate analysis of a sample spiked with target analytes at a concentration in the working range of the method. This data may be compiled from a series of existing blank spikes or laboratory control samples. Accuracy (percent recovery) of the replicate analysis is averaged and compared to established method performance limits. Values within method limits indicate an acceptable performance demonstration. Precision and accuracy date is also used to annually demonstrate analytical capability for individual analysts. Annual demonstration of capability data is archived in individual training files.

Alternative methods for demonstrating may be analysis of a PT sample, quadruplicate analysis of a known standard, and/or review of the test with the analyst for determination of understanding of the method. Capability is demonstrated using the PT samples if the results meet the PT provider's acceptance criteria. For quadruplicate analysis, capability is demonstrated if the standard deviation of the analyses falls within the method acceptance criteria. If no standard deviation criteria exist, the supervisor reviews the results and make a judgement regarding acceptability of the standard deviation results. Additionally, the supervisor may determine capability by review of the test with the analyst to establish the analyst's understanding of the procedure.

8.11 ***Method Performance Limits***. Quality control data from each method is compiled in the laboratory information management system (LIMS) and used to develop performance limits and control limits for selected quality parameters. The pooled data is statistically evaluated and updated annually. Upper and lower control limits are developed for these parameters, which replace the limits from the previous year. The new limits are used by the analysts to assess performance and determine compliance with the quality criteria of the method being used. The analyst is responsible for making decisions regarding corrective action using this information during the method set-up, sample analysis, and data review steps.

8.12 ***Method Sources & References.*** The Quality Assurance Staff maintains a list of active methods used for the analysis of samples. This list includes valid method references from sources such as USEPA, ASTM or Standard Methods designations and the current version and version date.

Updated versions of approved reference methodology are placed into use as changes occur. The Quality Assurance Director informs operations management of changes in method versions as they occur. The operations management staff selects an implementation date. The operations staff is responsible for completing all method use requirements prior to the implementation date. This includes modification of SOPs, completion of MDL and precision and accuracy studies and staff training. Documentation of these activities is provided to the QA staff that retains this information on file. The updated method is placed into service on the implementation date and the old version is de-activated.

Multiple versions of selected methods may remain in use to satisfy client specific needs. In these situations, the default method version becomes the most recent version. Client specific needs are communicated to the laboratory staff using method specific analytical method codes, which clearly depict the version to be used. The old method version is maintained as an active method until the specified client no longer requires the use of the older version.

Accutest will not use methodology that represents significant departures from the reference method unless specifically directed by the client. If clients direct the laboratory to use a method modification that represents a significant departure from the reference method, the request will be documented in the project file.

Analytical Capabilities. Appendix III provides a detailed listing of the methodology employed for the analysis of test samples.

9.0 SAMPLE MANAGEMENT, LOGIN, CUSTODY, STORAGE AND DISPOSAL

Requirement: The laboratory must employ a system which ensures that client supplied product (the sample) is adequately evaluated, acknowledged, and secured upon delivery to the laboratory. The system also assures that product chain of custody is maintained and that sample receipt conditions and preservation status are documented and communicated to the client and internal staff. The login procedure assigns, documents, and maps the specifications for the analysis of each unique sample to assure that the requested analysis is performed on the correct sample and enables the sample to be tracked throughout the laboratory analytical cycle. The system includes procedures for reconciling defects in sample condition or client provided data, which are identified at sample arrival. The system specifies the procedures for proper sample storage, transfer to the laboratory, and disposal after analysis. The system is also documented in standard operating procedures.

9.1 *Order Receipt and Entry*

Order Receipt and Entry. New orders are initiated and processed by Client Services or Sample Management (See Chapter 14, Procedures for Executing Client Specifications). The new order procedure includes mechanisms for providing bottles to clients, which meet the size, cleanliness, and preservation specifications for the analysis to be performed.

New orders are communicated to the laboratory verbally, by FAX using the sample supply requisition form, or by email using the on-line requisition system. This form provides critical project details to the sample management staff, which are used to prepare and assemble the sample bottles for shipment to the client prior to sampling.

The bottle order is assembled using bottles that meet USEPA specifications for contaminant free sample containers. Accutest uses a combination of commercially supplied pre-cleaned bottles and bottles that have been tested for residual contamination and verified to meet USEPA specifications prior to use. Sterile bottles for microbiological samples are purchased from commercial sources.

Bottles, which are not purchased pre-cleaned, are checked to assure that they are free of contamination from targeted analytes before being released for use. Sterile bottles are checked for contamination with each lot. Acceptable cleanliness is demonstrated by the absence of the analyte(s) of interest at the detection limit of the method to be used for sample analysis.

Preservative solutions that are specified for the analysis requested are dispensed into the sample bottle prior to shipment. All preservative solutions are prepared in the laboratory or purchased from commercial suppliers. Each solution is checked to assure that it is free of contamination from the compounds being analyzed before being released for use.

Reagent water for trip and field blanks is poured into appropriately labeled containers. One temperature blank per cooler is provided. All bottles are packed into ice chests with blank chain of custody forms and the original bottle order form. Completed bottle orders are delivered to clients using Accutest couriers or commercial carriers for use in field sample collection.

9.2 ***Sampling***. Documented procedures are employed by the field staff for field sample collection and are accessible during sample collection activities. Field activities are documented in controlled notebooks which detail relevant field conditions, site data and the results of field measurements. Appropriate custody procedures for collected samples are initiated by the field staff at the time of sample collection. Samples are documented, labeled and preserved according to the specifications of the method and/or regulatory program prior to being shipped to the laboratory.

9.3 ***Sample Receipt and Custody***. Samples are delivered to the laboratory using a variety of mechanisms including Accutest couriers, commercial shippers, and client self-delivery. Documented procedures are followed for arriving samples to assure that custody and integrity are maintained and handling/ preservation requirements are documented and maintained.

Sample custody documentation is initiated when the individual collecting the sample collects field samples. Custody documentation includes all information necessary to provide an unambiguous record of sample collection, sample identification, and sample collection chronology. Initial custody documentation employs either Accutest or client generated custody forms.

Accutest generates a chain of custody in situations where the individuals who collected the sample did not generate custody documentation in the field.

Accutest defines sample custody as follows:

1 The sample is in the actual custody or possession of the assigned responsible person,

2 The sample is in a secure area.

The Accutest facility is defined as a secure facility. Perimeter security has been established, which limits access to authorized individuals only. Visitors enter the facility through the building lobby and must register with the receptionist prior to entering controlled areas. While in the facility, visitors are required to wear a visitor's badge and must be accompanied by their hosts at all times. Building access is controlled using a computerized password keypad system. This system limits building access to individuals with a pre-assigned authorization status. Clients delivering samples after hours must make advanced arrangements through client services and sample management to assure that staff is available to take delivery and maintain custody.

Upon arrival at Accutest, the sample custodian reviews the chain of custody for the samples received to verify that the information on the form corresponds with the

samples delivered. This includes verification that all listed samples are present and properly labeled, checks to verify that samples were transported and received at the required temperature, verification that the sample was received in proper containers, verification that sufficient volume is available to conduct the requested analysis, and a check of individual sample containers to verify test specific preservation requirements including the absence of headspace for volatile compound analysis.

The following items are verified upon receipt:

* Sample Matrix
* Sample Collection Information (Sample Point).
* Signature of samplers and individuals relinquishing custody.
* Date and time of sample collection.
* The sample volume and preservative used for each bottle.
* Sample Integrity (breakage, headspace for volatiles, etc.).
* Signatures of the individual accepting sample custody.
* The analysis to be performed.
* The client's name, contact, address, phone number.
* Any special requirements, such as turnaround, or reporting deliverables.
* Sample(s) or cooler temperature. (documented upon receipt)
* Residual chlorine for wastewater and drinking water samples (as applicable).

Sample conditions and other observations are documented on the chain of custody by the sample custodian prior to completing acceptance of custody and in an online database that creates a permanent record of all sample login activities. The sample custodian accepts sample custody upon verification that the custody document is correct. Discrepancies or non-compliant situations are documented and communicated to the Client Services staff, who contacts the client for resolution. Temperature violations are recorded in a memo (form SM003), which is sent to the client. The resolution is documented and communicated to sample management for execution.

The sample management staff maintains an electronic sample receipt log. This log details all sample-related information in a searchable database that is updated upon data entry and backed up daily. The log records include critical date information, numbers of samples, numbers of bottles for each parameter, descriptions of bottles for each parameter, preservation conditions, bottle refrigerator location, and bottle conditions. Data entry into the log is secured using individual usernames.

During initial login, each bottle is assigned a unique number and is labeled with a barcode corresponding to that number. A bar-coding and scanning system electronically tracks sample custody transfers between individuals within the laboratory. Internal custody documentation may be required for compliance with regulatory agency or contractual specifications. A documented, chronological record of each sample transfer identifying each individual having possession of the sample is created in the laboratory information management system, which can be printed and included in data reports to demonstrate continuous custody.

9.4 ***Sample Tracking Via Status Change.*** An automated, electronic LIMS procedure records sample exchange transactions between departments and changes in analytical status. This system tracks all preparation, analytical, and data reporting procedures to which a sample

is subjected while in the possession of the laboratory. Each individual receiving samples must acknowledge the change in custody and operational status in the LIMS. This step is required to maintain an accurate electronic record of sample status, dates of analytical activity, and custody throughout the laboratory.

Sample tracking is initiated at login where all chronological information related to sample collection dates and holding times are entered into the LIMS. This information is entered on an individual sample basis.

9.5 ***Sample Acceptance Policy***. Incoming samples must satisfy Accutest's sample acceptance criteria before being logged into the system. Sample acceptance is based on the premise that clients have exercised proper protocols for sample collection. This includes complete documentation, sufficient volume, proper chemical preservation, temperature preservation, sample container sealing and labeling, and appropriate shipping container packing. Accutest's Sample Acceptance Policy is provided in a separate document, and is reviewed annually by the Sample Management Staff.

The sample management staff will make every attempt to preserve improperly preserved samples upon arrival. However, if preservation is not possible, the samples may be refused unless the client authorizes analysis. No samples will be accepted if holding times have been exceeded or will be exceeded before analysis can take place unless the client authorizes analysis.

Sample acceptance criteria include proper custody and sample labeling documentation. Proper custody documentation includes an entry for all physical samples delivered to the laboratory with an identification code that matches the sample bottle and a date and signature of the individual who collected the sample and delivered them to the laboratory. All chain of custodies and sample labels must be written in indelible ink.

Accutest reserves the right to refuse any sample which in its sole and absolute discretion and judgment is hazardous, toxic and poses or may pose a health, safety or environmental risk during handling or processing. The company will not accept samples for analysis using methodology that is not performed by the laboratory or for methods that lab does not hold valid accreditations unless arrangements have been made to have the analysis conducted by a qualified subcontractor, or a valid accreditation is unnecessary.

The reason for sample rejection must be documented. This documentation should be included in the job folder. All jobs must therefore be provided a number – even if rejected – in order to provide the lab with the means of maintaining documentation of any sample rejections.

9.6 ***Assignment of Unique Sample Identification Codes***. Unique identification codes are assigned to each sample bottle to assure traceability and unambiguously identify the tests to be performed in the laboratory.

The sample identification coding process begins with the assignment of a unique alphanumeric job number. A job is defined as a group of samples received on the same day, from a specific client pertaining to a specific project. A job may consist of groups of samples received over multi-day period. The first character of the job number is an alpha-character that identifies the laboratory facility ("M" is for Massachusetts). The next

characters are numeric and sequence by one number with each new job.

Unique sample numbers are assigned to each bottle collected as a discrete entity from a designated sample point. This number begins with the job number and incorporates a second series of numbers beginning at one and continuing chronologically for each point of collection. The test to be performed is clearly identified on the bottle label. Multiple sample bottles collected for analysis of the same parameter are numbered bottle 1, 2, … etc.

Alpha suffixes may be added to the sample number to identify special designations such as subcontracted tests, in-house QC checks, or re-logs. Multiple sample bottles for a specific analysis are labeled Bottle 1, Bottle 2, etc.

9.7 ***Subcontracted Analysis***. Subcontract laboratories are employed to perform analysis not performed by Accutest. The Laboratory Director and Quality Assurance department evaluates subcontract laboratories to assure their quality processes meet the standards of the environmental laboratory industry prior to engagement. Throughout the subcontract process, Accutest follows established procedures to assure that sample custody is maintained and the data produced by the subcontractor meets established quality criteria.

9.8 ***Subcontracting Procedure*.** Subcontracting procedures are initiated through several mechanisms, which originate with project or sample management. Samples for analysis by a subcontractor are logged into the Accutest system using regular login and Accutest job number assigning procedures. The subcontract process is triggered if subcontract parameters are part of the project or sample management has received subcontracting instructions for a specific project. The Sample Manager or Client Services contacts an approved subcontractor to place the subcontract order. The subcontract chain of custody is forwarded to sample management. A copy is filed with the original COC. Sample management signs the subcontract chain of custody and ships the sample(s) to the subcontractor. The subcontract COC is filed with the original COC and the request for subcontract and maintained in sample management.

Sample receipt by the subcontract laboratory is confirmed and documented. The documentation may consist of an acknowledgement form from the sub lab or a copy of the signed COC. For samples delivered by an Accutest courier, a signed COC for the subcontracted analyses must be obtained. All documents are filed with the original client COC. The status of the data will be tracked routinely by the administrative assistant.

Clients are verbally notified of the need to subcontract analysis as soon as the need is identified by the client services staff. This may occur during the initial project setup or at the time of login if the project setup had not been initiated through the client services staff. Copies of the subcontract CoC and the original CoC, which are electronically distributed to clients, constitutes documented client notification of the laboratories intent to subcontract analysis.

Subcontractor data packages are reviewed by the Laboratory Director or Manager or QA Staff to assess completeness and quality compliance. If completeness defects are detected, the subcontractor is asked to immediately upgrade the data package. If data quality defects are detected, QA retains the package for further review. QA will pursue a corrective action solution before releasing defective data to the client.

Approved subcontract data is entered into the laboratory information management system (LIMS) if possible and incorporated into the final report. All subcontract data is footnoted to provide the client with a clear indication of its source. Copies of original subcontract data are included in the data report depending on the reporting level specified by the client. Applicable subcontractor accreditation information is provided with the subcontractor data.

Subcontractor laboratories must have an established and documented laboratory quality system that complies with DoD QSM requirements. The subcontractor laboratories must be approved by the specific DoD Component laboratory approval process (or if not this must be communicated to the DoD client for approval). Subcontractor laboratories must demonstrate the ability to generate acceptable results from the analysis of proficiency testing samples (as applicable). Subcontractor laboratories must receive project-specific approval from the DoD client before any samples are analyzed.

9.9 ***Subcontract Laboratory Evaluation*****.** The Laboratory Director or QA Officer evaluates subcontract laboratories prior to engagement. The subcontract laboratory must provide Accutest with a valid regulatory agency certification to perform the requested analysis, a copy of the laboratory quality assurance plan, copies of SOPs used for the subcontracted analysis (if possible), a copy of the most recent proficiency study (PT) results for the subcontracted parameter (optional - if applicable), a copy of the internal data integrity policy and copies of the most recent regulatory agency audit report (if possible). Certification verification, audit reports and PT data must be submitted to Accutest annually. If possible, the QA officer or Laboratory Director conducts a site visit to the laboratory to inspect the quality system. Qualification of a subcontract laboratory may be bypassed if the primary client directs Accutest to employ a specific subcontractor.

9.10 ***Sample Storage***. Following sample custody transfer, samples are assigned to refrigerated storage areas by the sample custodian depending upon the test to be performed and the matrix of the samples. The samples are stored in refrigerators maintained at 4.0^0 C ($2 - 6^0$ C) until analysis. Some volatile soil samples may be stored at <-10°C to -20°C. The temperature of each refrigerator or freezer is recorded each day (including weekends and holidays) using a dedicated thermometer calibrated against a NIST-traceable thermometer. The use of a maximum/minimum reading thermometer may by used to monitor refrigerator or freezer temperatures in lieu of a direct reading for weekends and holidays. Corrective action is taken immediately if the temperature is outside of the acceptable range. The location (refrigerator and shelf) of each sample is recorded on the chain of custody adjacent to the line corresponding to each sample number. Samples remain in storage until the laboratory technician requests that they be transferred into the laboratory for analysis.

Samples for volatile organics analysis are placed in storage in designated refrigerators by the sample custodian and immediately transferred to the organics group control. These samples are segregated according to matrix to limit opportunities for cross contamination to occur.

The laboratory staff is authorized to retrieve samples from these storage areas for analysis. When analysis is complete, the samples are placed back into storage.

9.11 ***Sample Login***. Following sample custody transfer to the laboratory, the documentation

that describes the clients analytical requirements are delivered to the sample login group for coding and entry to the Laboratory Information Management System (LIMS). This process translates all information related to collection time, turnaround time, sample analysis, and deliverables into a code which enables client requirements to be electronically distributed to the various departments within the laboratory for scheduling and execution.

The technical staff is alerted to client or project specific requirements through the use of the comments field in LIMS. The comments will direct the technical staff to controlled specifications documents detailing the unique requirements.

9.12 ***Sample Retrieval for Analysis.*** Internal Chain of Custody Procedure is instituted for all client projects. The purpose of an internal chain of custody is to document each date, time and reason a sample changes hands. When a sample changes hands the recipient is responsible for the security of the sample. Accutest uses an internal chain of custody form to document changes of custody in the laboratory. Internal transfer of samples is tracked by use of the bar code label.

Samples are scanned by the analyst/prep technician when removed from the storage location, and scanned back in once the sample is returned.

9.13 ***Sample Disposal***. Accutest retains all samples under proper storage for a minimum of 30 days following completion of the analysis report. Longer storage periods are accommodated on a client specific basis if required. Samples may also be returned to the client for disposal.

Accutest disposes of all laboratory wastes following the requirements of the Resource Conservation and Recovery Act (RCRA). The Company has obtained and maintains a waste generator identification number, MAV00001277487780.

Sample management generates a sample disposal dump sheet from the LIMS tracking system each week, which lists all samples whose holding period has expired. Data from each sample is compared to the hazardous waste criteria established by RCRA Hazard Class Limits.

Samples containing constituents at concentrations above the criteria are labeled as hazardous and segregated into several separate waste categories for disposal as follows:

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- Organic extracts: Chlorinated and Non-Chlorinated solvents
- Chlorinated and Non-Chlorinated used solvent vials.
- Mixed flammable solvents (hexane, acetone, toluene)
- Chlorinated solvents (methylene chloride)
- Waste oil
- Soil (solids)/Oily solid waste
- Aqueous
- Metals/liquid/acid
- Sludges (semi-solids)
- Hexane/acid waste
- Mercury waste
- PCB waste

Non-hazardous aqueous samples are diluted and disposed directly into the laboratory sink. All aqueous liquids pass through a neutralization system before entering the municipal system.

Non-hazardous solids are disposed of by the hazardous waste contractor in drums labeled as non-hazardous solids. Laboratory wastes are collected by waste stream in designated areas throughout the laboratory. Waste streams are consolidated daily or as necessary and transferred to stream specific drums for disposal through a permitted waste management contractor. Filled, consolidated drums are tested for hazardous characteristics if suspect contamination may be present and scheduled for removal from the facility.

Glass and plastic bottles from aqueous and solid samples are segregated for recycling. Recycled materials are collected by a commercial contractor and transported to a fully-permitted Total Recycling Facility. The material is sorted and processed. The glass is processed through a hammermill and broken down. The plastic containers are ground, washed, and extruded into new products.

9.14 ***Sample Collection.*** Refer to the Accutest SOP MSM293 for detail on sample collection procedures.

10.0 LABORATORY INSTRUMENTATION AND MEASUREMENT STANDARDS

Requirement: The laboratory has established procedures, which assure that instrumentation is performing to a pre-determined operational standard prior to the analysis of any samples. In general, these procedures follow the regulatory agency requirements established in promulgated methodology. The instrumentation selected to perform specified analysis are capable of providing the method specified uncertainty of measurement needed. These procedures are documented and incorporated into the standard operating procedures for the method being executed.

10.1 ***Mass Tuning – Mass Spectrometers***. The mass spectrometer tune and sensitivity is monitored to assure that the instrument is assigning masses and mass abundances correctly and that the instrument has sufficient sensitivity to detect compounds at low concentrations. This is accomplished by analyzing a specific mass tuning compound at a fixed concentration. If the sensitivity is insufficient to detect the tuning compound, corrective action must be performed prior to the analysis of standards or samples. If the mass assignments or mass abundances do not meet criteria, corrective action must be performed prior to the analysis of standards or samples.

10.2 ***Wavelength Verification – Spectrophotometers***. Spectrophotometer detectors are checked on a regular schedule to verify proper response to the wavelength of light needed for the test in use. If the detector response does not meet specifications, corrective action (detector adjustment or replacement) is performed prior to the analysis of standards or samples.

10.3 ***Inter-element Interference Checks (Metals)***. Inductively Coupled Plasma Emission Spectrophotometers (ICP) are subject to a variety of spectral interferences, which can be minimized or eliminated by applying interfering element correction factors and background correction points. Interfering element correction factors are checked on a specified frequency through the analysis of check samples containing high levels of interfering elements. Analysis of single element interferant solutions is also conducted on an as-needed basis.

If the check indicates that the method criteria have not been achieved for any element in the check

standard, the analysis is halted and data from the affected samples are not reported. Sample analysis is resumed after corrective action has been performed and the correction factors have been re-calculated.

New interfering element correction factors are calculated and applied whenever the checks indicate that the correction factors are no longer meeting criteria. At a minimum, correction factors are replaced once a year.

10.4 ***Calibration and Calibration Verification***. Many tests require calibration using a series of reference standards to establish the concentration range for performing quantitative analysis. Instrument calibration is performed using standards that are traceable to national standards. Method specific procedures for calibration are followed prior to any sample analysis.

Calibration is performed using a linear regression calculation or calibration factors calculated from the curve. The calibration must meet method specific criteria for linearity or precision. If the criteria are not achieved, corrective action (re-calibration or instrument maintenance) is performed. The instrument must be successfully calibrated before analysis of samples can be conducted.

Initial calibration for metals analysis performed using inductively coupled plasma (ICP) employs the use of a single standard and a calibration blank to establish linearity. The calibration blank contains all reagents that are placed into the calibration standard with the exception of the target elements. Valid calibration blanks must not contain any target elements.

Initial calibrations must be initially verified using a single concentration calibration standard from a second source (i.e. separate lot or different provider). The continuing validity of existing calibrations must be regularly verified using a single calibration standard. The response to the standard must meet pre-established criteria that indicate the initial calibration curve remains valid. If the criteria are not achieved corrective action (re-calibration) is performed before any additional samples may be analyzed.

Calibration verification is also performed whenever it appears that the analytical system is out of calibration or no longer meets the calibration requirements. It is also performed when the time period between calibration verification has expired.

10.5 ***Linear Range Verification and Calibration (ICP Metals)***. Linear range verification is performed for all ICP instrumentation. The regulatory program or analytical method specifies the verification frequency. A series of calibration standards are analyzed over a broad concentration range. The data from these analyses are used to determine the valid analytical range for the instrument. ICP instrument calibration is routinely performed using a single standard at a concentration within the linear range and a blank.

Some methods or analytical programs require a low concentration calibration check to verify that instrument sensitivity is sufficient to detect target elements at the reporting limit. The analytical method or regulatory program defines the criteria used to evaluate the low concentration calibration check. If the low calibration check fails criteria, corrective action is performed and verified through reanalysis of the low concentration calibration check before continuing with the field sample analysis.

10.6 ***Retention Time Development and Verification (GC).*** Chromatographic retention time windows are developed for all analysis performed using gas chromatographs with conventional detectors. An initial experimental study is performed, which establishes the width of the retention window for each compound. The retention time range of the window defines the time ranges for elution of specified target analytes on the primary and confirmation columns. Retention times are regularly updated through the analysis of an authentic standard during calibration verification (or the

midpoint of an initial calibration). New studies are performed when major changes, such as column replacement are made to the chromatographic system.

10.7 ***Equipment List.*** See Appendix III for a listing of all equipment used for measurement and/or calibration in laboratory processes.

11.0 INSTRUMENT MAINTENANCE

Requirement. Documented procedures have been established for conducting equipment maintenance. The procedure includes maintenance schedules if required or documentation of daily maintenance activities. All instrument maintenance activities are documented in instrument specific logbooks.

11.1 ***Routine, Daily Maintenance***. Routine, daily maintenance is required on an instrument specific basis and is performed each time the instrument is used. Daily maintenance includes activities to insure a continuation of good analytical performance. This may include performance checks that indicate if non-routine maintenance is needed. If performance checks indicate the need for higher level maintenance, the equipment is taken out of service until maintenance is performed. Analysis cannot be continued until all performance checks meet established criteria and a return to operational control has been demonstrated and documented. The individual assigned to the instrument is responsible for daily maintenance.

11.2 ***Non-routine Maintenance***. Non-routine maintenance is initiated for catastrophic occurrences such as instrument failure. The need for non-routine maintenance is indicated by failures in general operating systems that result in an inability to conduct required performance checks or calibration. Equipment in this category is taken out of service, tagged accordingly and repaired before attempting further analysis. Before initiating repairs, all applicable safety procedures for safe handling of equipment during maintenance, such as lock-out/tag-out are followed. Analysis is not resumed until the instrument meets all operational performance check criteria, is capable of being calibrated and a return to operational control has been demonstrated and documented. Section supervisors are responsible for identifying non-routine maintenance episodes and initiating repair activities to bring the equipment on-line. This may include initiating telephone calls to maintenance contractors if necessary. They are responsible for documenting all details related to the occurrence and repair.

11.3 ***Scheduled Maintenance***. Modern laboratory instrumentation rarely requires regular preventative maintenance. If required, the equipment is placed on a schedule, which dictates when maintenance is needed. Examples include annual balance calibration by an independent provider or ICP preventative maintenance performed by the instrument manufacturer. Section supervisors are responsible for initiating scheduled maintenance on equipment in this category. Scheduled maintenance is documented using routine documentation practices.

11.4 ***Maintenance Documentation***. Routine and non-routine maintenance activities are documented in logbooks assigned to instruments and equipment used for analytical measurements. The logbooks contain either a daily checklist for instruments requiring daily maintenance (eg. semivolatiles) or a general section to describe non-routine maintenance (volatiles and semivolatiles). The analyst who performs the maintenance is

required to check the activity upon its completion and initial the form. Maintenance logbooks must include the following information: instrument ID and type, manufacturer name and model, serial number, date received and put into use (if available).

11.5 ***General Maintenance Procedures.*** Refer to the below table for general maintenance procedures. For additional detail refer to the analytical SOP and/or instrument manual. The below table is not inclusive of all procedures.

Instrument Maintenance Table

	Frequency		Frequency
GCMS		**GC**	
Source Cleaning	As Necessary	Septa Change	As Necessary
Septa Change	As Necessary	Column Clip	As Necessary
Column Clip	As Necessary	Liner Replacement/Clean	As Necessary
Liner Replacement/Clean	As Necessary	Gold Seal Replacement	As Necessary
Gold Seal Replacement	As Necessary	Guard Column Replacement	As Necessary
Sparge Tube Cleaning	As Necessary	Y Connector Replacement	As Necessary
Clean Autosampler/Conc. Lines	As Necessary	Sparge Tube Cleaning	As Necessary
Jet Separator Clean/Replace	As Necessary	Clean Autosampler/Conc. Lines	As Necessary
		Clean Detector	As Necessary
		Jet Separator Clean/Replace	As Necessary
ICP (check/repair/adjust)		**LACHAT**	
Pump	Daily	Tubing Replacement	As Necessary
Injector	Daily	O-Ring Replacement	As Necessary
Plasma Torch	Daily		
Nebulizer	Daily		
Spray Chamber	Daily		
Autosampler Tube	Daily		
Internal Standard Tube	Daily		
Argon Cool Flow	Daily		
TOC Analyzer		**Hg Analyzer (check/repair/clean/adjust)**	
Catalyst Replacement	As Necessary	Drier	Daily
Rinse Bottles Filled	As Necessary	Rinse Tube	Daily
Absorber Replacement	As Necessary	Sample Tube	Daily
		Tin Chl Tube	Daily
		Pump cassette	Daily
		Optical Cells	Daily
IR		**LC**	
Change Dessicator	As Necessary	Replace In-Line Filter (Frit)	As Necessary
		Replace Cartridge Before Column	As Necessary

12.0 QUALITY CONTROL PARAMETERS, PROCEDURES, AND CORRECTIVE ACTION

Requirement: All procedures used for test methods incorporate quality control parameters to monitor elements that are critical to method performance. Each quality parameter includes acceptance criteria that have been established by regulatory agencies for the methods in use. Criteria may also be established through client dictates or through the accumulation and statistical evaluation of internal performance data. Data obtained for these parameters must be evaluated by the analyst, and compared to the method criteria in use. If the criteria are not achieved, the procedures must specify corrective action and conformation of control before proceeding with sample analysis. QC parameters, procedures, and corrective action must be documented within the standard operating procedures for each method. In the absence of client specific objectives the laboratory must define qualitative objectives for completeness and representativeness of data.

12.1 ***Procedure***. Bench analysts are responsible for methodological quality control and sample specific quality control. Each method specifies the control parameters to be employed for the method in use and the specific procedures for incorporating them into the analysis. These control parameters are analyzed and evaluated with every designated sample group (batch).

The data from each parameter provides the analyst with critical decision making information on method performance. The information is used to determine if corrective action is needed to bring the method or the analysis of a specific sample into compliance. These evaluations are conducted throughout the course of the analysis. Each control parameter is indicative of a critical control feature. Failure of a methodological control parameter is indicative of either instrument or batch failure. Failure of a sample control parameter is indicative of control difficulties with a specific sample or samples.

Sample Batch. All samples analyzed in the laboratory are assigned to a designated sample batch, which contains all required quality control samples and a defined maximum number of field samples that are prepared and/or analyzed over a defined time period. The maximum number of field samples in the batch is 20. Accutest has incorporated the NELAP batching policy as the sample-batching standard. This policy incorporates the requirement for blanks and spiked blanks as a time based function as defined by NELAP. Accordingly, the specified time period for a sample batch is 24 hours. Matrix spike/matrix spike duplicate, matrix spikes and duplicates are defined as sample frequency based functions and may be applied to several batches until the frequency requirement has been reached. A matrix spike/matrix spike duplicate, matrix spikes and/or duplicate is required every 20 samples.

Client criteria that defines a batch as a time based function which includes a matrix spike/matrix spike duplicates as a contractual specification will be honored. The typical batch contains a blank and a laboratory control sample (LCS or spiked blank). Batch documentation includes lot specifications for all reagents and standards used during preparation of the batch.

12.2 ***Methodological Control Parameters and Corrective Action***. Prior to the analysis of

field samples the analyst must determine that the method is functioning properly. Specific control parameters indicate whether critical processes meet specified requirements before continuing with the analysis. Method specific control parameters must meet criteria before sample analysis can be conducted. Each of these parameters is related to processes that are under the control of the laboratory and can be adjusted if out of control.

Method Blank. A method blank is analyzed during the analysis of any field sample. The method blank is defined as a sample. It contains the same standards (internal standards, surrogates, matrix modifiers, etc.) and reagents that are added to the field sample during analysis, with the exception of the sample itself. If the method blank contains target analytes(s) at concentrations that exceed method or client requirements (typically defined as method detection limit or reporting limit concentrations), the source of contamination is investigated and eliminated before proceeding with sample analysis. Systematic contamination is documented for corrective action and resolved following an analysis of consecutive blanks or system performance checks until contamination is corrected. In specific cases, contamination detected in the method blank may be acceptable if the concentrations do not exceed regulatory limits or client defined reporting limits.

Laboratory Control Samples (LCS or Spiked Blanks). A laboratory control sample (spiked blank or commercially prepared performance evaluation sample) is analyzed along with field samples to demonstrate that method accuracy is within acceptable limits. These spike solutions may be from different sources than the sources of the solutions used for method calibration depending upon the method requirements. The performance limits are derived from published method specifications or from statistical data generated from the analysis of laboratory method performance samples. Spiked blanks are blank matrices (reagent water or clean sand) spiked with target parameters and analyzed using the same methods used for samples. Accuracy data is compared to laboratory derived limits to determine if the method is in control. Laboratory control samples (LCS) are commercially prepared spiked samples in an inert matrix. Performance criteria for recovery of spiked analytes are pre-established by the commercial entity preparing the sample. The sample is analyzed in the laboratory as an external reference.

Accuracy data is compared to the applicable performance limits. If the spike accuracy exceeds the performance limits, corrective action, as specified in the SOP for the method is performed and verified before continuing with field sample analysis. In some cases, decisions are made to continue with sample analysis if performance limits are exceeded, provided the unacceptable result has no negative impact on the sample data.

Marginal exceedence (ME) values are calculated for methods containing more than eleven (11) targeted analytes. The ME is calculated as $\pm$ 4 standard deviations about the mean. MEs are considered for multi-analyte methods because of the increasing likelihood of an LCS failure as the number of analytes in the method increase. The number of allowable MEs is based on the number of target analytes in the method. Analytes that regularly fall into the ME category are treated as systematic problems, which are resolved using established trend monitoring and corrective action procedures.

Blanks and spikes are routinely evaluated before samples are analyzed. However, in situations where sample analysis is performed using an autosampler, they may be evaluated after sample analysis has occurred. If the blanks and spikes do not meet criteria, sample analysis is repeated.

Proficiency Testing. Proficiency test samples (PTs) are single or double blind spikes, introduced to the laboratory to assess method performance. PTs may be introduced as double blinds submitted by commercial clients, single or double blinds from regulatory agencies, or internal blinds submitted by the QA group.

A minimum of two single blind studies must be performed each year for every parameter in aqueous and solid matrices for each field of testing for which the laboratory maintains accreditation. Proficiency samples must be purchased as blinds from an NIST/NVLAP accredited vendor. Data from these studies are provided to the laboratory by the vendor and reported to accrediting agencies. If unsatisfactory performance is noted, corrective action is performed to identify and eliminate any sources of error. A new single blind must be analyzed if required to demonstrate continuing proficiency.

PT samples performed for accrediting agencies or clients, which do not meet performance specifications, require a written summary that documents the corrective action investigation, findings, and corrective action implementation.

Single or double blind proficiency test samples may be employed for self-evaluation purposes. Data from these analyses are compared to established performance limits. If the data does not meet performance specifications, the system is evaluated for sources of acute or systematic error. If required, corrective action is performed and verified before initiating or continuing sample analysis.

Analysis of Proficiency Samples must be performed in the same manner as client samples. Proficiency samples may not receive special treatment.

Trend Analysis for Control Parameters. The quality assurance staff is responsible for continuous analytical improvement through quality control data trend analysis. Accuracy data for spiked parameters in the spiked blank are statistically evaluated daily for trends indicative of systematic problems. Data from LCS parameters and surrogates are pooled on a method, matrix, and instrument basis. This data is evaluated by comparison to existing control and warning limits. Trend analysis is performed automatically as follows:

- Any point outside the control limit
- Any three consecutive points between the warning and control limits
- Any eight consecutive points on the same side of the mean.
- Any six consecutive points increasing or decreasing

The results of the trend analysis are printed daily for supervisory evaluation prior to sample analysis. Trends that indicate the potential loss of statistical control are further evaluated to determine the impact on data quality and to determine if corrective action is necessary. If corrective action is indicated, the supervisor informs the analysts of the corrective actions to be performed. Return to control is demonstrated before analysis resumes.

Non-Conformance Tracking. QA generates a spreadsheet detailing all QC failures on a regular basis. The spreadsheet is generated to include a specified period of time. The spreadsheet is evaluated for any significant QC trends, which are entered into the

corrective action system for resolution.

12.3 ***Sample Control Parameters and Corrective Action***. The analysis of samples can be initiated following a successful demonstration that the method is operating within established controls. Additional controls are incorporated into the analysis of each sample to determine if the method is functioning within established specifications for each individual sample. Sample QC data is evaluated and compared to established performance criteria. If the criteria are not achieved the method or the SOP specifies the corrective action required to continue sample analysis. In many cases, failure to meet QC criteria is a function of sample matrix and cannot be remedied. Each parameter is designed to provide quality feedback on a defined aspect of the sampling and analysis episode.

Duplicates. Duplicate sample analysis is used to measure analytical precision. This can also be equated to laboratory precision for homogenous samples. Precision criteria are method dependent. If precision criteria are not achieved, corrective action or additional action may be required. Recommended action must be completed before sample data can be reported.

Laboratory Spikes & Spiked Duplicates. Spikes and spiked duplicates are used to measure analytical precision and accuracy for the sample matrix selected. Precision and accuracy criteria are method dependent. If precision and accuracy criteria are not achieved, corrective action or additional action may be required. Recommended action must be completed before reporting sample data.

Serial Dilution (Metals). Serial dilutions of metals samples are analyzed to determine if analytical matrix effects may have impacted the reported data. If the value of the serially diluted samples does not agree with the undiluted value within a method-specified range, the sample matrix may be causing interference, which may lead to either a high or low bias. If the serial dilution criterion is not achieved, it must be flagged to indicate possible bias from matrix effects.

Post Digestion Spikes. Digested samples are spiked and analyzed to determine if matrix interferences are biasing the results when the pre-digestion spike (matrix spike) recovery falls outside the control limits. It may also be used to determine potential interferences per client's specification. The sample is spiked at the concentration specified in the method SOP. No action is necessary if the post digestion spike is outside of the method criteria, unless a preparation problem is suspected with the spike, in which case the post digestion spike should be remade and reanalyzed.

Surrogate Spikes (Organics). Surrogate spikes are organic compounds that are similar in behavior to the target analytes but unlikely to be found in nature. They are added to all quality control and field samples to measure method performance for each individual sample. Surrogate accuracy limits are derived from published method specifications or from the statistical evaluation of laboratory generated surrogate accuracy data. Accuracy data is compared to the applicable performance limits. If the surrogate accuracy exceeds performance limits, corrective action, as specified in the method or SOP is performed before sample data can be reported.

Internal Standards (Organic Methods). Internal standards are retention time and instrument response markers added to every sample to be used as references for

quantitation. Their response is compared to reference standards and used to evaluate instrument sensitivity on a sample specific basis. Internal standard retention time is also compared to reference standards to assure that target analytes are capable of being located by their individual relative retention time.

If internal standard response criteria are not achieved, corrective action or additional action may be required. The recommended action must be completed before sample data can be reported.

If the internal standard retention time criteria are not achieved corrective action or additional action may be required. This may include re-calibration and re-analysis. Additional action must be completed before sample data is reported.

Microbiological Quality Control. Microbiological Quality Control procedures are described in detail in the individual analytical standard operating procedures (SOPs) and the Microbiological Quality Control SOP .

12.4 ***Laboratory Derived Quality Control Criteria.*** Control criteria for in-house methods and client specific modifications that exceed the scope of published methodology are defined and documented prior to the use of the method. The Quality Assurance Officer is responsible for identifying additional control criteria needs. Control parameters and criteria, based on best technical judgment are established using input provided by the operations staff. These control parameters and criteria are documented and incorporated into the method.

The laboratory-derived criteria are evaluated for technical soundness on spiked samples prior to the use of the method on field samples. The technical evaluation is documented and archived by the Quality Assurance Staff.

When sufficient data from the laboratory developed control parameter is accumulated, the data is statistically processed and the experimentally derived control limits are incorporated into the method. This process is performed on at least an annual basis.

12.5 ***Bench Review & Corrective Action***. The bench chemists are responsible for all QC parameters. Before proceeding with sample analysis, they are required to successfully meet all instrumental QC criteria. They have the authority to perform any necessary corrective action before proceeding with sample analysis. Their authority includes the responsibility for assuring that departures from documented policies and procedures do not occur.

The bench chemists are also responsible for all sample QC parameters. If the sample QC criteria are not achieved, they are authorized and required to perform the method specified corrective action before reporting sample data.

12.6 **Data Qualifiers.** An alpha character coding system is employed for defining use limitations for reported data. These limitations are applied to analytical data by the analyst to clarify the usefulness of the reported data for data user. Common data qualifiers and their definitions are as follows (refer to form QA108 for additional qualifiers):

Organics.

J: Indicates an estimated value. Applied to calculated concentrations for tentatively identified compounds and qualitatively identified compounds whose concentration is below the reporting limit, but above the MDL.

N: Indicates qualitative evidence of a tentatively identified compound whose identification is based on a mass spectral library search and is applied to all TIC results.

B: Used for analytes detected in the sample and its associated method blank.

E: Applied to compounds whose concentration exceeds the upper limit of the calibration range.

Metals and Inorganics.

B: Applied if the reported concentration value was less than the reporting limit but greater than the MDL.

U: Applied if the reading is less than the MDL (or IDL if IDL reporting is being used).

N: Spike sample recovery not within control limits.

*: Duplicate or matrix spike duplicate analysis not within control limits.

12.7 ***QA Monitoring***. The QA staff conducts a spot review of completed data packages prior to client release for specified projects. This review includes an examination of QC data for compliance and trends indicative of systematic difficulties. If non-conformances are detected, the QA staff places an immediate stop on the release of the data and initiates corrective action to rectify the situation. The data package is released when the package becomes compliant with all quality requirements. If compliance is not possible, the data is qualified and an appropriate case narrative is generated for inclusion in the data package.

If the review reveals trends indicative of systematic problems, QA initiates an investigation to determine the cause. If process defects are detected, a corrective action is implemented and monitored for effectiveness.

Additionally, QA must conduct a review of a minimum of 10% of DoD data packages annually. This review may occur prior to release of the data package or may be performed on closed projects.

Performance Limits. The Quality Assurance Officer is responsible for compilation and maintenance of all precision and accuracy data used for performance limits. Quality control data for all test methods are accumulated and stored in the laboratory information management system (LIMS). Parameter specific QC data is extracted annually and statically processed to eliminate outliers and develop laboratory specific warning limits and control limits. The new limits are reviewed and approved by the supervisory staff prior to their use for data assessment. The new limits are used to evaluate QC data for

compliance with method requirements for a period of one year. Laboratory generated limits appear on all data reports.

12.8 ***Data Package Review***. Accutest employs multiple levels of data review to assure that reported data has satisfied all quality control criteria and those client specifications and requirements have been met. Each production department has developed specific data review procedures, which must be completed before data is released to the client.

Analytical Review. The analyst conducts the primary review of all data. This review begins with a check of all instrument and method quality control and progresses through sample quality control, concluding with a check to assure that the client's requirements have been executed. Analyst checks focus on a review of qualitative determinations and checks of precision and accuracy data to verify that existing laboratory criteria have been achieved. Checks at this level may include comparisons with project specific criteria if applicable. The analyst has the authority and responsibility to perform corrective action for any out-of-control parameter or nonconformance at this stage of review.

Analysts who have met the qualification criteria for the method in use perform secondary, peer level data reviews. Analyst qualification requirements include a valid demonstration of capability and demonstrated understanding of the method SOP. Section supervisors may perform secondary review in-lieu of a peer review. Supervisors review 100% of the data produced by their department. It includes a check of all manual calculations; an accuracy check of manually transcribed data from bench sheets to the LIMS, a check of calibration and continuing calibration, a check of all QC criteria and a comparison of the data package to client specified requirements. Also included are checks to assure the appropriate methodology was applied and that all anomalous information was properly flagged for communication in the case narrative. Supervisors have the authority to reject data and initiate re-analysis, corrective action, or reprocessing.

The Client Services staff performs a full tertiary review of the data package following its assembly by the administrative assistant. This review includes an evaluation of QC data against acceptance criteria and a check of the data package contents to assure that all analytical requirements and specifications were executed.

All laboratory data requiring manual entry into LIMS system is double-checked by the analysts performing initial data entry and the section supervisor. Verification of supervisory review is indicated on the raw data summary by the supervisor's initials and date.

Electronic data that is manually edited at the bench by the primary analyst is automatically flagged by the instrument data system indicating an override by the analyst. All manual overrides must be verified and approved by a supervisor who initials and dates all manual changes.

Hard copies of manually integrated chromatographic peaks are printed that clearly depict the manually drawn baseline. The hard copy is reviewed and approved by the section supervisor (initialed and dated) and included in the data package of all full tier reports or the archived batch records of commercial report packages.

A manager or supervisor only has permission to edit electronic data that has been committed to the LIMS. These edits may be required if needs for corrections are

indicated during the final review. A GALP audit record for all electronic changes in the LIMS is automatically appended to the record.

Report Generation Review. For full deliverable or certain designated projects the report generation group reviews all data and supporting information delivered by the laboratory for completeness and compliance with client specifications. Missing deliverables are identified and obtained from the laboratory. The group also reviews the completed package to verify that the delivered product complies with all client specifications. Non-analytical defects are corrected before the package is sent to the client. The Report Generation staff performs QA review of the packages, and works closely with the QA department.

Quality Assurance Review. Spot-check reviews are performed by the Quality Assurance Officer. This review focuses on all elements of the deliverable including the client's specifications and requirements, analytical quality control, sample custody documentation and sample identification. QA reviews at this step in the production process are geared towards systematic process defects, which require procedural changes to effect a corrective action. However, if defects are identified that can be corrected prior to data release, the QA officer returns the package to the laboratory for corrective action. The QA officer is responsible for overall quality control coordination for the laboratory.

Data Reporting. Analytical data is released to clients following a secondary review by the Department Manager. Data release at this stage of the process is limited to electronic information, which is released to clients through a secure, encrypted, password protected, Internet connection.

Hard copy data is compiled by the administrative assistant or project manager/quality assurance and assembled into the final report. The report is sent to the client following review by the Laboratory Director (and sometimes QA).

Results which exceed the maximum contaminant levels (MCLs) or reportable concentrations are reported to the client within 24 hours of obtaining valid (reviewed and approved) data. Quality Assurance prints out a report from LIMS daily detailing any MCL or reportable concentration exceedences. The Client Service Representative, Lab Manager, or Quality Assurance may report the results to the client.

All data reports include specified information, which is required to identify the report and its contents. This information includes a title, name and address of the laboratory, a unique report number, total number of pages in the report, clients name and address, analytical method identification, arriving sample condition, sample and analysis dates, test results with units of measurement, authorized signature of data release, statement of applicability, report reproduction restrictions and NELAC requirements certification.

Below is a description of the contents of a report:

A cover page is included in all analytical reports. This cover page includes:

- Client and Project names
- Accutest job number
- Sampling date

- Address to which the report is to be sent
- Total number of pages
- Lab Director Signature
- Client service contact
- NELAC logo
- Certifications and associated certification/accreditation numbers

Several types of data package formats are reported by the laboratory including but not limited to results only, results and QC summaries, and full deliverable data packages which include results, narrative, COC, QC summaries, calibration summaries, and all associated raw instrument data and applicable client correspondence. Different state packages (which require specialized data packages) may also be reported such as the MA DEP MCP data package and the CT DEP RCP data package which include results, narrative, COC, QC summaries, and applicable state forms. For the MA DEP, current certification information is provided with the data package. Data that does not conform to the requirements of the applicable party is footnoted appropriately.

All data packages include the following information:

- Cover page
- Sample summary
- Chain of custody (COC)
- Results page

The Results page includes the following information (where available):

- Client sample ID
- Lab sample ID
- Matrix
- Analytical method
- Sample and receipt dates
- Project name
- File ID
- Batch number
- Dilution factor
- Prep and analytical dates
- Compound name and CAS number
- Unit of measurement
- Result
- Reporting limit
- Method detection limit (upon request)
- Qualifier Definitions

Additional data may be also be reported per client/program and the above list may not be inclusive.

12.9 ***Electronic Data Reduction.*** Raw data from sample analysis is entered into the laboratory information management system (LIMS) using automated processes or manual entry. Final data processing is performed by the LIMS using procedures developed by the

Company.

All LIMS programs are tested and validated prior to use to assure that they consistently produce correct results. The Information Technology Staff performs software validation testing. The testing procedures are documented in an SOP. Software programs are not approved for use until they have demonstrated that they are capable of performing the required calculations.

12.10 ***Representativeness***. Data representativeness is based on the premise that qualitative and quantitative information developed for field samples is characteristic of the sample that was collected by the client and analyzed in the laboratory. The laboratory objective for representativeness defines data as representative if the criteria for all quality parameters associated with the analysis of the sample are achieved.

12.11 ***Comparability***. Analytical data is defined as comparable when data from a sample set analyzed by the laboratory is representatively equivalent to other sample sets analyzed separately regardless of the analytical logistics. The laboratory will achieve 100% comparability for all sample data which meets the criteria for the quality parameters associated with its analysis using the method requested by the client.

13.0 CORRECTIVE ACTION SYSTEM

Requirement. The laboratory employs polices and procedures for correcting defective processes, systematic errors, and quality defects enabling the staff to systematically improve product quality. The system includes procedures for communicating items requiring corrective action to responsible individuals, corrective action tracking procedures, corrective action documentation, monitoring of effectiveness, and reports to management. The system is fully documented in a standard operating procedure. Individual corrective actions and responses are documented in a dedicated database.

13.1 ***Procedure***. Corrective action is the step that follows the identification of a process defect. The type of defect determines the level of documentation, communication, and training necessary to prevent re-occurrence of the defect or non-conformance. The formal system is maintained by the quality assurance department. Operations management is responsible for working within the system to resolve identified deficiencies.

Routine Corrective Action. Routine corrective action is defined as the procedures used to return out of control analytical systems back to control. This level of corrective action applies to all analytical quality control parameters or analytical system specifications.

Bench analysts have full responsibility and authority for performing routine corrective action. The resolution of defects at this level does not require a procedural change or staff re-training. The analyst is free to continue work once corrective action is complete and the analytical system has been returned to control. Documentation of routine corrective actions is limited to logbook comments for the analysis being performed.

Process Changes. Corrective actions in this category require procedural modifications. They may be the result of systematic defects identified during audits, the investigation of client inquiries, failed proficiency tests, product defects identified during data review, or

method updates. Resolution of defects of this magnitude requires formal identification of the defect, development and documentation of a corrective action plan, and staff training to communicate the procedural change.

Technical Corrective Action. Technical corrective action encompasses routine corrective action performed by bench analysts for out of control systems and corrective actions performed for data produced using out of control systems. Technical corrective action for routine situations is conducted using the procedures detailed above.

Non-routine corrective actions apply to situations where the bench analysts failed to perform routine corrective action before continuing analysis. Supervisors and Department Managers perform corrective action in these situations. Documentation of all non-routine corrective actions is performed using the corrective action system.

Sample re-analysis is conducted if sufficient sample and holding time remain to repeat the analysis using an in-control system. If insufficient sample or holding time remains, the data is processed and qualifiers applied that describe the out of control situation. The occurrence is further documented in the case narrative and in the corrective action response. The corrective action must include provisions for retraining the analysts who failed to perform routine corrective action.

13.2 ***Documentation & Communication***. Routine corrective actions are documented as part of the analytical record. Notations are made in the comments section of the analytical chronicle or data sheet detailing the nonconformance and corrective action. Continuation of the analysis indicates that return to control was successful.

Corrective actions for process changes are documented, tracked and monitored for effectiveness. Supervisors or senior staff members may initiate corrective actions by generating a corrective action using the corrective action database application.

The corrective action database is an Access application. The initiator generates the corrective action investigation form, which is documented, tracked, distributed to responsible parties and archived through the application. The application assigns a tracking number initiation data and due date to each corrective action initiated and copies the corrective action form to the corrective action database. An E-mail message containing the form is automatically distributed to the responsible parties for resolution.

The responsible party identifies the root cause of the defect, initiates the immediate fix and develops and implements the procedural change. Existing documentation such as SOPs are edited to reflect the change. The affected staff is informed of the procedural change through a formal training session (as applicable). The training is documented and copies are placed into individual training files. The corrective action form is completed by the responsible party and returned to the QA staff via e-mail using the database application.

Initial and completed corrective action forms are maintained in the corrective action database. This entire database is backed-up and archived daily. The corrective action tracking form is maintained as an active report in the database.

Monitoring. The QA Staff monitors the implemented corrective action until it is evident that the action has been effective and the defect has been eliminated. The corrective

action database is updated by QA to reflect closure of the corrective action. The QA staff assigns an error code to the corrective action for classification of the type of errors being committed. Additional monitoring of the corrective action is conducted during routine laboratory audits.

If QA determines that the corrective action response has not effectively remedied the deficiency, the process continues with a re-initiation of the corrective action. Corrective action continues until the defect is eliminated. If another procedural change is required, it is treated as a new corrective action, which is documented and monitored using established procedures.

Client Notification. Defective processes, systematic errors, and quality defects, detected during routine audits may have negative impacts on data quality. In some cases, data that has been released to clients may be affected. If defective data has been released for use, Accutest will notify the affected clients of the defect and provide specific details regarding the magnitude of the impact to their data.

Corrective Action Code Definitions.

A = Analytical Error (an error involving analytical results such as not-acceptable PT result, compound mis-identification, incorrect result, etc.)

O = Omission Error (an error involving an inadvertent omission of a procedure or documentation).

R = Random Error (an error that is random in nature and not likely to re-occur).

S = Systemic (an error that is caused by a defect to the system)

T = Training (an error that is caused by a lack of or need for training)

N = No error identified

13.3 Management of Change

Management and Control of Change is intended to ensure that all major laboratory procedural changes affecting any aspect of the laboratories operation are reviewed and approved by the appropriate management staff before the proposed change is implemented. Supporting documentation for changes is maintained on file.

Major changes to laboratory procedures are documented throughout the change period. The initiator of the change documents the need for the change, which is forwarded to the Lab Director for approval. The approved form is returned to the initiator of the change request, who is responsible for planning, implementing, and monitoring the change. Prior to implementation, the initiator is responsible for communicating the change to the affected staff. All training and procedural changes are documented in individual training files.

Refer to the Management of Change SOP (MQA233) for system details.

13.4 Preventative Action

Needed improvement and potential sources of nonconformances, either technical or concerning the quality system are identified by preventative actions. Preventative actions include internal audits, instrument maintenance, data review, participation in Proficiency Testing (PT) studies, Trending Analysis, and any other action which is used to prevent potential non-conformance.

14.0 PROCEDURES FOR EXECUTING CLIENT SPECIFICATIONS

Requirement. Systems have been established for evaluating and processing client specifications for routine and non-routine analytical services. The systems enable the client services staff to identify, evaluate, and document the requested specifications to determine if adequate resources are available to perform the analysis. The system includes procedures for communicating the specifications to the laboratory staff for execution and procedures for verifying the specifications have been executed.

14.1 ***Client Specific Requirements.*** The Laboratory Director or Client Services (project manager) are the primary contacts for clients requesting laboratory services. Client specifications are communicated using several mechanisms. The primary source of information is the client's quality assurance project plan (QAPP) which details analytical and quality control specifications for the project. In the absence of a QAPP, projects specifications can also be communicated using contracts, letters of authorization, or letters of agreement, which may be limited to a brief discussion of the analytical requirements and the terms and conditions for the work. These documents may also include pricing information, liabilities, scope of work, in addition to the analytical requirements. QAPPs include detailed analytical requirements and data quality objectives, which supersede those found in the referenced methods. This information is essential to successful project completion.

All client specifications are reviewed by QA. A memo describing any exceptions or alternative methods is generated by QA and provided to Client Services for submittal to the client. The review memo is maintained by QA under the specific client file.

The managerial and client services staff provides additional assistance to clients who are unsure of the specifications they need to execute the sampling and analysis requirements of their project. They provide additional support to clients who require assistance in results interpretation as needed, provided they possess the expertise required to render an opinion.

The Laboratory Director or project manager is responsible for obtaining project documents, which specify the analytical requirements. Following an initial evaluation, copies are distributed to the QA Officer and the appropriate departmental managers for review and comment. The original QAPP is numbered with a document control number and filed in a secure location.

14.2 ***Requirements for Non-Standard Analytical Specifications***. Client requirements that specify departures from documented policies, procedures, or standard specifications must be submitted to Accutest in writing. These requirements are reviewed and approved by

the technical staff before the project is accepted. Once accepted, the non-standard requirements become analytical specifications, which follow the routine procedure for communicating client specifications. Departures from documented policies, procedures, or standard specifications that do not follow this procedure are not permitted.

14.3 ***Evaluation of Resources.*** A resource evaluation is completed prior to accepting projects submitted by clients. The evaluation is initiated by the Lab Director or project manager who communicates the logistical requirements of the project to the appropriate managers. Logistical specifications for new projects are summarized in writing for evaluation by the affected departments or discussed during the weekly manager's meeting. The specifications are evaluated by the department manager from a scheduling and hardware resources perspective. The project is not accepted unless the department managers have the necessary resources to execute the project according to client specifications.

14.4 ***Documentation***.

New projects are initiated by the Client Services department. All of the information needed to correctly enter the specifications for each client sample into the laboratory information management system (LIMS) is obtained by client services. This information includes data reporting requirements, billing information; data turnaround times, QA level, state of origin, and comments for detailing project specific requirements. This information is obtained prior to sample arrival and login.

Sample receipt triggers project creation and the login process. The information on the sample supply requisition form and the chain of custody is entered into the LIMS immediately prior to logging in the first sample. The sample supply requisition form may be accompanied by a quotation, which details the analytical product codes and sample matrices. These details are entered into the LIMS during login.

Special information is distributed to the laboratory supervisors and login department in electronic or hardcopy format upon project setup. All, project specific information is retained by the administrative assistant in a secure file. The project manager maintains a personal telephone log, which details conversations with the client regarding the project.

Quality Assurance prepares a summary sheet (may be a PDF of the requirement table or a manually-prepared spreadsheet) that details the client specific analytical or QC requirements for each test. The special requirements are communicated to the analyst in the comments field of the LIMS. Client specific analytical or QC requirements are located on the QA server, and are accessible to all analysts.

14.5 ***Communication***. A pre-project meeting is held between the Lab Director, project manager, and the operations managers to discuss the specifications described in the QAPPs, contract, and/or related documents. Project logistics are discussed and finalized and procedures are developed to assure proper execution of the client's analytical specifications and requirements. Questions, raised in the review meeting, are discussed with the client for resolution. Exceptions to any requirements, if accepted by the client, are documented and incorporated into the QAPP or project documentation records.

Non-standard specifications for individual clients are documented in the LIMS at the

client account level or program level. Simple specifications are documented as comments for each project. Once entered into the LIMS, these specifications become memorialized for all projects related to the client account.

Client specifications are detailed in the Client Technical Specifications folder on the QA server (write-protected). Each client technical specification is stored separately under its own folder. Depending on the complexity of the specifications – these specifications may be further broken down for each department. The analysts are directed for follow a client specification by comments documented in the LIMS during login, and all analysts are trained on the use of this system. QA is responsible for updating the Client Technical Specifications folder as needed.

14.6 ***Operational Execution.*** A work schedule is prepared for each analytical department on a daily basis. Analytical specifications from recently arrived samples have now been entered into the LIMS database. The database is sorted by analytical due date and holding time into product specific groups. Samples are scheduled for analysis by due date and holding time. The completed schedule, which is now defined as a work list, is printed. The list contains the client requested product codes and specifications required for the selected sample(s). Special requirements are communicated to the analyst using the comments section or relayed through verbal instructions provided by the supervisor. The bench analyst assumes full responsibility for performing the analysis according to the specifications printed on the work sheet.

14.7 ***Verification***. Prior to the release of data to the client, laboratory section managers and the report generation staff review the report and compare the completed product to the client specifications documentation to assure that all requirements have been met. Project managers perform a spot check of projects with unique requirements to assure that the work was executed according to specifications.

15.0 CLIENT COMPLAINT RESOLUTION AND DATA INQUIRY PROCEDURE

Requirement. The laboratory follows a formal system for managing and reconciling client complaints and data inquiries. The system includes procedures for documenting client complaints/data inquiries and communicating the complaint or inquiry to the appropriate department for resolution. The system also includes a quality assurance evaluation to determine if the complaint is related to systematic defects requiring process changes.

15.1 ***Procedure***. Client complaints or inquiries are communicated to the Laboratory Director, Laboratory Manager, client services representative, quality assurance staff, or senior management staff for resolution. QA retains the responsibility for documentation and communicating the nature of the complaint to the responsible department(s) for resolution. QA uses the Data Inquiry application for documentation and resolution tracking. The responsible party addresses the complaint. The resolution is communicated to quality assurance (QA) or documented in the application, and the originator for communication to the client. QA reviews the complaint/inquiry and resolution to determine if systematic defects exist. If systematic defects are present, QA initiates a corrective action for the responsible party who develops and implements a response that eliminates the defect.

15.2 ***Documentation***. Client's complaints/inquiries are documented by the individual receiving the complaint using a telephone conversation log or by receipt of an email or letter. The complaint is distributed to all appropriate parties, and always the Lab Director. Documentation of the complaint is filed with the Quality Assurance Officer (in the application), and/or may be filed with the project (as applicable), Lab Director, or project manager.

15.3 ***Corrective Action***. Responses to complaints are required from the responsible party or QA. At a minimum, the response addresses the query and provides an explanation to the complaint. Formal corrective action may focus on the single issue expressed in the complaint. Corrective action may include reprocessing of data, editing of the initial report, and re-issue to the client. If the QA review indicates a systematic error, process modification is required. The defective process at the root of the complaint is changed. SOPs are either created or modified to reflect the change. The party responsible for the process implements process changes.

QA Monitoring. Process changes, implemented to resolve systematic defects, are monitored for effectiveness by QA. If monitoring indicates that the process change has not resolved the defect, QA works with the department management to develop and implement an effective process. If monitoring indicates that the defect has been resolved, monitoring is slowly discontinued and the corrective action is closed. Continued monitoring is incorporated as an element of the annual system audit.

16.0 CONTROL OF NONCONFORMING PRODUCT

Requirement: Policies and procedures have been developed and implemented that describe the procedures employed by the laboratory when any aspect of sample analysis or data reporting do not conform to established procedures or client specifications. These procedures include steps to ensure that process defects are corrected and affected work is evaluated to assess its impact to the client.

Procedure. Nonconforming product is identified through routine internal review and audit practices or through client inquiry. The individuals who identify the nonconformance or receiving a nonconformance inquiry immediately inform the Laboratory Director and the Quality Assurance Officer. The Laboratory Director initiates an evaluation of the nonconformance through the Quality Assurance Department and takes full responsibility for managing the process and identifying the course of action to take, initiating corrective action and mitigating the impact of the nonconformance to the client.

16.1 ***Corrective Action.*** The outcome of the evaluation dictates the course of action. This includes client notification when the quality of data reported has been impacted and may also include corrective action if applicable. Immediate corrective action is performed using the procedures specified in Accutest SOP MQA234. However, additional action may be required including cessation of analysis and withholding and or recalling data reports. If the evaluation indicates that nonconforming data may have been issued to clients, the client is immediately notified and data may be recalled. If work has been stopped because of a nonconformance, the Laboratory Director is the only individual

authorized to direct a resumption of analysis.

Nonconformance caused by systematic process defects require retraining of the personnel involved as an element of the corrective action solution.

17.0 CONFIDENTIALITY PROTECTION PROCEDURES

Requirements: Policies and procedures have been developed to protect client data from release to unauthorized parties or accidental release of database information through accidental electronic transmission or illegal intrusion. These policies have been communicated to clients and staff. Electronic systems are regularly evaluated for effectiveness.

17.1 ***Client Anonymity***. Information related to the Company's clients is granted to employees on a "need to know" basis. An individual's position within the organization defines his "need to know". Individuals with "need to know" status are given password access to systems that contain client identity information and access to documents and document storage areas containing client reports and information. Access to client information by individuals outside of the Company is limited to the client and individuals authorized by the client.

Individuals outside of the Company may obtain client information through subpoena issued by a court of valid jurisdiction. Clients are informed when subpoenas are received ordering the release of their information.

Client information may be released directly to regulatory agencies without receiving client authorization under specified circumstances. These circumstances require that the regulatory agency have statutory authority under the regulations for laboratory certification and that Accutest's operations fall under the purview of the regulation. In these situations, Accutest will inform the client of the regulatory agencies request for information pertaining to his data and proceed with the delivery of the information to the regulatory agency.

17.2 ***Documents***. Access to client documents is restricted to employees in need to know positions. Copies of all client reports are stored in secure electronic archives with restricted access. Reports and report copies are distributed to individuals who have been authorized by the client to receive them. Data reports or data are not released to third parties without verbally expressed or written permission from the client.

17.3 ***Electronic Data***.

Database Intrusion. Direct database entry is authorized for employees of Accutest only on a need to know basis. Entry to the database is restricted through a user specific multiple password entry system. Direct access to the database outside of the facility is possible through a dial-up connection. A unique password is required for access to the local area network. A second unique password is required to gain access to the database. The staff receives read or write level authorization on a hierarchical privilege basis.

Internet Access. Access to client information is through an HTTP Web application only.

It does not contain a mechanism that allows direct access to the database. Clients can gain access to their data only using a series of Accutest assigned client and user specific passwords. The viewable data, which is encrypted during transmission, consists of an extraction of database information only.

Client Accessibility. Accessibility to client data delivered via electronic means follows strict protocols to insure confidentiality. Clients accessing electronic data are assigned a company account. The account profile, which is established by the MIS staff, grants explicit access to specific information pertaining to the client's project activity. Passwords are assigned on an individual basis within a client account. These accounts can be activated or deactivated by the MIS staff only.

17.4 ***Information Requests***.

Client specific data or information is not released to third parties without verbally expressed or written permission from the client. Written permission is required from third parties, who contact the Company directly for the release of information. Verbal requests will be honored only if they are received directly from the client. These requests must be documented in a record of communication maintained by the authorized recipient.

17.5 ***Transfer of Records***. Archived data, which has previously been reported and transmitted to clients, is the exclusive property of Accutest Laboratories. In the event of a cessation of business activities due to business failure or sale, The Company's legal staff will be directed to arrange for the final disposition of archived data.

The final disposition of archived data will be accomplished using the approach detailed in the following sequence:

1. All data will be transferred to the new owners for the duration of the required archive period as a condition of sale.

2. If the new owners will not accept the data or the business has failed, letters will be sent to clients listed on the most recent active account roster offering them the option to obtain specific reports (identified by Accutest Job Number) at their own expense.

3. A letter will be sent to the NELAC accrediting authority with organizational jurisdiction over the company offering them the option to obtain all unclaimed reports at their own expense.

4. All remaining archived data will be recycled using the most expedient means possible.

18.0 QUALITY AUDITS AND SYSTEM REVIEWS

Requirement: The quality assurance group conducts regularly scheduled audits of the laboratory to assess compliance with quality system requirements, technical requirements of applied methodology, and adherence to documentation procedures. The information gathered during these audits is used to provide feedback to senior management and perform corrective action where needed for quality improvement purposes.

18.1 ***Quality System Reviews***. Quality system reviews are performed annually by the Quality Assurance Officer and presented to the Laboratory Director and Quality Assurance Director. In this review, the laboratory is evaluated for compliance with the laboratory Quality Systems Manual (QSM) and the quality system standards of the National Environmental Laboratory Accreditation Conference. Findings, which indicate non-compliance or deviation from the QSM, are flagged for corrective action. Corrective actions require either a return to compliance or a plan change to reflect an improved quality process. The Quality Assurance Officer is responsible for making and documenting changes to the QSM. These changes are reviewed by the Corporate Quality Assurance and Laboratory Director prior to the approval of the revised system. The Quality Systems Manual is reviewed (for accuracy and adequacy) and updated (as necessary) by the Quality Assurance staff on at least an annual basis. This review is documented by the dated signatures of the Laboratory Director, Laboratory Manager, Quality Assurance Officer, and Technical Directors. Minor revisions may be accomplished by attaching an addendum in lieu of creating a new version of the document. Major revisions should result in a new version of the document.

18.2 ***Quality System Audits.*** Quality system audits are conducted to evaluate the effectiveness and laboratory compliance with individual quality system elements. These audits are conducted on an established schedule. Audit findings are documented and communicated to the management staff and entered into the corrective action system for resolution. If necessary, retraining is conducted to assure complete understanding of the system requirements.

18.3 ***Test Method Assessments.*** Test Method Assessments are performed throughout the year following an established schedule. Selected analytical procedures are evaluated for compliance with standard operating procedures (SOPs) and method requirements. If non-conformances exist, the published method serves as the standard for compliance. SOPs are edited for compliance if the document does not reflect method requirements. Analysts are trained to the new requirements and the process is monitored by quality assurance. Analysts are retrained in method procedures if an evaluation of bench practices indicates non-compliance with SOP requirements.

18.4 ***Documentation Audits***. Documentation audits are conducted as needed. This audit includes a check of measurement processes that require manual documentation. It also includes checks of data archiving systems and a search to find and remove any inactive versions of SOPs that may still be present in the laboratory and being accessed by the analysts. Non-conformances are corrected on the spot. Procedural modifications are implemented if the evaluation indicates a systematic defect.

18.5 ***Corrective Action Monitoring***. Defects or non-conformances that are identified during client or internal audits are documented in the corrective action systems and corrected through process modifications and/or retraining. Once a corrective action has been designed and implemented, it is monitored for compliance on a regular basis by the QA staff. Spot corrections are performed if the staff is not following the new procedure. Monitoring of the corrective action continues until satisfactory implementation has been verified.

18.6 ***Preventive Action.*** Laboratory systems or processes, which may be faulty and pose the potential for nonconformances, errors, confusing reports or difficulties establishing traceability may be identified during internal audits. These items are highlighted for systematic change using the corrective action system and managed to resolution using the procedures for corrective action identified in the SOP MQA234.

18.7 ***Client Notification*****.** Defective processes, systematic errors, and quality defects, detected during routine audits may have negative impacts on data quality. In some cases, data that has been released to clients may be affected. If defective data has been released for use, Accutest will notify the affected clients of the defect and provide specific details regarding the magnitude of the impact to their data.

18.8 ***Management Review.***

An annual review of the Quality System is performed by the Quality Assurance Officer. A report detailing the general condition of the Quality Systems, findings, and suggestions for improvement is generated from this review and submitted to the Laboratory Director and Corporate Quality Assurance Director. QA and management ensure that any actions determined from the review are recorded and are carried out within an appropriate and agreed upon time frame. Documentation of resolution of any action items should be maintained as appropriate.

The annual Quality System Review includes but may not be limited to the following areas:

1. Suitability of policies and procedures
2. Reports from managerial and supervisory personnel
3. The outcome of recent internal audits
4. Corrective and Preventative Actions
5. Assessments by external auditors (government and private entities)
6. The results of PT studies
7. Changes in volume/type of work
8. Client feedback and complaints
9. General QA activities
10. Resources and training
11. Goals, objectives, and action plans for the upcoming year; Continuous Improvement

Managerial meetings are held on a weekly or monthly basis. The minutes for these meetings are kept and maintained by QA. Topics of discussion for these meetings include but are not limited to:

1. General lab and Department status and current workload
2. New projects and current in-house projects
3. Personnel changes
4. Troubleshooting
5. PT studies
6. Audit findings and scheduled audits
7. Client complaints, inquiries, and feedback
8. Improvements

18.9 Continuous Improvement

Accutest takes regular measures throughout the year in order to continually improve laboratory operations. These measures include but are not limited to procurement of new laboratory equipment, addition of staff, cross-training of current staff, addition of new training programs, development of new analytical methods, addition of new accreditations/certifications, addition or renovation of lab space, development of new services or products, and implementation of new automated procedures or other software improvements.

Major improvements will be managed and tracked for resolution within the Management of Change (MOC) program.

Any improvements during the year will be included in the annual managerial review, and regular managerial staff meetings. The management review will include a list of items that improve the quality of the laboratory operations. Additionally, employees will be informed of any major improvements via memo or department meetings.

19.0 HEALTH AND SAFETY

Requirement. The company operates a formal health and safety program that complies with the requirements of the Occupational Health and Safety Administration. The program consists of key policies and practices that are essential to safe laboratory operation. All employees are required to receive training on the program elements. Job specific training is conducted to assure safe practices for specific tasks. All employees are required to participate in the program, receive initial and annual training, and comply with the program requirements. All plan and program requirements are detailed in the Health and Safety Program Manual.

19.1 Policy. Accutest Laboratories will provide a safe and healthy working environment for its employees and clients while protecting the public and preserving the Company's assets and property. The company will comply with all applicable government regulations pertaining to safety and health in the laboratory and the workplace.

The objective of the Accutest Health and Safety Program is to promote safe work practices that minimize the occurrence of injuries and illness to the staff through proper health and safety training, correct laboratory technique application and the use of engineering controls.

19.2 Responsibilities. The Health and Safety Program assists managers, supervisors and non-

supervisory employees in control of hazards and risks to minimize the potential for employee and client injuries, damage to client's property and damage or destruction to Accutest's facility.

The Health, Safety and Facilities Manager is responsible for implementing the Program's elements and updating its contents as necessary. He also conducts periodic audits to monitor compliance and assess the program's effectiveness. The Health, Safety and Facilities Manager is also responsible for creating and administering safety training for all new and existing employees.

The employee is responsible for following all safety rules established for their protection, the protection of others and the proper use of protective devices provided by the Company. The employee is also expected to comply with the requirements of the program at all times. Department Managers and Supervisors are responsible for ensuring the requirements of the Safety Program are practiced daily. The Company President retains the ultimate responsibility for the program design and implementation.

19.3 **Program Elements.** The Accutest Health and Safety Program include key program elements that compliment the company's health and safety objective. These elements form the essence of the health and safety policy and assure that the objectives of the program are achieved.

Safety Education and Training and Communication. Training is conducted to increase the staff's awareness of laboratory hazards and their knowledge of the safety practices and procedures required to protect them from those hazards. It is also used to communicate general safety procedures required for safe operation in a chemical laboratory.

Initial health and safety training for new employees is conducted during orientation. The training focuses on the Accutest Safety and Health Program and includes specific training for the hazards that may be associated with the employee's duties. Training is also conducted for all program elements focusing on general, acceptable, laboratory safety procedures. Targeted training is conducted to address hazards or safety procedures that are specific to individual employee's work assignments. All training activities are documented and archived in individual training folders. A health and safety training inventory is maintained in the training database.

Safety Committee. The safety committee provides the employee with an opportunity to express their views and concerns on safety issues in a forum where those concerns will be addressed. This committee meets quarterly to assure that the interests of the company and the well being of the employee are protected. They also serve as a catalyst for elevating the level of safety awareness among their peers.

Hazard Identification and Communication. The hazard communication program enables employees to readily identify laboratory hazards and the procedures to protect themselves from those hazards. This program complies with OSHA's Hazard Communication Standard, Title 29 Code of Federal Regulations 1910.1200 that requires the company to adopt and adhere to the following key elements:

- Material Safety Data Sheets (MSDS) must be available to any employee wishing to view them,

- The Company must maintain a Hazardous Chemicals Inventory (by location), which is updated on an annual basis,
- Containers are properly labeled,
- All employees must be provided with annual Hazard Communication and Right to Know training,

Identification of Workplace Hazards. The workplace hazard identification procedures have been designed to assure that hazards that have the potential to cause personnel injury or destruction of property are identified, managed and/or systematically eliminated from the operation. This system eliminates hazards, limits the potential for injury and increases the overall safety of the work environment.

Employee Exposure Assessment. Employee exposure assessment is performed to identify and evaluate potential exposure hazards associated with the employees work station. The exposure assessment data is used to determine if changes or modifications to the work station are needed to limit exposure to laboratory conditions that could negatively affect an employee's existing medical conditions.

Bloodborne Pathogens. Accutest has implemented the OSHA Bloodborne Pathogen Standard, 29CFR1910.1030 to reduce occupational exposure to Hepatitis B Virus (HBV), Human Immunodeficiency Virus (HIV) and other bloodborne pathogens that employees may encounter in their workplace.

Respiratory Protection Plan. The respiratory protection plan assures that Accutest employees are protected from exposure to respiratory hazards. This program is used in situations where engineering controls and/or safe work practices do not completely control the identified hazards. In these situations, respirators and other protective equipment are used. Supplemental respiratory protection procedures are applied to specified maintenance personnel, employees who handle hazardous wastes in the hazardous waste storage area, and any employee that voluntarily elects to wear a respirator.

Chemical Hygiene Plan. The Chemical Hygiene Plan complies with the requirements of the Occupational Safety and Health Administration's Occupational Exposure to Hazardous Chemicals in the Laboratory Standard, 29 CFR 1910.1450. This plan establishes procedures, identifies safety equipment, personal protective equipment, and work practices that protect employees from the potential health hazards presented by hazardous chemicals in the laboratory if properly used and/or applied.

Chemical Spill Response Plan. The chemical spill response plan has been designed to minimize the risks from a chemical spill or accidental chemical release in the laboratory. Risk minimization is accomplished through a planned response that follows a defined procedure. The staff has been trained to execute spill response procedures according to the specifications of the plan, which identifies the appropriate action to be taken based on the size of the spill.

Emergency Action & Evacuation Plan. The Emergency Action and Evacuation Plan

details the procedures used to protect and safeguard Accutest's employees and property during emergencies. Emergencies are defined as fires or explosions, gas leaks, building collapse, hazardous material spills, emergencies that immediately threaten life and health, bomb threats and natural disasters such as floods, hurricanes or tornadoes. The plan identifies and assigns responsibility for executing specific roles in situations requiring emergency action.

Lockout/Tagout Plan. Lockout/tagout procedures have been established to assure that laboratory employees and outside contractors take steps to render equipment inoperable and/or safe before conducting maintenance activities. The plan details the procedures for conducting maintenance on equipment that has the potential to unexpectedly energize, start up, or release energy or can be operated unexpectedly or accidentally resulting in serious injury to employees. The plan ensures that employees performing maintenance render the equipment safe through lock out or tag out procedures.

Personal Protection Policy. Policies have been implemented which detail the personal protection requirements for employees. The policy includes specifications regarding engineering controls, personal protective equipment (PPE), hazardous waste, chemical exposures, working with chemicals and safe work practices. Safety requirements specific to processes or equipment are reviewed with the department supervisor or the Health and Safety Manager before beginning operations.

Emergency Preparedness Plan. This plan identifies the actions to be taken by Accutest Laboratory's staff in the event of terrorism or terroristic actions, to ensure the safety of the employees and the facility. The plan describes the building security actions coinciding with the "Alert Condition", designated by the Department of Homeland Security.

Visitor and Contractor Safety Program. A safety brochure is given to all visitors and contractors who visit or conduct business at the facility. The brochure is designed to inform anyone who is not an employee of Accutest Laboratories of the laboratories safety procedures. The brochure directs them to follow all safety programs and plans while on Accutest property. This program also outlines procedures for visitors and contractors in the event of an emergency. Visitors are required to acknowledge receipt and understanding of the Accutest policy annually.

Appendix I

Glossary of Terms

GLOSSARY OF TERMS

Acceptance Criteria: specified limits placed on characteristics of an item, process, or service defined in requirement documents.

Accuracy: the degree of agreement between an observed value and an accepted reference value. Accuracy includes a combination of random error (precision) and systematic error (bias) components which are due to sampling and analytical operations; a data quality indicator.

Analyst: the designated individual who performs the "hands-on" analytical methods and associated techniques and who is the one responsible for applying required laboratory practices and other pertinent quality controls to meet the required level of quality.

Audit: a systematic evaluation to determine the conformance to quantitative *and qualitative* specifications of some operational function or activity.

Batch: environmental samples that are prepared and/or analyzed together with the same process and personnel, using the same lot(s) of reagents. A preparation batch is composed of one to 20 environmental samples of the same NELAC-defined matrix, meeting the above mentioned criteria and with a maximum time between the start of processing of the first and last sample in the batch to be 24 hours. An analytical batch is composed of prepared environmental samples (extracts, digestates or concentrates) which are analyzed together as a group.

Blank: a sample that has not been exposed to the analyzed sample stream in order to monitor contamination during sampling, transport, storage or analysis. The blank is subjected to the usual analytical and measurement process to establish a zero baseline or background value and is sometimes used to adjust or correct routine analytical results.

Blind Sample: a sub-sample for analysis with a composition known to the submitter. The Analyst/Laboratory may know the identity of the sample but not its composition. It is used to test the analyst's or laboratory's proficiency in the execution of the measurement process.

Calibration: to determine, by measurement or comparison with a standard, the correct value of each scale reading on a meter, instrument, or other device. The levels of the applied calibration standard should bracket the range of planned or expected sample measurements.

Calibration Curve: the graphical relationship between the known values, such as concentrations, of a series of calibration standards and their instrument response.

Calibration Method: a defined technical procedure for performing a calibration.

Calibration Standard: a substance or reference material used to calibrate an instrument.

Certified Reference Material (CRM): a reference material one or more of whose property values are certified by a technically valid procedure, accompanied by or traceable to a certificate or other documentation, which is issued by a certifying body.

Chain of Custody: an unbroken trail of accountability that ensures the physical security of samples and includes the signatures of all who handle the samples.

Confirmation: verification of the identity of a component through the use of an approach with a

different scientific principle from the original method. These may include, but are not limited to second column confirmation, alternate wavelength, derivatization, mass spectral, interpretation, alternative detectors or, additional cleanup procedures.

Continuous Improvement: Any procedure, capitol improvement, expansion, or addition that improves the quality of operations of the laboratory.

Corrective Action: the action taken to eliminate the causes of an existing nonconformity, defect or other undesirable situation in order to prevent recurrence.

Data Reduction: the process of transforming raw data by arithmetic or statistical calculations, standard curves, concentration factors, etc., and collation into a more useable form.

Demonstration of Capability: a procedure to establish the ability of the analyst to generate acceptable accuracy.

Document Control: the act of ensuring that documents (and revisions thereto) are proposed, reviewed for accuracy, approved for release by authorized personnel, distributed properly and controlled to ensure use of the correct version at the location where the prescribed activity is performed.

Duplicate Analyses: the analyses or measurements of the variable of interest performed identically on two sub-samples of the same sample. The results from duplicate analyses are used to evaluate analytical or measurement precision but not the precision of sampling, preservation or storage internal to the laboratory.

Field of Testing: NELAC's approach to accrediting laboratories by program, method and analyte. Laboratories requesting accreditation for a program-method-analyte combination or for an up-dated/improved method are required to submit to only that portion of the accreditation process not previously addressed (see NELAC, section 1.9ff).

Laboratory Control Sample (such as laboratory fortified blank, spiked blank, or QC check sample): a sample matrix, free from the analytes of interest, spiked with verified known amounts of analytes from a source independent of the calibration standards or a material containing known and verified amounts of analytes. It is generally used to establish intra-laboratory or analyst specific precision and bias or to assess the performance of all or a portion of the measurement system.

Matrix: the component or substrate that contains the analyte of interest. For purposes of batch and QC requirement determinations, the following matrix distinctions shall be used:

Aqueous: any aqueous sample excluded from the definition of Drinking Water matrix or Saline/Estuarine source. Includes surface water, groundwater, effluents, and TCLP or other extracts.

Drinking Water: any aqueous sample that has been designated a potable or potential potable water source.

Saline/Estuarine: any aqueous sample from an ocean or estuary, or other salt-water source such as the Great Salt Lake.

Non-aqueous Liquid: any organic liquid with <15% settleable solids.

Solids: includes soils, sediments, sludges and other matrices with >15% settleable solids.

Chemical Waste: a product or by-product of an industrial process that results in a matrix not previously defined.

Air: whole gas or vapor samples including those contained in flexible or rigid wall containers and the extracted concentrated analytes of interest from a gas or vapor that are collected with a sorbant tube, impinger solution, filter, or other device.

Biota: animal or plant tissue, consisting of entire organisms, homogenates, and/or organ or structure specific subsamples.

Matrix Spike (spiked sample or fortified sample): a sample prepared by adding a known mass of target analyte to a specified amount of matrix sample for which an independent estimate of target analyte concentration is available. Matrix spikes are used, for example, to determine the effect of the matrix on a method's recovery efficiency.

Matrix Spike Duplicate (spiked sample or fortified sample duplicate): a second replicate matrix spike prepared in the laboratory and analyzed to obtain a measure of the precision of the recovery for each analyte.

Method Blank: a sample of a matrix similar to the batch of associated samples (when available) that is free from the analytes of interest, which is processed simultaneously with and under the same conditions as samples through all steps of the analytical procedures, and in which no target analytes or interferences are present at concentrations that impact the analytical results for sample analyses.

Method Detection Limit: the minimum concentration of a substance (an analyte) that can be measured and reported with 99% confidence that the analyte concentration is greater than zero and is determined from analysis of a sample in a given matrix containing the analyte.

National Environmental Laboratory Accreditation Conference (NELAC): a voluntary organization of State and Federal environmental officials and interest groups purposed primarily to establish mutually acceptable standards for accrediting environmental laboratories. A subset of NELAP.

National Environmental Laboratory Accreditation Program (NELAP): the overall National Environmental Laboratory Accreditation Program of which NELAC is a part.

NELAC Standards: the plan of procedures for consistently evaluating and documenting the ability of laboratories performing environmental measurements to meet nationally defined standards established by the National Environmental Laboratory Accreditation Conference.

Performance Audit: the routine comparison of independently obtained *qualitative and* quantitative measurement system data with routinely obtained data in order to evaluate the proficiency of an analyst or laboratory.

Precision: the degree to which a set of observations or measurements of the same property, obtained under similar conditions, conform to themselves; a data quality indicator. Precision is

usually expressed as standard deviation, variance or range, in either absolute or relative terms.

Preservation: refrigeration and/or reagents added at the time of sample collection (or later) to maintain the chemical and/or biological integrity of the sample.

Proficiency Testing: a means of evaluating a laboratory's performance under controlled conditions relative to a given set of criteria through analysis of unknown samples provided by an external source.

Proficiency Test Sample (PT): a sample, the composition of which is unknown to the analyst and is provided to test whether the analyst/laboratory can produce analytical results within specified acceptance criteria.

Quality Assurance: an integrated system of activities involving planning, quality control, quality assessment, reporting and quality improvement to ensure that a product or service meets defined standards of quality with a stated level of confidence.

Quality Control: the overall system of technical activities whose purpose is to measure and control the quality of a product or service so that it meets the needs of users.

Quality Manual: a document stating the management policies, objectives, principles, organizational structure and authority, responsibilities, accountability, and implementation of an agency, organization, or laboratory, to ensure the quality of its product and the utility of its product to its users.

Quality System: a structured and documented management system describing the policies, objectives, principles, organizational authority, responsibilities, accountability, and implementation plan of an organization for ensuring quality in its work processes, products (items), and services. The quality system provides the framework for planning, implementing, and assessing work performed by the organization and for carrying out required QA and QC.

Reporting Limits: the maximum or minimum levels, concentrations, or quantities of a target variable (e.g., target analyte) that can be quantified with the confidence level required by the data user.

Reagent Blank (method reagent blank or method blank): a sample consisting of reagent(s), without the target analyte or sample matrix, introduced into the analytical procedure at the appropriate point and carried through all subsequent steps to determine the contribution of the reagents and of the involved analytical steps.

Reference Material: a material or substance one or more properties of which are sufficiently well established to be used for the calibration of an apparatus, the assessment of a measurement method, or for assigning values to materials.

Reference Method: a method of known and documented accuracy and precision issued by an organization recognized as competent to do so.

Reference Standard: a standard, generally of the highest metrological quality available at a given location, from which measurements made at that location are derived.

Replicate Analyses: the measurements of the variable of interest performed identically on two or

more sub-samples of the same sample within a short time interval.

Sample Duplicate: two samples taken from and representative of the same population and carried through all steps of the sampling and analytical procedures in an identical manner. Duplicate samples are used to assess variance of the total method including sampling and analysis.

Spike: a known mass of target analyte added to a blank sample or sub-sample; used to determine recovery efficiency or for other quality control purposes.

Standard: the document describing the elements of laboratory accreditation that has been developed and established within the consensus principles of NELAC and meets the approval requirements of NELAC procedures and policies.

Traceability: the property of a result of a measurement whereby it can be related to appropriate standards, generally international or national standards, through an unbroken chain of comparisons.

Validation: the process of substantiating specified performance criteria.

Work Cell: A defined group of analysts that together perform the method analysis. Members of the group and their specific functions within the work cell must be fully documented. A "work cell" is considered to be all those individuals who see a sample through the complete process of preparation, extraction, or analysis. The entire process is completed by a group of capable individuals. Each member of the work cell demonstrates capability for each individual step in the method sequence.

Appendix II

Analytical Capabilities

Method Type	Method Number	Regulatory Program
Organics – GC/MS:		
Volatile Organics	EPA 524.2	Clean Water Act
Volatile Organics	EPA 624	Clean Water Act
Semi-Volatile Organics	EPA 625	Clean Water Act
Volatile Organics	SW846 – 8260B + C	RCRA
Semi-Volatile Organics	SW846 – 8270C + D	RCRA
Volatile Organics	MA DEP APH	MCP
Volatile Organics	EPA TO-15	Clean Air Act
Organics- HPLC:		
Formaldehyde	SW846 – 8315A	RCRA
NitroAromatics & Amines (Explosives)	SW846 –8330 + A	RCRA
Polynuclear Aromatics (PAH)	SW846- 8310	RCRA
Organics – GC:		
EDB and DBCP – DW	EPA 504.1/8011	Safe Drinking Water Act
Chlorinated Acid Herbs–DW	EPA 515.1	Safe Drinking Water Act
Purgeable Aromatics	EPA 602	Clean Water Act
Chlorinated Pesticides & PCBs	EPA 608	Clean Water Act
Volatile Aromatic/Halocarbons	SW-846 – 8021B	RCRA
Organochlorine Pesticides	SW-846 – 8081A + B	RCRA
Polychlorinated Biphenyls	SW-846 – 8082 + A	RCRA
Volatile Petro. Hydrocarbons	MA DEP	MCP
Extract. Petro. Hydrocarbons	MA DEP	MCP
Connecticut ETPH	CT DEP	None – State Specific
Diesel Range Organics (DRO)	8015B + C	RCRA
Gasoline Range Organics (GRO)	8015B + C	RCRA
Maine GRO	ME DHS	None – State Specific
Maine DRO	ME DHS	None-State Specific
CT ETPH	CT DPH	None-State Specific
Methane	8015B	RCRA
Chlorinated Acid Herbicides	SW-846 – 8151A	RCRA
RSK-175 (methane, ethane, ethene, CO2)	RSK-175	Clean Water Act
Wisconsin GRO	Wisconsin GRO	None – State Specific
Wisconsin DRO	Wisconsin DRO	None – State Specific
Washington EPH	Washington EPH	None – State Specific
Washington VPH	Washington VPH	None – State Specific
METALS:		
Total Recov. Metals Digestion	EPA 200.7	Clean Water Act

ICP: General – EPA WW	EPA 200.7, May 1994	Clean Water Act
Method Type	Method Number	Regulatory Program
ICP: General – EPA DW	EPA 200.7, May 1994	Safe Drinking Water Act
Cold Vapor Mercury - DW	EPA 245.1	Safe Drinking Water Act
Cold Vapor Mercury	EPA 245.1	Clean Water Act
Non-Pot. Water Digest: ICP	SW846 3010A	RCRA
Digestion of Soils for ICP	SW846 3050B	RCRA
ICP General – SW846	SW846 6010C	RCRA
Cold Vapor Mercury - Soils / Liquids	SW846-7471A/7470A	RCRA
Wet Chemistry:		
Specific Gravity	ASTM 1429	ASTM Standard
Oxidation-Reduction Potential	ASTM D1498-76	ASTM Standard
Tetraethyl Lead in Soil & Water	ASTM D3341-87 M-Solids.	ASTM Standard
Neutral Leaching	ASTM E3987	ASTM Standard
Specific Conductance	EPA 120.1/SM2510B	Clean Water Act
Color, Apparent	EPA 110.2/SM2120B	Clean Water Act
Hardness	EPA 130.2	Clean Water Act
Odor	EPA 140.1/SM2150B	Clean Water Act
PH by electrode (Waters)	EPA 150.1/SM21 4500H+B	Clean Water Act
Total Dissolved Solids	EPA 160.1/SM18 2540C	Clean Water Act
Total Suspended Solids	EPA 160.2/SM2540D	Clean Water Act
Volatile Suspended Solids	EPA 160.2/160.4	Clean Water Act
Total Solids	EPA 160.3/SM21 2540B	Clean Water Act
Total Solids	EPA 160.3 M-Solids./SM21 2540B M	None – Solids Modification
Percent Solids	EPA 160.3M-Solids	Clean Water Act
Total Volatile Solids	EPA 160.4	Clean Water Act
Total Volatile Solids	EPA 160.4 M-Solids.	None – Solids Modification
Settleable Solids	EPA 160.5/SM2540F	Clean Water Act
Turbidity	EPA 180.1	Clean Water Act
Acidity	EPA 305.1/SM21 2310B	Clean Water Act
Alkalinity	EPA 310.1/SM18 2320B	Clean Water Act
Chloride – Titrametric	EPA 325./SM21 4500 CL-C + D	Clean Water Act
Chloride – Titrametric	EPA 325.3/SW9252A M-Solids	None – Solids Modification
Total Residual Chlorine	EPA 330.4/SM18 4500CLF	Clean Water Act
CN[amen] Amenable to Chlorination	EPA 335.1/2, SW846 9010	CWA or RCRA
Cyanide	EPA 335.2/EPA 335.4 (AQ) /EPA 335.4 (DW)	Clean Water Act/Safe Drinking Water Act
Fluoride	EPA 340.2/SM4500 F-C	Clean Water Act
Fluoride	EPA 340.2 M-Solids.	None – Solids Modification

Method Type	Method Number	Regulatory Program
Ammonia	EPA 350.2/EPA 350.1/SM21 4500 NH3 B+C	Clean Water Act
Ammonia	EPA 350.2M-Solids/ SM21 4500 NH3 B+C	None – Solids Modification
Total Kjeldahl Nitrogen	EPA 351.2	Clean Water Act
Total Kjeldahl Nitrogen	EPA 351.2 M-Solids.	None – Solids Modification
Nitrate/Nitrite (Lachat)	EPA 353.2	Clean Water Act
Nitrate/Nitrite (Lachat)	EPA 353.2 M-Solids.	None – Solids Modification
Nitrogen, Nitrite	EPA 354.1/EPA 353.2\SM18 4500NO2B	Clean Water Act
Nitrogen, Nitrite	EPA 354.1/EPA 353.2/SM18 4500NO2B M	None – Solids Modification
Dissolved Oxygen	EPA 360.1	Clean Water Act
Orthophosphate	EPA 365.3/SM19 4500PE	Clean Water Act
Orthophosphate	EPA 365.3/SM19 4500PE M	None – Solids Modification
Total Phosphates	EPA 365.3/EPA 365.4	Clean water Act
Total Phosphates	EPA 365.3/EPA 365.4 M-Solids.	None – Solids Modification
Dissolved Silica	EPA 370.1/SM21 4500 SiO2C	Clean Water Act
Sulfate (Gravimetric)	EPA 375.3/SM 4500 SO4 C+D	Clean Water Act
Sulfate (Gravimetric)	EPA 375.3 M-Solids.	None – Solids Modification
Sulfate (Turbidimetric)	EPA 375.4/ASTM D516-90-02	Clean Water Act
Sulfate (Turbidimetric)	EPA 375.4M	None – Solids Modification
Sulfide	EPA 376.1/SM21 4500 S2-F + E	Clean Water Act
Sulfide	EPA 376.1 M-Solids.	None – Solids Modification
Sulfite	EPA 377.1/SM 4500 SO3 B	Clean Water Act
Temperature	SM2550B	Clean Water Act
BOD	EPA 405.1/SM5210B	Clean Water Act
Chemical Oxygen Demand	SM 18 5220C	Clean Water Act
Chemical Oxygen Demand	SM 18 5220C M-Solids	None – Solids Modification
Oil & Grease, Gravimetric – AQ	EPA 413.1/EPA 1664A	Clean Water Act
Inorganic Carbon	EPA 415.1 M-Solids	None – Solids Modification
Total Organic Carbon/DOC –Soils	SW846 9060	RCRA
Total Organic Carbon/DOC –Soils	Lloyd Khan	RCRA
Total Organic Carbon/DOC – AQ	EPA 415.1/SM21 5310B C D	Clean Water Act
Petroleum Hydrocarbons – AQ	EPA 418.1/EPA 1664A/SW846 9070A	Clean Water Act
Petroleum Hydrocarbons (Soils)	EPA 418.1M-Solids/SW846 9071B	None – Solids Modification
Phenols	EPA 420.1 + 4	Clean Water Act
Phenols - chloroform extraction	EPA 420.1 + 4	Clean Water Act
Bromide	SM18 4500 Br	None – Standard Method
Bromide	SM18 4500 Br M-Solids	None – Standard Method
Calcium Hardness	EPA 130.1/SM18 2340B or C	None – Standard Method
Salinity	SM18 2520B	None – Standard Method
Ferrous Iron	SM18 3500 FE-B	None – Standard Method
Free CO_2 by Titrametric Method	SM18 4500 CO2 C	None – Standard Method

Method Type	Method Number	Regulatory Program
Bicarbonate, Carbonate, CO_2	SM18 4500 CO2D	None – Standard Method
Chloride in Drinking Waters	SM18 4500Cl-D	None – Standard Method
Total Organic Nitrogen	SM18 4500N	None – Standard Method
Total Nitrogen Total Organic Nitrogen	SM18 4500N SM18 4500N M-Solids	None – Standard Method None – Standard Method
Total Nitrogen	SM18 4500N, M-Solids.	None – Standard Method
Hydrogen Sulfide	SM18 4500S2-F	None – Standard Method
MBAS (Anionic Surfactants as)	SM18 5540C	None – Standard Method
TCLP Leaching Procedure	SW846 1311	RCRA
SPLP Extraction	SW846 1312	RCRA
Hexa - Chromium/soils	SW846 3060/7196A	None- Modification
Hexavalent Chromium (Waters)	SW846 7196A	RCRA
Hexavalent Chromium (Waters)	SM 3500 CR - B	RCRA
Corrosivity & pH – aqueous	SW846 9040B + C	RCRA
Soil and Waste pH, Corrosivity	SW846 9045B, Chpt. 7	RCRA
Phenols - chloroform extraction	SW846 9065/9066	RCRA
Phenols	SW846 9065/9066	RCRA
Oil & Grease, Gravimetric (Soils)	SW846 9071A M-Solids	RCRA
Cation Exchange Capacity	SW846 9081	RCRA
Paint Filter Test	SW846 9095	RCRA
Sulfide and Cyanide Reactivity	SW846 Chapter 7	RCRA
Sulfide Reactivity – Analysis	SW846 Chapter 7, 9034	RCRA
Ignitability	SW846 Chp 7, SW1010A/1020A	RCRA
Silica	SM4500 Si D	None – Standard Method
Chlorine	EPA 325.2	CWA
Cyanide (Total and Amenable)	EPA 9012/9014/9010	RCRA

Appendix III

Laboratory Equipment

Accutest Laboratories Major Instrument List

Instrument	Manufacture & Description	Serial Number	Purchase
GC/MS E	HP5972 MSD/HP5890 GC/OI 4551A AS/OI Eclipse 4660 Conc	3507A02625/ 3336A59916	1997
GC/MS F	HP5973 MSD/HP6890 GC/HP7683 AS	US80210969/ US00021408	1998
GC/MS G	HP5973 MSD/HP6890 GC/OI 4551A AS/OI 4560 Conc.	US91411732/ US00028322	1999
GC/MS H	HP5973 MSD/HP6890 GC/OI 4551A AS/OI 4560 Conc.	US93112038/ US00031336	2000
GC/MS I	Agilent 5973N MSD/Agilent 6890N GC/Agilent 7683B Conc.	US10442139/ US10150048	2002
GC/MS J	Agilent 5973N MSD/Agilent 6890N GC/Entech 7016CA AS/Entech 7100A Pre Conc.	US30945108/ CN10305006	2003
GCMS K	Agilent 5973N MSD/Agilent 6890N GC/OI 4551A AS/OI 4560 Conc.	US41746643/ CN10416096	2004
GCMS L	Agilent 5973N MSD/Agilent 6890 GC/ Tekmar SOLATek 72 AS/Tekmar Velocity XPT Conc.	US43146823/ CN10432009	2004
GC/MS M	Agilent 5973N MSD/Agilent 6890N GC/OI 4552/Tekmar Velocity XPT Conc.	US44647285/ CN10514047	2005
GC/MS N	Agilent 5973 MSD/Agilent 6890N GC/ Tekmar Aquatek 70 AS/Tekmar Velocity XPT Conc.	US44647359/ CN10518015	2005
GC/MS P	Agilent 5975 MSD/Agilent 6890N GC/Tekmar SOLAtek 72 AS/Tekmar Velocity XPT Conc.	US52420296/ CN10526064	2005
GC/MS Q	Agilent 5975 MSD/Agilent 6890N GC/Entech 7016CA AS/ Entech 7100A Pre Conc.	US53921409/ CN10544040	2005
GCMS R	Agilent 5975 MSD/Agilent 6890N GC/ OI 4551A AS /OI Eclipse 4660 Conc.	US71225925/ CN10717006	2007
GCMS S	Agilent 5975C MSD/Agilent 7890A GC/Agilent 7683B Conc.	US80838995/ CN10816024	2008
GCMS T	Agilent 5975C MSD/Agilent 7890A GC/OI 4551A AS /OI Eclipse 4660 Conc.	US80838990 CN10816005	2008
GCMS U	Agilent 5973 MSD/Agilent 6890A GC/Agilent 7683B Conc.	US10440609 US00042179	2011
GCMS V	Agilent 5975C MSD/Agilent 7890A GC/OI 4552/ OI Eclipse 4660 Conc.	US11142810 CN11131009	2011
GCMS W	Agilent 5973N MSD/Agilent 6890N GC/Agilent 7683 Conc.	US21863096 US10150099	2012
GCMS X	Agilent 5975C MSD/Agilent 7890A GC/EST Centurion AS/Dual ENCON Evolution Conc.	US81839986 CN10829086	2012
GC 17	HP5890 GC/ Agilent 7683 Conc. AS/ Dual FID	3225A50073	1998
GC 19	Agilent GC 6890/OI 4551A AS/OI 4560 Conc./PID/FID	US00039860	2001
GC 20	Agilent GC 6890A/Agilent 7683 AS/ Dual ECD	US00041387	2001
GC 21	Agilent GC 6890/OI 4551A AS/OI 4560 Conc./PID/FID	US10136093	2002
GC 22	Agilent GC 6890 FID/TCD	US10204132	2002
GC 23	Agilent GC 6890N/Agilent 7683B AS/ Dual ECD	CN10433089	2004
GC 24	Agilent GC 6890N/Agilent 7683B AS/Dual FID	CN10515075	2005
GC 25	Agilent GC 6890N/OI 4551A AS/ OI 4560 Conc./PID/FID	US10515051	2005
GC 26	Agilent GC 6890N/Agilent 7683B AS/Dual ECD	CN10614019	2006
GC27	Agilent GC 7890A/Agilent 7683B AS/Dual FID	CN10816091	2008
GC28	Agilent GC 7890/ OI 4551A AS/ OI Eclipse 4660 Conc./PID/FID	CN10815135	2008
GC29	Agilent GC 7890A/Agilent 7693 AS/PID/FID	CN10241070	2010
GC30	Agilent GC 7890A/Agilent 7693 AS/PID/FID	CN10461112	2010
GC31	Agilent GC 7890A/Agilent 7693 AS/PID/FID	CN10471014	2010
Sampler	Tedlar Bag Sampler Model 7032A-LB	1010	2003

HPLC 2	Hewlett-Packard 1100 Automated LC System	Multiple	2000
HPLC 3	Agilent 1260 Infinity Automated LC System	Multiple	2011
HPLC 4	Agilent 1200 Automated LC System	Multiple	2011
Balance	Fisher XL-300	9681	1990
	Software		
Data System	Hewlett-Packard/Enviroquant		1997

Metals & Wet Chemistry Instrumentation

Instrument	Manufacture & Description	Serial Number	Purchase
ICP SS1	ICap 6500 Duo Thermo Scientific/CETAC ASX – 520 AS	20070805	2007
ICP SS2	ICap 6500 Duo Thermo Scientific/CETAC ASX – 520 AS	20114308	2011
ICP-MS	ICP-MS Thermo Scientific 7500 CX Series/CETAC ASX – 500 AS	JP51202142	2012
Hg Analyzer – HG1	Leeman Labs, Inc. Hydra AA (Hg)	2006	1990
Hg Analyzer – HG2	Leeman Labs, Inc. Hydra AA (Hg)	2046	2002
Sonifier	Branson 450	B190039	1990
Sonifier	Fisher 550	F1790	1997
Balance	Fisher XI-300	10939	1990
Balance	Fisher A-160 (Analytical Balance)	25819	1990
Auto Analyzer	Lachat QuikChem FLA+ 8000 Series/XYZ Sampler	A83000-2044	2002
Auto Analyzer	Lachat Micro Dist	A2000-875	2007
Spectrophotometer	Milton Roy/Spectronic 20D	3321017021	1990
TOC Analyzer	Shimadzu TOCL CPH/CPN	H54114900158AE	2012

Instrument	Manufacture & Description	Serial Number	Purchase
O_2 Meter	YSI 54 Arc	90C014142	1990
PH Meter	Orion 720A	1545	1990
PH Meter	Orion 520A	1828	1990
Conductivity	Fisher		1998
Seta Flash	Stanhope-Seta 13741-2	G1206	1990
Turbidometer	VWR		1999
Autoclave	NAPCO 8000 DSE	603033111	2003
Incubator 1	Fisher 146	-	-
Incubator 2	Precision 815	601071745	2003

Organic Preparation Equipment

Instrument	Manufacture & Description	Serial Number	Purchase
ASE I	ASE 200 Auto Accelerated Solvent Extractor	099070528	1999
ASE II	ASE 200 Auto Accelerated Solvent Extractor	96100201	1999
ASE III	ASE 200 Auto Accelerated Solvent Extractor	04120615	2004
Zymark I	Zymark Turbovap LV	TV0320N11719503	5/03
Zymark II	Zymark Turbovap	TV9922N8930	1999
Zymark III	Zymark Turbovap	TV9915N8828	2000
Zymark IV	Zymark Turbovap	TV0823N14951	2008
Zymark V	Zymark Turbovap	TV1026N15943	2010
Zymark VI	Zymark Turbovap	TV1032N16029	2010
S-EVAP (3)	S-EVAP Organomation	-	-
N-EVAP (3)	N-EVAP Organomation	-	-

Balance	Ohaus Adventurer	D158218050028	1990
MARS	Microwave Accelerated Reaction System		2010
Autoclave	Napco 800-DSE	603033111	2003
Colony Counter	Mini Light Box	15597	2003
Colony Counter	Leica Darkfield Quebec Colony Counter		2003
Incubator	Fisher Model 146	-	1985
Incubator	Precision 815	601071745	2003
Ultraviolet Lamp	UVP	-	2003
Ultraviolet Lamp	UVP	-	2003
Microscope	Binocular SPI Microscope No. 20934	-	2003
Illuminator	Clay Adams E&G Illuminator No. A-1494	Z6145	2003
Water Bath	Napco 230A	-	1990
Balance	Adventurer Pro	8026391102	2005
Balance	Adventurer Pro	8029361043	2008
Balance	Adventurer Pro AV212	8029361042	2008
Conductivity Met.	Fisher Digital Conductivity Meter	-	1998
De-Ionized Water	US Filter	-	1990

Analytical Software

Manufacturer	Version
Enviroquant	G1701BA Rev. B.01.00
	G1701CA Rev. C.00.00
	G1701DA Rev. D.00.01.27 and D.00.01.38
	G1701AA A.03.00
LC Chemstation	Rev. 06.03 and 07.01
MSD Chemstation	D.02.00.237
	D.02.00.275
	Build 75

Appendix IV

Active SOP List

(Supplied on request)

APPENDIX B-1
EA
Standard Operating Procedures - Field

Standard Operating Procedure No. 001
for
Sample Labels

Prepared by

EA Engineering, Science, and Technology, Inc.
225 Schilling Circle, Suite 400
Hunt Valley, Maryland 21031

Revision 0
August 2007

CONTENTS

1. SCOPE AND APPLICATION

The purpose of this Standard Operating Procedure is to delineate protocols for the use of sample labels. Every sample will have a sample label uniquely identifying the sampling point and analysis parameters. An example label is provided below. Other formats with similar levels of detail are acceptable.

PROJECT NAME _____ PROJECT NUM. _____
SAMPLE LOCATION/SITE ID _____
DATE: ____/____/____ TIME: _____:_____
ANALYTES: METALS VOC EXPLOSIVES ORGANICS OTHER
FILTERED: [NO] [YES]
PRESERVATIVE: [NONE] [HNO_3] [OTHER _____]
SAMPLER: _____

2. MATERIALS

The following materials may be required: sample label and indelible laboratory marker.

3. PROCEDURE

The following sections describe how to use the sample labeling system.

3.1 LABEL INFORMATION

As each sample is collected/selected, fill out a sample label. Enter the following information on each label:

- Project name
- Project number
- Location/site identification—Enter the media type (i.e., well number, surface water, soil, etc.) sampling number, and other pertinent information concerning where the sample was taken
- Date of sample collection

- Time of sample collection
- Analyses to be performed (NOTE: Due to number of analytes, details of analysis should be arranged with laboratory *prior to start of work)*
- Whether filtered or unfiltered (water samples only)
- Preservatives (water samples only)
- Number of containers for the sample (e.g., 1 of 2, 2 of 2).

3.2 ROUTINE CHECK

Double-check the label information to make sure it is correct. Detach the label, remove the backing, and apply the label to the sample container. Cover the label with clear tape, ensuring that the tape completely encircles the container.

3.3 RECORD INFORMATION

Record the sample number and designated sampling point in the field logbook, along with the following sample information:

- Time of sample collection (each logbook page should be dated)
- Location of the sample
- Organic vapor meter or photoionization meter readings for the sample (when appropriate)
- Any unusual or pertinent observations (oily sheen on groundwater sample, incidental odors, soil color, grain size, plasticity, etc.)
- Number of containers required for each sample
- Whether the sample is a quality assurance sample (split, duplicate, or blank).

3.3.1 Logbook Entry

A typical logbook entry might look like this:

- 7:35 a.m. Sample No. MW-3. PID = 35 ppm
- Petroleum odor present. Sample designated MW-3-001.

NOTE: Duplicate samples will be given a unique sample designation rather than the actual sample number with an added prefix or suffix. This will prevent any indication to the laboratory that this is a duplicate sample. This fictitious sample number will be listed in the logbook along with the actual location of the sample.

3.4 SHIPMENT

Place the sample upright in the designated sample cooler. Make sure there is plenty of ice in the cooler at all times.

4. MAINTENANCE

Not applicable.

5. PRECAUTIONS

5.1 INCIDENTAL ODORS

Note that although incidental odors should be noted in the logbook, it is unwise from a safety and health standpoint to routinely "sniff test" samples for contaminants.

5.2 DUPLICATE SAMPLE

No indication of which samples are duplicates is to be provided to the laboratory.

6. REFERENCES

U.S. Environmental Protection Agency. 1980. Interim Guidelines and Specifications for Preparing Quality Assurance Project Plans. QAMS-005/80.

Standard Operating Procedure No. 002
for
Chain-of-Custody Form

Prepared by

EA Engineering, Science, and Technology, Inc.
225 Schilling Circle, Suite 400
Hunt Valley, Maryland 21031

Revision 0
August 2007

CONTENTS

Page

1. SCOPE AND APPLICATION

The purpose of this Standard Operating Procedure is to delineate protocols for use of the chain-of-custody form. An example is provided as Figure SOP002-1. Other formats with similar levels of detail are acceptable.

2. MATERIALS

The following materials may be required: chain-of-custody form and indelible ink pen.

3. PROCEDURE

- Give the site name and project name/number.
- Enter the sample identification code.
- Indicate the sampling dates for all samples.
- List the sampling times (military format) for all samples.
- Indicate “grab” or “composite” sample with an “X.”
- Specify the sample location.
- Enter the total number of containers per cooler.
- List the analyses/container volume.
- Obtain the signature of sample team leader.
- State the carrier service and airbill number, analytical laboratory, and custody seal numbers.
- Sign, date, and time the “relinquished by” section.
- Upon completion of the form, retain the shipper copy, and affix the other copies to the inside of the sample cooler, in a zip-seal bag to protect from moisture, to be sent to the designated laboratory.

4. MAINTENANCE

Not applicable.

5. PRECAUTIONS

None.

6. REFERENCES

U.S. Environmental Protection Agency (U.S. EPA). 1980. Interim Guidelines and Specifications for Preparing Quality Assurance Project Plans, QAMS-005/80.

———. 1990. Sampler's Guide to the Contract Laboratory Program. EPA/540/P-90/006, Directive 9240.0-06, Office of Emergency and Remedial Response, Washington, D.C. December.

———. 1991. User's Guide to the Contract Laboratory Program. EPA/540/O-91/002, Directive 9240.0-01D, Office of Emergency and Remedial Response. January.

Company Name:

Project Manager or Contact:

Phone:

Project No.

Dept.: Task:

Project Name:

Sample Storage Location:

ATO Number:

Page of

Report #:

Parameters/Method Numbers for Analysis

Chain of Custody Record

EA Laboratories
19 Loveton Circle
Sparks, MD 21152
Telephone: (410) 771-4950
Fax: (410) 771-4077

Report Deliverables:
1 2 3 4 D E

EDD: Yes/No

DUE TO CLIENT: __________

Date	Time	Water	Soil	Sample Identification 19 Characters	**No. of Containers**														**EA Labs Accession Number**	Remarks
				ξ ξ ξ ξ ξ ξ ξ ξ ξ ξ ξ ξ ξ ξ ξ ξ ξ ξ																**LPM**:
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Samples by: (Signature)	Date/Time	Relinquished by: (Signature)	Date/Time	Received by: (Signature)	Date/Time
Relinquished by: (Signature)	Date/Time	Received by Laboratory: (Signature)	Date/Time	Airbill Number:	Sample Shipped by: (Circle) Fed Ex. Puro. UPS Hand Carried Other:
Cooler Temp. _____C pH: ___ Yes ___ No Comments:				**Custody Seals Intact ___ Yes ___ No**	
NOTE: Please indicate method number for analyses requested. This will help clarify any questions with laboratory techniques.					

Standard Operating Procedure No. 003
for
Subsurface/Utility Clearance

Prepared by

EA Engineering, Science, and Technology, Inc.
225 Schilling Circle, Suite 400
Hunt Valley, Maryland 21031

Revision 0
August 2007

CONTENTS

1. SCOPE AND APPLICATION

1.1 PURPOSE

The purpose of this Standard Operating Procedure is to prevent injury to workers and damage to subsurface structures (including tanks, pipe lines, water lines, gas lines, electrical service, etc.) during ground disturbance activities (including drilling, augering, sampling, use of direct-push technologies, excavation, trenching, concrete coring or removal, fence post installation, grading, or other similar operations).

1.2 LIMITATIONS

The procedures set forth in this document are the suggested procedures but may not be applicable to particular sites based on the site-specific considerations. The Project Manager is responsible for making a site-specific evaluation of each site to determine whether the Subsurface/Clearance Procedures should be utilized or require modification. If safety or other site-specific considerations require a modified or different procedure, the Project Manager should review the modified procedure with the Business Unit Director, Profit Center Manager, or Senior Technical Reviewer.

1.3 SCOPE

This procedure provides minimum guidance for subsurface clearance activities, which must be followed prior to and during ground disturbance activities at EA project sites. Even after completing the subsurface clearance activities required in this procedure, all ground disturbance activities should proceed with due caution.

Deviations from this procedure may be provided on an exception basis for specific situations, such as underground storage tank systems removals, verified aboveground/overhead services/lines, undeveloped land/idle facilities, shallow groundwater conditions, soil stability, or well construction quality assurance/quality control concerns, etc.

EA or its subcontractors are responsible for, and shall ensure that, all ground disturbance activities are completed safely, without incident, and in accordance with applicable federal, state, and local regulations.

This procedure shall not override any site-specific or consultant/contractor procedures that are more stringent or provide a greater degree of safety or protection of health or the environment.

2. PROCEDURES

The EA Project Manager or his designee must complete the Subsurface Clearance Procedure Checklist (Appendix A) in conjunction with the following procedures. The checklist must be completed before initiating any ground disturbance activities. The completed checklist must be submitted to the appropriate team individuals, subcontractors, and/or the client and included in the project files.

2.1 SAFETY

A Health and Safety Plan must be available onsite and followed by all contractors and subcontractors.

All work areas shall be defined and secured with safety cones, safety tape, construction fence, other barriers, or signs as appropriate.

Site work permits must be obtained as required by site procedures. Based on site conditions or classification, the use of intrinsically-safe equipment may be required.

To ensure the safety of all onsite personnel and subsurface structure integrity, consideration should be given to de-energizing and locking out selected site utilities or temporarily shutting down a portion of or the entire facility.

2.2 PREPARATION TASKS

Objective—To gather all relevant information about potential subsurface structures prior to the actual site visit.

2.2.1 Obtain Permits and Site Access

The consultant/contractor is responsible for following all applicable laws, guidance, and approved codes of practice; obtaining all necessary permits and utility clearances; and securing site access permission.

2.2.2 Historic Site Information

Obtain most recent as-built drawings and/or site plans (including underground storage tank, product, and vent lines) as available.

NOTE: As-built drawings may not accurately depict the locations and depths of improvements and subsurface structures and should, therefore, not be **solely** relied upon.

EA should obtain any other site information such as easements, right-of-ways, historical plot plans, fire insurance plans, tank (dip) charts, previous site investigations, soil surveys, boring logs, and aerial photographs, etc. as relevant to the planned ground disturbance activities.

Where applicable, EA should also contact contract personnel who may have historic site knowledge.

2.2.3 Mark-Outs

Objective—To identify location of subsurface structures on surface.

EA must ensure that a thorough mark-out at the site is completed to locate electrical, gas, telephone, water, sewer, low voltage electric lines, product delivery pipelines, fiber optic, and all other subsurface utilities/services.

- Where available, public utility companies must be contacted to identify underground utilities. (This can be accomplished through the One-Call system in most instances.)

- In addition, where available and warranted by site conditions, a private utility/pipeline mark-out company should be contracted to perform an electronic subsurface survey to identify the presence of suspected hazardous or critical underground utilities and subsurface structures. In some cases, this is necessary to confirm public utility mark-outs in the vicinity of planned ground disturbance activities.

EA will review all available site plan subsurface information with the private mark-out company to assist in locating utilities and other subsurface structures.

NOTE: Mark-outs may not accurately depict the exact locations of improvements and subsurface structures and should, therefore, not be **solely** relied upon.

Where possible, EA personnel are encouraged to be onsite at the time of subsurface mark-outs. This is to ensure accuracy and understanding of subsurface structures identified and provides an opportunity to exchange information with mark-out company personnel regarding planned work activities.

Subsurface structures should be marked throughout the entire work area(s) with adequate materials (e.g., site conditions may require paint and tape/flags). Ground disturbance activities must be started within 30 days of mark-out, unless local ordinances specify a shorter time period. If activities are not started within required time period or markings have faded, mark-outs must be redone.

EA personnel will record time and date of mark-out request and list all companies contacted by the service and confirmation number. This should be available for review onsite and checked off after visual confirmation of markings.

2.2.4 Initial Site Visit

Objective—To compare the site plan to actual conditions based on information gathered in Procedures 2 and 3 above, obtain additional site information needed, and prepare a vicinity map.

EA will document all findings and update the site plan with this information. On third party sites, close coordination with the site owner's representatives for mark-outs, review of as-builts, and other information reviews should be conducted prior to work. Project Managers are encouraged to provide updated as-built information to the client.

In some regions, it may be more effective and efficient to conduct the site visit at the same time the contractor and drill rig are mobilized to the site. The inspection should include the following activities and may include others as determined by the consultant/contractor and the Project Manager.

2.2.5 Utilities

EA shall perform a detailed site walk-through for the purpose of identifying all aboveground indicators of subsurface utilities/services that may be leading to or from buildings within the planned work area. The inspection shall include, but not be limited to, the following:

- Utility mark-outs
- Aboveground utilities
- Area lights/signs
- Phones
- Drains
- Junction boxes
- Natural gas meters or connections
- Other utilities including: fire hydrants, on/below grade electrical transformers, splice cages, sewer lines, pipeline markers, cable markers, valve box covers, clean-outs/traps, sprinkler systems, steam lines (including insulated tanks that may indicate steam lines), and cathodic protection on lines/tanks
- Observe paving scars (i.e., fresh asphalt/concrete patches, scored asphalt/concrete).

NOTE: In many cases, the onsite location of low-voltage electrical lines and individual property water and sewer line branches may be approximated by using the following technique:

- Locate the entry/connection location at the facility building

- Attempt to identify utility connections for the mains (water sewer, etc.) by locating clean-outs, valve manways, etc. The location path of the utility is likely with the area between the main connection and facility building connection. Subsurface electrical line locations from the facility building to signs, lamps, etc. can be estimated with the same process.

2.2.6 Other Subsurface Systems

Some other subsurface systems to be cognizant of during subsurface activities include product delivery systems (i.e., at gas stations) and existing remediation systems.

2.2.7 Selection of Ground Disturbance Locations

EA will utilize the information collected to this point in combination with regulatory requirements and project objectives to select ground disturbance locations. Ground disturbance locations should also consider the location of overhead obstructions (e.g., power lines). Work at active gasoline retail locations must consider several special considerations that should be outlined in the site-specific safety and health plan.

2.2.8 Review of Selected Locations with the Client

EA will review the selected ground disturbance locations with the client. EA will not proceed with the subsurface activities until the plan has been discussed with the client. During execution of the project, subsurface activities are required outside of the area previously approved by the client. EA will submit these changes to the client for approval prior to execution.

2.2.9 Ground Disturbance Activity Sequence

EA will plan ground disturbance activities starting at the point farthest from the location of suspected underground improvements. This is done to determine the natural subsurface conditions and to allow EA site personnel to recognize fill conditions.

Experience has shown that the following warning signs may indicate the presence of a subsurface structure:

- Warning tape (typically indicative of underground services).

- Pea gravel/sand/non-indigenous material (typically indicative of tanks or lines).

- Red concrete (typically indicative of electrical duct banks).

- The abrupt absence of soil recovery in a hand auger. This could indicate pea gravel or sand that has spilled out of the auger. This may not be indicative in areas where native soil conditions typically result in poor hand auger recoveries.

- Any unexpected departure from the native soil or backfill conditions as established by prior onsite digging.

If any of these conditions is encountered by EA site personnel, digging should stop and the client should be contacted.

3. SUBSURFACE CLEARANCE METHODS

The method used to delineate the subsurface should be compatible with the inherent associated risk given the type of facility/property, soil stratigraphy, and the location of the ground disturbance activity, such that required delineation is obtained. It should be noted that in areas where there is paving, sufficient paving should be removed to allow clear visibility of the subsurface conditions during clearance activities. The following is a list of potential clearance methods that may be used on a job site:

- Vacuum digging
- Probing
- Hand digging
- Hand augering
- Post-hole digging.

EA personnel will evaluate the potential for electrical shock or fire/explosion for each subsurface disturbance project and will evaluate as necessary the use of non-conductive or non-sparking tools (i.e., fiberglass hand shovels, and thick electrically insulating rubber grips on hand augers or probes). The potential need for the use of non-conductive materials, electrical safety insulated gloves, and footwear will also be evaluated on a case-by-case basis.

3.1 SUBSURFACE CLEARANCE PROCEDURES FOR DRILLING, DIRECT-PUSH TECHNOLGY, AUGERING, FENCE POST INSTALLATION, OR OTHER BOREHOLE INSTALLATION ACTIVITIES

The area to be delineated will exceed the diameter of the largest tool to be advanced and sufficiently allow for visual inspection of any obstructions encountered.

3.2 SUBSURFACE CLEARANCE PROCEDURES FOR TRENCHING/ EXCAVATION ACTIVITIES

Appropriate subsurface clearance methods should be conducted along the length and width of the excavation at a frequency sufficient to ensure adequate precautions have been applied to the entire work area. The frequency and density of investigations will be based on site knowledge, potential hazards, and risks of the work area to surrounding locations (e.g., proximity to a residential area or school).

Whenever subsurface structures are exposed, EA will cease work and mark the area (e.g., flags, stakes, cross bracing) to ensure the integrity of these exposed structures is maintained during subsequent trenching/excavation/backfilling.

Uniform color codes for marking of underground facilities are provided in Appendix B.

Appendix A

Subsurface Clearance Procedure Checklist

Subsurface Clearance Procedure Checklist

Site Identification: _____

Project Consultant/Contractor: _____

Section 1: Safety, Preparation Tasks, and Mark-Outs

Activity	Yes	No	N/A	Comments including Justification if Response Is No or Not Applicable
Health and Safety Plan is available and all contractors and subcontractors are familiar with it.				
All applicable local, state, and federal permits have been obtained.				
Site access/permission has been secured.				
Most recent as-built drawings and/or site plans (including underground storage tank, product, and vent lines) obtained.				
Reviewed site information to identify subsurface structures relevant to planned site activities (easements, rights-of-way, historical plot plans, fire insurance plans, tank dip charts, previous site investigations, soil surveys, boring logs, aerial photographs, etc.).				
Utility mark-outs have been performed by public utility company(s). Mark-outs clear/visible.				
Subsurface structure mark-outs performed by private mark-out company. Mark-outs clear/visible.				
Additional Activities: Were dig locations reviewed with site representative?				

Section 2: Initial Site Visit and Selecting Ground Disturbance Locations

Activity	Yes	No	N/A	Comments including Justification if Response Is No or Not Applicable
Location of all aboveground indicators of subsurface utilities/services that may be leading to or from buildings within the planned work area has been identified.				
Location of utility mark-outs by all utility companies previously contacted has been identified within required time period.				
Location of all subsurface structure mark-outs by private mark-out company has been identified within required time period.				
Location of area lights/signs and associated subsurface lines identified.				
Location of all phones and associated subsurface lines identified.				
Location of all drains and associated interconnecting lines identified.				
Location of all electrical junction boxes and associated interconnecting lines identified				
Location of all natural gas meters or connections and all interconnecting lines identified				

Completed by: _____
Name

Signature: _____ _____
Company Date

Appendix B

Uniform Color Codes for Excavation

APWA UNIFORM COLOR CODE

WHITE - Proposed Excavation

PINK - Temporary Survey Markings

RED - Electric Power Lines, Cables, Conduit and Lighting Cables

YELLOW - Gas, Oil, Steam, Petroleum or Gaseous Materials

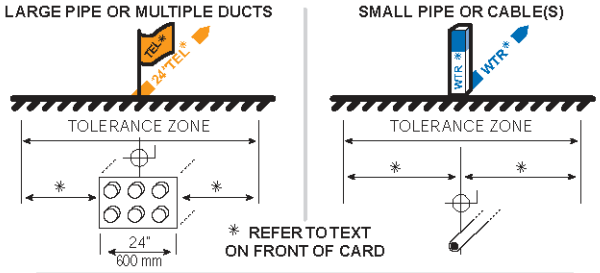
ORANGE - Communication, Alarm or Signal Lines, Cables or Conduit

BLUE - Potable Water

PURPLE - Reclaimed Water, Irrigation and Slurry Lines

GREEN - Sewers and Drain Lines

TYPICAL MARKING



Customize with your center's phone and address information

GUIDELINES FOR UNIFORM TEMPORARY MARKING OF UNDERGROUND FACILITIES

This marking guide provides for universal use and understanding of the temporary marking of subsurface facilities to prevent accidents and damage or service interruption by contractors, excavators, utility companies, municipalities or any others working on or near underground facilities.

ONE-CALL SYSTEMS
The One-Call damage prevention system shall be contacted prior to excavation.

PROPOSED EXCAVATION
Use white marks to show the location, route or boundary of proposed excavation. Surface marks on roadways do not exceed 1.5" by 18" (40 mm by 450 mm). The facility color and facility owner identity may be added to white flags or stakes.

USE OF TEMPORARY MARKING
Use color-coded surface marks (i.e., paint or chalk) to indicate the location or route of active and out-of-service buried lines. To increase visibility, color coded vertical markers (i.e., stakes or flags) should supplement surface marks. Marks and markers indicate the name, initials or logo of the company that owns or operates the line, and width of the facility if it is greater than 2" (50 mm). Marks placed by other than line owner/operator or its agent indicate the identity of the designating firm. Multiple lines in joint trench are marked in tandem. If the surface over the buried line is to be removed, supplementary offset markings are used. Offset markings are on a uniform alignment and clearly indicate the actual facility is a specific distance away.

TOLERANCE ZONE
Any excavation within the tolerance zone is performed with non-powered hand tools or non-invasive method until the marked facility is exposed. The width of the tolerance zone may be specified in law or code. If not, a tolerance zone including the width of the facility plus 18" (450 mm) measured horizontally from each side of the facility is recommended.

ADOPT UNIFORM COLOR CODE
The American Public Works Association encourages public agencies, utilities, contractors, other associations, manufacturers and all others involved in excavation to adopt the APWA Uniform Color Code, using ANSI standard Z535.1 Safety Colors for temporary marking and facility identification.

Rev. 4/99

Standard Operating Procedure No. 004
for
Sample Packing and Shipping

Prepared by

EA Engineering, Science, and Technology, Inc.
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Revision 0
August 2007

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1. SCOPE AND APPLICATION

The purpose of this Standard Operating Procedure (SOP) is to delineate protocols for the packing and shipping of samples to the laboratory for analysis.

2. MATERIALS

The following materials may be required:

Clear tape	Plastic garbage bags
Custody seals	Sample documentation
Ice	Waterproof coolers (hard plastic or metal)
Metal cans with friction-seal lids (e.g., paint cans)	Zip-seal plastic bags
Packing material[1]	

3. PROCEDURE

Check cap tightness and verify that clear tape covers label and encircles container. Wrap sample container in bubble wrap or closed cell foam sheets. Enclose each sample in a clear zip-seal plastic bag.

Place several layers of bubble wrap, or at least 1 in. of vermiculite on the bottom of the cooler. Line cooler with open garbage bag, place all the samples upright inside a garbage bag, and tie the bag.

Double bag and seal loose ice to prevent melting ice from soaking the packing material. Place the ice outside the garbage bags containing the samples.

Pack shipping containers with packing material (closed-cell foam, vermiculite, or bubble wrap). Place this packing material around the sample bottles or metal cans to avoid breakage during shipment.

Enclose all sample documentation (i.e., Field Parameter Forms, chain-of-custodies) in a waterproof plastic bag and tape the bag to the underside of the cooler lid. If more than one cooler is being used, each cooler will have its own documentation.

Seal the coolers with signed and dated custody seals so that if the cooler were opened, the custody seal would be broken. Place clear tape over the custody seal to prevent damage to the seal.

1. Permissible packing materials are: (a) (non-absorbent) bubble wrap or closed cell foam packing sheets, or (b) (absorbent) vermiculite. Organic materials such as paper, wood shavings (excelsior), and cornstarch packing "peanuts" will not be used.

Refer to SOP Nos. 001, 002, 016, and 039.

Tape the cooler shut with packing tape over the hinges and place tape over the cooler drain. Ship all samples via overnight delivery on the same day they are collected if possible.

4. MAINTENANCE

Not applicable.

5. PRECAUTIONS

Any samples suspected to be of medium/high contaminant concentration or containing dioxin must be enclosed in a metal can with a clipped or sealable lid (e.g., similar to a paint can). Label the outer metal container with the sample number of the sample inside.

6. REFERENCES

U.S. Environmental Protection Agency (U.S. EPA). 1980. Interim Guidelines and Specifications for Preparing Quality Assurance Project Plans, QAMS-005/80.

———. 1990. Sampler's Guide to the Contract Laboratory Program. EPA/540/P-90/006, Directive 9240.0-06, Office of Emergency and Remedial Response, Washington, D.C. December.

———. 1991. User's Guide to the Contract Laboratory Program. EPA/540/O-91/002, Directive 9240.0-01D, Office of Emergency and Remedial Response. January.

Standard Operating Procedure No. 005
for
Field Decontamination

Prepared by

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CONTENTS

1. SCOPE AND APPLICATION

All personnel or equipment involved in intrusive sampling, or which enter a hazardous waste site during intrusive sampling, must be thoroughly decontaminated prior to leaving the site to minimize the spread of contamination and prevent adverse health effects. This Standard Operating Procedure describes the normal decontamination of sampling equipment and site personnel.

2. MATERIALS

The following materials may be required:

0.01N HCl	Non-phosphate laboratory detergent (liquinox)
0.10N nitric acid	Plastic garbage bags
Aluminum foil or clean plastic sheeting	Plastic sheeting, buckets, etc. to collect wash water and rinsates
Approved water	Pressure sprayer, rinse bottles, brushes
High performance liquid chromatography (HPLC)-grade water[1]	Reagent grade alcohol[2]

3. PROCEDURE

3.1 SAMPLE BOTTLES

At the completion of each sampling activity, the exterior surfaces of the sample bottles must be decontaminated as follows:

- Ensure the bottle lids are on tight.
- Wipe the outside of the bottle with a paper towel to remove gross contamination.

3.2 PERSONNEL DECONTAMINATION

Review the project Health and Safety Plan for the appropriate decontamination procedures.

1. For the purposes of this Standard Operating Procedure, HPLC-grade water is considered equivalent to "deionized ultra filtered water," "reagent-grade distilled water," and "deionized organic-free water." The end product being water which is pure with no spurious ions or organics to contaminate the sample. The method of generation is left to the individual contractor.
2. For the purposes of this Standard Operating Procedure, the term "reagent grade alcohol" refers to either pesticide grade isopropanol or reagent grade methanol.

3.3 EQUIPMENT DECONTAMINATION

3.3.1 Water Samplers

3.3.1.1 Bailers

After each use, polytetrafluoroethelyne (PTFE) double check valve bailers used for groundwater sampling will be decontaminated as follows:

- Discard all ropes used in sampling in properly marked sealable container, or as directed by the Health and Safety Plan. NOTE: No tubing is to be used in conjunction with a bailer in collecting samples.
- Scrub the bailer to remove gross (visible) contamination, using appropriate brush(es), approved water, and non-phosphate detergent.
- Rinse off detergent three times with approved water.
- Rinse bailer with reagent grade alcohol.
- Rinse bailer three times with HPLC-grade water.
- Rinse bailer with 0.10N nitric acid solution.
- Rinse bailer three times with HPLC-grade water.
- Allow bailer to air dry.[3]
- Wrap bailer in aluminum foil or clean plastic sheeting, or store in a clean, dedicated polyvinyl chloride or PTFE storage container.
- Dispose of used decontamination solutions with drummed purge water.
- Rinse bailer with HPLC-grade water immediately prior to re-use.

3.3.1.2 Pumps

Submersible pumps will be decontaminated as follows:

3. If the bailer has just been used for purging and is being decontaminated prior to sampling, do not air dry. Double rinse with HPLC-grade water and proceed to collect samples.

- Scrub the exterior of the pump to remove gross (visible) contamination, using appropriate brush(es), approved water, and non-phosphate detergent. (Steam cleaning may be substituted for detergent scrub.)

- Calculate the volume of pump plus any tubing which is not disposable and not dedicated to a single well. Pump three volumes of non-phosphate laboratory detergent solution to purge and clean the interior of the pump.

- Rinse by pumping no less than nine volumes of approved water to rinse.

- Rinse pump exterior with reagent grade alcohol.

- Rinse pump exterior with HPLC-grade water.

- Allow pump to air dry.

- Wrap pump in aluminum foil or clean plastic sheeting, or store in a clean, dedicated polyvinyl chloride or PTFE storage container.

- Prior to reusing pump rinse exterior again with HPLC-grade water. (Double rinse in Bullet 5 above may be substituted for this step).

3.3.1.3 Dip Samplers

All dip samplers, whether bucket, long-handled, or short-handled, will be decontaminated in the same manner as provided in Section 3.3.1.1.

3.3.1.4 Labware

Labware, such as beakers, which are used to hold samples for field measurements, water chemistry, etc. will be decontaminated according to the procedures in Section 3.3.1.1.

3.3.1.5 Water Level Indicators

Electric water level indicators, weighted measuring tapes, or piezometers used in the determination of water levels, well depths, and/or non-aqueous phase liquid levels will be decontaminated in accordance with Section 3.3.1.1. Clean laboratory wipes may be substituted for brushes. Tapes, probes, and piezometers should be wiped dry with clean laboratory wipes, and coiled on spools or clean plastic sheeting rather than allowed to air dry.

3.3.2 Solid Materials Samplers

Solid materials samplers include soil sampling probes, augers, trowels, shovels, sludge samplers, and sediment samplers, which will be decontaminated as follows:

- Scrub the sampler to remove gross (visible) contamination, using appropriate brush(es), approved water, and non-phosphate laboratory detergent.
- Rinse off detergent with approved water.
- Rinse sampler with reagent grade alcohol.
- Rinse sampler with HPLC-grade water.
- For non-metallic samplers only, rinse sampler with 0.10N nitric acid solution.
- For non-metallic samplers only, rinse sampler with HPLC-grade water.
- Allow sampler to air dry.
- Wrap sampler in aluminum foil clean plastic sheeting, or store in a new zipseal bag (size permitting) or clean, dedicated polyvinyl chloride or PTFE storage container.
- Dispose used decontamination solutions properly according to the site-specific Health and Safety Plan.
- Rinse sampler with HPLC-grade water immediately prior to re-use.

3.3.3 Other Sampling and Measurement Probes

Soil gas sampling probes will be decontaminated as solids sampling devices.

Temperature, pH, conductivity, redox, and dissolved oxygen probes will be decontaminated according to manufacturer's specifications. If no such specifications exist, remove gross contaminant and triple rinse probe with HPLC-grade water. A summary of the decontamination procedures to be used must be included in the instrument-specific standard operating procedure.

Measuring tapes that become contaminated through contact with soil during field use will be decontaminated as follows:

- Wipe tape with a clean cloth or laboratory wipe that has been soaked with non-phosphate laboratory detergent solution to remove gross contamination. Rinse cloth in the solution and continue wiping until tape is clean.
- Wipe tape with a second clean, wet cloth (or laboratory wipe) to remove soap residues.
- Dry tape with a third cloth (or laboratory wipe) and rewind into case, or re-coil tape.

3.3.4 Drilling Rigs and Other Heavy Equipment

All drilling rigs and associated equipment such as augers, drill casing, rods, samplers, tools, recirculation tank, and water tank (inside and out) will be decontaminated prior to site entry after over-the-road mobilization and immediately upon departure from a site after drilling a hole. Supplementary cleaning will be performed prior to site entry when there is a likelihood that contamination has accumulated on tires and as spatter or dust enroute from one site to the next.

- Place contaminated equipment in an enclosure designed to contain all decontamination residues (water, sludge, etc.).
- Steam clean equipment until all dirt, mud, grease, asphaltic, bituminous, or other encrusting coating materials (with the exception of manufacturer-applied paint) have been removed.
- Water used will be taken from an approved source.
- Containerize in 55-gal drums; sample; characterize; and, based on sample results, dispose of all decontamination residues properly.

Other heavy equipment includes use of backhoes, excavators, skid steers, etc. If heavy equipment is utilized during field activities, i.e., a backhoe for test pitting, the bucket should not come in contact with soil to be sampled. If the bucket contacts the soil to be sampled, then it should be decontaminated between sample locations, following the same procedures as listed above for a drill rig.

3.3.5 High Performance Liquid Chromatography-Grade Water Storage

Dedicated glass storage containers will be used solely for dispensing HPLC-grade water. New HPLC-grade water containers will be decontaminated as follows:

- Clean with tap water from approved source and non-phosphate laboratory detergent while scrubbing the exterior and interior of the container with a stiff-bristled brush.
- Rinse thoroughly with approved water.
- Rinse with 0.01N nitric acid.
- Rinse with approved water.
- Rinse thoroughly with HPLC-grade water.
- Fill clean container with HPLC-grade water. Cap with one layer of PTFE-lined paper and one layer of aluminum foil. Secure cap with rubber band and date the container.

Used HPLC-grade water containers will be decontaminated as follows:

- Clean the exterior with tap water from an approved source, non-phosphate laboratory detergent, and a stiff-bristled brush.
- Rinse the exterior thoroughly with HPLC-grade water.
- Rinse the interior twice with pesticide-grade isopropanol.
- Rinse interior thoroughly with HPLC-grade water.
- Fill clean container with HPLC-grade water. Cap with one layer of PTFE-lined paper and one layer of aluminum foil. Secure cap with rubber band and date the container.

3.3.6 Ice Chests and Reusable Shipping Containers

- Scrub exterior/interior with approved brush and liquinox detergent.
- Rinse off detergent three times with approved water.
- Let air dry and properly store until re-use.

NOTE: If container/ice chest is severely contaminated, clean as thoroughly as possible, render unusable, and properly dispose.

4. MAINTENANCE

HPLC-grade water will be stored only in decontaminated glass containers with aluminum foil lids as stipulated above. The water may not be stored for more than nor used more than 3 days after manufacture.

HPLC-grade water will be manufactured onsite. An approved tap water source will be used as the influent to the system. Procedures for system setup, operation, and maintenance will conform to manufacturer's specifications.

5. PRECAUTIONS

Dispose of all wash water, rinse water, rinsates, and other sampling wastes (tubing, plastic sheeting, etc.) in properly marked, sealable containers, or as directed by the Health and Safety Plan.

Once a piece of equipment has been decontaminated, be careful to keep it in such condition until needed.

Do not eat, smoke, or drink onsite.

6. REFERENCES

Site-specific Health and Safety Plan.

Standard Operating Procedure No. 010
for
Water Level and
Well Depth Measurements

Prepared by

EA Engineering, Science, and Technology, Inc.
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Revision 0
August 2007

CONTENTS

1. SCOPE AND APPLICATION

The purpose of this Standard Operating Procedure (SOP) is to delineate protocols for measuring water level and well depth. This procedure is applicable to the sampling of monitoring wells and must be performed prior to any activities which may disturb the water level, such as purging or aquifer testing.

2. MATERIALS

The following materials may be required:

Electric water level indicator (dipmeter) with cable measured at 0.01-ft increments OR weighted steel tape and chalk OR transducer and datalogger
Oil/water interface probe
Plastic sheeting
Photoionization detector or intrinsically safe flame ionization detector

3. PROCEDURE

3.1 PRELIMINARY STEPS

Locate the well and verify its position on the site map. Record whether positive identification was obtained, including the well number and any identifying marks or codes contained on the well casing or protective casing. Gain access to the top of the well casing.

Locate the permanent reference mark at the top of the casing. This reference point will be scribed, notched, or otherwise noted on the top of the casing. If no such marks are present, measure to the top of the highest point of the well casing and so note this fact in the field logbook. Determine from the records and record in the notebook the elevation of this point.

Record any observations and remarks regarding the completion characteristics and well condition, such as evidence of cracked casing or surface seals, security of the well (locked cap), and evidence of tampering.

Keep all equipment and supplies protected from gross contamination; use clean plastic sheeting. Keep the water level indicator probe in its protective case when not in use.

3.2 OPERATION

Sample the air in the well head for gross organic vapors by lifting the well cap only high enough for an organic vapor meter (photoionization detector or flame ionization detector) probe to be entered into the well casing. This will indicate the presence of gross volatile contaminants as well as indicating potential sampler exposure.

Remove cap. Allow well to vent for 60-90 seconds. Resample headspace. Record both readings. If the second reading is lower than the first, use the second reading to determining whether respiratory protection will be required during subsequent water level and well depth determinations, and sampling.

Note that all headspace sampling must be performed at arm's length and from the upwind side of the well if possible.

Refer to SOP Nos. 011, 023, or 024 as appropriate.

If non-aqueous phase liquid (NAPL) contamination is suspected[1], use an interface probe to determine the existence and thickness of NAPLs.

Open the probe housing, turn the probe on, and test the alarm. Slowly lower the probe into the well until the alarm sounds. A continuous alarm indicates a NAPL while an intermittent alarm indicates water. If a NAPL is detected, record the initial level (first alarm). Mark the spot by grasping the cable with the thumb and forefingers at the top of the casing. If a mark is present on the casing, use the mark as the reference point. If no mark is present, use the highest point on the casing as the reference point. Withdraw the cable sufficiently to record the depth.

Continue to slowly lower the probe until it passes into the water phase. Slowly retract the probe until the NAPL alarm sounds and record that level in the manner as described above.

Record the thickness of the light NAPL[2] (Section 3.3).

Continue to slowly lower the interface probe through the water column to check for the presence of dense NAPL.

Measure and record the thickness of the dense NAPL layer (if any) as described above.

Slowly raise the interface probe, recording the depth to each interface as the probe is withdrawn. If there is a discrepancy in depths, clean the probe sensors and re-check the depths.

NOTE: Air/liquid interface depth is more reliable if probe is lowered into liquid. NAPL/water depths are more accurate if probe is moved from water into NAPL.

Always lower and raise interface probe slowly to prevent undue mixing of media.

1. Interface probes will be used in all wells for first round sampling, regardless of site history. If no NAPLs are detected during the first round of sampling, this step may be omitted during subsequent sampling events **unless** conditions such as site history or headspace vapors would indicate otherwise.
2. If NAPL is viscous, such as coal tar or weather bunker oil, several confirmation measurements should be made after decontamination of the probe to verify that the NAPL is not sticking to the probe and causing erroneous readings. One way to accomplish this would be to partially fill a 5-gal bucket with water and dip the probe to ensure that decontamination has effectively removed the NAPL.

Always perform NAPL check in wells installed in areas with suspected NAPL contamination. Always perform NAPL check if headspace test reveals presence of volatiles. Always perform NAPL check the first time a well is sampled. **If** a well has been sampled previously **and** no NAPLs were present **and** none of the preceding conditions are met, the NAPL check may be omitted.

If no NAPL is present, use an electronic water level detector as follows:

- Remove the water level indicator probe from the case, turn on the sounder, and test check the battery and sensitivity scale by pushing the red button. Adjust the sensitivity scale until you can hear the buzzer.

- Slowly lower the probe and cable into the well, allowing the cable reel to unwind. Continue lowering until the meter buzzes. Very slowly, raise and lower the probe until the point is reached where the meter **just** buzzes. Marking the spot by grasping the cable with the thumb and forefingers at the top of the casing. If a mark is present on the casing, use the mark as the reference point. If no mark is present, use the highest point on the casing as the reference point. Withdraw the cable and record the depth.

Alternatively use a steel tape with an attached weight if aquifer gradients are lower than 0.05 ft/ft. Due to the possibility of adding unknown contaminants from chalk colorants, only white chalk is permitted.

Rub chalk onto the first 1 ft of the steel tape and slowly lower the chalked end into the well until the weighted end is below the water surface. (A small splash can be heard when the weighted end hits the water surface.)

Using the method described above read and record the length from the steel tape.

Remove the steel tape. The chalk will be wet or absent where the tape was below the water surface. Locate, read, and record this length. Subtract wetted length from total length and record the difference. This is the depth to water table.

Transducers and dataloggers will be used where water level fluctuations over time are to be measured, such as tidal fluctuation studies (SOP No. 045) and aquifer (hydraulic) tests (SOP No. 033). Note that transducers are inappropriate for measuring well depth.

Slowly lower the transducer into the well until it is below the lowest possible piezometric level (typically 2-3 ft below the water table).

Tape the umbilical to the protective casing to prevent the transducer from falling further.

Attach the umbilical leads to the datalogger.

Turn datalogger on.

To measure the well depth, lower electric water level indicator probe or tape until slack is noted. Very slowly raise and lower the cable until the exact bottom of the well is "felt." Measure (cable) or read the length (tape) and record the depth.

Note that if the electric water level indicator is used to determine depth of well, the offset distance between the tip of the probe and the electrode must be added to the reading to determine actual depth.

Withdraw the probe or tape. Decontaminate the probe(s) and cable(s).

3.3 DATA RECORDING AND MANIPULATION

Record the following computations:

- Date and time
- Weather
- Method of measurement
- Casing elevation
- NAPL surface elevation = casing elevation – depth to NAPL
- NAPL thickness = depth to bottom of NAPL – depth to top of NAPL
- Water level elevation = casing elevation – depth to water
- Well bottom elevation = casing elevation – depth to bottom (or read directly from tape).

Refer to SOP Nos. 005 and 016.

4. CALIBRATION

No calibration is needed.

5. PRECAUTIONS

Depending upon the device used, correction factors may be required for some measurements. Check instrument batteries prior to each use. Exercise care not to break the seals at the top of the electric water level indicator probe.

6. REFERENCES

McAlary, T.A. and J.F. Barker. 1987. Volatilization Losses of Organics during Groundwater Sampling from Low Permeability Materials in Groundwater Monitoring Review. Fall.

Thornhill, J.T. 1989. Accuracy of Depth to Groundwater Measurements in U.S. Environmental Protection Agency Superfund Groundwater Issue EPA/540/4-89/002.

Standard Operating Procedure No. 011
for
Photoionization Detector

Prepared by

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Page

1. SCOPE AND APPLICATION

The purpose of this Standard Operating Procedure (SOP) is to delineate protocols for field operations with the photoionization detector (MiniRae). The photoionization detector uses an ultraviolet emitting lamp designed to detect, measure, and display the total concentration of airborne ionizable gases and vapors. This information is used to determine control measures such as protection and action levels.

Use of brand names in this SOP is not intended as endorsement or mandate that a given brand be used. Alternate equivalent brands of detectors, sensors, meters, etc. are acceptable. If alternate equipment is to be used, the contractor will provide applicable and comparable SOPs for the maintenance and calibration of same.

2. MATERIALS

The following materials may be required:

Battery pack	Tedlar bag
Calibration gas (100 ppm isobutylene)	Tygon tubing
Microtip/MiniRae	Regulator

3. STARTUP/CALIBRATION PROCEDURE

Turn the instrument on by pressing the back of the power switch located on the handle of the instrument.

The message “Warming up now, please wait” will be displayed for up to 3 minutes. After normal display appears, the instrument is ready for calibration.

Fill a Tedlar bag with the desired calibration gas (usually 100 ppm Isobutylene).

Press SETUP button and select the desired Cal Memory using the arrow keys (normally set to 200 ppm). Press EXIT button to leave setup function.

Press CAL button and expose instrument to Zero Gas. (Usually clean outdoor air will be suitable. If any doubt exists as to the cleanliness of the background air a commercial source of zero gas should be used.)

The instrument then asks for the Span Gas concentration. Enter the known span gas concentration and then connect the Tedlar bag containing the Span Gas.

NOTE: The span gas concentration is dependent upon both the concentration of the span gas used and the rating of the UV lamp in the instrument at time of calibration. If using 100 ppm isobutylene and the standard 10.6 eV lamp, the span gas concentration will be 56 ppm.

Press enter and the instrument sets its sensitivity. Once the display reverts to normal, the instrument is calibrated and ready for use. Remove the Span Gas from the inlet probe. The instrument should be calibrated at least once a day.

4. BATTERY CHARGING

Ensure instrument is off. Set the voltage selector switch on the bottom of the battery charger to the appropriate AC line voltage. Press the release button on the bottom of the instrument and remove the battery pack by sliding it backwards. Plug charger into the battery pack and then into an AC outlet and allow the battery to charge for at least 8 hours. After charging, remove the charger, first from the outlet then from the battery pack, and slide the battery pack back onto the instrument.

5. PRECAUTIONS

Instrument does not carry an Intrinsic Safety Rating and must not be used in a hazardous location where flammable concentrations of gases or vapors are constantly present.

All calibration, maintenance, and servicing of this device, including battery charging, must be performed in a safe area away from hazardous locations.

Do not open or mutilate battery cells. Do not defeat proper polarity orientation between the battery pack and battery charger. Substitution of components may affect safety rating.

6. REFERENCES

Instrument User's Manual.

Standard Operating Procedure No. 013
for
Collection of Monitoring Well Samples

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Revision 0
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1. SCOPE AND APPLICATION

The purpose of this Standard Operating Procedure is to delineate protocols for the collection of groundwater samples from monitoring wells.

2. MATERIALS

The following materials may be required:

0.45 µM filters	Polyvinyl chloride bailer (for purging only)
Bladder pump (dedicated to one well only)	Sample bottles and labels
Conductivity meter	Stainless steel bailer (for purging and sampling)
Dissolved oxygen meter	Submersible pump and hose (for purging only)
Generator	Thermometer (optional)[1]
Logbook or book of field parameter forms	Transparent bailer with a double check valve
Peristaltic pump with tubing for filtering samples	Turbidity meter
pH meter with oxidation-reduction potential probe	Tygon tubing
Photoionization detector organic vapor analyzer.	Variable speed, low flow submersible pump (e.g., Grundfos MP1 groundwater sampling pump) (for purging and sampling)
Plastic sheeting	Water level indicator
Polypropylene rope	
Polytetrafluoroethelyne (PTFE) bailer with PTFE-coated stainless steel cable, double check valve top, and controlled flow bottom discharge attachment[2] for volatile organic compound (VOC) sampling (40-mL vials), and top discharge attachment for collecting larger samples (1-L bottles) (for purging and sampling)	

3. PROCEDURE

3.1 GENERAL

Groundwater sampling will follow these general steps:

- Arrive onsite
- Set up apparatus (generators, pumps, etc.)
- Glove
- Organic vapor check, water level, and well depth measurements

1. Temperature compensation and measurement capabilities are generally available as integral functions of pH meters and conductivity meters. If this is the case, a separate thermometer is not required.
2. Although use of a controlled flow bottom discharge valve is historically preferred, use of such a device can cause aeration of the sample.

- Sample non-aqueous phase liquids (NAPLs) (as required)

- Begin purge procedure

  — If using bailer to purge and sample, see Section 3.6
  — If using pump to purge and bailer to sample, see Section 3.7
  — If using bladder or low-flow pump to purge and sample, see Section 3.8

- Decontaminate/reglove

- Take samples

  — If with bailer, see Section 3.6
  — If with bladder or low flow pumps, see Section 3.8

- Decontaminate/dispose of wastes, move equipment to next site.

3.2 GENERAL RULES FOR GROUNDWATER FIELD PARAMETER LOGBOOK

Only one site or installation per logbook, and only one sampling location per page or form (if using pre-printed forms). The same book may be used for more than one sampling event. First five pages will be reserved for index, general notes, etc. Sign and date each entry. Last five pages will be reserved for recording calibration data for the pH, temperature, turbidity, oxidation-reduction potential, dissolved oxygen, and conductivity meters. Use the page number or a separately recorded "Cal Reference Number" to refer to each calibration. As appropriate, insert the cardboard flap under the form being filled out, so that writing does not go through to the pages below. As appropriate, fill in the forms from front to back of the logbook, tearing out the white copy for each sample when the sample has been collected. This copy goes in the cooler with the sample, directly to the laboratory. The original copy must be torn out before you write on the back of the duplicate form. As appropriate, duplicate copies, index pages, and calibration sheets remain intact.

3.3 GROUNDWATER SAMPLING GENERAL RULES

Groundwater samples will be collected from the least contaminated wells first, progressing to the most contaminated[3]. Upon arrival at the well site, immediately set up and organize the purging, sampling, and filtration equipment. If needed, due to muddy or contaminated ground, remoteness from sampling vehicle, and\or for placement of hose(s) and\or power cord if a pump is used, place clean plastic sheeting at, or around the well, to serve as a clean staging area for purging and sampling equipment, as conditions warrant. Care must be exercised not to step on plastic sheeting. If the well is remote from the sampling vehicle, set up the filtration equipment

3. First round samples are to be collected from upgradient wells first, moving to downgradient wells under the assumption that upgradient wells will be less contaminated than downgradient wells. Results of first round analysis may mandate a change in sampling sequence.

and place rope, wrapped bailer, and pre-labeled sample containers on the plastic sheet, from the well. When a pump is to be used, situate the portable generator on level ground approximately 15 ft away from and downwind from the well. All generator maintenance (oil and fueling) is to be performed offsite. If the hose(s) and/or power cord of the pump are not on a reel, place the pump with its hose and power cord on the plastic sheeting downhill from the well.

Check well headspace for organic vapor which may pose a health and safety hazard and indicate the presence of NAPL. Measure depth(s) to and thickness(es) of NAPL(s) as appropriate. Measure the depth to water and depth of well. From the water depth, well diameter, sand pack length, etc., calculate the equivalent volume (1 EV) of water in the well.

1 EV = volume in casing + volume in saturated sand pack. Therefore, if the water table lies below the top of the sandpack, use the following equation:

$$1\ EV = (\pi R_w^2 h_w) + (0.30\pi(R_s^2 - R_w^2)h_w) * (0.0043)$$

If the water table lies above the top of the sandpack use this equation:

$$1\ EV = [(\pi R_w^2 h_w) + (0.30\pi(R_s^2 - R_w^2)h_s)] * (0.0043)$$

where

R_s = Radius of sandpack in inches
R_w = Radius of well casing in inches
h_s = Height of sandpack in inches
h_w = Water depth in inches

0.0043 gal/in.^{3}
Assumed filter pack porosity = 30 percent.

Samples will always be collected in order of decreasing volatility (i.e., the samples to be analyzed for the volatile constituents should be collected first). Deliver the VOC sample to the vial by allowing the water to trickle down the inside wall of the vial at a rate no greater than approximately 100 ml/min. Other samples may be delivered at a faster rate. Sampling rates will at no time exceed 1 L/min. Procedures for each class of samples are contained in the site-specific Quality Assurance Project Plan.

When collecting samples for volatile analysis, care should be taken to prevent analyte loss by volatilization. The following procedures should be adhered to when collecting these samples:

- Avoid excessive aeration and agitation of sample.

- Fill vial so that a reverse meniscus is present by adjusting the flow rate from the sampling device.

- Place septum on vial so that the PTFE side is in contact with the sample. After the cap is on the bottle, check for air bubbles in the sample. If air bubbles are present, properly dispose of that sample and recollect the sample in the same vial.

- Make sure vial is labeled and immediately transfer the vial to the cooler with ice.

Filtered and unfiltered samples will be taken for inorganics (metals) analyses. The samples will be filtered through an in-line 0.45-µM filter (preferred method), or by gravity through a 0.45-µM membrane placed in a filter funnel. Use forceps to place the membrane into the funnel and pour sample through funnel until appropriate volumes have been filtered.

If necessary, due to slow filtering, a peristaltic pump may be used to filter the sample through an in-line filter. Connect the pump to the generator, attach tygon tubing to the bottom discharge valve on the bailer. Start pump and collect sample from the end of the in-line filter directly into the proper container, preserved, and placed in the cooler. Filtered samples will be preserved in the field with acid to a pH of less than 2. Make sure sample bottle is labeled and the cap is on tightly. Then place in cooler with ice immediately.

— OR —

If a low flow pump is used collect the samples, filtered samples will be taken by installing a 0.45-µM filter in-line and pumping the water through the filter. Collect sample from the end of the in-line filter directly into the proper container, preserved, and placed in the cooler. Filtered samples will be preserved in the field with acid to a pH of less than 2. Make sure sample bottle is labeled and the cap is on tightly. Then place in cooler with ice immediately.

Unfiltered samples will be collected by slowly pouring the sample water into the appropriate sample container, being careful not to agitate or cause bubbles to form. Do not overfill bottles. Make sure sample bottle is labeled and the cap is on tightly, then place the sample in cooler with ice immediately.

All samples will be delivered to the laboratory as soon as possible. If possible, samples will be shipped on the same day as they are collected. If samples must be retained due to weekend sampling (Friday through Sunday), the laboratory will be notified as to the time sensitive nature of the samples.

3.4 SAMPLING OF NON-AQUEOUS PHASE LIQUIDS

If NAPLs are detected in the well, a sample from all layers must be collected prior to any purging activities. NAPLs may be indicated by the presence of volatiles in the well headspace, and confirmed by the oil/water interface probe.

Collecting light non-aqueous phase liquid (LNAPL) will be accomplished using a transparent bailer with a double check valve. This bailer will be slowly lowered until the bottom of the bailer is 1-2 in. below the LNAPL-water interface, then slowly withdrawn. Verify that the interface was sampled by visual inspection of the bailer contents through the side of the bailer. Measure the thickness of the LNAPL in the bailer and note in the Field Logbook. Sample for laboratory analysis. An additional field verification may be performed by decanting the remainder of the contents of the bailer into a glass jar, adding a hydrophobic dye such as Sudan IV, or Redoil, shaking the sample and looking for coloration of NAPL. Alternate field tests are: examine the sample under ultraviolet light (many fluoresce), or allow the sample to stand overnight, and examine for interface and/or volatiles in the headspace the following day. Refer to following sections on purging and sample collection for setup and general operation.

Collecting dense non-aqueous phase liquids (DNAPLs) will be accomplished using a transparent bailer with a double check valve. The bailer must be lowered very slowly to the bottom of the well and raised slowly out of the well in a controlled fashion. Sample for analysis as above. The same field check described above may be employed for DNAPL. Refer to following sections on purging and sample collection for set up and general operation.

If NAPLs are present in the well, **and** a low-flow pump is to be used for purging and sampling, the well will be allowed to re-equilibrate prior to purging and sampling. This will be accomplished by allowing the well to stand undisturbed for at least 8 hours prior to purging and sample collection.

3.5 WELL PURGING GENERAL RULES

Water within the casing of a well will stagnate, degas, lose volatiles, possibly precipitate metals due to changes in redox potential, and may react with the screen and/or casing material. It is, therefore, necessary to purge a sufficient volume of this stagnant water from the well and/or casing to ensure that a representative sample of formation water can be obtained. Traditionally, the volume of water to be purged was arbitrarily set at 3-5 equivalent volumes. Recent advances in sampling technologies have caused a re-thinking of such arbitrary purge volumes. It is for this reason that monitoring of select chemical and physical properties of the sample medium will be used instead of strict volumes to determine when a representative sample may be taken from a well.

Acceptable purge/sampling devices include: bailers, high-discharge submersible pumps (purge only), and variable speed, low-flow pumps which include both submersible pumps (purge and sample) and dedicated bladder pumps (purge and sampling). It is recommended to purge and sample at similar rates with one type device per well. An acceptable exception to this general rule is to use a high-discharge submersible pump to purge a deep, fast-recharging well, and a bailer to sample the same well.

Peristaltic, gas-lift, and centrifugal pumps can cause volatilization, produce high pressure differentials, and can result in variability in the analysis of some analytes of interest. These types of pumps will not be used to purge or sample wells.

To prevent groundwater from cascading down the sides of the screen into an open hole, thereby aerating the sample, purge rates will closely match recharge rates. If the static water level is within the casing, the initial purge rates may be set high enough to lower the water level to the top of the screen, then reduced to maintain that level.

Purging will be accomplished with either a submersible pump, a low-flow (submersible or bladder) pump, or bailer. The choice of bailer or pump will be based on depth to water table, volume to be purged, and permeability of the aquifer. If the well recharges rapidly and/or has greater than 20 gal (estimated EV) to be purged, water may be removed with a submersible pump or a low-flow pump. If the well recharges slowly and/or has less than 20 gal to be purged, water will be removed with a bailer or a low-flow pump.

Purging will be accomplished with as minimal disturbance to the surrounding formation as possible.

Purge water will be containerized onsite until analysis of samples is completed. Based on sample results, accumulated purge water will be properly disposed.

If the water level is within the screened interval and the well recharge rate is less than 0.1 L/min, purge the well using a low-flow pump as follows:

1. Draw the water down to within 1 ft of the top of the pump.

2. Allow the well to recover.

3. Check and record field parameters.

4. Repeat Steps 1 through 3 then collect samples for metals analysis only[4].

5. Note the event in the Field Logbook, and report the problem to the Project Manager. If this extremely low recharge problem consistently occurs in a given well, the well may be considered for re-development and/or replacement.

6. If adjacent wells have elevated VOC levels, additional soil gas surveys will be considered in the vicinity of the low recharge well to help determine the need for replacement.

3.6 PURGING AND SAMPLING WITH BAILERS

Bailers may be used for both purging and sampling wells if: (a) the well recharge rate is less than 4 L/min, (b) depth to the water table is less than 50 ft, and (c) less than 20 gal are to be purged (5 EV < 20 gal)[5].

4. Analyte losses due to volatilization in a drained well are too high for valid VOC sampling (McAlary and Barker 1987).

When purging with a bailer, either a polyvinyl chloride, PTFE, or stainless steel bailer may be used. The bailer will be attached to either a spool of PTFE-coated stainless steel cable or polypropylene rope. If using cable, attach it to the bailer using stainless steel cable clamps. Thoroughly decontaminate the cable after each use, prior to rewinding cable onto spool. Cable clamps and raw cable ends may serve to trap contamination. Exercise particular caution in decontaminating these areas. If using rope, attach the rope to the bailer using a bowline knot, dispense the needed length (a few feet more than the well depth) and cut the remainder away; then, at the end opposite the bailer, make a slip knot and place it around the well casing or protective posts to prevent losing the bailer and rope down the well. The polypropylene rope will be not reused; it will be properly disposed of. Either type of bailer will be repeatedly lowered gently into the well until it fills with water, removed, and the water will be discharged into an appropriate container until purging is complete. Care must be taken not to unduly agitate the water, as this tends to aerate the sample, increase turbidity, makes stabilization of required parameters difficult to achieve, and generally prolongs purging.

After purging 2 EV, obtain a sample of groundwater and measure the following stabilization parameters: temperature, conductivity, pH, turbidity, redox potential (Eh), and dissolved oxygen level at each successive half-well volume. When three of these stabilization parameters are in agreement within approximately 10 percent in three consecutive half-well volume samples, sufficient water has been purged from the well. The results of these tests should be recorded in the sampling logbook. Should these parameters not reach agreement, no more than five well volumes will be purged.

Immediately upon completion of purging, collect samples for laboratory analysis using a PTFE bailer on a PTFE-coated stainless steel cable. The bailer will be equipped with double check valve top and controlled flow bottom discharge attachments for VOC sampling (40-mL vials), and top discharge attachment for collecting larger samples (1-L bottles).

Slowly, so as not to agitate the water, lower the bailer into the well, using a spool of PTFE-coated cable. Allow bailer to fill, withdraw smoothly. Refill bailer as needed.

If the controlled flow bottom discharge attachment is used for VOC sampling, attach it to the bottom of the bailer. Using the stopcock valve on the bailer to control the flow, fill sample vials as described above in Section 3.3.

Remove check valve top and pour unfiltered sample into inorganics sample bottles.

Collect filtered samples as described in Section 3.3. Decontaminate bailer and cable.

5. These numbers are based on the following assumptions: (1) In purging, it is preferable to remove water at approximately the recharge rate; (2) 4 L/min is estimated as the approximate maximum rate at which water can be removed with a bailer from depths of 20-50 ft; and (3) 20 gal is estimated to be at the limit of the sampler's endurance, at which point fatigue and sloppiness of technique begin.

3.7 PURGING WITH PUMP, SAMPLING WITH BAILER

If the recharge rate of the well is greater than 30 L/min, or the water level is deeper than 50 ft, or more than 20 gal of purge water will be generated (5 EV > 20 gal), then purging and sampling may be accomplished using a submersible pump/bailer combination.

When purging with a pump, gradually lower the intake until it is submerged within the screened interval. Lower an electronic water level probe to the top of the screen (as determined from completion records) to the monitor water level, start pump, and slowly lower the pump as the water level continues to fall. Care should be exercised to lower the water column to the top of the screened interval (water level probe will stop beeping) but not below the top of the screen if possible. This will ensure that the stagnant layer has been removed, but should minimize the detrimental effects of over pumping the well. Secure hose(s) and/or power cord to casing and place discharge hose into the proper container, downhill and as far away from the well as possible. Determine and record the discharge rate.

Discharge rate = volume of container/time to fill container

The discharge rate will be established at approximately equal to or just greater than the well's recharge rate (determined from well development). If well development records are incomplete, recharge rate can be determined by monitoring the rise/fall of the water level within the casing as one purges the well. If the water level is static at a given pumping rate, but fluctuates up or down as pumping rate is decreased or increased, the pumping rate at which the water level is static is the recharge rate.

After purging 2 EV, obtain a sample of groundwater and measure the following stabilization parameters: temperature, conductivity, pH, turbidity, redox potential (Eh), and dissolved oxygen level at each successive half-well volume. When three of these stabilization parameters are in agreement within approximately 10 percent in three consecutive half-well volume samples, sufficient water has been purged from the well. The results of these tests should be recorded in the sampling logbook. Should these parameters not reach agreement, no more than five well volumes will be purged.

Immediately upon completion of purging, collect samples for laboratory analysis using a PTFE bailer on a PTFE-coated stainless steel cable. The bailer will be equipped with double check valve top and controlled flow bottom discharge attachments for VOC sampling (40-mL vials), and top discharge attachment for collecting larger samples (1-L bottles). Filtration of metals samples will be accomplished using either an in-line filter attached to the bottom of the bailer, or a funnel and appropriate filter (Section 3.3).

Slowly, so as not to agitate the water, lower the bailer into the well, using a spool of PTFE-coated cable. Allow bailer to fill, withdraw smoothly, fill sample containers as described in Section 3.6. Decontaminate bailer and cable in and decontaminate pump.

3.8 PURGING AND SAMPLING WITH LOW-FLOW PUMP

To obtain representative samples, subsurface disturbances should be kept to a minimum, thereby preventing sample alteration due to sampling actions. The reasoning behind the use of low-flow pumps to purge and sample monitoring wells is that these pumps minimize physical disturbance (turbulence) at the sampling point and chemical changes (aeration) in the medium. For these reasons, the low-flow pump is the preferred method for both purging and sampling in most cases. For the purposes of this SOP, "low-flow pumps" are defined as either dedicated bladder pumps or variable speed submersible pumps. Practical operational flow rates for these sampling devices range from 0.1 L/min to 30 L/min.

Low-flow pumps may be used for purging and sampling any well having recharge greater than 0.1 L/min, which is the practical lower limit of pump performance. Below that pumping rate, pump inefficiencies and/or overheating may alter the physical and chemical properties of the sample. If the pump is continuously operated at sampling rates higher than the well recharge rate, the water level will be lowered in the well, possibly allowing aeration of the sample which is unacceptable sampling procedure. Low-flow pumps are suitable for sampling wells with recharge rates lower than 0.1 L/min if precautions are taken to avoid aeration of the sample.

Low flow submersible pumps will be used as follows:

- Lower the pump into the well, slowly so as not to agitate the water, until the pump is at the mid-point of the screened interval or the mid-point of the water column if the static water table lies below the top of the screen[6]

- Attach the pump's umbilical cord (which will consist of power cord and sampling tubing) to the protective casing, or lock the cord spool so that the pump cannot move vertically in the well during sampling.

- Lower the water level probe into the well behind the pump until it just touches water. This will allow the sampler to monitor the water level while purging and sampling, and prevent the inadvertent drying of the well.

6. This assumes a 10-ft screened interval. If the screened interval is greater than 10 ft, multiple samples should be taken as follows:
   - If the screen is 10-12 ft, sample the canter of the water column, as outlined above.
   - If the screen is longer than 12 ft, and the water column is 10 ft or less, sample the center of the water column.
   - If the screen is longer than 12 ft, and the water column fills the screen, or extends above the screen, sample at 1/3 and 2/3 the height of the water column, or about every 6 ft.

- Begin purging at the pump's lowest setting, then gradually increase rate[7] until the pumping rate matches the aquifer recharge rate. **If the water level is above the top of the screen**, the pumping rate may be allowed to slightly exceed recharge rate, lowering the water level to no less than 1 ft above the screen, then reduced until it matches recharge rate and purging continued. **If the water level is below the top of the screen**, always keep the purge rate lower than well's recharge rate.

- Monitor stabilization parameters listed in Section 3.6 beginning immediately, using an in-line monitoring system. Record parameters regularly, at a rate of one set of parameters per each 1-3 liters of water removed from the well. When these parameters stabilize to within 10 percent over three consecutive readings, reduce[8] flow rate to 0.1 L/min (if needed) and begin collecting VOC samples directly from the discharge line.

- If the well recharges at a rate less than 0.1 L/min, purge until the water level is even with the top of the screen, allow the well to recover, and sample immediately.

- Remove and decontaminate water level probe and pump.

4. MAINTENANCE

Refer to manufacturer's requirements for maintenance of pumps and generators.

5. PRECAUTIONS

Refer to the site-specific Health and Safety Plan for appropriate personal protective equipment.

6. REFERENCES

Garske, E.E. and M.R. Schock. 1986. An Inexpensive Flow-Through Cell and Measurement System for Monitoring Selected Chemical Parameters in Groundwater.

Gass, T.E., J.F. Barker, R. Dickhout, and J.S. Fyfe. 1991. Test Results of the Grundfos Groundwater Sampling Pump, in Proceedings of the Fifth National Symposium on Aquifer Restoration and Groundwater Monitoring.

7. Some sources indicate that the pumping rate should not exceed 1 L/min, with 0.5 L/min being preferable. The optimal purge rate is highly aquifer dependent, and may range from less than 0.5 L/min to greater than 10 L/min. The purge rate for a given well will, therefore, be a field decision, based on well development, purge, and sampling records rather than SOP mandate.
8. Sampling should occur at the same rate as purging as long as aeration of sample does not occur.

McAlary, T. A. and J.F. Barker. 1987. Volatilization Losses of Organics During Groundwater Sampling From Low Permeability Materials, in Groundwater Monitoring Review. Fall.

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Puls, R.W., R.M. Powell, D.A. Clark, and C.J. Paul. 1991. Facilitated Transport of Inorganic Contaminants in Groundwater: Part II Colloidal Transport, in EPA Environmental Research Brief. EPA/600/M-91/040. July.

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Standard Operating Procedure No. 13A

Groundwater Sampling with Passive Diffusion Bags

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Revision 0
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CONTENTS

1. SCOPE AND APPLICATION

The purpose of this Standard Operating Procedure (SOP) is to establish the protocol for collecting groundwater samples with passive diffusion bag samplers. The procedure is designed to permit the collection of representative groundwater samples for analysis of volatile organic compounds (VOCs).

2. MATERIALS

The following materials may be required:

Passive diffusion bag (PDB) samplers	Field notebook
Groundwater sampling equipment (SOP-13/SOP-48)	Decontamination supplies (SOP-05)
Electronic water level meter or interface probe (SOP-10)	Personnel protective equipment as required by the Accident Prevention Plan

Passive diffusion bag (PDB) samplers will be equivalent to those described in United States Geological Survey Water Investigations Report 01-0460, *User's Guide for Polyethylene-Based Passive Diffusion Bag Samplers to Obtain Volatile Organic Compound Concentrations in Wells* (United States Geological Survey 2001) and purchased from a certified manufacturer. A typical PDB sampler consists of a low-density polyethylene lay-flat tube closed at both ends and containing deionized water. Sampler lengths vary, but are typically 1.5 ft.

3. PROCEDURE

3.1 PASSIVE DIFFUSION BAG INSTALLATION

Prior to installation of PDB samplers, the well will be inspected for damage or evidence of tampering. Upon opening the well, VOCs will be measured with a photoionization detector instrument at the rim of the well and in the breathing zone. The depth to water and the total well depth will then be measured to the nearest hundredth of a foot using an electronic water level meter or interface probe per SOP 10.

PDB samplers will be prepared for deployment by attaching a dedicated stainless-steel weight to the bottom of the PDB and a dedicated stainless-steel cable to the top of the PDB. The assembly will be lowered into the well so that the sampler is positioned at the approximate midpoint of the well screen. The cable from which the sampler is hung will be attached to the well cap. The sampler will remain installed in the well, undisturbed, for a period of at least two weeks.

3.2 PASSIVE DIFFUSION BAG RETRIEVAL

After a minimum of two weeks, the PDB sampler will be retrieved using the attached cable. Care will be taken to not expose the sampler to heat or agitation. Upon removal from the well, the PDB sampler will be examined for evidence of algae, iron, or other coatings. If present, these will be noted in the field logbook. The bag will also be examined for tears in the membrane. If tears exist, the sample should be rejected. After the PDB sampler is removed from the weighted line, excess liquid will be removed from the outside of the bag to minimize

the potential for cross-contamination. The bag will be cut with decontaminated, stainless-steel, scissors. Groundwater samples will be collected by directly filling sample containers provided by the analytical laboratory. The remaining water in the PDB sampler will be poured into a separate container and water quality parameters will be measured and recorded using a multi-parameter water quality meter as per SOP-43.

Groundwater sample containers will be labeled, tracked via chain of custody forms, and packed and shipped to an offsite laboratory for analysis, as per SOPs 1, 2, and 4, respectively.

4. INVESTIGATIVE DERIVED WASTE

Investigative derived waste generated during groundwater sampling will be disposed of per SOP-42.

5. DECONTAMINATION

Non-dedicated sampling equipment will be decontaminated prior to use, between coring locations, and following completion of logging as per SOP 5.

6. PRECAUTIONS

Prior to collecting any samples, consult the Accident Prevention Plan for personal protective equipment required for sampling activities.

Decontaminate the sampling equipment and change gloves between samples to minimize the risk of cross-contamination.

Static water levels should be measured prior to PDB emplacement.

7. REFERENCES

United States Geological Survey. 2001. Water Investigations Report 01-0460, *User's Guide for Polyethylene-Based Passive Diffusion Bag Samplers to Obtain Volatile Organic Compound Concentrations in Wells*.

Standard Operating Procedure No. 015
for
Document Control System

Prepared by

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Revision 0
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1. SCOPE AND APPLICATION

The purpose of this Standard Operating Procedure is to delineate protocols for identifying and storing a complete set of documents relating to project tasks. Each document will receive a unique identification number comprised of elements describing the document.

2. MATERIALS

Not applicable.

3. PROCEDURE

Each project-related document will be given to the Document Control Officer. The Document Control Officer will record information for each document on a Document Control Sheet which will be retained as a backup record. The information from each Document Control Sheet will be maintained in a computer database.

The individual Document Control Number will be entered on the Document Log Sheet and will be written on the document.

The storage location for each document will be recorded on the Document Control Log Sheet and the documents will be stored in the recorded location.

The database file will be backed up on a regular basis to prevent accidental loss of the data.

4. MAINTENANCE

Not applicable.

5. PRECAUTIONS

None.

6. REFERENCES

None.

Standard Operating Procedure No. 016
for
Surface Water, Groundwater, and Soil/Sediment Field Logbooks

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1. SCOPE AND APPLICATION

The purpose of this Standard Operating Procedure (SOP) is to delineate protocols for recording surface water, groundwater, soil/sediment sampling information, instrument calibration data, and data from hydrologic testing in the field logbooks. Acceptable field logbooks are: bound, unprinted books such as a surveyor's field book, or a federal supply service No. 7530-00-222-3525 record book (or equivalent); or they may be company-proprietary, pre-printed forms bound into a field logbook. Example forms are provided herein. Alternate, equivalent forms are acceptable.

2. MATERIALS

The following material may be required: applicable field logbook and indelible ink pen.

3. PROCEDURE

Information pertinent to soil/sediment, groundwater, or surface water sampling will be recorded in the appropriate logbook. Each page/form of the logbook will be consecutively numbered. Entries will be made in indelible ink. Corrections will consist of line-out deletions that are initialed and dated. If using carbon paper or self-duplicating forms, before entering data in logbook, insert a sheet protector between form sets to isolate first blank form from remaining forms.

3.1 SOIL/SEDIMENT LOGBOOK (Requires Figures SOP016-1 and SOP016-3)

3.1.1 Field Parameter Form (Items on Figures SOP016-1 and SOP016-2)

1. HIGH CONCENTRATION EXPECTED?: Answer "Yes" or "No."

2. HIGH HAZARD?: Answer "Yes" or "No."

3. SITE: Record the complete name of the site.

4. AREA: Record the area designation of the sample site.

5. INST CODE: Record the 2-letter installation code appropriate for the installation or site. Correct abbreviations can be found on Pages 3-6 of the IRDMS User's Guide for chemical data entry.

6. FILE NAME: Record "CSO" for a soil sample or "CSE" for a sediment sample.

7. SITE TYPE: Record the abbreviation appropriate for where the sample was taken. Correct abbreviations can be found on Pages 18-21 of the IRDMS User's Guide for chemical data entry. This entry must match the Site Type on the map file form.

8. SITE ID: Record a code up to 10 characters or numbers which is unique to the site.

9. FIELD SAMPLE NUMBER: Record a code specific for the sample.

10. DATE: Enter the date the sample was taken.

11. TIME: Enter the time (12-hour or 24-hour clock acceptable as long as internally consistent) the sample was taken.

12. AM PM: Circle "AM" or "PM" to designate morning or afternoon (12-hour clock).

13. SAMPLE PROG: Record "GQA" (Groundwater Quality Assessment) or other appropriate sample program.

14. DEPTH (TOP): Record the total depth sampled.

15. DEPTH INTERVAL: Record the intervals at which the plug will be sampled.

16. UNITS: Record the units of depth (feet, meters)

17. SAMPLE MEASUREMENTS: Check the appropriate sampling method.

18. CHK: Check off each container released to a laboratory.

19. ANALYSIS: Record the type of analysis to be performed on each sample container.

20. SAMPLE CONTAINER: Record the sample container type and size.

21. NO.: Record the number of containers.

22. REMARKS: Record any remarks about the sample

23. TOTAL NUMBER OF CONTAINERS FOR SAMPLE: Record the total number of containers.

24. SITE DESCRIPTION: Describe the location where the sample was collected.

25. SAMPLE FORM: Record the form of the sample (i.e., clay, loam, etc.) using The Unified Soil Classification System.

26. COLOR: Record the color of the sample as determined from standard Munsell Color Charts.

27. ODOR: Record the odor of the sample or "none." See SOP No. 001 Section 5.

28. PID (HNu): Record the measured PID (HNu) values.

29. UNUSUAL FEATURES: Record anything unusual about the site or sample.

30. WEATHER/TEMPERATURE: Record the weather and temperature.

31. SAMPLER: Record your name.

3.1.2 Map File Form (Figure SOP016-3)

1. The map file logbook form will be located on the reverse of the field parameter logbook form, or on an adjoining page of the field logbook (if level book is used).

2. SITE ID: Record the Site ID from the field parameter form.

3. POINTER: Record the field sample number for the sample being pointed to.

4. DESCRIPTION/MEASUREMENTS: Describe the location where the sample was taken, along with distances to landmarks.

5. SKETCH/DIMENSIONS: Diagram the surroundings and record the distances to landmarks.

6. MAP REFERENCE: Record which U.S. Geological Survey Quad Map references the site.

7. COORDINATE DEFINITION: Write the compass directions the X- and Y-Coordinates of the map run.

8. COORDINATE SYSTEM: Write "UTM" (Universal Transverse Mercator).

9. SOURCE: Record the 1-digit code representing the Map Reference.

10. ACCURACY: Give units (e.g., write "1-M" for 1 meter).

11. X-COORDINATE: Record the X-Coordinate of the sample site location.

12. Y-COORDINATE: Record the Y-Coordinate of the sample site location.

13. UNITS: Record the units map sections are measured in.

14. ELEVATION REFERENCE: Record whether topography was determined from a map or a topographical survey.

15. ELEVATION SOURCE: Record the 1-digit code representing the elevation reference.

16. ACCURACY: Record the accuracy of the map or survey providing the topographical information.

17. ELEVATION: Record the elevation of the sampling site.

18. UNITS: Write the units in which the elevation is recorded.

19. SAMPLER: Write your name.

3.2 SURFACE WATER LOGBOOK (Requires Figures SOP016-2 and SOP016-3)

3.2.1 Field Parameter Form (Items Unique to Figure SOP016-3)

1. CAL REF: Record the calibration reference for the pH meter.

2. pH: Record the pH of the sample.

3. TEMP: Record the temperature of the sample in degrees Celsius.

4. COND: Record the conductivity of the water.

5. For all other sections, see Section 3.2.1.

3.3 GROUNDWATER SAMPLING LOGBOOK (Requires Figures SOP016-2, SOP016-3, and SOP016-4)

3.3.1 Field Parameter Form (Items on Figure SOP016-4)

1. WELL NO. OR ID: Record the abbreviation appropriate for where the sample was taken. Correct abbreviations can be found on Pages 18-21 of the IRDMS User's Guide for chemical data entry.

2. SAMPLE NO.: Record the reference number of the sample.

3. WELL/SITE DESCRIPTION: Describe the location where the sample was taken, along with distances to landmarks.

4. X-COORD and Y-COORD: Record the survey coordinates for the sampling site.

5. ELEV: Record the elevation where the sample was taken.

6. UNITS: Record the units the elevation was recorded in.

7. DATE: Record the date in the form MM/DD/YY.

8. TIME: Record the time, including a designation of AM or PM.

9. AIR TEMP.: Record the air temperature, including a designation of C or F (Celsius or Fahrenheit).

10. WELL DEPTH: Record the depth of the well in feet and inches.

11. CASING HT.: Record the height of the casing in feet and inches.

12. WATER DEPTH: Record the depth (underground) of the water in feet and inches.

13. WELL DIAMETER: Record the diameter of the well in inches.

14. WATER COLUMN HEIGHT: Record the height of the water column in feet and inches.

15. SANDPACK DIAM.: Record the diameter of the sandpack. Generally, this will be the same as the bore diameter.

16. EQUIVALENT VOLUME OF STANDING WATER: Use one of the following equations, to determine one equivalent volume (EV):

    1 EV = Volume in casing + volume in saturated sand pack. Or to restate:

    $1\ EV = (BR_w^2 h_w + 0.30B(R_s^2 - R_w^2)h_s) * (0.0043)$

    where
    R_s = Radius of sandpack in inches
    R_w = Radius of well casing in inches
    h_s = Height of sandpack in inches
    h_w = Water depth in inches

    $0.0043 = gal/in.^3$
    and filter pack porosity is assumed as 30 percent

    — OR —

    Volume in casing = $(0.0043\ gal/in.^3)(B)(12\ in./ft)(R_c^2)(W_h)$

    where
    R_c = Radius of casing in inches
    W_h = Water column height in feet

    Vol. in sandpack = $(0.0043\ gal/in.^3)(B)(12\ in./ft)(R_b^2 - R_c^2)(W_h)(0.30)$

    (if W_h is less than the length of the sandpack),

— PLUS —

Vol. in sandpack = $(0.0043 \text{ gal/in.}^3)(B)(12 \text{ in./ft})(R_b^2 - R_c^2)(S_h)(0.30)$

(if W_h is greater than the length of the sandpack).

where
R_b = Radius of the borehole
S_h = Length of the sandpack.

Show this calculation in the comments section.

17. VOLUME OF BAILER OR PUMP RATE: Record bailer volume or pump rate.

18. TOTAL NUMBER OF BAILERS OR PUMP TIME: Record the number of bailers required to remove 3 equivalent volumes (EV) of water from the well or the total purge time and volume as applicable.

19. WELL WENT DRY? Write "YES" OR "NO."

20. NUMBER OF BAILERS OR PUMP TIME: Record the number of bailers or pump time which made the well go dry.

21. VOLUME REMOVED: Record the volume of water (gal) removed before the well went dry.

22. RECOVERY TIME: Record the time required for the well to refill.

23. PURGE AGAIN?: Answer "YES" or "NO."

24. TOTAL VOL. REMOVED: Record the total volume of water (in gal) removed from the well.

25. CAL REF.: Record the calibration reference for the pH meter.

26. TIME: Record time started (INITIAL T[0]), 2 times DURING the sampling and the time sampling ended (FINAL).

27. pH: Record the pH at start of sampling (INITIAL), twice DURING the sampling and at the end of sampling (FINAL).

28. TEMP: Record the water temperature (Celsius) at the start of sampling, twice DURING the sampling and at the end of sampling (FINAL).

29. COND: Record the conductivity of the water at the start of sampling, twice DURING the sampling and at the end of sampling (FINAL).

30. D.O.: Record the dissolved oxygen level in the water at the start of sampling, twice DURING the sampling and at the end of sampling (FINAL).

31. TURBIDITY: Record the readings from the turbidity meter (nephelometer) and units at the start of sampling, twice DURING the sampling and at the end of sampling (FINAL).

32. ORD: Record the oxidation/reduction (RedOx) potential of the water sample at the start of sampling, twice DURING the sampling and at the end of sampling (FINAL).

33. HEAD SPACE: Record any positive readings from organic vapor meter reading taken in well headspace prior to sampling.

34. NAPL: Record the presence and thickness of any non-aqueous phase liquids (light or dense)

35. COMMENTS: Record any pertinent information not already covered in the form.

36. SIGNATURE: Sign the form.

3.4 FIELD CALIBRATION FORMS (Maintained as a separate logbook, or incorporated into sampling logbooks)

3.4.1 Items on Figure SOP016-5

1. Record time and date of calibration. Note whether 12- or 24-hour clock was used.

2. Record calibration standard reference number.

3. Record meter I.D. number

4. Record initial instrument reading, recalibration reading (if necessary), and final calibration reading on appropriate line.

5. Record value of reference standard (as required).

6. COMMENTS: Record any pertinent information not already covered on form.

7. SIGNATURE: Sign form.

3.5 GROUNDWATER HYDROLOGY TESTS LOGBOOK (Must include Figures SOP016-6 and SOP016-7 and/or SOP016-8, OR SOP016-9 or SOP016-10)

3.5.1 Field Permeability Test Data Sheet (Items on Figures SOP016-6)

1. CONTRACTOR: Organization performing the test.

2. SEQ. #: Enter page number of this set of forms (page # of #).

3. PROJECT NAME: Record the name assigned by the contractor's organization to the project.

4. PROJECT NO.: Record the contractor assigned project number or the contract number.

5. LOCATION: Specific location

6. CLIENT: Agency or company with the contract under which the work is being performed.

7. FIELD PARTY CHIEF: Printed name of the person responsible for this particular field test.

8. WELL #: Record the well number as it appears on the well completion tag, affixed to the protector casing or well completion records.

9. TEST TYPE: Short description of the type of test to be performed.

10. RISING/FALLING HEAD WITH SLUG: Check if the test involved the insertion/removal of and inert object.

11. RISING/FALLING HEAD WITHOUT SLUG: Check if the test involved the addition/removal of a quantity of water.

12. START DATE: Date on which the test was begun.

13. CLOCK TIME: Time each datum (depth to groundwater level) is collected. Note whether 12- or 24-hour clock was used.

14. ELAPSED TIME: Time since the last datum was collected.

15. DEPTH TO GWL (ft): Depth to the top of the groundwater table (Groundwater Level) as measured by manual methods.

16. REC. (ft): Water level as reported by transducer/datalogger (this is the depth of water above the transducer.

17. TIME: Time the discharge rate check was begun (addition or removal of water method). Note whether 12- or 24-hour clock was used.

18. FLOW METER (Addition or removal of water method): The amount of water added or removed as registered by the flowmeter, in gal of liters.

19. DISCHARGE RATE: Flowmeter reading divided by time interval (gal/min or liters/min).

20. SIGNATURE: The person completing this form must sign the form at the end of the test.

21. DATE: Date the form was signed.

3.5.2 Groundwater Levels – Single Well (Items on Figure SOP016-7)

1. CONTRACTOR: Organization performing the test.

2. SEQ. #: Enter page number of this set of forms (page # of #).

3. PROJECT NO.: Record the contractor assigned project number or the contract number.

4. WELL #: Record the well number as it appears on the well completion tag, affixed to the protector casing or well completion records.

5. PROJECT NAME: Record the name assigned by the contractor's organization to the project.

6. LOCATION: Specific location.

7. FIELD PARTY CHIEF: Printed name of the person responsible for this particular field test.

8. CLIENT: Agency with the contract under which the work is being performed.

Well Data

9. STICKUP: Enter the length of well casing extending above the average ground surface at the base of the protective casing.

10. MEASURED UP(+)/DOWN(-) FROM: Describe the starting point for the previous measurement.

11. MP ELEVATION: Enter the elevation of the measuring point here. NOTE: This datum may require reference to tables and/or maps and may be added after completing the day's field work.

12. DATUM = MSL OR: Is the datum for the previous elevation Mean Sea Level? If not, what? Also tell whether it was derived from a map elevation (write "MAP") or survey data (write "SURVEY").

13. MEASURING POINT DESCRIPTION: Describe the point used as the origin for all down-hole (water table) measurements. NOTE: Remedial investigation wells are required to have a permanently marked reference (measuring) point (refer to SOP No. 019).

14. REMARKS: Record any pertinent observations about the site/well conditions not specifically required in the preceding.

15. DATE: Date of each water level reading

16. TIME: Time of each water level reading. Note whether 12- or 24-hour clock was used.

17. ELAPSED TIME: Time since test was begun.

18. DEPTH TO WATER: Measured depth to the groundwater table.

19. WATER ELEVATION: Elevation of the top of the groundwater table (use datum listed above).

20. MEAS. METH.: Method used to measure the water level in the well (see abbreviation key at the bottom of the data sheet).

21. TAPE NO.: The unique identification number of the traceable standard tape used to calibrate the measuring device.

22. WELL STATUS: Condition of the well at the time of measuring (see abbreviation key at the bottom of the data sheet).

23. REMARKS: Any additional pertinent comments not specifically required above.

24. INITIALS: Initials of person completing this data entry.

25. ABBREVIATION KEYS: Self explanatory.

26. SIGNATURE: The person completing this form must sign the form at the end of the test.

27. DATE: Date the form was signed.

3.5.3 Groundwater Levels – Single Well (Items on Figure SOP016-8)

1. CONTRACTOR: Organization performing the test.

2. SEQ. #: Enter page number of this set of forms (page # of #).

3. PROJECT NO.: Record the contractor assigned project number or the contract number.

4. WELL #: Record the well number as it appears on the well completion tag, affixed to the protector casing or well completion records.

5. PROJECT NAME: Record the name assigned by the contractor's organization to the project.

6. LOCATION: Specific location.

7. FIELD PARTY CHIEF: Printed name of the person responsible for this particular field test.

8. CLIENT: Agency with the contract under which the work is being performed.

WELL DATA

9. STICKUP: Enter the length of well casing extending above the average ground surface at the base of the protective casing.

10. MEASURED UP(+)/DOWN(-) FROM: Describe the starting point for the previous measurement.

11. MP ELEVATION: Enter the elevation of the measuring point here. NOTE: This datum may require reference to tables and/or maps and may be added after completing the day's field work.

12. DATUM = MSL OR: Is the datum for the previous elevation Mean Sea Level? If not, what? Also tell whether it was derived from a map elevation (write "MAP") or survey data (write "SURVEY").

13. MEASURING POINT DESCRIPTION: Describe the point used as the origin for all down-hole (water table) measurements. NOTE: All Rhode Island wells are required to have a permanently marked reference (measuring) point (refer to SOP No. 019).

14. REMARKS: Record any pertinent observations about the site/well conditions not specifically required in the preceding.

15. DATALOGGER: This section is record of pertinent datalogger information.

16. MANUFACTURER: Record the manufacturer/brand name as stated on the datalogger.

17. MODEL: Enter the model number of the datalogger.

18. S/N: Enter the serial number of this datalogger.

19. TAG PROGRAMMED IN LOGGER: What is the identifier used in the datalogger's program to indicate that this unit was used to record a given data set?

20. TRANSDUCER: This section is a listing of pertinent information about the transducer used.

21. MANUFACTURER: Record the manufacturer/brand name as stated on the transducer.

22. MODEL: Enter the model number of the transducer.

23. S/N: Enter the serial number of this transducer.

24. INPUT/UNITS: What are the units this transducer uses?

25. RANGE: Record the pressure or depth range over which this transducer is certified.

CALIBRATION

26. PRESSURE RATING: This is taken from the manufacturer's specifications for a given transducer. (Usually in psi, or kpa).

27. "SUBMERGENCE = ___ (V) / (MV)": Record the voltage returned by the transducer at a given depth of submergence. Indicate whether the reading is in volts (v), or millivolts (mv).

28. VOLUME WATER ADDED/REMOVED: (Applicable if inert object insertion/removal method was not employed.) Record the volume of water added to or removed from the well.

29. DISCHARGE RATE: If z (above) is filled, enter the rate at which this water was added or removed.

30. INITIAL WATER LEVEL (ft): Enter the water level in the well at the beginning of the test.

31. PRESSURE TRANSDUCER SUBMERGENCE: Record the depth to which the transducer is submerged at the beginning of the test and the depth to the transducer at the end if the test. All depths will be recorded to the nearest 0.01 ft.

32. TIME: Record the time the test is begun and ended. Note whether 12- or 24-hour clock was used.

33. OBSERVED CHANGES IN ADJACENT WELLS: Note any changes in water levels in nearby wells.

34. RESULTS RECORDED ON DISKETTE #: Tracking number of the diskette on which these data are archived.

35. DISKETTE FILE NAME: Name of the file(s).

36. SIGNATURE: The person completing this form must sign the form at the end of the test

37. DATE: Date the form was signed.

3.6 GROUNDWATER LEVELS – MULTIPLE WELLS (Items on Figure SOP016-9)

1. CONTRACTOR: Organization performing the test.

2. SEQ. #: Enter page number of this set of forms (page # of #).

3. PROJECT NO.: Record the contractor assigned project number or the contract number.

4. PROJECT NAME: Record the name assigned by the contractor's organization to the project.

5. LOCATION: Specific location.

6. FIELD PARTY CHIEF: Printed name of the person responsible for this particular field test.

7. CLIENT: Agency with the contract under which the work is being performed.

8. REMARKS: Any pertinent observations not specifically required above.

9. WELL: Record the well number as it appears on the well completion tag, affixed to the protector casing or well completion records.

10. DATE: Date this measurement was made.

11. TIME: Time this measurement was made. Note whether 12- or 24-hour clock was used.

12. DEPTH TO WATER: Depth from MP to top of groundwater table.

13. STICKUP: Enter the length of well casing extending above the average ground surface at the base of the protective casing.

14. MP ELEV.: Enter the elevation of the measuring point here. NOTE: This datum may require reference to tables and/or maps and may be added after completing the day's field work.

15. MEAS. METH.: Method used to measure the water level in the well (see abbreviation key at the bottom of the data sheet).

16. REMARKS/MP: Describe the location and nature of the measuring point.

17. INITIALS: Initials of the person completing this form.

18. ABBREVIATION KEYS: Self explanatory.

19. SIGNATURE: The person completing this form must sign the form at the end of the test.

20. DATE: Date the form was signed.

3.7 GROUNDWATER LEVELS – DATALOGGERS (Items on Figure SOP016-10)

1. CONTRACTOR: Organization performing the test.

2. SEQ. #: Enter page number of this set of forms (page # of #).

3. PROJECT NO.: Record the contractor assigned project number or the contract number.

4. WELL #: Record the well number as it appears on the well completion tag, affixed to the protector casing or well completion records.

5. PROJECT NAME: Record the name assigned by the contractor's organization to the project.

6. LOCATION: Specific location.

7. FIELD PARTY CHIEF: Printed name of the person responsible for this particular field test.

8. CLIENT: Agency with the contract under which the work is being performed.

WELL DATA

9. STICKUP: Enter the length of well casing extending above the average ground surface at the base of the protective casing.

10. MEASURED UP(+)/DOWN(-) FROM: Describe the starting point for the previous measurement.

11. MP ELEVATION: Enter the elevation of the measuring point here. NOTE: This datum may require reference to tables and/or maps and may be added after completing the day's field work.

12. DATUM = MSL OR: Is the datum for the previous elevation Mean Sea Level? If not, what? Also tell whether it was derived from a map elevation (write "MAP") or survey data (write "SURVEY").

13. MEASURING POINT DESCRIPTION: Describe the point used as the origin for all down-hole (water table) measurements. NOTE: All Rhode Island wells are required to have a permanently marked reference (measuring) point (refer to SOP No. 019, Section 3.4).

14. REMARKS: Record any pertinent observations about the site/well conditions not specifically required in the preceding.

DATALOGGER (This section is a record of pertinent datalogger information)

15. MANUFACTURER: Record the manufacturer/brand name as stated on the datalogger.

16. MODEL: Enter the model number of the datalogger.

17. S/N: Enter the serial number of this datalogger.

18. TAG PROGRAMMED IN LOGGER: What is the identifier used in the datalogger's program to indicate that this unit was used to record a given data set?

TRANSDUCER (This section is a listing of pertinent information about the transducer used)

19. MANUFACTURER: Record the manufacturer/brand name as stated on the transducer.

20. MODEL: Enter the model number of the transducer.

21. S/N: Enter the serial number of this transducer.

22. INPUT/UNITS: What are the units this transducer uses?

23. RANGE: Record the pressure or depth range over which this transducer is certified.

CALIBRATION

24. PRESSURE RATING: This is taken from the manufacturer's specifications for a given transducer (usually in psi, or kpa).

25. "SUBMERGENCE = ___ (V) / (MV)": Record the voltage returned by the transducer at a given depth of submergence. Indicate whether the reading is in volts (v), or millivolts (mv).

26. DATE: Date of each water level reading

27. TIME: Time of each water level reading. Note whether 12- or 24-hour clock was used.

28. LOGGING TIME INTERVAL: Time since test was begun.

29. WL FEET BELOW MP: Measured depth to the groundwater table from measuring point.

30. SUBMERGENCE: Depth of water above the transducer.

31. MEAS.METHOD: What device/method was used to measure the water level.

32. TAPE NO.: Record the tape identification number.

33. TRANSDUCER MOVED?: Was the transducer moved since the last water level reading?

34. REMARKS: Any pertinent remarks not otherwise specified.

35. INITIALS:

DATA TRANSFER TO DISKETTE:

36. DATE: Date data were archived onto diskette.

37. TIME: Time stamp the computer assigns the data file.

38. FILE NAME: Name assigned the data file.

39. SOFTWARE USED FOR TRANSFER: Any special software, or computer operating system used to write the files to diskette. NOTE: If a "shareware" archiver which compresses files was used, and the archived file is not self-extracting, a copy of the unarchive program should be copied onto the diskette also.

40. OUTPUT FORMAT: What is the format of the output file? (DOS, UNIX, Binary, Compressed?)

41. INITIALS: Initials of the person who copied the data to diskette.

42. ABBREVIATION KEY: Self-explanatory.

4. MAINTENANCE

Not applicable.

5. PRECAUTIONS

None.

6. REFERENCES

U.S. Environmental Protection Agency. 1984. User's Guide to the Contract Laboratory Program. July.

FIGURE SOP016-1
FIELD PARAMETER LOGBOOK
SOIL AND SEDIMENT SAMPLES

HIGH CONCENTRATION EXPECTED? HIGH HAZARD?

INSTALLATION/SITE _____ AREA _____

INST CODE FILE NAME _____

SITE TYPE SITE ID
FIELD SAMPLE NUMBER

DATE (MM/DD/YY) / / TIME AM PM SAMPLE PROG.

DEPTH (TOP) DEPTH INTERVAL UNIT _____

SAMPLING METHOD:

SPLIT SPOON AUGER SHELBY TUBE SCOOP OTHER

CHK	ANALYSIS	SAMPLE CONTAINER	NO.	REMARKS

TOTAL NUMBER OF CONTAINERS FOR SAMPLE

DESCRIPTION OF SITE AND SAMPLE CONDITIONS
SITE DESCRIPTION:_____
SAMPLE FORM _____ COLOR _____ ODOR _____
PID (HNu) _____ UNUSUAL FEATURES _____
_____
WEATHER/TEMPERATURE
SAMPLER

HIGH CONCENTRATION EXPECTED? HIGH HAZARD?

FIGURE SOP016-2
FIELD PARAMETER LOGBOOK
GROUNDWATER AND SURFACE WATER SAMPLES

INSTALLATION/SITE AREA
INST CODE FILE NAME SITE TYPE
SITE ID FIELD SAMPLE NUMBER
DATE (MM/DD/YY) / / TIME AM PM SAMPLE PROG.
DEPTH (TOP) DEPTH INTERVAL UNITS

SAMPLING MEASUREMENTS

CAL REF.	pH	TEMPERATURE C	CONDUCTIVITY	OTHER

CHK	ANALYSIS	SAMPLE CONTAINER	NO.	REMARKS

TOTAL NUMBER OF CONTAINERS FOR SAMPLE

DESCRIPTION OF SITE AND SAMPLE CONDITIONS
SITE DESCRIPTION
SAMPLING METHOD
SAMPLE FORM COLOR ODOR
PID (HNu)
UNUSUAL FEATURES
WEATHER/TEMPERATURE _____ SAMPLER _____

FIGURE SOP016-3
MAP FILE LOGBOOK

SITE ID POINTER _____
DESCRIPTION/MEASUREMENTS
SKETCH/DIMENSIONS:

MAP REFERENCE
COORDINATE DEFINITION (X is Y is)
COORDINATE SYSTEM SOURCE ACCURACY
X-COORDINATE Y-COORDINATE UNITS
ELEVATION REFERENCE
ELEVATION SOURCE ACCURACY ELEVATION
UNITS

SAMPLER

FIGURE SOP016-4
MAP FILE AND PURGING LOGBOOK
GROUNDWATER SAMPLES

WELL COORD. OR ID SAMPLE NO._____
WELL/SITE
DESCRIPTION_____
_____
X-COORD. Y-COORD. _____ ELEV. UNITS
DATE ____/____/____ TIME AIR TEMP.

WELL DEPTH _____ ft _____ in. CASING HT. _____ ft _____ in.
WATER DEPTH _____ ft _____ in. WELL DIAMETER _____ in.
WATER COLUMN HEIGHT _____ ft _____ in. SANDPACK DIAM. _____ in.
EQUIVALENT VOLUME OF STANDING WATER _____ (gal) (L)
VOLUME OF BAILER _____ (gal) (L) or PUMP RATE _____ (gpm) (lpm)
TOTAL NO. OF BAILERS (5 EV)_____ or PUMP TIME _____ MIN.
WELL WENT DRY? [Yes] [No] NUM. OF BAILERS _____ or PUMP TIME_____MIN
VOL. REMOVED _____ (gal) (L) RECOVERY TIME _____ MIN
PURGE AGAIN? [Yes] [No] TOTAL VOL. REMOVED _____ (gal) (L)

Date and Time	Quantity Removed	Time Required	pH	Cond	Temp	ORD	Turb	DO	Character of water (color/ clarity/odor/partic.)
(before)									
(during)									
(during)									
(during									
(after)									

COMMENTS:_____
_____
_____
_____

_____
SIGNATURE

FIGURE SOP016-5
FIELD CALIBRATION: pH, CONDUCTIVITY, TEMPERATURE, TURBIDITY, OXIDATION-REDUCTION POTENTIAL, AND DISSOLVED OXYGEN METERS

INITIAL CALIBRATION	FINAL CALIBRATION
DATE:	DATE:
TIME:	TIME:

pH METER CALIBRATION

CALIBRATION STANDARD REFERENCE NO: _____

METER ID _____

pH STANDARD	INITIAL READING	RECALIB. READING	FINAL READING
7.0			
10.0			
4.0			

CONDUCTIVITY METER CALIBRATION

CALIBRATION STANDARD REFERENCE NO: _____

METER ID _____

COND. STANDARD	INITIAL READING	RECALIB. READING	FINAL READING

TEMPERATURE METER CALIBRATION

METER ID _____

TEMP. STANDARD	INITIAL READING	RECALIB. READING	FINAL READING
ICE WATER			
BOILING WATER			
OTHER			

FIGURE SOP016-5 *(continued)*

TURBIDITY METER CALIBRATION

CALIBRATION STANDARD REFERENCE NO: _____

METER ID _____

STANDARD	INITIAL READING	RECALIB. READING	FINAL READING

ORD METER CALIBRATION

CALIBRATION STANDARD REFERENCE NO: _____

METER ID _____

STANDARD	INITIAL READING	RECALIB. READING	FINAL READING

DISSOLVED OXYGEN METER CALIBRATION

CALIBRATION STANDARD REFERENCE NO: _____

METER ID _____

STANDARD	INITIAL READING	RECALIB. READING	FINAL READING

COMMENTS:_____

_____

_____

_____

_____

SIGNATURE

FIGURE SOP016-6
FIELD PERMEABILITY TEST DATA SHEET

Contractor: **Seq. #** /

Project Name:	Project #:
Location:	Client:
Party Chief:	Contractor:
Observation Well:	
Test Type: Rising/Falling Head w/Slug	Rising/Falling Head w/out Slug
Start Date:	Discharge Rate

Clock Time	Elapsed Time (min)	Depth to GWL (ft)	Rec (ft)	Clock Time	Elapsed Time	Depth to GWL (ft)	Rec (ft)	Time	Flow Meter	Discharge Rate

Signature: _____ **Date:** _____

FIGURE SOP016-7
GROUNDWATER LEVELS – SINGLE WELL

Contractor: **Seq. #** /

Project No.:
Project Name:
Field Party Chief:

WELL DATA:

Stickup: (ft)
MP Elevation:
Well No.: Site: Area:
Site: Area:
Area:

up (+)/down (-) from: Datum = MSL or:
Datum = MSL or:

Measuring Point Description:

Datalogger:
Manufacturer: Model: S/N:
Tag No. Programmed in Logger:

Transducer: Manufacturer: Model: S/N:
Input/Units: Range:

Calibration:
Pressure Rating:
0 ft submergence = (v) / (mv) ft submergence = (v) / (mv)

Volume Water Added/Removed:
Discharge Rate:
Initial Water Level (ft):

Pressure Transducer Submergence
Initial (ft): Final(ft): Time:Start: End:
Observed Changes in Adjacent Wells:

Results Recorded on Diskette #:
Diskette File Name:

Signature: _____ Date: _____

FIGURE SOP016-8
GROUNDWATER LEVELS – MULTIPLE WELLS

Contractor: **Seq. #** /

Project No.:
Project Name:
Field Party Chief:

WELL DATA:

Stickup: (ft)
MP Elevation:

Measuring Point Description:
Remarks:
Well No.:
Site: Area: up (+)/down (-) from:
up (+)/down (-) from:
up (+)/down (-) from:
Datum = MSL or:

Date	Time	Elapsed Time	Depth to Water	Water Elevation	Meas. Meth.	Tape No.	Well Status	Remarks	Initials

Measurement Method:
A = Airline
C = Chalk and tape
E = Electric tape
T = Tape with popper
X = Other (describe in remarks)

Well Status:
D = Dry
F = Flowing
P = Pumping
RP = Recently pumped
NP = Nearby well pumping
NRP = Nearby well recently
X = Obstructed

Signature: _____ Date: _____

FIGURE SOP016-8 *(continued)*

Contractor: **Seq. #** /

Project No.:
Project Name: Location:
Field Party Chief: Client:

Well	Date	Time	Depth to Water	Stickup	MP Elev.	Meas. Meth.	Tape No.	Remarks/MP	Initials

Measurement Method:
A = Airline
C = Chalk and tape
E = Electric tape
T = Tape with popper
X = Other (describe in remarks)

Well Status:
D = Dry
F = Flowing
P = Pumping
RP = Recently pumped
NP = Nearby well pumping
NRP = Nearby well recently
X = Obstructed

FIGURE SOP016-9
GROUNDWATER LEVELS DATALOGGERS

Contractor
Project No.: **Well No.:**
Project Name: **Site:**
Field Party Chief: **Area:**

WELL DATA: Stickup: (**ft**) **up (+)/down (-) from:**
MP Elevation: **Datum = MSL or:**

Measuring Point Description:
Remarks:

Datalogger:
Manufacturer: Model: S/N:
Tag No. Programmed in Logger:
Transducer: Manufacturer: Model: S/N:
Input/Units: Range:
Calibration: Pressure Rating:
0 ft submergence = (v) / (mv) ft submergence = (v)

Logging	Date	Time	Logging Time Interval	WL, ft Below MP	Submergence (logger reading)	Meas. Method	Tape No.	Well Status	Transducer Moved	Remarks	Initials
Start											
Stop											
Start											
Stop											

Data Transfer to Disk

Date	Time	File Name	Software Used for Transfer	Output Format	Initials

Measurement Method:
A = Airline
C = Chalk and tape
E = Electric tape
T = Tape with popper
X = Other (describe in remarks)

Well Status:
D = Dry
F = Flowing
P = Pumping
RP = Recently
NP = Nearby well pumping
NRP = Nearby well recently pumped
X = Obstructed

Signature **Date**

Standard Operating Procedure No. 019
for
Monitoring Well Installation

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CONTENTS

1. SCOPE AND APPLICATION

The installation of monitoring wells is contingent upon the existing conditions at the project site. The purpose of this Standard Operating Procedure is to delineate the quality control measures required to ensure the accurate installation of monitoring wells. The applicable Work Plan should be consulted for specific installation instructions. The term "monitoring wells," as used herein, is defined to denote any environmental sampling well. An example well log form is provided in Appendix A. Alternate, equivalent forms are acceptable.

2. MATERIALS

2.1 DRILLING EQUIPMENT

The following drilling equipment may be required:

- Appropriately sized drill adequately equipped with augers, bits, drill stem, etc.
- Steam cleaner and water obtained from approved source for decontaminating drilling equipment.
- Photoionization Detector: Microtip HL-200 (or equivalent)
- Water level indicator
- Weighted steel tape measure
- Lower explosive limit – oxygen monitor
- Steel drums for intrusion derived wastes (drill cuttings, contaminated personal protective equipment, decontamination solutions, etc.)
- Source of approved water
- Heavy plastic sheeting
- Sorbent pads and/or log.

2.2 WELL INSTALLATION MATERIALS[1]

The following well installation materials may be required:

- Well screen:[2]
  - — Polyvinyl chloride (PVC): JOHNSON (or equivalent); PVC 0.010 slot; Schedule 40; flush-threaded (leak-proof) joints; PVC complies with American Society for Testing and Materials (ASTM) D2665, ASTM D1784, and ASTM F480; free of ink markings; cleaned and prepackaged by manufacturer.
  - — Stainless steel: JOHNSON (or equivalent); stainless steel 0.010 slot; 304 stainless steel[3]; ASTM F480 flush threads; cleaned, wrapped, and heat sealed by manufacturer.
- Riser pipe:
  - — PVC: JOHNSON (or equivalent); STD; PVC; Schedule 40; flush-threaded (leak-proof) joints; PVC complies with ASTM D2665, ASTM D1784, and ASTM F480; free of ink markings; cleaned and prepackaged by manufacturer.
  - — Stainless steel: JOHNSON (or equivalent); Schedule 5; 304 stainless steel; ASTM Type A312 material; 4-in. diameter; cleaned, wrapped, and heat sealed by manufacturer.
- Plugs/caps: JOHNSON (or equivalent); standard PVC or stainless steel.
- Filter pack: MORIE, 100 well gravel (or equivalent). NOTE: Final gradation may vary as a function of the gradation of the formation.[2]
- Fine Ottawa sand.
- Bentonite seal: BAROID, bentonite pellets (3/8-in. diameter)
- Cement: Type II Portland Cement (table below).

1. Technical information on all installed materials (screens, riser pipe, filter pack, bentonite, cement, etc.) and representative samples of the proposed filter pack, bentonite powder, and bentonite pellets will be supplied to the Project Manager.
2. Well screen slot size and filter pack gradation will be determined from sieve analysis of aquifer materials. Screen and casing material type will be determined based on field tests of groundwater chemistry and contaminants.
3. Unless the sum of Cl-, F-, and Br- is >1,000 ppm, in which case Type 316 should be used.

Cement Type	Special Characteristics	Recommended Usage
I	No special properties	General use as grout mix or cement plug (if sulfates <250 ppm), surface pad.
IA	Air-entraining Type I (Note that air entrainment properties can be achieved by chemical admixtures)	Air entrainment gives cement greater freeze-thaw resistance. Recommended for surface pads.
II	Moderate sulfate resistance, low heat of hydration	General use as grout mix or cement plug where groundwater sulfate >250 ppm and <1,500 ppm, surface pad.
IIA	Air-entraining Type II	See Type IA.
III	High early strength, high heat of hydration	Elevated temperature can damage well casing and fracture grout/cement plugs. NOT RECOMMENDED.
IIIA	Air-entraining Type III	NOT RECOMMENDED.
IV	Low heat of hydration	General use as grout mix or cement plug preferred type for well abandonment to ensure intact grout/cement plug.
V	High Sulfate resistance	Use when groundwater sulfate levels >1,500 ppm.

- Bentonite powder: BAROID, Aquagel Gold Seal.
- Steel protective casing: BRAINARD-KILMAN (or equivalent) zinc-plated steel, lockable, painted.[4]
- Geotextile: MIRAFI (or equivalent); GTF 130; non-woven; 4 oz.
- Coarse (blanket) gravel: Crushed stone aggregate.
- Containers for purged water, as required.
- Submersible pump or bailer of appropriate capacity, and surge block sized to fit well.
- Hach DREL 2000 portable laboratory (or equivalent).
- Conductivity, pH, oxidation-reduction potential (ORP), turbidity, dissolved oxygen, and temperature meters.
- Electric well sounder and measuring tape.
- Portland Type II cement (see previous table).
- Steel Posts (pickets), painted (see footnote).

4. All painted components (protector casing, steel pickets) will be painted high-visibility orange and allowed to dry completely prior to being brought onsite.

2.3 DOCUMENTATION

The following document may be provided:

- Copy of appropriate Work Plan
- Copy of approved Health and Safety Plan
- Copies of well and excavation permits
- Boring log forms
- Well completion diagram form
- Well development form.

2.4 GEOLOGIST'S PERSONAL EQUIPMENT

The following equipment may be required for the geologist:

- 10X handlens
- Unified Soil classification System chart
- Munsell color chart
- Sieve set (Keck model SS-81 or equivalent)
- Personal protective equipment as required by the Health and Safety Plan.

3. PROCEDURE

3.1 MATERIALS APPROVAL

Water sources for drilling, grouting, sealing, filter placement, well installation, and equipment decontamination must be approved by the Project Manager prior to arrival of the drilling equipment. Information required for the water source includes: water source, manufacturer/ owner, address and telephone number, type of treatment and filtration prior to tap, time of access, cost per gallon (if applicable), dates and results associated with all available chemical analyses over the past 2 years, and the name and address of the analytical laboratory (if applicable).

Pure sodium bentonite with no additives (bentonite) will be the only drilling fluid additive allowed, and its use must be approved by the Project Manager prior to the arrival of the drilling equipment. The information required for evaluation includes: brand name, manufacturer, manufacturer's address and telephone number, product description, and intended use for the product.

Granular Filter Pack material must be approved by the Project Manager prior to drilling. A 1-pint representative sample must be supplied to the Project Manager. Information required includes: lithology, grain size distribution, brand name, source, processing method, and slot size of intended screen.

Portland Type II cement will be used for grout (see previous table).

3.2 DRILLING

The objective of the selected drilling technique is to ensure that the drilling method provides representative data while minimizing subsurface contamination, cross-contamination of aquifers, and drilling costs. The preferred drilling method is with a hollow-stem auger. Other drilling methods[5] are approved as conditions warrant, and will not require variances be issued by the U.S. Environmental Protection Agency. The method used at a specific site will be proposed in the work plan and evaluated by the Project Manager. Any drilling method not listed herein will require approval on a case by case basis by the U.S. Environmental Protection Agency.

A Site Geologist will be present during all well drilling and installation activities and will fully characterize all tasks performed in support of these activities into the monitoring well logbook. The Site Geologist will be responsible at only one operating rig for the logging of samples, monitoring of drilling operations, recording of water losses/gains and groundwater data, preparing the boring logs and well diagrams, and recording the well installation procedures of the rig. The Site Geologist will have onsite sufficient equipment in operable condition to perform efficiently his/her duties as outlined in the contractual documents. Items in the possession of each Site Geologist will include the approved Health and Safety Plan, this Standard Operating Procedure, a hand lens (10X), a standard color chart, grain-size chart, and a weighted (with steel or iron) steel tape long enough to measure the deepest well, heavy enough to reach that depth, and small enough to fit readily within the annulus between the well and drill casing. The Site Geologist will also have onsite, a water level measuring device, preferably electrical.

Only solid vegetable shortening (e.g., Crisco®) without flavoring or additives may be used on downhole drilling equipment. Additives containing either lead or copper will not be allowed. In addition, polychlorinated biphenyls will not be permitted in hydraulic fluids or other fluids used in the drilling rig, pumps, and field equipment/vehicles.

5. If the design depth of the well is <100 ft, open, hollow-stem augers will be used to drill the well unless "running sands" preclude the use of open augers. In that case, an inert "knockout" plug may be used in the bottom of the auger string. This plug will be driven out of the augers and left at the bottom of the hole when the well is installed.

   If the design depth of the well is >100 ft, rotary drilling methods may be used to install wells. The following drill fluids and methods are approved in the order listed: (1) rotary drilling with water from an approved source as drilling fluid (clays from the formations will tend thicken the fluid and coat the walls of the borehole and this is acceptable); (2) rotary drilling with water as a fluid, advancing a temporary casing with the bit to maintain an open hole; and (3) mud rotary using water with additives as drill fluid. Due to the potential for aquifer contamination and plugging, mud rotary drilling is not recommended for monitoring wells. If, however, "running sands" are encountered and the aquifer is expected to have a relatively high flow rate, then mud rotary is considered an approved method. Pure sodium bentonite is the only approved additive. Mud rotary drilling must be halted at the last aquitard above the target aquifer. Casing must be set, all bentonite-bearing fluids flushed from the hole and drill rig, and drilling may be resumed using water only as the drill fluid until the target depth is reached.

Surface runoff or other fluids will not be allowed to enter any boring or well during or after drilling/construction.

Antifreeze used to keep equipment from freezing will not contain rust inhibitors and sealants. Antifreeze is prohibited in areas in contact with drilling fluid. The ground surface at the well site will be protected from possible coolant, fuel, and hydraulic fluid spills and/or leakage by placement of plastic sheeting with raised edges, draining into a lined catch basin large enough to contain spills and/or leakage from motors, radiators, or vehicle tanks. Sorbent pillows will be placed to catch obvious leaks from the drill rig. Sorbent logs may be used instead of, or in conjunction with, a lined catch basin to contain spills.

An accurate measurement of the water level will be made upon encountering water in the borehole and later upon stabilization. Levels will be periodically checked throughout the course of drilling. Any unusual change in the water level in the hole, such as a sudden rise of a few inches may indicate artesian pressure in a confined aquifer, will be the basis for cessation of drilling. The geologist will immediately contact the Project Manager[6]. Particular attention for such water level changes will be given after penetrating any clay or silt bed, regardless of thickness, which has the potential to act as a confining layer.

Anticipated depths of wells are given in well specific work plans. In case the previously defined criteria have not been met before the depth range for a given hole is reached, the geologist will stop the drilling and confer with the Project Manager. The current boring conditions (depth, nature of the stratigraphic unit, and water table depth) will be compared to those of other wells nearby to decide to continue drilling or to terminate and complete the well.

If the well is to be installed in the surficial aquifer, drilling will be terminated before penetrating the basal aquitard. The basal aquitard is defined as the first 2 ft-thick clay below the water table, or below 5 ft in the case of a shallow aquifer.

If the well is to be installed in a lower, confined aquifer:

- Penetrations of aquifers located lower than the water table aquifer will be limited to avoid cross-contamination.

- Placement of new upper confined aquifer wells will be initially limited to those areas where contamination has been confirmed.

- The location of upper confined aquifer wells will be based upon the findings of the water table aquifer investigation. Areas of known contamination will be targeted for installing upper confined aquifer wells for the purposes of delineating vertical contamination.

6. The contract technical oversight will also be contacted for guidance.

- Where possible, upper-confined aquifer wells will be located such that they afford triangulation with other wells within the same aquifer to allow for a determination of groundwater flow direction.

- Some upper-confined aquifer wells will be installed approximately 10-15 ft from water table wells to enable the accurate assessment of vertical hydraulic gradients. If the direction of groundwater flow is known, wells within a group will be located sidegradient of each other.

- The boring will be advanced until the base of the surficial aquifer is reached (Section 3.2).

- An outer, surface casing will be set 2-5 ft into the confining layer to minimize the potential for cross-contamination from the unconfined aquifer during drilling activities.

- The surface casing will be driven into the confining bed and grouted into place. Grout will be tremied into the annulus around the outside of the casing to within 5 ft of the ground surface. A grout plug at least 2 ft thick will be tremied into the bottom of the surface casing. The grout will be permitted to cure for 24 hours. All drilling fluids within the surface calling will then be removed, and the casing will be flushed with clean potable water.

- The drilling equipment will be decontaminated, a smaller bit or auger selected, and the hole will be continued through the grout plug into the confined aquifer.

- If deeper aquifers are to be screened, repeat preceding steps until total depth is reached.

If dense non-aqueous phase liquid (DNAPL) contamination is detected during drilling, the well will be terminated and completed at the base of the aquifer. Drilling will not continue through the confining unit.

Stainless steel screens will be used in DNAPL wells. Screen size selection will be according to criteria set forth in Section 3.4. The formation grain size will be multiplied by the higher factor (6) to determine filter pack grain size. This will ensure that the filter pack is sufficiently coarse to permit DNAPL to pass freely from the formation into the coarser filter pack, then into the open well (Cohen and Mercer 1993).

DNAPL sampling cups are prohibited. The well screen will be capped, and set 0.3 ft (0.5 ft max.) into the top of the confining bed and rest on the bottom of the hole or bentonite backfill (if used). No sand will be placed below the screen. The remainder of the well installation and completion will be accomplished according to Section 3.4.

3.3 LOGGING

All borings for monitoring wells will be logged by a geologist. Logs will be recorded in a field logbook and/or a boring log. If the information is recorded in a logbook, it will be transferred to Boring Log Forms on a daily basis. Field notes are to include, as a minimum:

- Boring number
- Material description (as discussed below)
- Weather conditions
- Evidence of contamination
- Water conditions (including measured water levels)
- Daily drilling footage and quantities (for billing purposes)
- Notations on man-placed materials
- Drilling method and borehole diameter
- Any deviations from established field plans
- Blow counts for standard penetration tests
- Core and split-spoon recoveries.

Material description for soil samples must include:

- Classification
- Unified Soil Classification symbol
- Secondary components and estimated percentages
- Color
- Plasticity
- Consistency
- Density
- Moisture content
- Texture/fabric/bedding and orientation
- Grain angularity
- Depositional environment and formation
- Incidental odors
- Photoionization detector reading(s)
- Staining.

Material description for rock samples must include:

- Classification
- Lithologic characteristics
- Bedding/banding characteristics
- Color
- Hardness
- Degree of cementation
- Texture
- Structure and orientation

- Degree of weathering
- Solution or void conditions
- Primary and secondary permeability
- Sample recovery
- Incidental odors
- Photoionization detector reading(s)
- Staining.

3.4 WELL CONSTRUCTION AND INSTALLATION

After the hole is drilled and logged, backfill hole as required for proper screen placement. The integrity of the aquitard will be restored by placing a bentonite plug of an appropriate thickness, either to the top of the aquitard (normal well installation) or to within 0.3 ft of the top of the aquitard (DNAPL well). Aquifer fill will be clean filter pack.

Normal screen placement for the water table (surficial) aquifer will be within 2 ft of the screen extending above the static water level. The bottom of the screen will rest no more than 6 in. from the bottom of the hole or backfill material, whichever is applicable.

NOTE: The end cap in DNAPL wells will rest on the bottom of the bottom of the hole, or bentonite backfill if applicable (Section 3.2).

Screen placement for a confined aquifer well will normally be at the top of the confined aquifer.

Screen lengths will not normally exceed 10 ft. If it appears advantageous in a given situation (e.g., to screen an entire aquifer which is thicker than 10 ft), approval must be sought on a case-by-case basis from the appropriate regulatory agency. Otherwise, wells will be screened as follows:

Thickness of Aquifer	Action
<10 ft	Screen entire aquifer
>10 ft <30 ft	Screen top 10 ft consider vertically nested well cluster
>30 ft	Install vertically nested well cluster

The installation of monitoring wells in uncased or partially cased holes will begin within 12 hours of completion of drilling, or if the hole is to be logged, within 12 hours of well logging, and within 48 hours for holes fully cased with temporary drill casings. Once installation has begun, work will continue until the well has been grouted and the drill casing has been removed.

Well screens, casings, and fittings will conform to National Sanitation Foundation Standard 14 or ASTM equivalent for potable water usage. These materials will bear the appropriate rating logo. If the logos are not present, a written statement from the manufacturer/supplier stating that the materials contain the appropriate rating must be obtained. Material used will be new and essentially chemically inert to the site environment.

Well screen and casing should be inert with respect to the groundwater; therefore, the selection of screen and casing material will be based on select field tests of aquifer chemistry and potential contaminants. The screen will be capped without sediment trap or DNAPL sampling cup, and lowered into the hole. The well casing will be pre-cut to extend 2-2.5 ft above ground surface. Prior to placement of the last piece of well casing, a notch or other permanent reference point will be cut, filed, or scribed into the top edge of the casing.

Screen slot size will be appropriately sized to retain 90-100 percent of the filter pack material, the size of which will be determined by sieve analysis of formational material (Section 3.4).

The tops of all well casing will be capped with covers composed of materials compatible with the products used in the well installation. Caps may either be vented, or a telescopic fit, constructed to preclude binding to the well casing caused by tightness of fit, unclean surfaces, or weather conditions. In either case, it should be secure enough to preclude the introduction of foreign material into the well, yet allow pressure equalization between the well and the atmosphere.

Filter pack material will be placed, lightly tamped, and leveled. Filter pack will extend from the bottom of the hole to a height of 1-2 ft above the top of the screen. The filter pack will be capped with a minimum of 1 ft of fine (Ottawa) sand to prevent the bentonite seal from infiltrating the filter pack. If the bentonite seal is placed as a slurry, a minimum of 2 ft of fine sand will be required.

If the hole is less than 20-ft deep, the filter pack may be poured into the annulus directly. If the hole is deeper than 20 ft, the filter pack must be tremied into place.

Granular filter packs will be chemically and texturally clean, inert, and siliceous.

Filter pack grain size will be based on formation grain-size analysis. The D30 (70 percent retained) sieve size multiplied by a factor of not less than 3 nor greater than 6 will be used to determine the appropriate grain size.

Calculations regarding filter pack volumes will be entered into the Field Logbook along with any discrepancies between calculated and actual volumes used. If a discrepancy of greater than 10 percent exists between calculated and actual volumes exists, an explanation for the discrepancy will also be entered in the Field Logbook.

Bentonite seals will be no less than 2-ft thick nor more than 5-ft thick as measured immediately after placement. The normal installation will include a 5-ft seal. Thinner seals may be used in special cases. The final depth to the top of the bentonite seal will be measured and recorded.

3.4.1 Grout

Grout used in construction will be composed by weight of:

- 20 parts cement (Portland cement, type II) (see previous table)
- 0.4-1 part (maximum) (2-5 percent) bentonite
- 8-gal (maximum) approved water per 94-lb bag of cement.

Neither additives nor borehole cuttings will be mixed with the grout. Bentonite will be added after the required amount of cement is mixed with the water.

All grout material will be combined in an aboveground container and mechanically blended to produce a thick, lump-free mixture. The mixed grout will be recirculated through the grout pump prior to placement. Grout placement will be performed using a commercially available grout pump and a rigid, side discharge tremie pipe.

The following will be noted in the Field Logbook: (1) calculations of predicted grout volumes; (2) exact amounts of cement, bentonite, and water used in mixing grout; (3) actual volume of grout placed in the hole; and (4) any discrepancies between calculated and actual volumes used. If a discrepancy of greater than 10 percent exists between calculated and actual volumes exists, an explanation for the discrepancy will also be entered in the Field Logbook.

Well protective casings will be installed around all monitoring wells on the following day as the initial grout placement around the well. Any annulus formed between the outside of the protective casing and the borehole will be filled to ground surface with cement.

The construction of each well will be depicted as built in a well construction diagram. The diagram will be attached to the boring log and will graphically denote:

- Screen location, length
- Joint location
- Granular filter pack
- Seal
- Grout
- Cave-in
- Centralizers
- Height of riser
- Protective casing detail.

3.5 MONITORING WELL COMPLETION

Assemble appropriate decontaminated lengths of pipe and screen. Make sure these are clean and free of grease, soil, and residue. Lower each section of pipe and screen into the borehole, one at a time, screwing each section securely into the section below it. No grease, lubricant, polytetrafluoroethelyne tape, or glue may be used in joining the pipe and screen sections.

If a well extends below 50 ft, centralizers will be installed at 50 ft and every 50 ft thereafter except within screened interval and bentonite seal. Centralizer material will be PVC, polytetrafluoroethelyne, or stainless steel. Determination of centralizer material will be based on the same criteria as screen and calling selection.

Cut the riser with a pipe cutter approximately 2-2.5 ft above grade. All pipe cuts MUST be square to ensure that the elevation between the highest and lowest point of the well casing is less than or equal to 0.02 ft Notch, file, or otherwise permanently scribe a permanent reference point on the top of the casing.

Torches and saws may not be used to cut the riser. Care must be taken that all filings or trimmings cut from the reference point fall outside the riser rather than into the well. **Under no circumstances will a permanent marker or paint pencil be used to mark the reference point**.

In some locations, safety requirements may mandate that a well be flush-mounted with no stick-up. If a flush-mounted well is required at a given location, an internal pressure cap must be used instead of a vented cap to ensure that rainwater cannot pool around the wellhead and enter the well through the cap.

When the well is set to the bottom of the hole, temporarily place a cap on top of the pipe to keep the well interior clean.

Place the appropriate filter pack (Section 3.4). Monitor the rise annulus with a weighted tape to assure that bridging is not occurring.

After the pack is in place, wait 3-5 minutes for the material to settle, tamp and level a capped PVC pipe, and check its depth weighted steel tape.

Add a 1-2 ft cap of fine-grained (Ottawa) sand to prevent infiltration of the filter pack by overlying bentonite seal. See Section 3.4 for guidance on appropriate thickness of fine sand layer.

Install the bentonite seal (2- to 5-ft thick) by dropping bentonite pellets into the hole gradually. If the well is deeper than 30 ft, a tremie pipe will be used to place either bentonite pellets or slurry. Tamp and level pellets. If the well is 30 ft, tamp with a capped PVC pipe, if >30 ft, tamping bay be accomplished with the weighted end of the tape. In either case, check the depth to the top of the seal with a weighted tape as above.

If the bentonite pellets are of poor quality, they may have a tendency to hydrate and swell inside the tremie pipe and bridge. This situation may be solved by the following procedure:

1. Use a different brand of pellets. Different brands may have longer hydration times.

2. Freeze the pellets[7]. Note that this will require a longer wait time to allow proper hydration after the pellets thaw.

3. Place the bentonite seal as a slurry using a side-discharge tremie pipe as though installing grout. Note (Section 3.4) this will require that a minimum of 2 ft of fine sand be placed as a cap on top of the filter pack material.

Wait for the pellets to hydrate and swell. Hydration times will be determined by field test or by manufacturer's instructions. Normally this will be 30-60 minutes. Document the hydration time in the field notebook. If the pellets are above the water level in the hole, add several buckets of clean water to the boring. Document the amount of water added to the hole.

Mix an appropriate cement-bentonite slurry (Section 3.4). Be sure the mixture is thoroughly mixed and as thick as is practicable.

Lower a side discharge tremie pipe into the annulus to the level of the pellet seal.

Pump the grout slurry into the annulus while withdrawing the tremie pipe and temporary casing.

Stop the grout fill at 5 ft below the ground surface. Allow to cure for not less than 12 hours. If grout settles more than 6 in., add grout to bring level back up to within 5 ft of ground surface. Place approximately 2 ft of bentonite pellets (minimum 0.5 ft) in annulus. Seat the protective casing in the bentonite seal, allowing no more than 0.2 ft between the top of the well casing and the bottom of the protective casing cap. Fill inner annulus (between well casing and protective casing) with bentonite pellets to the level of the ground surface. Cover bentonite pellets with 1 ft of clean granular material (coarse sand or pea gravel filter pack). Fill the outer annulus (between the protective casing and the borehole) with neat cement. Allow the cement to mound above ground level and finish to slope away from the casing. Lock the cap.

— OR —

Continue the grout fill to the ground surface. Seat the protective casing in the grout, allowing no more than 0.2 ft between the top of the well casing and the bottom of the protective casing cap. Lock the cap.

— AND —

Allow the grout slurry to set overnight.

7. Bentonite pellets may be "flash-frozen" by brief immersion in liquid nitrogen (LN2). This can be accomplished by pouring LN2 over a small quantity (0.25-0.5 bucket) of pellets, allowing the LN2 to boil off, then pouring the pellets into the tremie pipe. **NOTE:** Use of LN2 is an additional jobsite hazard and must be addressed in the contractor's Health and Safety Plan. This contingency must be covered before drilling starts in order to avoid delays in well installation.

Fill the outer annulus (between the casing and the borehole) with neat cement. Allow the cement to mound above ground level and finish to slope away from the casing.

Slope the ground surface away from the casing for a distance of 2 ft, at a rate of no less than 1 in. in 2 ft. Surface this sloping pad with a geotextile mat covered by 3 in. of coarse gravel.

— OR —

Frame and pour a 4-ft square × 6-in. thick (4 ft × 4 ft × 6 in.) concrete pad centered around the protective casing.

— AND —

Set pre-painted protective steel pickets (3 or 4) evenly around and 4 ft out from well. These pickets will be set into 2 ft deep holes, the holes will then be filled with concrete; and if the pickets are not capped, they will also be filled with concrete.

3.6 WELL DEVELOPMENT

Well development is the process by which drilling fluids, solids, and other mobile particulates within the vicinity of the newly installed monitoring well have been removed while restoring the aquifer hydraulic conductivity. Development corrects any damage to or clogging of the aquifer caused by drilling, increases the porosity of the aquifer in the vicinity of the well, and stabilizes the formation and filter pack sands around the well screen.

Well development will be initiated after 48 consecutive hours but no longer than 7 calendar days following grouting and/or placement of surface protection.

Two well development techniques, over pumping and surging, will be employed in tandem. Over pumping is simply pumping the well at a rate higher than recharge. Surging is the operation of a plunger up and down within the well casing similar to a piston in a cylinder.

3.6.1 Materials Required

The following materials will be required for well development:

- Well Development Form
- Boring Log and Well Completion Diagram for the well
- Submersible pump or bailer of appropriate capacity, and surge block
- Conductivity, pH, ORP, turbidity, dissolved oxygen, and temperature meters
- Electric well sounder and measuring tape
- Containers for purged water, if required.

3.6.2 Summary of Procedures and Data Requirements

Pump or bail the well to ensure that water flows into it, and to remove some of the fine materials from the well. Removal of a minimum of one equivalent volume is recommended at this point. The rate of removal should be high enough to stress the well by lowering the water level to approximately half its original level. If well recharge exceeds 15 gpm, the requirement to lower the head will be waived.

Slowly lower a close-fitting surge block into the well until it rests below the static water level, but above the screened interval. (NOTE: This latter is not required in the case of a light non-aqueous phase liquid well.)

Begin a gentle surging motion which will allow any material blocking the screen to break up, go into suspension, and move into the well. Continue surging for 5-10 minutes, remove surge block, and pump or bail the well, rapidly removing at least one equivalent volume.

Repeat previous step at successively lower levels within the well screen until the bottom of the well is reached. Note that development should always begin above, or at the top of, the screen and move progressively downward to prevent the surge block from becoming sand locked in the well casing. As development progresses, successive surging can be more vigorous and of longer duration as long as the amount of sediment in the screen is kept to a minimum.

Development is expected to take at least 2 hours in a small well installed in a clean sand, and may last several days in large wells, or in wells set in silts with low permeabilities.

Development will continue until little or no sediment can be pulled into the well, and target values for parameters listed below are met.

At a minimum, development will remove 3-5 well volumes of water. One development volume (DV) is defined as (1) equivalent volume, plus (1) the amount of fluid lost during drilling, plus (1) the volume of water used in filter pack placement.

1. Monitor water quality parameters before beginning development procedures, and after removing 2, 2.5, and 3 well volumes of water.

2. If these parameters have stabilized over the three readings, the well will be considered developed.

3. If the parameters have not stabilized after these three readings, continue pumping the well to develop, but stop surging. Monitor the stabilization parameters every half DV.

4. When the parameters have stabilized over three consecutive readings at half DV intervals, the well will be considered developed.

All water removed must be disposed of as directed by the Work Plan.

Record all data as required on a Well Development Record Form (Appendix A), which is made a part of the complete Well Record. These data include:

- Depths and dimensions of the well, casing, and screen obtained from the well diagram.
- Water losses and uses during drilling, obtained from the boring log for the well.
- Measurements of the following indicator parameters: turbidity, pH, conductivity, ORP potential, dissolved oxygen, and temperature.
- Target values for the indicator parameters listed above are as follows: pH – stabilize, conductivity – stabilize, ORP – stabilize, dissolved oxygen –- stabilize, temperature – stabilize, turbidity – 5 nephelometric turbidity units or stabilize. A value is considered to have stabilized when three consecutive readings taken at half DV intervals are within 10 percent of each other.
- Notes on characteristics of the development water.
- Data on the equipment and technique used for development.
- Estimated recharge rate and rate/quantity of water removal during development.

4. MAINTENANCE

Not applicable.

5. PRECAUTIONS

Refer to the site-specific Health and Safety Plan for discussion of hazards and preventive measures during well development activities.

6. REFERENCES

Aller, L. et al. 1989. Handbook of Suggested Practices for the Design and Installation of Groundwater Monitoring Wells, National Water Well Association.

American Society for Testing and Materials (ASTM). D2487-92 Standard Classification of Soils for Engineering Purposes (Unified Soil Classification System).

———. D5092-90 Standard Practice for Design and Installation of Groundwater Monitoring Wells in Aquifers.

Cohen, R.M. and J.W. Mercer. 1993. DNAPL Site Evaluation, CRC Press, Inc.

Nielsen, D.M. 1993. Correct Well Design Improves Monitoring, in *Environmental Protection*, Vol.4, No.7. July.

U.S. Army Toxic and Hazardous Materials Agency. 1987. Geotechnical Requirements for Drilling, Monitoring Wells, Data Acquisition and Reports. March.

U.S. Environmental Protection Agency. 1989. Groundwater Handbook.

Appendix A

Field Record of Well Development Form

EA Engineering, P.C.
EA Science and Technology

FIELD RECORD OF WELL DEVELOPMENT

Project Name:	Project No:	Date:
EA Personnel:	Development Method:	
Weather/Temperature/Barometric Pressure:		Time:

Well No.:	Well Condition:
Well Diameter:	Measurement Reference:
Well Volume Calculations	
A. Depth To Water (ft):	D. Well Volume/ft:
B. Total Well Depth (ft):	E. Total Well Volume (gal)[C*D]:
C. Water Column Height (ft):	F. Five Well Volumes (gal):

Parameter	Beginning	1 Volume	2 Volumes	3 Volumes	4 Volumes	5 Volumes
Time (min)						
Depth to Water (ft)						
Purge Rate (gpm)						
Volume Purged (gal)						
pH						
Temperature (°F)						
Conductivity (µmhos/cm)						
Dissolved Oxygen						
Turbidity (NTU)						
ORP (mV)						

Parameter	6 Volumes	7 Volumes	8 Volumes	9 Volumes	10 Volumes	End
Time (min)						
Depth to Water (ft)						
Purge Rate (gpm)						
Volume Purged (gal)						
pH						
Temperature (°F)						
Conductivity (µmhos/cm)						
Dissolved Oxygen						
Turbidity (NTU)						
ORP (mV)						

NOTE: NTU = Nephelometric turbidity unit.
ORP = Oxidation-reduction potential.

COMMENTS AND OBSERVATIONS: _____

EA Engineering, P.C.
EA Science and Technology

FIELD RECORD OF WELL DEVELOPMENT

Project Name:	Project No:	Date:
EA Personnel:	Development Method:	
Weather/Temperature/Barometric Pressure:		Time:

Well No.:	Well Condition:
Well Diameter:	Measurement Reference:

Parameter	Beginning	1 Volume	2 Volumes	3 Volumes	4 Volumes	5 Volumes
Time (min)						
Depth to Water (ft)						
Purge Rate (gpm)						
Volume Purged (gal)						
pH						
Temperature (°F)						
Conductivity (µmhos/cm)						
Dissolved Oxygen						
Turbidity (NTU)						
ORP (mV)						

Parameter	6 Volumes	7 Volumes	8 Volumes	9 Volumes	10 Volumes	End
Time (min)						
Depth to Water (ft)						
Purge Rate (gpm)						
Volume Purged (gal)						
pH						
Temperature (°F)						
Conductivity (µmhos/cm)						
Dissolved Oxygen						
Turbidity (NTU)						
ORP (mV)						

Standard Operating Procedure No. 19A

Bedrock Coring and Well Installation

Prepared by

EA Engineering, Science, and Technology, Inc.
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Revision 0
December 2013

CONTENTS

1. SCOPE AND APPLICATION

The purpose of this Standard Operating Procedure (SOP) is to describe the installation of bedrock cores and bedrock monitoring wells. The term "monitoring wells," as used herein, is defined to denote any environmental sampling well.

2. MATERIALS

In addition to materials required for drilling through overburden as detailed in SOP-19, conducting aquifer tests as described in SOP-33A, the following materials are needed to complete rock coring:

Hollow stem auger drilling equipment (SOP-19)	Groundwater sampling equipment (SOP-13 / SOP-48)
Roller Bit	Decontamination supplies (SOP-05)
NQ/HQ wireline coring equipment	Personnel protective equipment as required by the Accident Prevention Plan

3. PROCEDURES FOR BEDROCK CORING

The bedrock coring location will be staked in the field and utility clearance will be performed as per SOP 3. The proposed location will be adjusted in the field based on utility clearance. Based upon the presence and absence of visual contamination or subsurface debris, or field observations, the proposed location may be shifted or stepped out to delineate the extent of site-related impacts.

Hollow stem augers (6.25-in. internal diameter) will be used to advance the borehole through the overburden to the top of bedrock, as detailed in SOP-19. Upon reaching bedrock, a roller bit will be used to drill approximately 2 feet into bedrock and a $5^{7/8}$-in. diameter rock socket will be set. Four-inch internal diameter casing will be placed into the borehole and grouted in place to seal off the overburden. Coring will commence at each of the locations no sooner than 24 hours after the casing is grouted in place. After casing is set, drilling methods will change to fluid rotary and NQ/HQ wireline coring to the target depth. Rock cores will be collected in five-foot core runs using N-size double-tube core barrels.

Bedrock coring will be performed under the full-time supervision of an EA field geologist. At a minimum, the following information will be recorded by the field geologist:

- Date/times drilling occurred
- Rock quality density (RQD) Roll
- Description of fractures (including angle, spacing, degree of weathering, mineralization
- Lithology description
- Headspace photoionization detector (PID) readings
- Any unusual characteristics (e.g., odor, sheens, staining, etc.)
- Addition or loss of water
- Drill rig behavior and penetration rate (ft/min)
- Borehole depth information

If at any time, evidence of potentially mobile DNAPL is observed, drilling should only proceed in accordance with a DNAPL contingency plan that minimizes the potential mobilization of DNAPL, in consultation with the project manager.

4. PROCEDURES FOR INSTALLING BEDROCK MONITORING WELLS

The open rock holes will be left open to complete the downhole geophysical activities as detailed in SOP-100. After the geophysical activities are completed, bedrock monitoring wells will be installed where needed following the procedures for monitoring well installation in SOP-33. The wells will be secured with a locking J-plug and a boltdown flush-mount cover or an above grade locking steel protective casing. A concrete pad will be emplaced around the protective casing. Well development will be completed as detailed in SOP-33.

5. ABANDONMENT

If a monitoring well is not installed at a bedrock coring location, the corehole will be abandoned following the procedures detailed in SOP-28.

6. INVESTIGATIVE DERIVED WASTE

Investigative derived waste generated during drilling activities, bedrock coring, monitoring well development, and sampling will be disposed of per SOP-42.

7. DECONTAMINATION

Drilling equipment will be decontaminated prior to use, between coring locations, and following completion of coring as detailed in SOP-05.

8. PRECAUTIONS

Prior to collecting any samples, consult the Accident Prevention Plan for personal protective equipment required for sampling activities.

Decontaminate the sampling equipment and change gloves between samples to minimize the risk of cross-contamination.

Standard Operating Procedure No. 024
for
Photoionization Detector (Microtip HL-200)

Prepared by

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August 2007

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Page

1. SCOPE AND APPLICATION

The purpose of this Standard Operating Procedure is to delineate protocols for field operations with a photoionization detector (PID). PIDs use an ultraviolet emitting lamp designed to detect, measure, and display the total concentration of airborne ionizable gases and vapors. This information is used to determine control measures such as protection and action levels.

Use of brand names in this Standard Operating Procedure is not intended as endorsement or mandate that a given brand be used. Alternate equivalent brands of detectors, sensors, meters, etc. are acceptable. If alternate equipment is to be used, the contractor will provide applicable and comparable standard operating procedure for the maintenance and calibration of same.

2. MATERIALS

The following materials may be required:

Battery back	Regulator
Calibration gas (100ppm Isobutylene)	Tedlar bag
PID (i.e., Microtip HL-200)	Tygon tubing

3. STARTUP/CALIBRATION PROCEDURE

The following describes startup and calibration procedures:

- Turn the instrument on by pressing the back of the power switch located on the handle of the Microtip.
- The message “Warming up now, please wait” will be displayed for up to 3 minutes. After normal display appears, the Microtip is ready for calibration.
- Fill a Tedlar bag with the desired calibration gas (usually 100 ppm isobutylene).
- Press SETUP button and select the desired Cal Memory using the arrow keys (normally set to 200 ppm). Press EXIT button to leave setup function.
- Press CAL button and expose Microtip to Zero Gas. (Usually clean outdoor air will be suitable. If any doubt exists as to the cleanliness of the background air, a commercial source of zero gas should be used.).
- The Microtip then asks for the Span Gas concentration. Enter the known span gas concentration and then connect the Tedlar bag containing the Span Gas.

NOTE: THE SPAN GAS CONCENTRATION IS DEPENDENT UPON BOTH THE CONCENTRATION OF THE SPAN GAS USED AND THE RATING OF THE ULTRAVIOLET LAMP IN THE MICROTIP AT TIME OF CALIBRATION. IF USING 100 ppm ISOBUTYLENE AND THE STANDARD 10.6 eV LAMP, THE SPAN GAS CONCENTRATION WILL BE 56 ppm.

- Press enter and the Microtip sets its sensitivity. Once the display reverts to normal, the Microtip is calibrated and ready for use. Remove the Span Gas from the inlet probe. The instrument should be calibrated at least once a day.

4. BATTERY CHARGING

The following is a summary of battery charging procedures:

- Ensure Microtip is off.
- Set the voltage selector switch on the bottom of the battery charger to the appropriate AC line voltage.
- Press the release button on the bottom of the Microtip and remove the battery pack by sliding it backwards.
- Plug charger into the battery pack and then into an AC outlet and allow the battery to charge for at least 8 hours.
- After charging, remove the charger, first from the outlet then from the battery pack, and slide the battery pack back onto the Microtip.

5. PRECAUTIONS

The following is a summary of precautions while using the Microtip:

- Microtip does not carry an Intrinsic Safety Rating and must not be used in a hazardous location where flammable concentrations of gases or vapors are constantly present.
- All calibration, maintenance, and servicing of this device, including battery charging, must be performed in a safe area away from hazardous locations.
- Do not open or mutilate battery cells.
- Do not defeat proper polarity orientation between the battery pack and battery charger.
- Substitution of components may affect safety rating.

6. REFERENCES

Microtip HL-200 User's Manual. February 1990.

Standard Operating Procedure No. 025
for
Soil Sampling

Prepared by

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CONTENTS

1. SCOPE AND APPLICATION

The purpose of this Standard Operating Procedure is to delineate protocols for sampling surface and subsurface soils. Soil samples give an indication of the area and depth of site contamination, so a representative sample is very important.

2. MATERIALS

The following materials may be required:

Bucket auger or push tube sampler	Split-spoon, Shelby tube, or core barrel sampler
Drill rig and associated equipment	Stainless steel bowl
Personal protective equipment as required by the Health and Safety Plan	Stainless steel spoon, trowel, knife, spatula (as needed)

3. PROCEDURE

3.1 SUBSURFACE SAMPLES

Don personal protective equipment. Collect split-spoon, core barrel, or Shelby Tube samples during drilling. Upon opening sampler, or extruding sample, immediately screen soil for volatile organic compounds using either a photoionization detector or flame ionization detector. If sampling for volatile organic compounds, determining the area of highest concentration, use a stainless steel knife, trowel, or laboratory spatula to peel and sample this area. Log the sample in the Field Logbook while it is still in the sampler. Peel and transfer the remaining sample in a decontaminated stainless steel bowl. Mix thoroughly with a decontaminated stainless steel spoon or trowel. Place the sample into the required number of sample jars. Preserve samples as required. Discard any remaining sample into the drums being used for collection of cuttings. Decon sampling implements. All borings will be abandoned.

NOTE: If sample recoveries are poor, it may be necessary to composite samples before placing them in jars. In this case, the procedure will be the same, except that two split-spoon samples will be mixed together. The Field Logbook should clearly state that the samples have been composited, which samples were composited, and why the compositing was done.

Samples taken for geotechnical analysis will be undisturbed samples, collected using a thin-walled (Shelby tube) sampler.

3.2 SURFICIAL SOIL SAMPLES

Don personal protective equipment. Remove vegetative mat. Collect a sample from under the vegetative mat with a stainless steel trowel, push tube sampler, or bucket auger. If a representative sample is desired over the depth of a shallow hole or if several shallow samples are to be taken to represent an area, composite as follows:

- As each sample is collected, place a standard volume in a stainless steel bowl.
- After all samples from each hole or area are in the bucket, homogenize the sample thoroughly with a decontaminated stainless steel spoon or spatula.

If no compositing is to occur, place sample directly into the sample jars. Place the leftover soil in the auger borings and holes left by sampling. If necessary, add clean sand to bring the subsampling areas back to original grade. Replace the vegetative mat over the disturbed areas. Samples for volatile organic compounds will not be composited. A separate sample will be taken from a central location of the area being composited and transferred directly from the sampler to the sample container. Preserve samples as required. Decon sampling implements.

4. MAINTENANCE

Not applicable.

5. PRECAUTIONS

Refer to the Health and Safety Plan.

Soil samples will not include vegetative matter, rocks, or pebbles, unless the latter are part of the overall soil matrix.

6. REFERENCES

ASTM International. Method D1586-84, Penetration Test and Split-Barrel Sampling of Soils.

———. Method D1587-83, Thin Walled Sampling of Soils.

Department of the Army, Office of the Chief of Engineers. 1972. Engineer Manual 1110-2-1907 Soil Sampling. 31 March.

Standard Operating Procedure No. 028
for
Well and Boring Abandonment

Prepared by

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August 2007

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Page

1. SCOPE AND APPLICATION

The purpose of this Standard Operating Procedure is to establish the protocols by which all wells and borings will be safely abandoned. The primary objective of well abandonment is to ensure that the abandoned well or boring does not provide a conduit for the vertical migration of contamination between aquifers.

2. MATERIALS

The following materials may be required:

Drill rig	Bentonite pellets (seal)
Filter pack material	Cement (Portland Type II)
Pure sodium bentonite with no additives (bentonite) powder (grout)	Approved water

3. PROCEDURE

The procedures used in boring abandonment will ideally accomplish two objectives: (1) protect aquifers from cross-contamination by sealing the borehole, and (2) restore the strata in the borehole to nearly original conditions by selective placement of fill material.

Any casing will be pulled, drilled out, or thoroughly pierced. Using tremie pipe, grout will be placed from the bottom of the hole to within 3 ft of the ground surface, and allowed to settle for 24 hours. The remainder of the hole will be filled with concrete. The surface of the concrete will be mounded, smoothed, and inscribed with "ABD," for abandoned, any assigned well or boring designation, and the date the hole was abandoned. All boring logs, samples, completion records, and abandonment procedures will be included in the records of work on the site or cluster.

If the hole is within 10 ft of a monitoring well in the same aquifer, or a replacement well is to be installed within 10 ft of the well, any temporary casing will be pulled, drilled out, or thoroughly pierced. Using tremie pipe, the hole will then be backfilled with filter pack material opposite sand strata and bentonite or grout opposite substantial (2 ft or thicker) clay and silt strata. Where sand as backfill approaches the ground surface, 2 ft of bentonite will be placed above the sand and a 3-ft concrete plug will be placed at the surface. Otherwise, backfill materials will be placed from the bottom of the hole to within 3 ft of the ground surface. These materials will be allowed to settle for 24 hours. The remainder of the hole will be filled with concrete. The surface of the concrete will be mounded, smoothed, and inscribed with "ABD," for abandoned, any assigned well or boring designation, and the date the hole was abandoned. All boring logs, samples, completion records, and abandonment procedures will be included in the records of work on the site cluster.

If the well is not within 10 ft of another monitoring well, or if there are no substantial, continuous sand bodies, and no replacement well is planned within 10 ft of the original well location, then the hole may be grouted from the bottom to the top.

3.1 GROUT

Grout used in construction will be composed by weight of:

- 20 parts cement (Portland cement, Type II or V)
- 0.4-1 part (maximum) (2-5 percent) bentonite
- 8 gal (maximum) approved water per 94-lb bag of cement.

Neither additives nor borehole cuttings will be mixed with the grout. Bentonite will be added after the required amount of cement is mixed with the water.

All grout material will be combined in an aboveground container and mechanically blended to produce a thick, lump-free mixture. The mixed grout will be recirculated through the grout pump prior to placement.

Grout placement will be performed using a commercially available grout pump and a rigid tremie pipe removal and grouting will be accomplished in stages, aquifer by aquifer, sealing the boring from the bottom to ground surface. This will be accomplished by placing a grout pipe to the bottom and pumping grout through the pipe until undiluted grout reaches the bottom of the next higher section of casing or, for the top-most section, until grout flows from the boring at ground surface. Efforts will be made to grout incrementally as the temporary casing is removed.

After 24 hours, the abandoned drilling site will be checked for grout settlement. On that day, any settlement depression will be filled with grout and rechecked 24 hours later. This process will be repeated until firm grout remains at the ground surface.

3.2 BORINGS

The term “Borings” as used in this Standard Operating Procedure applies to any drilled hole made during the course of a remedial investigation which is not completed as a well. This includes soil test borings, soil sampling borings, and deep stratigraphic borings. Whether completed to the planned depth or aborted for any reason prior to reaching that depth, borings will be grouted and normally closed within 4 hours, or within 4 hours or completion of logging of completion of logging.

3.2.1 Shallow Borings not Penetrating Water Table

Shallow borings made for the collection of subsurface soil samples will be abandoned by backfilling the hole with cuttings from the hole, **if and only if the boring does not penetrate the water table.** Clean sand will be used to make up any volume not filled by the cuttings.

3.2.2 Borings Penetrating the Water Table

Shallow borings made for the collection of subsurface soil samples **which penetrate the water table** will be abandoned by grouting the hole from the bottom to the top.

3.2.3 Deep Stratigraphic Borings

Deep stratigraphic borings will normally be located in areas which, by virtue of the historical record, are presumed relatively uncontaminated. Therefore, these borings are usually over 100 ft from any sampling well locations. Any boring located within 10 ft of a proposed well location, or located directly upgradient or downgradient (on anticipated flow line) of a proposed well location, will be abandoned by placing clean sand in the aquifer intervals and bentonite or grout in aquitard intervals as described above. If the boring is over 10 ft from and/or not upgradient of a proposed well location, the boring will be completely filled with grout.

3.3 WELLS

The following procedure applies to wells aborted prior to completion and existing wells determined to be ineffective or otherwise in need of closure.

Prior to abandoning any developed well, the proper well licensing body will be provided written notification along with an abandonment plan for that well.

If the well is within 10 ft of another monitoring well in the same aquifer, or a replacement well is to be installed within 10 ft of the well, casing will be pulled, drilled out, or thoroughly pierced. Using tremie pipe, the hole will then be backfilled with filter pack material opposite sand strata and bentonite or grout opposite substantial (2 ft or thicker) clay and silt strata. Where sand as backfill approaches the ground surface, 2 ft of bentonite will be placed above the sand and below the concrete plug near the surface. Backfill materials will be placed from the bottom of the hole to within 3 ft of the ground surface. These materials will be allowed to settle for 24 hours. The remainder of the hole will be filled with concrete. The surface of the concrete will be mounded, smoothed, and inscribed with “ABD,” for abandoned, any assigned well or boring designation, and the date the hole was abandoned. All boring logs, samples, completion records, and abandonment procedures will be included in the records of work on the site cluster.

If the well is not within 10 ft of another monitoring well, and is not to be replaced by another well within 10 ft of the original location, casing will be pulled, drilled out, or thoroughly pierced. Using tremie pipe, grout will be placed from the bottom of the hole to within 3 ft of the ground surface, and allowed to settle for 24 hours. The remainder of the hole will be filled with concrete. The surface of the concrete will be mounded, smoothed, and inscribed with “ABD,” for abandoned, any assigned well or boring designation, and the date the hole was abandoned. All boring logs, samples, completion records, and abandonment procedures will be included in the records of work on the site cluster.

4. REPLACEMENT WELLS

Replacement wells (if any) will normally be offset at least 10 ft from any abandoned well in a presumed upgradient or crossgradient groundwater direction. Site-specific conditions may necessitate variation to this placement.

5. PRECAUTIONS

None.

Standard Operating Procedure No. 031
for
Sample Container Cleaning

Prepared by

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CONTENTS

1. SCOPE AND APPLICATION

The purpose of this Standard Operating Procedure is to define laboratory protocols to be used in cleaning and preparing containers used to collect environmental samples.[1]

2. MATERIALS

The following materials may be required:

5 percent sodium hydroxide	Bottle caps
5 percent Ultrex nitric acid	Deionized water
40-ml vials	Hexane (Nanograde or equivalent)
Acetone	Methylene chloride
Alconox detergent	Polyethylene bottles
Amber glass bottles	Polytetrafluoroethelyne liners

3. PROCEDURE

3.1 POLYETHYLENE BOTTLES

Rinse bottles and lids sequentially with 5 percent sodium hydroxide, deionized water, and 5 percent nitric acid with deionized water. Drain and allow to air dry.

3.2 AMBER GLASS BOTTLES AND 40 ml VIALS

- Wash bottles in detergent and rinse with copious amounts of distilled water
- Rinse with acetone
- Rinse with methylene chloride
- Rinse with hexane
- Allow bottles to air dry
- Place bottles in a drying oven and heat to 200°C
- Allow bottles to cool prior to sealing with clean caps and polytetrafluoroethelyne liners.

3.3 BOTTLE CAPS

- If applicable, remove paper liners from caps
- Wash caps with detergent, followed by a distilled water rinse
- Dry caps in drying oven at 40°C.

1. This Standard Operating Procedure is included for completeness only. It is anticipated that sample containers will either be provided by the laboratory or that the sampling contractor will purchase new, certified clean sample containers.

3.4 POLYTETRAFLUOROETHELYNE LINERS

- Always handle liners with forceps or tweezers; never use fingers.
- Wash liners with detergent, followed by distilled water rinse.
- Rinse the liners with acetone, followed by hexane (Nanograde or equivalent).
- Allow liners to air dry prior to placing in clean caps, then heat liner and caps in drying oven at 40°C for 2 hours.
- Allow caps and liners to cool prior to placing on clean bottles.

A statistically representative number of randomly selected clean sample containers will be analyzed for Target Analyte List/Target Compound List analytes. Results of these analyses will be provided to the client.

4. MAINTENANCE

Not applicable.

5. PRECAUTIONS

None.

6. REFERENCES

None.

Standard Operating Procedure No. 033
for
Aquifer (Hydraulic) Testing

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1. SCOPE AND APPLICATION

The purpose of this Standard Operating Procedure (SOP) is to define various hydraulic test methods which may be used, to specify how these tests are to be performed, and to provide appropriate methodologies for data reduction and interpretation. This SOP assumes a high degree of technical competency on the part of the investigator, in that certain assumptions and interpretations must be made in the selection of the test and data analysis to achieve valid results.

Aquifer testing is a process performed on selected wells to characterize the **Hydraulic Conductivity**, **Transmissivity**, and **Storativity** of the aquifer into which those wells are installed. Aquifer tests fall into two broad categories: pumping tests and slug tests. Pumping tests and slug tests are relatively inexpensive when compared to the remedial investigation budget as a whole, but it should be noted that, as with many *in situ* tests, aquifer tests may yield non-unique solutions.

Pumping tests are typically performed on wells installed in highly permeable materials, confined aquifers, and in areas of little or no suspected contamination in the groundwater. The principle of pumping tests is to remove water from the aquifer at a sufficient rate and for a sufficiently long period of time to stress the aquifer and cause measurable drawdown in the pumped well and adjacent (10 to several hundreds of feet) observation well(s). The aquifer characteristics can then be calculated by substituting inter-well distances, drawdown and well discharge data into appropriate equations, employing curve matching techniques, or using computer programs to reduce the data.

- Advantages of performing pumping tests are: (1) they encompass large areas, (2) test results are more accurate, (3) they can resolve complex aquifer conditions (e.g., boundaries), (4) time periods and pump rates can be varied, and (5) pumping tests represent remedial actions.

- Disadvantages of pumping tests are: (1) large quantities of potentially contaminated water are generated, (2) the contaminant plume may be moved by the test, (3) they are very costly, and (4) the data produced are averages over large areas.

Single-Well Permeability Tests (Slug Tests) are conducted to determine the characteristics of an aquifer in materials whose conductivity is too low to perform a pumping test, or in aquifers which are highly contaminated. Slug tests consist of inserting and/or removing either a slug of inert material of known volume, or a "slug" of water of known volume. Either method will cause an instantaneous rise or fall and subsequent recovery of the water table within the aquifer.

- Advantages of slug tests are: (1) they provide location-specific data, (2) they are small-scale and unlikely to move the contaminant plume, (3) no contaminated water (other than decontaminated solutions) is generated, (4) they are low cost, therefore (5) high data density is feasible, and (6) they can be used as an aid in selecting an appropriate area to perform pumping test(s).

- Disadvantages of performing slug tests are: (1) they provide less precise estimates of parameters; (2) they may not yield values for storativity; (3) they cannot resolve complex geometries; and (4) since they are short-term tests, they cannot resolve long-term events.

2. MATERIALS

2.1 ALL TESTS

The following materials may be required for all tests:

Copy of the site Health and Safety Plan	Health and Safety monitoring equipment, and personal protective equipment as required by the Health and Safety Plan
Calculator	Portable computer[1]
Containers for investigation-derived materials	Program diskettes
Data diskettes	Stopwatches
Decontamination equipment and supplies	

2.2 PUMPING TESTS

The following materials may be required for pumping tests:

Generators (2), fuel, extension cords and/or other source of onsite electrical power	Stainless steel submersible pump with associated tubing, clamps, and wiring
Logbook	Steel register flowmeter or stopwatch and bucket, graduated cylinder, or rain gauge
Pump	Transducer or other water level indicator[1]
Recording barometer or other source of local barometric readings (e.g., local airport or National Weather Service recording station)	Type curves for curve-matching analyses
Semilogarithmic paper-arithmetic vertical scale and logarithmic horizontal scale, or log-log paper	

2.3 SLUG TESTS

The following materials may be required for slug tests:

1. Although removal (or insertion) of larger volumes of water may increase the recovery time(s) of the aquifer to the point that use of electrical water level meters or steel tapes is feasible, piezometers and data loggers are preferred because they tend to provide more complete records with less maintenance and operator error.

Test Conducted with Inert Cylinder	
Transducer[2]	Inert, negatively buoyant cylinder of known volume
Logbook and/or field data sheets (examples provided in SOP No. 016)	Type curves for curve-matching analyses
Semilogarithmic paper-arithmetic vertical scale and logarithmic horizontal scale	Slug device (solid stainless steel or a sealed polvyinyl chloride cylinder filled with sand or similar material)
Test Conducted with Input/Output of Water	
Teflon bailer with Teflon-coated stainless steel leader and rope or pump	
— OR —	
Stainless-steel submersible pump or centrifugal pump	Pump wiring
Teflon or polyethylene flexible piping	Steel register flowmeter
Generator and fuel or other onsite source of electricity	American Society for Testing and Materials (ASTM) Type II water
Stainless-steel hose clamps	
— AND —	
Logbook	Type curves for curve-matching analyses
Semilogarithmic paper	Approved water and/or containers for removed water as required in SOP No. 042
Transducer or other water level indicator[1]	

3. PROCEDURE

Regardless of the test method chosen, the following general procedures must be considered:

- All well intrusive equipment must be decontaminated prior to and after use.
- All water removed from the test wells is considered, and must be treated, as purge water.
- The accuracy of the reading(s) from pressure transducer (piezometer) and data logger output should be verified prior to beginning any test, periodically during the test, and immediately after the test by measuring the groundwater level with one of the aforementioned mechanical devices.
- All water level tapes and meters should be calibrated against one master tape which is traceable to the National Institute of Standards and Technology. This calibration should be recorded in the field logbook.
- Repeated measurements at any one well should be made using the same tape.

3.1 SELECTION OF TEST METHOD

Before beginning any aquifer test, the investigator should have a good conceptual model of the site's hydrogeologic condition. This is essential because of the assumptions made in each

2. Field portable computer and associated equipment are considered as optional. Access to a computer will be required to down-load dataloggers.

analysis method. If the site conditions do not correspond with the assumptions in a given model, the analysis will be invalid. The conceptual understanding of the hydrogeology of the site can be developed from driller's logs and/or borehole geophysical logs of the wells to be tested, or from previous reports on the hydrogeology of the area. Table SOP033-1 is a decision tree which can be used with this conceptual model to determine the appropriate test/analysis method(s) which may be used at a given site.

3.2 PUMPING TESTS

Pumping tests can be divided into two broad categories: (1) those in which the pumping (discharge) rate is kept constant, and (2) those in which the pumping rate varies over time. All water removed from pumping wells must be disposed of appropriately.

3.2.1 Constant Discharge

These methods require that the discharge or injection rate in the pumping well be kept constant. Of these, the Theis method is the most widely referenced and applied, and serves as the basis for the solution of other, more complex boundary condition problems. Both the Cooper & Jacob and the Jacob modifications to the Theis method recognize that if pumping times are long and/or the distances to control wells are small, the Theis solution will yield a straight line plot on semilogarithmic paper, thereby simplifying the Theis equation. The Thiem method, unlike the Theis/Modified Theis methods, assumes that steady-state (equilibrium) conditions can be achieved in a confined aquifer.

The Theis method is detailed in Section 5.1.1 and ASTM D4106, the modified Theis method in Section 5.1.2 and ASTM D4105, and the Thiem method in Section 5.1.3.

The preceding methods assume that the aquifer being tested is confined. If the aquifer is unconfined or semi-confined (leaky), the preceding methods are invalid. DeGlee developed an equation which assumes that the tested aquifer is either overlain or underlain by a continuous, leaky confining layer which has uniform properties, and that leakage from the aquitard is proportional to the hydraulic gradient across the aquitard. Hantush and Jacob derived the same equation. Hantush later observed that a simpler approximation is possible if the ratio of the distance to monitoring well/leakage across the aquitard is <0.05. As in all preceding cases, radial flow is assumed. The Hantush-Jacob method assumes that no storage occurs in the aquitard. The DeGlee equation and Hantush approximation is provided in Section 5.3.2.

Both Neuman and Witherspoon, and Hantush have proposed methods that take into account storage in the aquitard. The Neuman/Witherspoon method is provided in Section 5.3.1.

3.2.1.1 Field Operations

1. Arrange for all nearby extraction wells and automatic pump controls to be inoperative during the test period.

2. Measure and record the distance(s) between observation well(s) and pumping well.

3. Install pressure transducers in the pumping well and each observation well; submerge the transducer in the well to a sufficient depth to provide effective performance. Well bottom sediment plugging of the transducer must be avoided.

4. Perform a two-point calibration of each device as part of the installation, cross-checking data with a calibrated manual tape measure. Note that water levels must be noted and recorded to the nearest 0.01 ft.

5. Start dataloggers at each well several days before the test to record background fluctuations in the groundwater table. It is recommended that one observation well be selected beyond the expected influence of the test to monitor these background water table fluctuations during the actual test.

6. Using either the field barometer of data from a local meteorological station (Section 2.2), record precipitation and barometric pressure before, during and after the test.

7. Calibrate pump, flowmeter, and any other field instruments such as pH meters, etc.

8. Suspend pump at mid-point of pumping well screen and record water level.

    - If the recharge rate of the well is not known, conduct a step-drawdown test (Section 3.2.3) to determine the sustainable yield for the constant discharge test.

    - If the previous step is performed, the aquifer must be allowed to recover prior to performing the constant discharge test. At a minimum, the recovery period between step-test and pump test must be equal to the duration of the step test.

    - Manually check and record the water levels in all test wells before conducting the step test, at the end of the step test, and at 4-hour intervals (minimum) during the recovery period; recording these values in the field logbook.

9. Program the datalogger data acquisition rate as follows:

    - If the only mode of data acquisition is a fixed rate, program the datalogger(s) to record water levels every 5 seconds.

    - If a logarithmic data acquisition rate is possible, use this option.

    - If the data acquisition rate is programmable on the data logger, record data at incrementally lengthening frequencies. The following table is provided as an example, the actual rates at which data are acquired at a given site should be determined based on prior field tests.

Time after Aquifer Test Begins	Frequency of Measurements
0-60 minutes	Every 5-10 seconds
61-65 minutes	Every 15 seconds
65-75 minutes	Every 30 seconds
75-120 minutes	Every 5 minutes
120-180 minutes	Every 10 minutes
180 minutes – end of test	Every 30 minutes

10. Collect a complete round of static water levels, verifying transducer readings with a tape or electric water level meter.

11. Start data logger(s).

12. Start pump at previously selected rate. Monitor flow rate using flowmeter or stopwatch and bucket (rain gauge, graduated cylinder, etc.) half-hourly. Record this flow rate and adjust as necessary. Minimize fluctuations in flow rate, especially during the early stages of the test.

13. Periodically download data from the loggers and plot drawdown as a function of time to assess the status of the test in real time. Under no circumstances should the flow rate be varied during the test.[3] If drawdown is either falling substantially above or below predicted levels, then a decision should be made to continue the test at that pumping rate, terminate the test, allow the aquifer to recover, or restart the test at a different pumping rate.

14. Continue pumping and recording water levels for a total period of 72-96 hours (3-4 days). After the drawdown portion of the test, download the data onto data diskettes using the portable computer.

15. Reprogram the dataloggers to record a reading every 5 seconds or a logarithmic or variable interval if available on the datalogger.

16. Turn off the pump and allow the groundwater to recover to within 90 percent of static conditions. The pump should be equipped with a foot valve to prevent backflow of the column pipe fluid.

17. Periodically download data from the logger and plot recovery as a function of time to assess the status of the test in real time.

3. After a period of at least 12 hours into the test, brief interruptions (less than 5 minutes) in pumping due to mechanical failure are acceptable without re-starting the test. All critical equipment should have onsite backups as a contingency against equipment failures. Inasmuch as refueling an onsite generator every 4-10 hours while it is running is considered an unsafe practice, two generators should be provided at the pumping well site to maintain power to the pump during the test.

18. After recovery to 90 percent of static conditions, remove the dataloggers, pressure transducers, and cables from all the observation wells. Download the data onto data diskettes using the portable computer.

19. Collect a complete round of water levels from all wells in the monitoring network.

3.2.2 Variable Discharge

Variable discharge methods have been presented by numerous researchers. These methods are performed as a series of constant-rate, stepped changes in discharge rate. These changes in discharge rate may be linear or exponential. Type curves are derived for control wells. These methods can be applied in extensive leaky aquifers, but are generally used in confined aquifers. The only requirement is that the response to a unit stress be known. The step-test is incorporated as a preliminary measure to determine the optimal sustainable discharge rate for a given aquifer (Section 3.2.1.1). Otherwise, variable discharge methods are included herein for the sake of completeness. They are not widely used outside the research environment for aquifer characterization. No further details on variable pump rate tests are provided in this SOP.

Install a variable speed submersible pump and a pressure transducer into the pumping well.

Install pressure transducers into the nearby (closest) observation wells to provide preliminary indications of expected drawdown during the constant-discharge test.

Allow water level(s) to stabilize to original water level after installing the pump and transducer(s).

Program a datalogger to collect readings at 5- to 10-second intervals. If logarithmic programming is available, this collection period may be expanded to 1-minute increments by the completion of each step, where upon 5- to 10-second intervals will again be required to coincide with the start of the next step.

Measure static water levels from all wells expected to be influenced by the test, and calibrate pressure transducers. Record this information in the field logbook and field data sheets.

Select the pumping rates for the step-test. Four 100-minute steps should be run at steadily increasing flow rates. The flow rates should be selected, based on a preliminary estimate of a sustainable rate (ESR) as follows:

Step	Duration (minutes)	Pumping Rate (%)
1	100	25
2	100	50
3	100	100
4	100	125

Program the dataloggers, calibrate the pump to the initial flow rate, and initiate the test at 25 percent ESR. At the completion of the first step, increase the pumping rate to 50 percent ESR as instantaneously as possible. The pump should not be shut off between steps. At the completion of the second step, increase the flow rate to 100 percent ESR as instantaneously as possible. At the completion of the third step, increase the flow rate to 125 percent ESR as instantaneously as possible. At the completion of the fourth step, shut off the pump and record the recovery of water levels to at least 90 percent of static conditions.

During the test, plot drawdown in the pumping well on both arithmetic and semi-logarithmic graph paper to assess the performance of the test in real time. Make any adjustments to the test as appropriate, i.e., the addition of a fifth step at a higher rate if the aquifer does not appear to be adequately stressed, or termination of the test if the well is drawn dry.

At the conclusion of the test, analyze the data and select a pumping rate for the constant-discharge test. This rate should be sustainable for the anticipated duration of the test, and place the maximum stress on the aquifer.

Calibrate the variable speed pump to the selected rate so that at the start of the constant discharge test early fluctuations in flow rates will be minimized.

Allow the aquifer to fully recover for a period equal to or greater than the duration of the step-test prior to initiating the constant-discharge test.

3.3 SLUG TESTS

Slug tests involve the use of a single well, and evaluating its response to an instantaneous raising and/or lowering of the water level within the casing. If the well is poorly designed or poorly developed, the test may end up evaluating the performance of the well screen and/or filter pack rather than the aquifer.

Slug tests are usually of short duration, usually less than 5 minutes. The first 30 seconds are the most important in respect to data collected. Piezometers and digital data loggers are, therefore, a must.

3.4 DATA REDUCTION

If the data were recorded by the datalogger as feet of water above the pressure transducer, reduce these data to potentiometric head (in feet), relative to the initial water level as measured from the top of the casing. Record this with the respective changes in time. For each piezometer or well, tabulate the pre- and post-test water levels, dates, clock times, and times since pumping started or stopped.

Tabulate measurements of the rate of discharge at the control well, date, clock time, time since pumping started, and method of measurement.

Prepare a written description of each well, describing the measuring point, giving its elevation and the method of obtaining the elevation, and the distance of the measuring point above the mean land surface.

Once the data are collected and reduced, a variety of methods may be used to calculate aquifer parameters from pumping test data. Refer to Section 5 for analytical methods applied to test data.

Data can also be reduced electronically when downloaded from the transducers into software such as AQTESOLV®; this is software designed to calculate hydraulic conductivity, storativity and other aquifer properties from data sets collected during slug and aquifer (pumping) tests.

Text files, which are generated by commonly used pressure transducers, can be imported into the software and data can also be manually entered or pasted from a spreadsheet. After importing, the raw data can be manipulated using mathematical functions. For example, hydraulic head data can be converted to drawdown data. The program will also produce visual and automatic curve matching methods for confined, unconfined and leaky aquifers. Visual curve matching is analogous to traditional methods of aquifer test analysis with graph paper and type curves. The software is also capable of producing error logs which enable the user to quickly identify any deficiencies or inconsistencies detected in the data set.

4. FIELD DATA RECORDS

4.1 LOGBOOK

Only one site or installation per logbook, and only one slug test per data table (see below). The first page must include the well number, location, date of test, persons conducting the test, and reference plane for drawdown measurements. Next page(s) must be in table format with the columns designating time/date, water volume withdrawn/added or displaced by inert cylinder, water levels, etc.

Test data must be entered in a table as data are acquired. Data must include sufficient information to indicate that the water level was stable before the test, during equilibrium, and after the test(s).

5. INTERPRETATION OF DATA

5.1 CONFINED AQUIFER METHODS

5.1.1 Theis Method

The Theis test method involves pumping a well (pumped well) at a constant rate (Q) and measuring drawdown (s) in an adjacent observation well. Theis assumed that groundwater flow in an aquifer is analogous to heat flow in a solid and derived the following equation:

$$s=\frac{Q}{4\pi T}\int_{r^2\frac{S}{4Tt}}^{\infty}\left(\frac{e^{u}}{u}\right)du$$

Equation 1

where

s = Drawdown.
r = Radial distance to observation well.
Q = Pumping (discharge) rate.
T = Transmissivity (K × aquifer thickness).
K = Conductivity.
S = Storativity.
t = Time (since pumping began).

and

$$u=\frac{r^2S}{4Tt}$$

Equation 2

If the integral is expressed as the well function W(u), then the equation can be written as:

$$s=\frac{Q}{4\pi T}W(u)$$

Equation 3

which can be also written as:

$$T=\frac{Q}{4\pi s}W(u)$$

Equation 4

or

$$S=\frac{4Tt}{r^2}u$$

Equation 5

5.1.1.1 Assumptions

To permit an analytical solution for non-steady, radial flow into the well, the Theis method makes the following assumptions. Most of these assumptions are incorporated in the other analysis methods detailed herein. Only exceptions or additions to these assumptions will be noted in each method.

- The aquifer has seemingly infinite areal extent compared to the well, whose diameter is assumed infinitesimally small.
- The aquifer is homogeneous, isotropic, of uniform thickness, and horizontal.
- The head is uniform and constant prior to the test.
- Darcy's Law is valid.
- The well is pumped at a constant rate.
- Water is discharged from storage instantaneously.
- The well screen fully penetrates the aquifer.
- Flow within the aquifer is radial to the well and strictly horizontal.
- Drawdown data are taken from an adjacent observation well.

One additional assumption is made in this SOP, which was not made by Theis: The pumping well has been previously sampled and analysis of the groundwater at that site is not grossly contaminated (e.g., no non-aqueous phase liquids).

5.1.1.2 Procedure (see also ASTM D4106-91 and D4050-91)

Field Operations – Constant Discharge Test (Refer to Section 3.2.1)

Data Plots

1. Prepare a type curve of W(u) over 1/u on log-log paper. Figure SOP033-1 is an example of a type curve. Tables of data used to generate this curve may be found in ASTM D4106-91, ASTM D5270-92, or in most hydrology texts such as Fetter or Dominico[4]. It is recommended that this plot be copied onto tracing paper, drafting film, or an overhead viewer film copy be made to facilitate later steps. NOTE: W(u) over u can be plotted if preferred, but will require additional computational steps if used.

2. Plot drawdown over time for each observation well on log-log paper which has the same scale as the type curve (above). Note that for a single observation well, drawdown can be plotted as a function of time (t). However, for multiple observation wells, drawdown can be plotted as a function of t/r2, where r = radial distance from the pumping well to the observation well in which the measurements were made. This is done to normalize the data and allow comparison between wells.

4. See Section 6 for complete citations of these references.

3. Superimpose the type curve and the plot of observation well data. **Keeping both the X and Y axis of each plot parallel**, position the two curves to obtain the best match when overlain. NOTE: If only paper copies of both plots are available, a light table or brightly lit window will be required for this step.

4. Select a match point on the overlapping portion of the two graph papers such that the values for that point simplify the calculations (e.g., even log cycles of [W(u), 1/u] — [1,1]; [1,10] etc.).

5. Read coordinates for W(u), 1/u, s, and t.

6. Substitute the match point values for the appropriate variable in Equations 3, 4, and 5.

7. Repeat for each observation well.

5.1.2 Modified Theis (Cooper & Jacob; Jacob) Method (see also ASTM D4105-91)

5.1.2.1 General

From Equation 1, we have:

$$\int_u^{\infty}\left(\frac{e^u}{u}\right)du = W(u) = -0.577216 - \log_e u + u - \frac{u^2}{2!2} + \frac{u^3}{3!3} - \frac{u^4}{4!4} + \ldots$$

Equation 6.

Jacob noted that as values of u become small, the value of the series to the right of logeu becomes insignificant. That is the series becomes equal to or less than 0.01. The value of u decreases as the value of t (time) increases, and as the value of r (radial distance to observation well) decreases. Therefore, for long pumping times and/or observation wells reasonably close to the pumping well, the Theis equation can be stated as:

$$s = \frac{Q}{4\pi T}\left[-0.577216 - \ln\left(r^2\frac{S}{4Tt}\right)\right]$$

Equation 7.

Lohman was then able to show the following relationships:

$$T = \frac{2.3Q}{4\pi\Delta s/\Delta\log_{10} t}$$

Equation 8

and

$$T = \frac{2.3Q}{4\pi\Delta s/\Delta\log_{10} r}$$

Equation 9

where

Δs/Δlog10t = The drawdown (measured or projected) over one log cycle of time.
Δs/Δlog10r = The drawdown (measured or projected over one log cycle of radial distance from the control well.

5.1.2.2 Procedure

Field Operations (see Field Operations under Section 3.2.1.1)

Data Plots

1. Plot drawdown over time (log scale) on semilog paper.

2. Draw a best fit straight line through the later portion of the data and project it back to s = 0.

3. Read t0 as the time corresponding to the s = 0 point.

4. Solve for T using:

$$T=\frac{2.3Q}{4\pi\Delta s}$$

Equation 10

5. Solve for S using:

$$S=\frac{2.25Tt_0}{r^2}$$

Equation 11

where

t0 = the intercept of the line when extended to the zero drawdown axis.

6. Solve for K (K = T/aquifer thickness). Aquifer thickness = screened interval (see assumptions in Section 5.1.1.1).

7. Repeat for each monitoring well.

5.1.3 Thiem (Steady State Flow) Method

5.1.3.1 Assumptions

All of the Theis assumptions hold except that equilibrium has been reached between discharge and drawdown. Note that this condition is theoretically impossible in a confined aquifer where all discharge is derived from storage.

5.1.3.2 Equations

When these assumptions are met, the following equation expresses groundwater flow in the confined aquifer:

$$Q=\frac{2\pi T(s_1-s_2)}{\ln(\frac{r_2}{r_1})}$$

Equation 12

where

Q = Well discharge.
T = Aquifer transmissivity.
r1 and r2 are respective distances of OW-1 and OW-2 from the pumping well.
s1 and s2 are respective steady-state drawdowns in the observation wells.

Note that the Thiem equation is designed to solve for transmissivity only, and cannot be used to solve for storativity.

5.1.3.3 Procedure

Field Operations

1. Follow Steps 1 through 9 in Section 3.2.1.

2. Continue pumping until steady-state conditions are reached. This equilibrium is defined as the time when variations of drawdown with respect to time are negligible. Note that this may require considerably more time than with either the Theis or Modified Theis methods described above.

Data Plots Method 1

1. Substitute the steady-state drawdown of the two observation wells into Equation 12 along with the values of r and Q. Solve for T.

2. Repeat with all possible combinations of observation wells to obtain the mean transmissivity of the aquifer.

Data Plots Method 2

1. Plot the observed steady-state drawdown of each observation well over distance (log scale) to the pumping well.

2. Construct a best-fit straight line through the plotted points.

3. Determine the maximum drawdown per log cycle.

4. Substitute this value of (s1 - s2) in Equation 12 along with Q and solve. Note that the term ln(r2-r1) is taken to ln(10) equal to 2.30.

5.2 BOUNDED, NON-LEAKY, CONFINED AQUIFER

The test methods described in this section are essentially duplicates of the Theis and Modified Theis methods which are detailed above. The principal differences are that, by definition, a

bounded aquifer is limited in its areal extent by a fully-penetrating linear boundary, which is either a constant head (e.g., stream or lake) or a no-flow boundary (e.g., impermeable, or significantly less permeable geologic formation). These conditions are illustrated on Figure 2.

As stated, the equations used to evaluate data derived from bounded wells are modifications to the basic Theis equations. Drawdown(s) at any point in the aquifer is defined as the sum of the drawdown due to the real ($_r$) and image ($_i$) wells such that:

$$s_0 = s_r \pm s_i \qquad \text{Equation 13}$$

so that Equation 1 can be rewritten as:

$$s = \frac{Q}{4\pi T}[W(u_r) \pm W(u_i)] = \frac{Q}{4\pi T}\sum W(u) \qquad \text{Equation 14}$$

where

$$u_r = \frac{r_r^2 S}{4Tt} \qquad \text{Equation 15}$$

and

$$u_i = \frac{r_i^2 S}{4Tt} \qquad \text{Equation 16}$$

so that:

$$u_i = \left(\frac{r_i}{r_r}\right)^2 u_r \qquad \text{Equation 17}$$

or

$$u_i = k_i^2 u_r \qquad \text{Equation 18}$$

where

$$K_i = \frac{r_i}{r_r} \qquad \text{Equation 19.}$$

NOTE: K_1 is a constant of proportionality and should not be confused with the hydraulic conductivity.

5.2.1 Assumptions

All assumptions listed under the Theis method apply with these exceptions:

- The non-leaky aquifer is of infinite areal extent except where limited by linear boundaries.
- The boundaries are vertical planes of infinite length, which fully penetrate the aquifer.
- The hydraulic boundaries are perfect. Impermeable boundaries yield no water to the aquifer; recharge boundaries are in perfect hydraulic connection with the aquifer.

5.2.2 Procedure (see also ASTM D5270-92)

Field Operations (see Section 3.2.1.1)

Data Plots

1. Generate a family of type curves for the solution of the modified Theis formula (Kl). This family of curves should include curves for both discharging and recharging image wells. Plot the coordinates of ∑W(u) on the vertical axis and 1/u (Figure 3). It is recommended that this plot be copied onto tracing paper, drafting film, or an overhead viewer film copy be made to facilitate later steps.

2. Plot drawdown (s) over t/r2 for each observation well on log-log paper which has the same scale as the type curve (above). NOTE: **t=time, r=radial distance from pumping well.**

3. Superimpose the type curves and the plot of observation well data. **Keeping both the X and Y axis of each plot parallel**, position the two curves to obtain the best match when overlain. NOTE: If only paper copies of both plots are available, a light table or brightly lit window will be required for this step.

4. Select a match point on the overlapping portion of the two graph papers such that the values for that point simplify the calculations (e.g., even log cycles of [∑W(u), 1/up] — [1,1]; [1,10] etc.).

5. Read coordinates for ∑W(u), 1/up, s, and t/r2.

6. Substitute the match point values for the appropriate variable in the equations below:

Transmissivity = $T=\frac{Q}{4\pi s}\sum W(u)$ Equation 20

Storativity = $S=4T(\frac{t}{r^2})u$ Equation 21

7. For each OW, determine the distance from the image well (r_i) using the following:

$r_i=K_f r$ Equation 22

8. Repeat for each observation well.

5.2.3 Modified Theis Non-Equilibrium Method

As in the case of a non-bounded aquifer, the hydraulic parameters can also be determined using a Modified Theis equation and a straight line (semi-log) plot of s and log10t.

5.2.3.1 Procedure

1. Refer to Section 5.1.2 (above) and ASTM D4105-91 for details on plotting the data and the equations to be used in calculating transmissivity and storativity using this method. Note that the data will define two rather than one straight line portions. This is due to the image well effect of the boundary conditions.

2. Select a convenient value of s within the initial straight-line part of the plot. Drawdown represented by this portion of the curve has not been affected by the boundary. Therefore, s = sr and the corresponding value of tr corresponds to sr.

3. Graphically extend the initial straight-line part of the curve to the right. The departure of the measured drawdown from the extended line represents the drawdown due to the presence of the boundary. This effect is also referred to as the image well drawdown, si.

4. Select a point on the second straight-line such that si = sr. Note the value of time ti which corresponds to si.

5. Since tr and ti are selected such that si = sr, then ur = ui and (Equation 23)

$$\frac{r_r^2 S}{4Tt_r} = \frac{r_i^2 S}{4Tt_i}$$

so that:

$$K_i = \frac{r_i}{r_r} = \sqrt{\frac{t_i}{t_r}} \qquad \text{(Equation 24)}$$

6. Determine the distance to the image well using Equation 22.

7. Repeat this calculation for each observation well.

Determine the location of the boundary as follows:

- Accurately plot the locations of the control and observation wells on a map.

- With a compass, using each observation well as the center point, draft a circle which has a radius equal to the distance from that well to the boundary.

- The image well is located at the intersection of these circles. If the circles do not intersect exactly, the probable well location is at the centroid of the intersections, or the polygonal area bounded by the circles in the case of no overlap.

- Draw a straight line from the pumping well to the image well. The boundary is defined as the perpendicular bisector to this line at the image well.

5.2.4 Limitations

The following caveats apply to either of the above two methods:

- In cases where this test method is employed to locate an unknown boundary, a minimum of three observation wells is required to accurately locate the image well which is the boundary. Two observation wells will yield two possible locations for the image well. One observation well will indicate the presence of a boundary, and the distance (radius) from the observation well, but the image well will be located somewhere on that surface.

- The effects of a constant head (recharging) boundary are indistinguishable from those of a leaky aquifer. It is, therefore, imperative that care be taken in developing the conceptual model of the geohydrologic system being studied prior to testing.

5.3 SEMI-CONFINED (LEAKY) AQUIFER

In some instances, the confining beds either above or below the aquifer will not be completely impermeable. In these cases, the aquifer is said to be "leaky." This condition can be readily determined from the Theis s over t plot on log-log paper. In the initial phase of pumping, the plot will look like the Theis "type" curve. As pumping continues and the aquifer is depressurized (piezometric head decreases), a gradient within the overlying and/or underlying aquitard(s) is induced. Instead of the expected type curve, the plot will be somewhat flattened, and values for s may actually decrease over time if the vertical component of groundwater flow through the aquitard(s) is sufficiently high. The log-log plot will yield a considerably flattened curve. This is one of the reasons it is recommended that these log-log plots be done in the field, so that such conditions can be detected early, and steps be taken to minimize any adverse environmental impact of aquifer cross contamination.

5.3.1 Neuman and Witherspoon Method

The Neuman and Whitherspoon approach to solving the problem of evaluating a leaky aquifer is two-fold. First, they assumed that if the distance between observation well and pumping well is minimized, the area of aquitard subjected to stress and possible leakage is minimized, and the Theis method could be employed. Next, they assumed that if only early time drawdown data were used, the effects of leakage could be further reduced. This is accomplished by closely monitoring the response curve(s) for transducers both in the aquifer and in the aquitard(s) themselves. When the s over t curve begins to flatten, indicating leakage, this is defined as the end of valid aquifer data. Data acquired beyond that point contains components of horizontal (aquifer) flow, and vertical (aquitard) flow. In order to accurately calculate the aquitard parameters, an undisturbed sample of the aquitard must be taken (ASTM D1587-83). The laboratory tests will provide values for storage of the aquitard(s). Conductivity within the aquitard(s) is provided from the s over t plots for transducers located within the aquitard.

5.3.1.1 Assumptions

The same assumptions as in the Theis method hold with the following differences:

- The aquifer is leaky.
- The aquifer and aquitard both have a seemingly infinite areal extent.
- Flow in the aquitard is vertical.
- Drawdown is negligible in both aquifer and aquitard.
- The aquitard has storage.
- The overlying and/or underlying aquifer(s) is capable of releasing water to the pumped aquifer through a decline in head.

5.3.1.2 Procedure

Field Operations

1. See field operations in Section 3.2.1.1.
2. In addition, set piezometers within the aquitard at 0.25 and 0.50 aquitard thickness. These should be set at essentially the same location as the observation well, either in the observation well or in a separate borehole adjacent to the observation well, and the same radial distance from the pumping well.

Data Plots

1. Prepare log-log plots of s over t for the observation well and transducers placed in the aquitard.
2. Use the valid early-time data and the Theis method to calculate the pumped aquifer parameters.
3. Calculate several s'/s ratios for the early time period used in b above.
4. Calculate the parameter tD — a "dimensionless" time parameter — using the following equation:

$$t_D = \frac{Kt}{Sr^2}$$

Equation 25

where

t = Time.
r = Radial distance to observation well (and transducers).
K = Pumped aquifer hydraulic conductivity.
S = Pumped aquifer storativity.

5. Using the value tD calculated in d (above), and s'/s, determine a value for t'D using Figure 4.

5.3.2 DeGlee Method and Hantush Approximation

DeGlee developed the following equation for steady-state drawdown within an aquifer with leakage from an aquitard proportional to the hydraulic gradient across the aquitard:

$$s=\frac{Q}{2\pi T}(K_0)\frac{r}{L}$$ Equation 26

where

T = Transmissivity.
s = Steady-state drawdown in observation well at distance r from pumping well.
Q = Discharge rate of pumping well.
L = Tc = leakage factor.
c = D'/K = hydraulic resistance of the aquitard.
D' = Saturated thickness of the aquitard.
K' = Hydraulic conductivity of the aquitard for vertical flow.
K0(x) = Modified bessel function of the second kind and of zero order (Hankel function).

Hantush observed that if r/L is small (0.05), Equation 14 can be estimated by:

$$s=2.30\frac{Q}{2\pi T}(\log 1.12\frac{L}{r})$$ Equation 27

5.3.2.1 Assumptions

See Section 5.3.1.1.

- Flow to the pumping well is in steady state.
- L is greater than 3D, where D is the saturated thickness of the aquifer.

5.3.2.2 Procedure

Field Operations

Field methods are identical to the Thiem method in Section 5.13.

Data Plots

Plot s over r (log scale) on semi-log paper where r/L is small, the points fall in a straight line plot. Where r/L is large, the curve approaches the zero-drawdown axis asymptotically.

5.3.3 Other Methods for Calculating Leaky Aquifer Parameters

The Neuman and Witherspoon Unsteady-state flow method, the Hantush Curve-Fitting Method, the Hantush Inflection Point Method, and the Walton Method. As before, these are mentioned for the sake of completeness, but not detailed herein.

5.4 UNCONFINED AQUIFERS

Flow to a pumping well in an unconfined (phreatic or water table) aquifer occurs in three phases. In the first phase, pumping has just begun, and the aquifer acts like a confined aquifer. Water is derived from storage (expansion of the water, compression of the aquifer). The time over drawdown plot for this phase closely mimics the Theis type curve. During the second phase, delayed yield occurs. This phenomenon results as water remaining in the pores is drained by gravity (specific yield), replenishing the portion of the aquifer supplying water to the well during the first phase, and results in a reduction in rate of drawdown over the first phase, and a flattening of the time-drawdown plot. The third phase brings equilibration in the rate of drawdown and the time-drawdown plot again looks like the Theis curve.

The duration of the first two phases is a function of the ratio of storage (S) to specific yield (Sy). If the ratio is in the range of 10-1 to 10-2, S is relatively large and the first phase drawdown should be significant. This condition is typical of saturated fine-grained sediments such as silts, clays, and fine-grained sands. If the ratio S/Sy is in the range of 10-4 to 10-6, Sy is relatively large, the second phase phenomenon is expected to be dominant, and coarser-grained sediments (sands and gravels) are indicated.

In addition to S/Sy, the distance between pumping well and observation well also affects the time-drawdown plot. As the distance to the observation well increases, the effects of S decrease.

5.4.1 Neuman Method

Flow to a pumping well in an unconfined (phreatic, or water table) aquifer occurs in three stages. During the first stage, the phreatic aquifer behaves like a confined aquifer, instantaneously releasing water from storage (expansion of the water, compression of the aquifer). This is

illustrated in Figure 5, where the early portion of the family of drawdown curves closely matches the Theis curve (1/uA). The second phase is termed a period of "delayed yield" or "delayed response," in which the rate of drawdown is lower than that predicted by the Theis curve. During this phase, specific yield, or gravity drainage of water remaining in the pore spaces in the vicinity of the pumping well replenishes the water being removed. During the third phase, the rates of yield and drawdown equilibrate, and the time-drawdown plots again converge on the Theis solution (1/uB).

5.4.1.1 Assumptions

- The same assumptions as listed in Section 5.1.1.1.
- At least one observation well located at r/b ≤ 1, where r = distance between pumping well and observation well, and b = aquifer thickness.

Drawdown in the observation well s ≤0.25 b.

5.4.1.2 Procedure

Field Operations

See Section 3.2.1.

Data Plots

1. Prepare A and B curves (Figure 5); tables of these values can be found in many hydrology texts such as Fetter
2. Plot s over t on log-log paper at the same scale as the Type A and B curves
3. Superimpose the late-time drawdown data over the B curves. Note the value for the curve which best matches the field data.
4. Select a match point which has a value of 1 for as many of the variables as possible.
5. At the selected match point, read values for s, t, W(uB,β) and 1/uB
6. Repeat steps a-e, superimposing the early time data over the A curve which has the same β value as the B curve.
7. Read values for s, t, W(uA,β) and 1/uA
8. Substitute these values in the following equations:

$$T_B = \frac{Q}{4\pi s}(W(u_B,\beta))$$

Equation 28. Transmissivity B curve.

$$T_A = \frac{Q}{4\pi s}(W(u'_A\beta))$$

Equation 29. Transmissivity A curve.

$$S_y = \frac{4T_B t u_B}{r^2}$$

Equation 30. Specific Yield.

NOTE: Transmissivities should be within ±10 percent of each other. If they are, their average should be used in the remaining equations, otherwise use the T value from the B curve.

5.5 SLUG TESTS

5.5.1 Preliminary to Operation (All Slug Tests)

Prior to conducting any tests, water level meters, transducers, dataloggers, and other materials should be examined for cleanliness and checked for defects.

Batteries should be checked in the calculator(s), water level meter(s), and datalogger(s).

Decontaminate all intrusive equipment prior to and after use at each location.

Lay plastic sheeting on the ground around the well casing.

Record the well number and other project and site information in the field logbook.

Check the well headspace for the presence of volatile organic compounds using applicable instruments. Record the results in the field logbook.

Measure and record the initial water level in the well and total depth of the well.

NOTE: If the static water level and water levels caused during testing are above the top of the screened, or the well consists of an open hole with no casing, then both rising-head and falling-head tests should be conducted.

If the static water level is at or below the top of the screened or open-hole interval, a rising-head test only should be conducted (i.e., falling-head slug tests are invalid for this situation).

5.5.2 Option 1 – Inert Object Insertion

This procedure describes the use of a solid slug to change the water level in a well.

Select an appropriate transducer for the range of water level change anticipated in the slug test.

Submerge the transducer in the well to a sufficient depth to provide effective performance. The range of the transducer must be considered in the determination of the submersion depth. Well bottom sediment plugging of the transducer must be avoided as well as transducer interference by the inert object.

Monitor water level until it returns to original level as measured in Section 5.5.2 before initiating the test.

Tie off the line to a decontaminated, inert cylinder (slug) prior to lowering it into the well. All intrusive equipment must be decontaminated.

"Instantaneously," but smoothly, lower the cylinder into the well, displacing the water and thereby raising the water level.

Measure and record water levels in the well initially. Record the water level response during cylinder insertion and every 5-10 seconds with the cylinder in place. Record the falling water level and time of each measurement in the field logbook and/or Field Permeability Test Data Sheet.

NOTE: If transducer and datalogger are employed, care must be taken to create backup copies and/or hardcopies of these data as soon as practicable.

Record data until water level has stabilized, or approximately 90 percent of the change in the water level has dissipated. The time for this to occur may range from less than 1 minute to more than a day. Usually, it is not necessary to continue measurements for more than a few hours because longer periods indicate extremely low hydraulic conductivity. Choose the time interval between measurements according to how rapidly the water level approaches the static level. From 10 to 30 measurements should be obtained at approximately equal time intervals during the recovery.

"Instantaneously," but smoothly, remove the cylinder from the well.

Conduct a rising head slug test by measuring the response of the water level to the removal of the cylinder. Record water levels and time until the water level equilibrates to 90 percent of the initial level, and record data as in Section 5.5.2.

Calculate the aquifer hydraulic conductivity using appropriate equations.

Decontaminate the slug and the tape or meter.

5.5.3 Option 2 – Adding or Removing Water

This procedure describes the use of a pump to change water level in a well and a datalogger and pressure transducer to measure the water level. The technique is intended for use in wells installed in highly permeable materials where the use of a slug may not induce a measurable

change in the water level. However, this technique can also be used in wells installed in low permeability materials. The methods described for the transducer/datalogger are also appropriate when using a solid slug or bailer for inducing a change in the water level in a well.

Select an appropriate transducer for the range of water level change anticipated in the slug test.

Submerge the transducer in the well to a sufficient depth to provide effective performance—usually this is to a depth at which its pressure rating is not exceeded but no less than 5 ft of water is above the transducer. The range of the transducer must be considered in selection of the submersion depth.

Check the transducer calibration at two different depths in the well. The transducer should be at least 1 ft above the bottom of the well to prevent bottom sediment from fouling the transducer and preventing accurate readings.

Turn on the datalogger to view the water level value (either in depth of submergence or depth to water).

IF REMOVING WATER: Insert the pump piping with check valve (if using a centrifugal pump) or submersible pump with check valve into the well at least 4 t below the water table but above the transducer. Attach pump discharge to treatment system, portable tanks, or drums to containerize the effluent if it is known or suspected to be contaminated.

— OR —

IF ADDING WATER: Insert piping from water source to a depth of about 1 ft below the surface of the water table (this will prevent undue aeration of the water column and possible anomalous readings).

Monitor water level until it returns to original level as measured in Section 3.1.2 before initiating the water addition/extraction test.

View water level value on datalogger. Values (either depth of submergence or depth to water) should be stable and approximately the same range as in Step C; if not, wait until the water level equilibrates to the initial value.

Begin logging and record the time.

Turn pump on until 4-5 ft of drawdown occurs or the well (at the depth of the poly pipe or pump) goes dry.

— OR —

Turn on water at supply tank until the water level in the well rises a minimum of 5 ft.

Shut off pump or water supply at the tank. Remove piping or pump to a position above the initial water level (water removal) or remove from the well (water addition or removal).

Record water levels and water volumes removed/added during the entire test.

Record water levels until approximately 90 percent of the change in the water level has dissipated. The time for this to occur may range from less than 1 minute to more than a day. Usually, it is not necessary to continue measurements for more than a few hours, because longer periods indicate extremely low hydraulic conductivity.

Periodically collect water levels manually using an electronic water level meter of tape to verify the datalogger values.

End logging and record the time. Remove the poly pipe and/or pump and pressure transducer and transfer data in datalogger to a computer disk. Make a backup copy of the file and record the file information in the field logbook.

Decontaminate intrusive equipment.

Calculate the aquifer hydraulic conductivity using appropriate equations.

5.5.4 Data Plots

Record the water level in the well immediately after the inert object emplacement/withdrawal (Option 1) or at the equilibrium of the water table (Option 2). This is the initial water level reading.

Following the initial water level reading, the water level in the well is continuously recorded along with the time of the level measurement.

The ratio of the initial water level to the change in head is plotted with respect to time.

The ratio is plotted on the arithmetic scale and time is plotted along the logarithmic scale.

The relationships of the initial water level to changes in the water level are a function of parameters shown on Figure 6 and the formation transmissivity. The values of the function relationship are plotted for a series of transmissivities and are depicted in Figure 5.

The resulting field data plot (curve) is compared to a series of type curves (Figure 5). The field-data curve is placed over the type curves with the arithmetic axis coincident. The field data curve is matched to the type curve that has the same curvature.

The formation transmissivity is determined.

The value of storativity is calculated.

6. REPORTS

After performing hydrologic tests, the contractor will generate a report which must contain a minimum of the following elements:

- A field data report including a site description, plots of water level and discharge with time, and a precursory analysis of data.

- The introduction should include the purpose of the test, dates and times water-level measurements were begun, dates and times discharge or injection was begun and ended, and the average rate of discharge or injection.

- Well logs (including construction diagrams) of all control wells, observation wells, and piezometers.

- Site map showing all well locations, distances between wells, and locations of all geologic boundaries or surface waterbodies which might affect the test.

- The locations of wells and boundaries which may affect the test generally need to be accurate within a radial distance of ±0.5 percent. For large-scale studies, it may be sufficient to locate wells from maps or aerial surveys. Small-scale studies require that well locations be surveyed. Additionally, other features such as faults, streams, and canals should be located. Well deviation surveys, which determine true horizontal distance between well screens, may be necessary if test wells are deep relative to their spacing.

7. MAINTENANCE

The transducers must be kept clean, operable, and thoroughly tested before emplacement in the well. A plugged or malfunctioning piezometer will give erroneous responses or fail to give any response.

8. PRECAUTIONS

In the case of slug tests, care should be exercised to maximize the efficiency of the well. If there is a great disparity between the conductivity of the aquifer and that of the well screen/filter pack, one may find that the slug test has accurately measured the conductivity of the well screen/filter pack rather than the aquifer.

Transducers should be double checked to ensure that they are calibrated in the correct water level range. Water level and well depth should be checked with an electronic water level meter or steel tape before and after placing the transducers.

Be sure that the wells used are well developed.

If the water removed is contaminated and cannot be discharged at the surface, a tank of sufficient size to hold the effluent of the pumping test must be available.

9. REFERENCES

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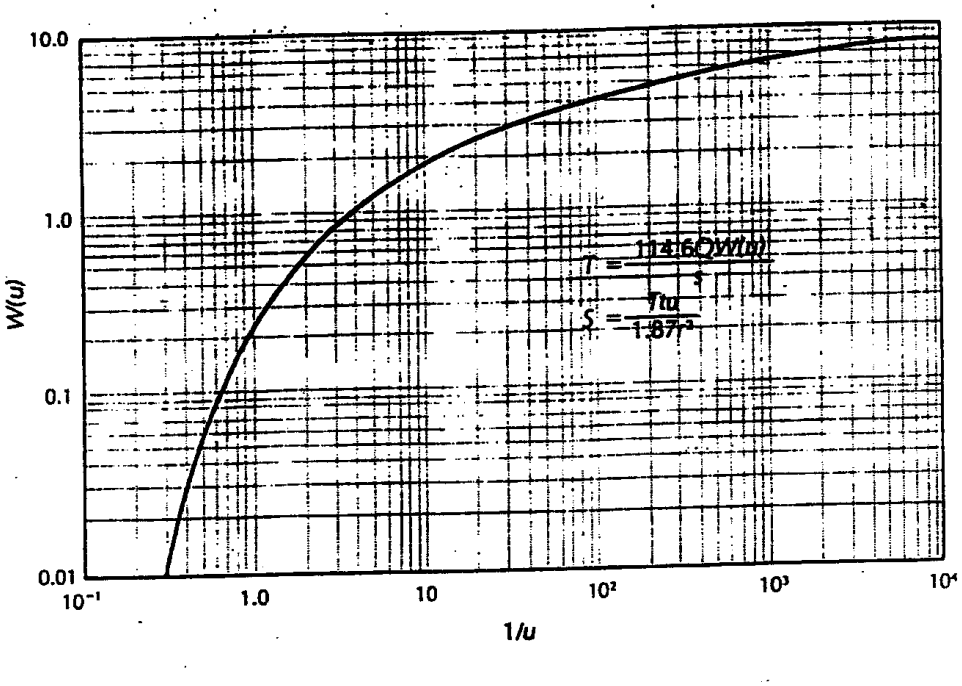

Figure SOP033-1. Theis type curve (after Fetter).

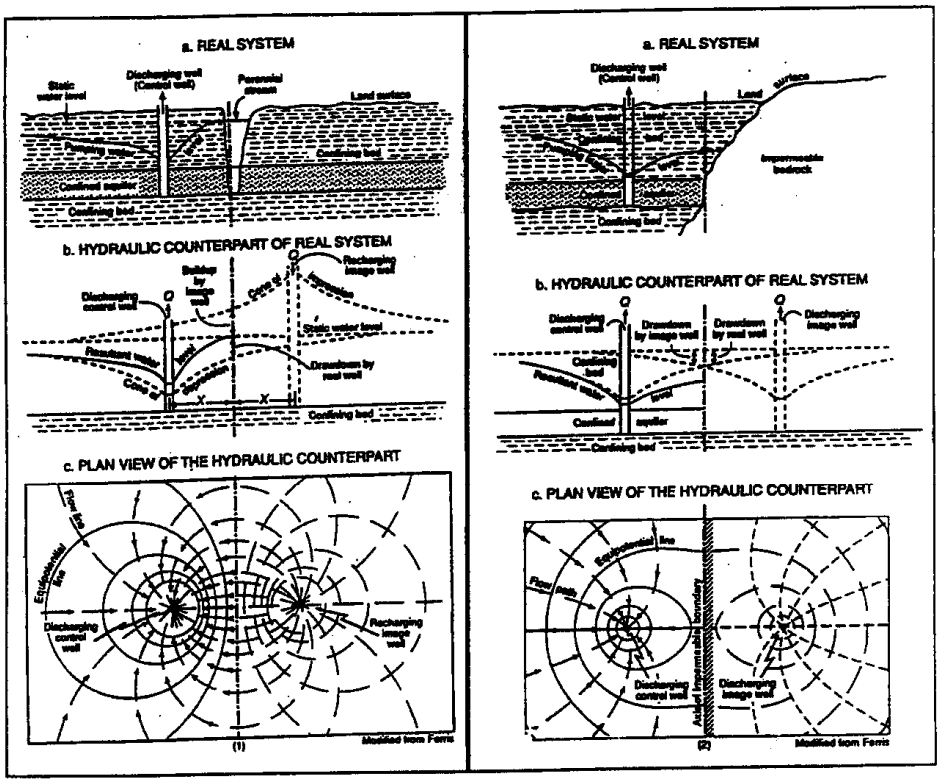

Figure SOP033-2. Illustration of boundary conditions
(after ASTM D-5270-92).

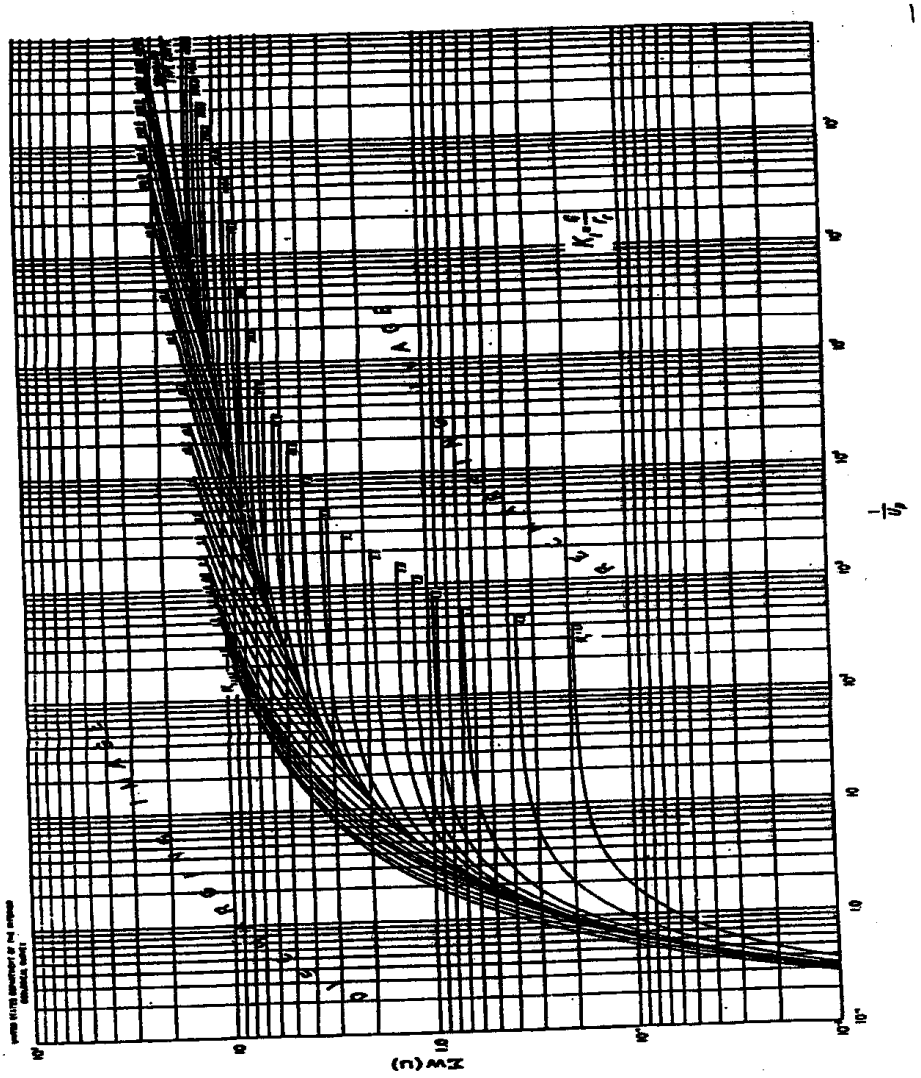

Figure SOP033-3. Family of type curves for the solution of the Modified Theis formula (after ASTM D-5270-92).

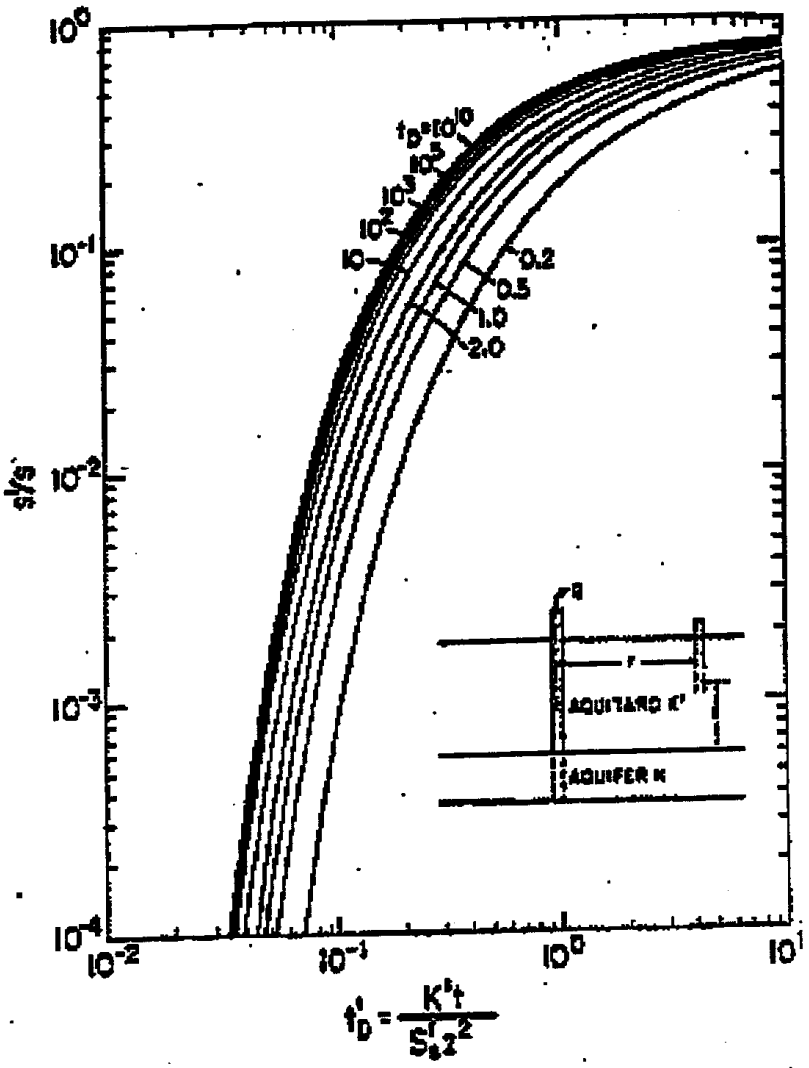

Figure SOP033-4. Variation of s'/s with t'_d for a Semi-Infinite Aquitard (after Reynolds).

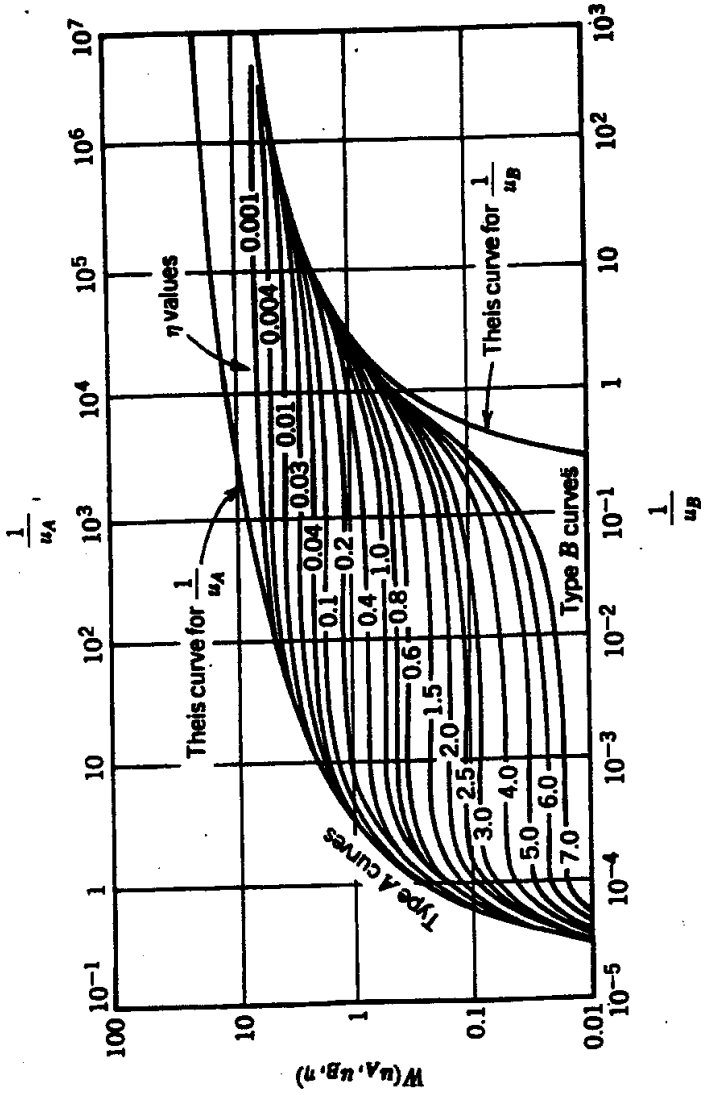

Figure SOP033-5. A and B type curves (after Dominico).

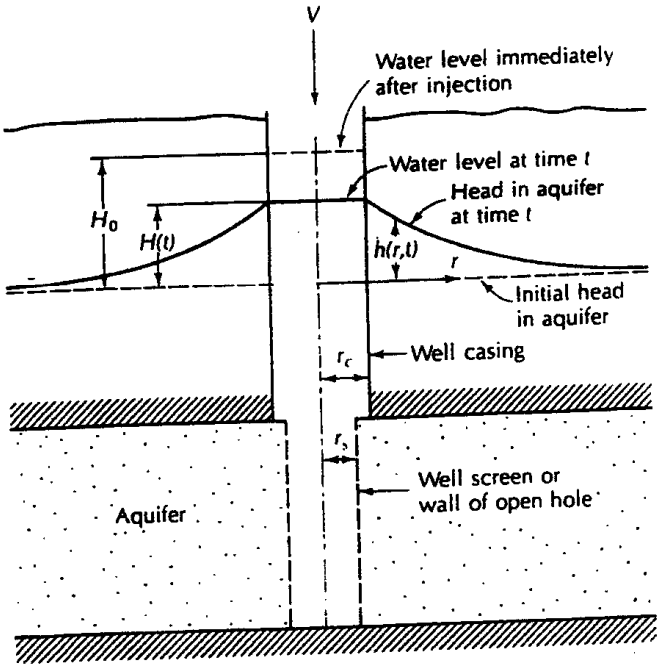

Figure SOP033-6. Well parameters – slug tests (after Fetter).

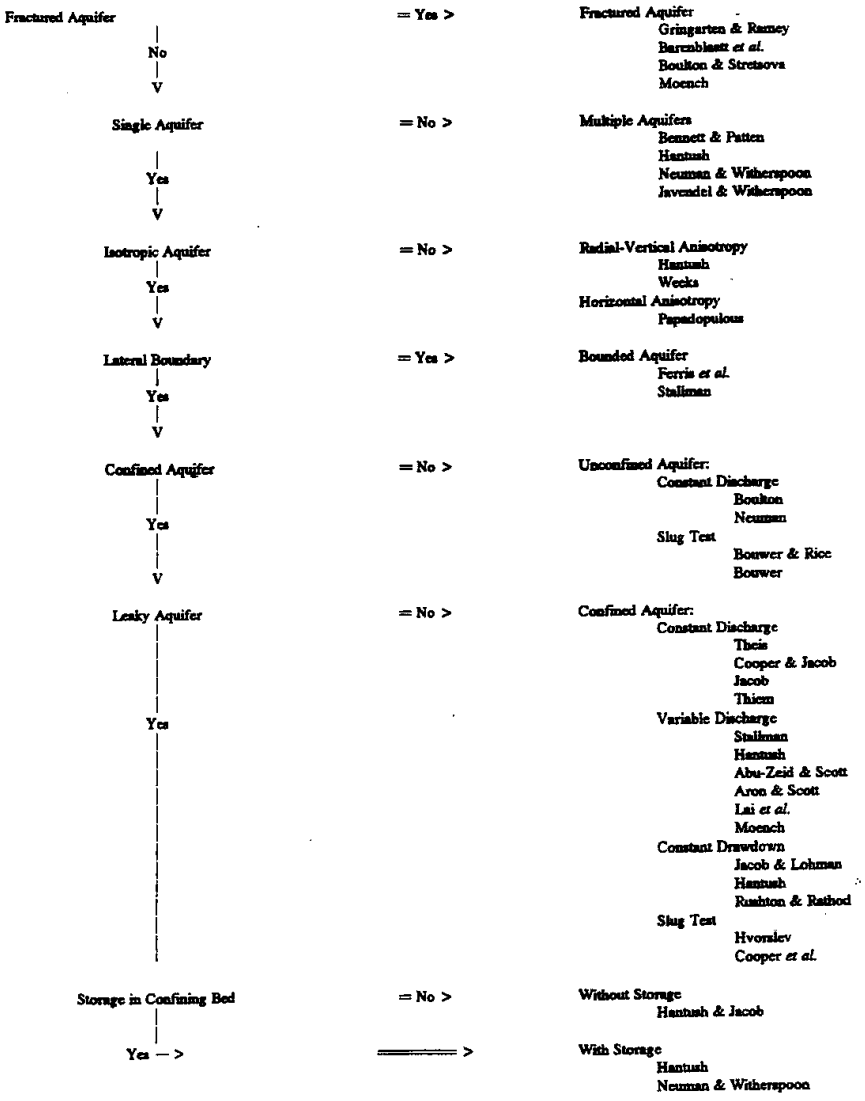

Table SOP033-1. Decision tree for selection of aquifer test method (after ASTM D-4043-91).

Standard Operating Procedure No. 33A

Packer Testing and Sampling

Prepared by

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Revision 0
December 2013

CONTENTS

1. SCOPE AND APPLICATION

The purpose of this Standard Operating Procedure (SOP) is to describe the use of a straddle packer assembly within bedrock cores. The methods are designed to enable completion of hydraulic testing using procedures detailed in SOP-33 and collection of discrete groundwater samples using sampling procedures as detailed in SOP-13 and/or SOP-48 from different bedrock intervals where the water is not mixed.

2. MATERIALS

In addition to materials required for conducting aquifer tests as described in SOP-33 and collecting groundwater samples from standard wells as described in SOP-13 and/or SOP-48, the following materials may be required that are associated with the straddle packer assembly:

SD Straddle Packer	Air compressor and associated air line
2-in. polyvinyl chloride (PVC) piping	Personnel protective equipment as required by the Accident Prevention Plan

3. PROCEDURES FOR INSTALLING THE PACKER SYSTEM

A single packer will be used to isolate the lowermost section of the borehole. If needed, an appropriate section of 2-in. screen (5-ft or 10-ft) will be placed at the end of the single packer to prevent the corehole from infilling or collapsing.

A multi-level straddle packer system will be used isolate desired areas within the upper and mid sections of the corehole. An appropriate section of 2-in. screen (5-ft or 10-ft) will be placed between packers.

- Following removal of the core barrel from the interval to be tested and/or sampled, the depth to water will be measured per SOP 10 to the nearest hundredth of a foot using an electronic water level meter. A consistent reference point will be used throughout the test (top of outer casing) and the distance from the reference point to ground surface will be recorded. The water level will be allowed to stabilize to obtain a static depth to water prior to inflating the packers, which will provide an average hydraulic head for the open interval.
- The corehole volume will be calculated for the interval to be tested and/or sampled, with volumes calculated from the packer to the bottom of the corehole for single packer systems or for the screened interval for multi-level packer systems.
- The packer system will be placed and inflated with compressed air (i.e., nitrogen, 40-80 PSI) at the desired interval (i.e. 5-10 ft above the bottom of the corehole, with the appropriate amount of 2-in. PVC casing to reach the desired depth). A submersible pump (i.e. Grundfos) will then be lowered through the packer assembly to the middle desired testing/sampling interval. If a pressure transducer with logging and display capabilities will be used during aquifer testing, the transducer will be lowered to a sufficient depth to provide effective performance (e.g., immediately above the top of the packer system).

4. PROCEDURES FOR CONDUCTING AQUIFER TESTS

Aquifer tests will be conducted in the selected bedrock interval following the procedures below and the appropriate field operations procedures in SOP-33.

- Following the inflation of the packer(s) using compressed gas (e.g., nitrogen), the water level will again be allowed to stabilize prior to recording the depth to water with an electronic water level meter. The water level above the packer will be monitored throughout pumping to ensure that leakage is not occurring into the desired testing/sampling interval.

- If a pressure transducer with logging and display capabilities will be used during aquifer testing, the data logger will be programmed and started.

- Pumping will be initiated at a relatively low discharge rate that can be maintained throughout the test (e.g., 500 mL per minute) and that yields a readily measurable drawdown (e.g., 4-6 in.). Observations related to the apparent openness of fractures (e.g., degree of weathering, iron staining, rock core density, water loss) made during coring should be taken into account to determine the initial pumping rate.

- The depth to water will be recorded over time using an electronic water level meter, at initially short intervals that decrease as the water levels begin to stabilize (e.g., every 30 seconds for the first 5 minutes, and every 3-5 minutes for the next 20 minutes). The pumping rate and volume of water removed will be recorded periodically during the test using a flowmeter or stopwatch and bucket. The pumping rate may need to be adjusted to allow measurable drawdown without dewatering the interval. Any adjustments in the pumping rate should be noted.

- Drawdown data (i.e., the difference between the static water level and the depth to water during pumping) from the test will be plotted vs. elapsed pumping time on log-log or semi-log graphs. Aquifer tests are anticipated to continue for 30 minutes; however, a test may continue beyond the anticipated 30 minutes, if required to approach pseudo- or steady state conditions.

- Note that for fractured rock systems, early pumping time data likely represent groundwater flow from the fractures, and therefore would overestimate the bulk hydraulic conductivity of the bedrock. Following this early pumping time, a transition occurs where the groundwater storage in rock matrix begins to supply water to the fractures and stabilize drawdown. In late pumping time data, groundwater would come out of storage from both the fractures and the matrix (Kruseman and DeRidder, 2000).

5. PROCEDURES FOR COLLECTING GROUNDWATER SAMPLES

Groundwater samples will be collected from tested intervals for laboratory analysis using the following procedures:

- At the end of the aquifer test, the rate of flow should be decreased to minimize volatilization.
- A total of 3-5 interval volumes (including the discharge line) will be purged prior to sample collection (including during aquifer testing). Field parameters (pH, temperature, conductivity, DO, ORP) will be monitored during the purge as detailed in SOP-43, and any sheens or odors will be noted.
- At the end of the aquifer test, the rate of flow should be decreased to minimize volatilization and a groundwater sample will be collected for VOC analysis as detailed in SOP-13 and/or SOP-48.
- Groundwater samples will be collected by directly filling sample containers provided by the analytical laboratory. Sample containers will be labeled, tracked via chain of custody forms, and packed and shipped to an offsite laboratory for analysis, as per SOPs 1, 2, and 4, respectively.

6. INVESTIGATIVE DERIVED WASTE

Investigative derived waste generated during purging and sampling will be disposed of per SOP-42.

7. DECONTAMINATION

Packer systems and non-dedicated sampling equipment will be decontaminated prior to use, between coring locations, and following completion of packer testing/sampling as detailed in SOP-05.

8. PRECAUTIONS

Prior to collecting any samples, consult the Accident Prevention Plan for personal protective equipment required for sampling activities.

Decontaminate the sampling equipment and change gloves between samples to minimize the risk of cross-contamination.

9. REFERENCES

Kruseman and DeRidder. 2000. “Analysis and evaluation of pumping test data.” International Institute for Land Reclamation and Improvement, June 1, 1990.

Standard Operating Procedure No. 039
for
Sample Preservation and Container Requirements

Prepared by

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Revision 1
April 2012

CONTENTS

Page

1. PURPOSE AND SCOPE

The purpose of this Standard Operating Procedure (SOP) is to define the preservatives and techniques to be employed in preserving environmental samples between collection and analysis.

2. MATERIALS

The following materials may be required:

Containers (see Section 3 for description)	NaOH
HNO_3	Ice chests
H_2SO_4	Ice

3. DEFINITION OF CONTAINER TYPES

Listed below are the definitions of various container types.

Type	Container	Closure	Septum
A	80-ounce (oz) amber glass, ring handle bottle/jug, 38-millimeter (mm) neck finish	White polypropylene or black phenolic, baked polyethylene cap, 38-430 size, 0.015-mm polytetrafluoroethelyne (PTFE) liner	
B	40-mililiter (mL) glass vial, 24-mm neck finish	White polypropylene or black phenolic, open top, screw cap, 15-mm opening, 24-400 size	24-mm disc of 0.005-in. PTFE bonded to 0.120-in. silicon for total thickness of 0.125 in.
C	1-L high density polyethylene, cylinder-round bottle, 28-mm neck finish	White polyethylene cap, white ribbed, 28-410 size; F217 polyethylene liner	
D	120-mL wide mouth glass vial, 48-mm neck finish	White polyethylene cap, 40-480 size; 0.015-mm PTFE liner	
E	250-mL Boston round glass bottle	White polypropylene or black phenolic, open top, screw cap	Disc of 0.005-in. PTFE bonded to 0.120-in. silicon for total thickness of 0.125 in.
F	8-oz short, wide mouth, straight-sided, flint glass jar, 70-mm neck finish	White polypropylene or black phenolic, baked polyethylene cap, 48-400 size; 0.030-mm PTFE liner	
G	4-oz tall, wide mouth, straight - sided, flint glass jar, 48-mm neck finish	White polypropylene or black phenolic, baked polyethylene cap, 48-400 size; 0.015-mm PTFE liner	

Type	Container	Closure	Septum
H	1-L amber, Boston round, glass bottle, 33-mm pour-out neck finish	White polypropylene or black phenolic, baked polyethylene cap, 33-430 size; 0.015-mm PTFE liner	
K	4-L amber glass ring handle bottle/jug, 38-mm neck finish.	White polypropylene or black phenolic, baked polyethylene cap, 38-430 size; 0.015-mm PTFE liner	
L	500-mL high-density polyethylene, cylinder bottle, 28-mm neck finish	White polypropylene, white ribbed, 28-410 size; F217 polyethylene liner	

4. PROCEDURE

All containers described in Section 3 must be certified clean, with copies of laboratory certification furnished upon request. There may be circumstances when alternative containers will be used (e.g., aluminum foil around tissue samples placed in plastic bags, plastic buckets for large soil/sediment samples, etc.) for which laboratory certification may not be available. Such containering should be appropriately decontaminated or verified appropriately clean prior to using.

Water samples will be collected into pre-preserved containers appropriate to the intended analyte as given in Quality Assurance Project Plan. Samples taken for volatile organic compounds will be collected in accordance with SOP No. 003, Section 3.3.8. Samples taken for metals analysis will be verified in the field to a pH <2. The container should be tightly capped, then swirled to thoroughly mix the sample. The cap will then be loosened to release any excess pressure this operation may have generated. Samples taken for total phosphorous content will be verified in the field to a pH <2. The container should be tightly capped and swirled to thoroughly mix the sample. The cap will then be loosened to release any excess pressure this operation may have generated. Samples taken for cyanide will be verified for a pH >12. Most other samples do not require added preservation; however, there are analytes that may require special preservation, i.e., sulfide which requires a zinc acetate preservation. Preservation must be performed as documented in the project-specific Quality Assurance Project Plan. These samples will be immediately placed on ice and cooled to 4±2°C.

Soil and sediment samples will be collected into containers appropriate to the intended analyte as given in the Quality Assurance Project Plan. Samples taken for volatile organic compound analysis will collected in accordance with the site-specific SOP. Samples taken for metals analysis will be tightly capped, placed on ice, and maintained at a temperature of 4°C. Samples taken for total phosphorous content will be tightly capped, placed on ice, and maintained at a temperature of 4°C. Under most circumstances, no preservatives will be added to any other soil samples; follow project-specific requirements as documented in the Quality Assurance Project Plan. These samples will be immediately placed on ice and cooled to 4±2°C.

5. MAINTENANCE

Not applicable.

6. PRECAUTIONS

Note that acidifying a sample containing cyanide may liberate HCN gas.

- Avoid breathing any fumes emanating from acidified samples.
- Acidify samples only in the open, rather than in closed spaces, i.e., a vehicle.
- Hold suspected HCN-generating sample away from body and downwind while manipulating it.
- See the Health and Safety Plan for other safety measures.

7. REFERENCES

U.S. Environmental Protection Agency (EPA). 1986. Test Methods for Evaluating Solid Waste, SW-846.

———. 1987. A Compendium of Superfund Field Operations Methods, EPA 540-P87-001.

———. 1991. A Compendium of ERT Soil Sampling and Surface Geophysics Procedures.

Standard Operating Procedure No. 042
for
Disposal of
Investigation-Derived Material

Prepared by

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Revision 0
August 2007

CONTENTS

1. SCOPE AND APPLICATION

The purpose of this Standard Operating Procedure is to define the required steps for disposing of investigation-derived material (IDM) generated during field activities.

IDM, as used herein, includes soil cuttings, drilling muds, purged groundwater, decontamination fluids, and disposable personal protective equipment. For the sake of clarity and ease in use, this Standard Operating Procedure is subdivided into procedures for disposal of liquid IDM and solid IDM as follows:

- Liquid IDM (Section 3.2) includes the following materials:
  - — Water from initial development of new wells and the redevelopment of existing wells.
  - — Purge water from groundwater sampling.
  - — Decontamination fluids (Section 3.4)
- Solid IDM (Section 3.3) consists of the following materials:
  - — Drill cuttings from monitoring well installation
  - — Grout, a mixture of cement and bentonite, generated during installation of monitoring wells
  - — Disposable personal protective equipment (Section 3.4).

2. MATERIALS

The following materials may be required:

Any additional equipment that may be dictated by project or site-specific plans	Hazardous waste labels
Bar codes	Permanent marker
Chain-of-custody forms	Field Logbook (bound)
Department of Transportation 17C spec. metal containers	Waste identification labels

3. PROCEDURE

3.1 GENERAL

No container will be labeled as a “Hazardous Waste” unless the contents are in fact known to be hazardous as defined by 40 CFR 261.

IDM may be disposed onsite if it is: (1) initially screened, or evaluated to determine whether it is contaminated; (2) not abandoned in an environmentally unsound manner; and (3) not inherently waste-like.

IDM are to be considered contaminated if they: (1) are visually or grossly contaminated, (2) have activated any field monitoring device which indicates that the level exceeds standard Level 1, (3) have previously been found to exhibit levels of contamination above environmental quality standards, and (4) the responsible party and/or appropriate regulator deem(s) that records of historical uses indicate that additional testing of the IDM is needed, or additional caution is warranted handling IDM from a given site.

3.2 PROCEDURES FOR LIQUID INVESTIGATION-DERIVED MATERIAL DISPOSAL

1. All water from initial development of new wells, and purge water generated during the first round of groundwater sampling will be containerized in Department of Transportation approved 55-gal drums. Decontamination fluids may be bulk-containerized until completion of field task.

2. Label all containers as to type of media, the date the container was sealed, the point-of-generation, and the points-of-contact. The well number and container number will be identified on the container.

3. The contractor/support personnel will log all media generated onsite into a bound Field Logbook. Media information should include the following: the date of generation, contents of containers, the number of containers with the same contents (if applicable), location of containers, the well number the media is associated with, personnel sampling the media, sampling dates, and sampling results.

4. Containers of well development water and purge water may be stored at the well site pending first round analytical results.

5. Laboratory turnaround time must be no greater than 30 days. Upon receipt of the analytical results, a copy will be furnished to the client within 3 working days. Both the client and contractor will evaluate the data to determine disposal requirements, per state and local regulations. A disposal decision is required within 10 days of receipt of sampling results. Appropriate disposal must be performed no later than 50 days from the decision date unless prevented by inclement weather (e.g., rain and muddy conditions may preclude site access, freezing weather may freeze media).

1. This value is defined as two times background, where “background” values are to be determined as follows: (1) regional background values will be used where they are available; and (2) if regional values are not available, background may be empirically determined at uncontaminated sampling sites using onsite sensors such as organic vapor analyzers (photoionization detector or flame ionization detector), scintillometers, etc.

Dispose of non-hazardous media in accordance with Step 6 et seq. through 8 et seq. of this procedure.

Dispose of hazardous waste in accordance with Step 9 et seq. of this procedure.

6. If the first round analytical data of the liquid media is below the Maximum Contaminant Levels established by the Federal Safe Drinking Water Act, the water may be gradually infiltrated into the ground at least 50 ft downgradient of the well.

   If the well location has no downgradient area, the water will be infiltrated into the ground in an area deemed appropriate by the client and the contractor/support personnel.

   Disposal locations must allow percolation of the water and prohibit "ponding."

   Upon completion of water discharge to ground, enter type of media, amount of media, date of disposal, and discharge point(s) in a bound Field Logbook and provide this information to the client.

   Empty containers are to be properly decontaminated, stored, and reused by the appropriate personnel.

   If the liquid media sampling results do not meet the required Maximum Contaminant Levels and cannot be discharged to the ground, then determine if the waste meets the sanitary sewer discharge criteria (National Pollutant Discharge Elimination System standards).

7. If at any time visual contamination of purge/development water is observed, or if organic vapor monitor readings (HNu, photoionization detector) register more than 5 ppm above background and/or rad meters register more than twice the background mrems, then the liquid will be drummed and a composite sample will be taken that day. A disposal decision will be based on the analytical results of this sample rather than the first round of analytical results.

3.3 PROCEDURE FOR SOLID INVESTIGATION-DERIVED MATERIAL DISPOSAL

1. If the conditions outlined in Section 3.1 are met, proceed to Section 3.3, Step 2; otherwise, go to Section 3.3, Step 7.

2. During drilling operations, the resulting cuttings and mud will be discharged onto the ground near the well if the following conditions are met: (1) no visual contamination is observed, (2) organic vapors are less than 5 ppm above background, (3) rad meter readings (if applicable) are under two times background, and (4) if the potential for metals contamination exists, the medium has been screened and found to be less than two times background.

Proper sediment and erosion control measures will be implemented as follows:

- Drill cuttings will be uniformly spread and contoured to blend with the surroundings of the site.
- If amount of solid IDM exceeds 5,000 ft^2 or 100 yd^3 of material, a Sediment and Erosion Control Plan is required.
- If the amount of solid IDM is under 5,000 ft^2 or 100 yd^3, the site will be stabilized as soon as possible. Stabilization includes mulch, seed, and tack.
- Critical areas require stabilization within 7 days from the date of well completion. Critical areas include swales, water sources, drainage ditches, etc.
- All other disturbed areas require stabilization within 14 days from the date of well completion.

3. If the well location is in or near a wetland, the drill cuttings will be drummed and transported away from the site for spreading.

4. Label all IDM containers that will not be spread on the day of generation. Each container should be labeled with the type of media, the date the container was sealed, the point-of-generation, and the name of the contact person. The well number and container number should be identified on the container.

5. The contractor/support personnel will log all media generated onsite into a bound Field Logbook. Media information should include: the date of generation, contents in containers, the number of containers with the same contents, location of containers, and the well number the media is associated with.

6. Containers will be staged at the well site until contractor/support personnel spread the cuttings in the appropriate locations, using proper sediment and erosion control measures per Section 3.3 et seq.

7. If drilling mud and cuttings show visible contamination, or organic vapor readings are more than 5 ppm above background levels, or rad meter readings (if applicable) show greater than two times background levels, media will immediately be containerized, labeled appropriately (Section 3.2), and sampled on the same day.

8. The solid IDM should be sampled and appropriate Toxicity Characteristic Leaching Procedure analyses conducted prior to determining disposition. Laboratory turn-around time must be no greater than 30 days. Upon receipt of analytical results, a copy will be furnished to the client within 3 working days. The contractor will evaluate the data to determine disposal requirements within 10 days. Appropriate disposal must be performed no later than 50 days after the decision date if weather permits (Section 3.2).

- If the solid IDM are determined to be non-hazardous and uncontaminated, go to Section 3.3.

- If the solid IDM are determined to be non-hazardous but contaminated , go to Section 3.3.

- If the solid IDM are found to be hazardous wastes, go to Section 3.3.

9. If the solid IDM are not a hazardous waste **and** analytical data shows contaminant concentrations below the U.S. Environmental Protection Agency Region 3 (or applicable Region where work is being performed) Risk-Based Concentrations, contact the appropriate federal, state, or local agency for approval to discharge onto the ground near the site of generation.

    - Follow steps detailed in Section 3.3, Step 2 et seq. (above) pertaining to sediment and erosion control.

    - Upon completion of the solid IDM discharge to the ground, enter type of media, amount of media, date of disposal, and discharge point(s) in a bound Field Logbook. This information must be provided to the client.

    - Empty containers are to be properly decontaminated, stored and reused by appropriate personnel.

10. If the intrusive media is not a hazardous waste but analytical data shows concentrations above the screening criteria, dispose of the IDM according to state and local regulations.

    - Ensure that the waste containers are properly labeled as applicable in accordance with Section 3.3, Step 4.

    - Inform the client of the type and amount of waste, and the location of the waste.

    - When the waste is removed, enter the type of waste, amount of waste, date of pickup, and the destination of the waste in a bound Field Logbook. This information must be provided to the client.

3.4 PROCEDURES FOR DECONTAMINATION SOLUTION AND PERSONAL PROTECTIVE EQUIPMENT DISPOSAL

Decontamination solutions include catch water from steam-cleaning operations performed on large sampling equipment, drill rigs, and drums, as well as smaller quantities of soapy water and rinse solutions used in decontaminating field sampling equipment. At the completion of the field event, a composite sample of the decontamination solution will be taken. The decontamination solution will be treated as liquid IDM pending results (Section 3.2 et seq.).

Personal protective equipment will be containerized onsite, appropriately labeled, and disposed of in a designated trash receptacle.

4. MAINTENANCE

Not applicable.

5. REFERENCES

Environment Article Section 7-201(t).

U.S. Environmental Protection Agency. 1991. Management of Investigation-Derived Wastes during Site Inspections PB91-921331, OERR Directive 9345.3-02. Office of Emergency and Remedial Response U.S. Environmental Protection Agency, Washington, D.C. May.

Standard Operating Procedure No. 043 for Multi-Probe Water Quality Monitoring Instruments

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Revision 1
April 2011

CONTENTS

1. PURPOSE AND SCOPE

The purpose of this Standard Operating Procedure is to delineate protocols for field operation of multi-probe water quality instruments. The instrument can monitor a variety of basic parameters including dissolved oxygen, percent saturation, temperature, pH, specific conductance, resistivity, salinity, total dissolved solids, oxidation reduction potential (ORP), level, and depth.

Use of brand names in this Standard Operating Procedure is not intended as endorsement or mandate that a given brand be used. Alternate equivalent brands of detectors, sensors, meters, etc. are acceptable. If alternate equipment is to be used, the contractor will provide applicable and comparable standard operating procedures for the maintenance and calibration of same.

2. MATERIALS

The following materials may be required:

- Multi-probe instrument
- Probe/sonde with appropriate cables
- Appropriate standards
- Accessories (batteries, charger, case, etc.)
- Instrument logbook
- Manufacturer's Operations Manual.

3. CALIBRATION PROCEDURE

Calibration must be performed daily at a minimum before using the instrument. Calibration may be performed in the laboratory or in the field. Detailed step-by-step calibration procedures for the equipment described below are provided in the most recent version of the manufacturer's Operations Manual. Documentation includes at a minimum: time, date, analyst, standard, primary standard lot number, secondary standard lot number, and expiration dates of standards.

Fill the calibration cup with the appropriate standard as follows:

- Temperature: None required
- Specific Conductance: Conductivity standards
- pH: pH 7 buffer plus pH 4 and/or pH 10 buffer
- Dissolved Oxygen: Saturated air or saturated water
- ORP: Quinhydrone (Zobell's Solution)
- Turbidity: Nephelometric turbidity unit (NTU) standards
- Salinity: Calibration for specific conductance
- Depth/Level: Set zero in air.

3.1 CONDUCTIVITY CALIBRATION

Conductivity meters are calibrated at least once per day to at least one standard. The standard should be selected in accordance with the range expected to be measured (e.g., 1.0 µS/cm standard should not be used to calibrate meters being used in saltwater). See manufacturer's recommendations in the Operations Manual for additional information on calibration standard selection. Calibration information is recorded in conjunction with the data collected for that sampling event.

3.2 pH CALIBRATION

The pH meters are calibrated at least once per day to a minimum of two standard buffers (pH 4 and 7, or pH 7 and 10) in accordance with the range expected to be measured. The calibration is verified using a fresh solution of pH 7 buffer post-calibration. Calibration information is recorded in conjunction with the data collected for that sampling event.

3.3 DISSOLVED OXYGEN CALIBRATION

Dissolved oxygen meters are air calibrated at least once per day. Calibration information is recorded in conjunction with the data collected for that sampling event.

3.4 OXIDATION REDUCTION POTENTIAL CALIBRATION

ORP meters are calibrated at least once per day to at least one standard. It is recommended that Zobell's Solution is used; however, another solution can be used as long as it meets the manufacturer's specifications for calibration. Calibration information is recorded in conjunction with the data collected for that sampling event.

3.5 TURBIDITY CALIBRATION

The turbidity meters are calibrated at least once per day to a minimum of two standards (0 NTU and 100 or 126 NTUs recommended) in accordance with the range expected to be measured. Calibration information is recorded in conjunction with the data collected for that sampling event.

3.6 DEPTH/LEVEL CALIBRATION

The depth and level calibration is performed with the depth sensor module in the air and not immersed in any solution. The appropriate correction for height above the water surface is inputted into the meter. Calibration information is recorded in conjunction with the data collected for that sampling event.

3.7 ADDITIONAL CALIBRATIONS

Additional measurements may be taken with the multi-probe water quality instruments. For any of these measurements, the calibration procedures will be conducted in accordance with the manufacturer's specifications. Calibration information is recorded in conjunction with the data collected for that sampling event.

4. FIELD OPERATION

4.1 SETUP OF MULTI-PROBE WATER QUALITY INSTRUMENT

Post-calibration and prior to sampling, the multi-probe water quality instrument will be set up for data collection. If the cables have been unattached, they will be reconnected to the transmitter (if applicable) and the display. Once all cables are attached, the meter will be turned on and allowed to warm up for a few seconds in order to allow the display screen to load.

4.2 SURFACE WATER

Prior to sampling, check the condition of the probes before each deployment. When sampling in surface water, the sensor must be in an amount of water sufficient for all probes to be submerged. Data values displayed on the display screen are recorded in the field logbook and accepted into the instrument's data logger. Post-data collection, the sensor will be retrieved and rinsed for use at the next sample location. If travel time between sample locations is great, the display is to be turned off. When all sampling is completed, disconnect all equipment and return it to its proper storage location.

4.3 GROUNDWATER

Prior to sampling, check the condition of the probes before each deployment. When sampling groundwater, mount sampler on a flow-through sampler cup. Start sampler pump and allow pump/hose system to be purged of air bubbles. Sampling rate should be set to record all parameters each time 1-3 liters (unless otherwise specified in the sampling plan) have been removed from the well. Record all the monitored values in the appropriate field logbook to ensure against inadvertent data loss.

5. MAINTENANCE

All maintenance should be performed in accordance with the manufacturer's Operations Manual.

6. PRECAUTIONS

Check the condition of the probes frequently between sampling. Do not force pins into connections, note keying sequence. If field readings are outside the expected range, check for bubbles on, or damage to, the probes. If there are no bubbles or damage, recalibrate the sensor.

7. REFERENCES

Manufacturer's Operations Manual.

Standard Operating Procedure No. 047
Direct-Push Technology Sampling

Prepared by

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Revision: 0
August 2007

CONTENTS

1. SCOPE AND APPLICATION

This Standard Operating Procedure (SOP) establishes the protocol for using direct-push technology (DPT) in media sampling and performing subsurface characterization. This SOP includes the following DPT methods: Geoprobe®, Hydropunch®, Cone Penetrometer Testing (CPT), and Site Characterization and Analysis Penetrometer System (SCAPS).

2. MATERIALS

The following materials may be required:

Appropriately sized, all-terrain vehicle-skid-or track-mounted; DPT equipment; and supplies (i.e., hydraulic derrick and hammer assembly)	Personal protective equipment
Bentonite grout and clean sand for DPT hole abandonment	Phosphate-free, laboratory-grade detergent (e.g., Liquinox, Alconox, etc.)
DPT stainless steel rods	Source of approved water
Heavy plastic sheeting	Steam cleaner/sprayer and water obtained from approved source for decontaminating DPT equipment
Logbook	Steel drums for intrusion derived wastes (e.g., contaminated personal protective equipment, decon solutions, etc.)
Long-handled bristle brushes	Wash and rinse tubs
Mini-bailer or tubing and peristaltic pump (groundwater sampling only)	

3. GEOPROBE® AND HYDROPUNCH®

3.1 MATERIALS

Water sources for Geoprobe® and Hydropunch® activities, grouting, sealing, filter placement, well installation, and equipment decontamination must be approved by the Project Manager prior to arrival of the Geoprobe® and Hydropunch® equipment. Information required for the water source includes: water source, manufacturer/owner, address and telephone number, type of treatment and filtration prior to tap, time of access, cost per gallon (if applicable), dates and results associated with all available chemical analysis over the past 2 years, and the name and address of the analytical laboratory (if applicable).

Pure sodium bentonite with no additives will be the only additive allowed, and its use must be approved by the Project Manager prior to the arrival of the Geoprobe® and Hydropunch® equipment. The information required for evaluation includes: brand name, manufacturer, manufacturer's address and telephone number, product number, product description, and intended use for the product.

Portland Type II cement will be used for grout (refer to SOP No. 019).

3.2 GROUNDWATER – HYDRAULIC PUSHING AND SAMPLING

The objective of the selected DPT sampling technique is to allow grab samples to be taken at a selected site to facilitate aquifer characterization and analysis of potential contaminants. The analytical results from sampling can also be used to determine the placement of monitoring wells.

A site geologist will be present during all sampling and installation procedures, and will fully document all procedures and soil characteristics in the Field Logbook (refer to SOP No. 016).

The site geologist will have on hand, at a minimum, a copy of the approved Health and Safety Plan, this SOP, the Field Investigation Work Plan, a hand lens (10X), a standard color chart, and a grain size chart.

Only solid vegetable shortening (e.g., Crisco®) without flavoring or additives may be used on downhole Geoprobe® and Hydropunch® equipment.

Surface runoff or other fluids will not be allowed to enter any DPT location or well during or after DPT activities.

The subcontractor will use the equipment specific guidelines for installation of the Geoprobe® DPT equipment. Probe rods will be forced into the ground by hydraulic means.

- Drive the sampler to the desired groundwater sampling interval. At the desired depth, insert extension rods down the inside diameter of the probe rods until the extension reaches the bottom of the screen. Remove the probe rods and sampler sheath while holding the screen in place.

- Collect the groundwater sample in the screen interval with a mini-bailer, peristaltic or vacuum pump, or other acceptable small diameter sampling device.

- The head of the rod may be equipped with a sensing device for characterization of soil properties or the contaminant content.

The subcontractor will use the equipment-specific guidelines for installation of the Hydropunch® equipment. Rods will be forced into the ground by hydraulic means.

- The Hydropunch® tool is a double cylinder, designed to be sealed until the desired sampling depth is reached. Upon reaching the desired sampling depth, the outer cylinder is pulled back, exposing a perforated, stainless steel sampling barrel covered with filter material.

- The water sample enters the barrel and the sample is retrieved by pulling the probe rods from the hole with the hydraulic derrick and hammer assembly. Groundwater is the only media that is sampled by Hydropunch® equipment.

- The head of the rod may be equipped with a sensing device for characterization of the soil properties or the contaminant content.

- The sample volume collected with this technique is approximately 500-1,000 ml. Larger sample volumes can be collected by inserting tubing attached to a peristaltic pump into the rods to obtain water samples.

If desired, a small diameter monitoring well may be installed at this point. Refer to SOP No. 019 (Monitoring Well Installation).

If a well will not be installed, the rods will be removed as the borehole is simultaneously filled with a bentonite/grout mixture. A polyvinyl chloride (PVC) tube fed into the rod casing will allow the addition of grout.

3.3 SUBSURFACE SOIL – HYDRAULIC PUSHING AND SAMPLING

The objective of the selected DPT sampling technique is to allow grab samples to be taken at a selected site for characterization of the stratigraphy and for analysis of potential contaminants. The analytical results from sampling can also be used to determine the placement of monitoring wells.

A site geologist will be present during all DPT sampling and soil characterization. All procedures and soil characteristics will be fully documented in the Field Logbook (refer to SOP No. 016).

The site geologist will have on hand, at a minimum, a copy of the approved Health and Safety Plan, this SOP, the Field Investigation Plan, a hand lens (10X), a standard color chart, and a grain-size chart.

Only solid vegetable shortening (e.g., Crisco®) without flavoring or additives may be used on downhole Geoprobe® equipment.

Surface runoff or other fluids will not be allowed to enter any DPT location or well during or after DPT activities.

The subcontractor will use the equipment specific guidelines for installation of the Geoprobe® DPT equipment. Probe rods will be forced into the ground by hydraulic means. Additional rods will be added in 3- to 4-ft increments until the leading edge of the sampler reaches the top of the desired sampling interval.

Once the desired sampling depth has been reached, insert extension rods down the inside diameter of the probe rods until it reaches the top of the sampler assembly. Attach the extension rod handle to the top extension rod. Turn the handle clockwise until the stop-pin detaches from the drive head. Remove the extension rods and the stop-pin. Attach a drive cap to the probe and drive the sampler approximately 2 ft using hydraulic derrick.

The DPT sampler can be retrieved by pulling the probe rods from the hole with the hydraulic derrick and hammer assembly.

The liner will be capped with Teflon® tape and vinyl end caps. The liners can be split open to remove samples for composition analysis or for transfer to other containers for shipment to the laboratory for analysis.

The head of the rod may be equipped with a sensing device for characterization of the soil properties or the contaminant content.

3.4 DECONTAMINATION

All Geoprobe® and Hydropunch® DPT equipment must be thoroughly cleaned before and after each use to allow retrieval of representative groundwater samples. Geoprobe® soil sample liners are disposed of after each use. Scrub all metal parts with a stiff, long bristle brush and a non-phosphate soap solution. Steam cleaning may be substituted where available. Rinse with distilled water and allow to air-dry before assembly.

After decontamination, a new clean liner will be installed and all parts will be inspected for wear or damage.

Refer to SOP No. 005 (Field Decontamination).

3.5 ABANDONMENT

Pure bentonite or a bentonite/grout mixture (20:1) will be used to fill the resulting borehole if the water table is penetrated. Boreholes that do not penetrate the water table will be backfilled with cuttings from the hole and topped with a bentonite seal. Clean sand will be used to fill any remaining volume in the borehole.

Abandonment of Geoprobe® and Hydropunch® generated DPT boreholes will meet the standards established under SOP No. 028 (Well and Boring Abandonment).

4. CONE PENETROMETER TESTING

4.1 MATERIALS

A CPT rig typically consists of an enclosed 20- to 40-ton truck equipped with vertical hydraulic rams that are used to force a sensor probe into the ground. The weight of the CPT rig is dependent upon the thrust required at the site. The majority of CPT rigs are mounted in heavy-duty trucks that are ballasted to a total dead weight of approximately 15 tons. Screw anchors are utilized to develop the extra reaction to reach the maximum thrust of 20 tons. The rig is separated into two separate workspaces: data acquisition and hydraulic push areas.

Water sources for CPT activities and decontamination must be approved by the Project Manager prior to arrival of the CPT equipment. Information required for the water source includes: water source, manufacturer/owner, address and telephone number, type of treatment and filtration prior to tap, time of access, cost per gallon (if applicable), dates and results associated with all available chemical analysis over the past 2 years, and the name and address of the analytical laboratory (if applicable).

Pure sodium bentonite with no additives will be the only additive allowed, and its use must be approved by the Project Manager prior to the arrival of the DPT equipment. The information required for evaluation includes: brand name, manufacturer, manufacturer's address and telephone number, product number, product description, and intended use for the product.

Portland Type II cement will be used for grout (refer to SOP No. 019).

4.2 SUBSURFACE CHARACTERIZATION

The objective of this technology is to collect stratigraphic information using CPT equipment to determine subsurface stratigraphy and geotechnical properties at a particular site. CPT activities will be in accordance with American Society for Testing and Materials D 3441-86 and American Society for Testing and Materials D 5778-95. The stratigraphic information gathered can be used to facilitate the selection of DPT sampling screen intervals. At the same time, it is possible to install a 0.25-in. diameter pre-packed PVC monitoring well.

CPT rods are used to hydraulically push the CPT probe into the subsurface. Probes cannot be pushed into hard rock, and significant gravel or cobble content in the formation may impede or preclude penetration of the probe. The depth of penetration achievable depends on the type of formation, type of sampling probe, and size of the hydraulic equipment used.

The CPT probe includes the following components:

- A conical tip to measure vertical resistance beneath the tip.
- A friction sleeve to measure frictional resistance on the side of the probe, as a function of depth.

- Two internal strain gauge-type load cells, which independently measure the vertical resistance and side friction.

- A cone pressure gauge to measure the water pressure as the probe is pushed into the ground.

- Inclinometer to determine potential drifting of the probe (optional).

- Seismic transducers to perform downhole seismic surveys (optional). Therefore, stratigraphic data collected with the CPT include: tip resistance, local friction, friction ratio, pore pressure, and resistivity.

Data will be transferred from the probe to the data acquisition system or logger through an electrical cable. The hole will be advanced continuously at a rate of 0.6-1.0 in. per second. The data will be logged at every 0.4-0.8 in. of penetration. Monitor the probe's stratigraphic position will be monitored as it advances downward. Perform pore water pressure dissipation tests in representative hydrostratigraphic intervals. Record dissipated pore water pressures to represent hydraulic head values.

Once the confining unit underlying the surficial aquifer or the required depth has been reached, the CPT is pulled from the ground. Target interval samples can be collected during CPT hole advancement using direct push sampling techniques, i.e., Geoprobe® or Hydropunch® (Section 3).

4.3 DECONTAMINATION

All CPT equipment must be thoroughly cleaned before arrival at the work site, between test holes, and prior to being moved out of a work area. Scrub all metal parts with a stiff, long bristle brush and a non-phosphate soap solution. Steam cleaning may be substituted where available. Rinse with distilled water and allow to air-dry before assembly.

Refer to SOP No. 005 (Decontamination).

4 .4 ABANDONMENT

If the push hole was developed for the stratigraphic test only, once the testing is completed, grout the hole from bottom to top. If the hole has not collapsed after removing the CPT, PVC piping will be used to grout the hole. If the hole has collapsed after removing the CPT, then hollow CPT rods and a sacrificial tip will be used to grout the hole. The PVC pipe or CPT rods will be pushed to the bottom of the hole. Grout will then be pumped to the bottom of the hole as the PVC pipe or CPT rods are withdrawn.

Refer to SOP No. 028 (Well and Boring Abandonment).

5. SITE CHARACTERIZATION AND ANALYSIS PENETROMETER SYSTEM

5.1 MATERIALS

SCAPS cone penetrometer and laser induced fluorescence (LIF) technology requires the use of a specialized 20-ton truck. The truck has two separate enclosed compartments. Each compartment is temperature controlled and monitored for air quality. The two rooms are the data acquisition and processing room, and the hydraulic ram/rod handling room. Approximately 20 ft of overhead clearance is required to fully extend the hydraulic ram and allow for leveling jack movement.

All materials required to complete SCAPS analysis are provided by the subcontractor to include cone penetrometer equipment. All hydraulic equipment, SCAPS rods, nitrogen lasers, etc. are included within the vehicle. A decontamination water source and a source of water for mixing the grout are required.

Water sources for equipment decontamination must be approved by the Project Manager prior to arrival of the SCAPS equipment. Information required for the water source includes: water source, manufacturer/owner, address and telephone number, type of treatment and filtration prior to tap, time of access, cost per gallon (if applicable), dates and results associated with all available chemical analysis over the past 2 years, and the name and address of the analytical laboratory (if applicable).

Pure sodium bentonite with no additives will be the only additive allowed, and its use must be approved by the Project Manager prior to the arrival of the SCAPS equipment. The information required for evaluation includes: brand name, manufacturer, manufacturer's address and telephone number, product number, product description, and intended use for the product.

Portland Type II cement will be used for grout (refer to SOP No. 019).

5.2 HYDRAULIC PUSHING AND SAMPLING

The objective of the SCAPS technique is to allow grab samples and stratigraphic information to be collected at a selected site to facilitate subsurface characterization and for analysis of potential contaminants. The analytical results obtained can also be used to determine the placement of monitoring wells. At the same time, it is possible to install a small diameter well for sampling purposes. Refer to SOP No. 019 (Monitoring Well Installation). If a well will not be installed, the borehole can be grouted as the equipment is removed.

A site geologist will be present during all installation and sampling procedures and will fully document all procedures and soil characteristics in the Field Logbook (refer to SOP No. 016).

The site geologist will have on hand, at a minimum, a copy of the approved Health and Safety Plan, this SOP, the Field Investigation Work Plan, a hand lens (10X), a standard color chart, and a grain-size chart.

Only solid vegetable shortening (e.g., Crisco®) without flavoring or additives may be used on downhole SCAPS equipment.

Surface runoff or other fluids will not be allowed to enter any DPT location or well during or after direct-push activities.

The subcontractor will use the equipment specific guidelines for installation of the SCAPS DPT equipment. Prior to SCAPS field activities, calibration soil samples will be collected and analyzed in order to determine the LIF sensor fluorescence threshold and detection limits for the site.

SCAPS LIF technology uses a pulsed nitrogen laser coupled with an optical detector to make fluorescence measurements via optical fibers. The LIF sensor is mounted on a cone penetrometer probe so that soil classification data and fluorescence data are collected simultaneously. The laser consumes nitrogen gas, which is supplied from cylinders stored on the accompanying trailer.

The SCAPS CPT sensors are used to gather stratigraphic information. See Section 4 for CPT operating procedures.

Target interval samples can be collected during SCAPS hole advancement using direct push sampling techniques such as Geoprobe® or Hydropunch® (Section 3).

5.3 DECONTAMINATION

Decontamination of SCAPS equipment is automated after initialization by a field team member. A pressurized hot water system is used to decontaminate the push rods as they are retracted from the ground. The SCAPS vehicle is equipped with a decontamination collar mounted to the bottom that cleans the rods. The decontamination water is removed by vacuum and transferred to a storage drum prior to disposal or treatment. A trailer attached to the back of the vehicle contains the water pump, heater for decontamination, and decontamination water containment drum.

Worker exposure is reduced by minimizing contact with contaminated media.

Refer to SOP No. 005 (Decontamination).

5.4 ABANDONMENT

SCAPS automatically grouts the penetrometer cavity as the rods are removed. The grout is pumped at high pressure through a 0.25-in. diameter tube in the center of the penetrometer rods. The tip is sacrificed at the bottom of the cavity to allow release of the grout.

A trailer attached to the back of the vehicle contains the 300-gal grout mixing bin and pump.

If the automatic grout feed does not work, the cavity will be manually filled with grout.

Abandonment of SCAPS generated borehole will meet the standards established under SOP No. 028 (Well and Boring Abandonment).

6. MAINTENANCE

Not applicable.

7. PRECAUTIONS

Refer to the site-specific Health and Safety Plan for discussion of hazards and preventive measures during intrusive activities.

8. REFERENCES

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Standard Operating Procedure No. 47A

Overburden Profiling

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CONTENTS

1. SCOPE AND APPLICATION

The purpose of this Standard Operating Procedure (SOP) is to establish the protocol for continuous logging of the overburden using a membrane interface probe (MIP), hydraulic profiling tool (HPT), and/or electrical conductivity (EC) profiling tool. Profiling will be completed in order to define vertical characteristics of the overburden and facilitate characterization of subsurface conditions.

2. MATERIALS

The following materials may be required:

Direct Push Drilling Equipment (SOP-47)	Soil sampling equipment (SOP-25 / SOP-47)
Membrane interface probe (MIP), Hydraulic Profiling Tool (HPT), and/or electrical conductivity (EC) profiling tool	Groundwater sampling equipment (SOP-13/SOP-48)
Photoionization Detector (PID) (SOP-11/ SOP-24)	Decontamination supplies (SOP-05)
Electron capture detector	Personnel protective equipment as required by the Accident Prevention Plan

The MIP will provide real-time field screening data. The tool will be equipped with a photoionization detector (PID) and an electron capture detector to provide real-time screening data for volatile organic compound (VOC) and relative chlorinated VOC concentrations. The MIP is equipped with a thin, permeable membrane impregnated with a stainless steel mesh that is heated to 100–120°C to facilitate discrete depth measurement of diffused gases. The gases are transported to the surface via a carrier gas (nitrogen or helium) and analyzed using an electron capture detector to generate a relative CVOC concentration profile.

The HPT sensors will provide real-time hydrostratigraphic and physiochemical data as the tooling is advanced, including relative hydraulic conductivity. HPT uses an injection and pressure transducer system to add clean water at a low flow rate (e.g., 300 mL/min). The matrix back-pressure response is monitored and provides an indication of soil permeability. The pressure response is inversely proportional to the soil permeability, with a higher back pressure indicating a low permeability and vice versa. A hydraulic head profile can also be determined for discrete intervals below the water table.

The EC profiling tool can be combined with either the HPT or MIP to provide electrical conductivity data that can be used to interpret water or clay content, because saturated soils and clay are more conductive.

3. PROCEDURE

Soil borings will be installed using direct-push drilling methods as per SOP-47, with the data acquisition tool (MIP, HPT, and/or EC) integrated with the direct-push equipment. Borings will be completed through overburden to the top of bedrock.

Boring locations will be staked in the field and utility clearance will be performed as per SOP 3. The proposed locations will be adjusted in the field based on utility clearance. Based upon the presence and absence of visual contamination or subsurface debris, or field observations, the proposed locations may be shifted or stepped out to delineate the extent of site-related impacts.

3.1 UNSATURATED ZONE SOIL SAMPLING

For MIP locations, an adjacent soil boring will be installed using direct-push drilling methods as per SOP-47 to allow comparison of the MIP readings to soil observations (e.g., soil classification, evidence of potential contamination including staining, sheens, elevated PID readings, and/or obvious odor). Soil samples will be collected for analysis of volatile organic compounds as based on MIP readings and soil observations per SOP-25 and SOP-47. Soil sample containers will be labeled, tracked via chain of custody forms, and packed and shipped to an offsite laboratory for analysis, as per SOPs 1, 2, and 4, respectively.

3.2 SATURATED ZONE GROUNDWATER SAMPLING

For HPT profiling locations, a minimum of one discrete *in situ* groundwater sample will be collected at the bedrock interface via the HPT integrated sampling screen/port. Additional samples will be collected as based on changes in hydrogeologic characteristics. To collect groundwater samples, hydraulic pressure will be used to fill a sample container and a nitrogen gas-drive pump will force the groundwater samples to the surface where physiochemical parameters (pH, ORP, DO, and specific conductance [SC]) will be recorded with a multi-parameter water quality meter, as per SOP-43. Purging will be conducted until all physiochemical parameters had stabilized (i.e., within ±10 percent of previous readings), ensuring that a representative groundwater sample will be collected for laboratory analysis.

Upon reaching stabilization, groundwater samples will be collected for analysis of volatile organic compounds. Groundwater samples will be collected by directly filling sample containers provided by the analytical laboratory. Sample containers will be labeled, tracked via chain of custody forms, and packed and shipped to an offsite laboratory for analysis, as per SOPs 1, 2, and 4, respectively.

4. ABANDONMENT

Following completion of the profiling point and sampling, the borehole advanced during profiling will either be abandoned per SOP-28, or selected as a location for bedrock coring per SOP-19A.

5. INVESTIGATIVE DERIVED WASTE

Investigative derived waste generated during soil boring installation will be disposed of per SOP-42.

6. DECONTAMINATION

Drilling equipment and non-dedicated sampling equipment will be decontaminated prior to use, between profiling locations, and following completion of profiling as per SOP 5.

7. PRECAUTIONS

Prior to collecting any samples, consult the Accident Prevention Plan for personal protective equipment required for sampling activities.

Decontaminate the sampling equipment and change gloves between samples to minimize the risk of cross-contamination.

Standard Operating Procedure No. 048
for
Low-Flow Sampling

Prepared by

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CONTENTS

1. GROUNDWATER SAMPLING BY LOW-FLOW PURGE AND SAMPLING METHOD USING DEDICATED PUMPS

1.1 SCOPE OF APPLICATION

The purpose of this Standard Operating Procedure (SOP) is to establish the protocol for collecting groundwater samples using dedicated pump systems. The procedure is designed to permit the collection of groundwater samples with minimum turbidity.

1.2 EQUIPMENT/MATERIALS

- Work Plan.
- Well construction data, location map, and field data from last sampling event.
- Field logbook and Field Record of Well Gauging, Purging, and Sampling forms (Figure SOP048-1).
- Electric water level measuring device, 0.01 ft accuracy for monitoring water level during pumping operations.
- Pumps: adjustable rate, submersible pumps constructed of stainless steel and Teflon®.
- Tubing: Teflon or Teflon-lined polyethylene must be used to collect samples for organic analysis. For samples collected for inorganics analysis, Teflon or Teflon-lined polyethylene tubing will be used.
- Flow measurement supplies (e.g., graduated cylinder and stop watch).
- Power source (generator, etc.).
- Water quality indicator parameter monitoring instruments—pH, turbidity, specific conductance, and temperature. Optional indicators—Eh and dissolved oxygen.
- Flow-through cell (preferred) or clean container for water quality probes.
- Decontamination supplies (for monitoring instrumentation).
- Sample bottles and sample preservation supplies (as required by the analytical methods).
- Sample tags or labels.
- Cooler with bagged ice for sample bottles.
- Drum for purge water containment.

1.3 PRELIMINARY SITE ACTIVITIES

The following site activities are required prior to performing well purging and groundwater sampling. Field logbooks and sampling forms should be filled out as the procedure is being performed, as noted:

- Enter the following information in the field logbook and sampling form, as appropriate: site name, project number, field personnel, well identification, weather conditions, date and time, equipment used, and quality assurance/quality control data for field instrumentation.

- Check well for damage or evidence of tampering, record pertinent observations in field logbook and sampling form.

- Lay out sheet of polyethylene for monitoring and sampling equipment.

- Unlock well and remove well cap (if applicable).

- Measure VOCs with an ionization detector (flame or photo) instrument at the rim of the well and in the breathing zone, and record the readings in the field logbook and the sampling form.

- Measure and record the height of protective casing above the concrete pad or ground surface, as appropriate. This reading is compared to that recorded during well installation as an indication of possible well damage or settling that may have occurred.

- Dedicated sampling pumps should be positioned with the pump intake mid-point in the screened interval. If non-dedicated equipment is used, care will be taken to position pump or sampling hose intake at the screen mid-point.

- Measure and record the depth to water (to 0.01 ft) in the well to be sampled before purging begins. If the well casing does not have a reference point (usually a v-cut or indelible mark in the well casing), make one. If a reference point is made, it will be noted in the field logbook. Care should be taken to minimize disturbance of any particulate attached to the sides or at the bottom of the well. The depth to well bottom will be measured following the completion of sampling because of the potential to stir up sediment at the bottom of the well.

- Prepare the pump by checking electrical connections, discharge tubing, and motor (Grundfos Redi-Flo2). Locate the generator (if applicable) downwind of the well; connect the power converter to the generator and to the pump.

1.4 WELL PURGING AND SAMPLING PROCEDURE

The following general procedure should be followed to obtain representative groundwater samples. Field logbooks and sampling forms should be filled out as the procedure is being performed, as noted:

- Enter the following information in the field logbook and sampling form, as appropriate, prior to purging: purge date and time, purge method, and total well depth.

- Connect the flow-through cell or clean container containing the instrumentation header to the pump discharge and begin purging the well at 0.2-0.5 L/min, unless a different purge rate has been previously established for that well. Fill the flow cell completely. Care should be taken not to cause entrapment of air in the system. Record the purge start time and purge rate.

- Establish that the water level has not dropped significantly such that the pump is dry (bubbles in discharge) or water is heard cascading down the inside of the well. Ideally, the pump rate should cause little or no water level drawdown in the well (>0.5 ft and the water level should stabilize). The water level should be monitored every 3-5 minutes (or as appropriate) during pumping. Record pumping rate adjustments and depths to water. Pumping rates should, if needed, be reduced to the minimum capabilities of the pump (e.g., 0.1-0.2 L/min) to avoid pumping the well dry and/or to ensure stabilization of indicator parameters. If water levels continue to drop with the pump on the lowest flow rate, the pump will be shut off and the well will be allowed to recharge to prevent the well from going dry. **The well will not be purged to dryness prior to sampling to prevent erroneous field parameters and groundwater samples.** Sampling will commence as soon as the well has recharged to a sufficient level to collect the appropriate volume of samples with the pump.

- During purging of the well, monitor the water quality indicator parameters (turbidity, temperature, specific conductance, pH, etc.) every 3-5 minutes (or as appropriate). Record purge rate, volume purged, depth to water, water quality indicator parameters values, and clock time at 3- to 5-minute intervals in field logbook and sampling record. Purging of the standing well water is considered complete when three consecutive readings of the water quality indicator parameters agree within approximately 10 percent. Turbidity readings consistently below 10 nephelometric turbidity units (NTU) are considered to represent stabilization of discharge water for this parameter. If the parameters have stabilized, but the turbidity is not in the range of the 10 NTU goal, the pump flow rate should be decreased and measurement of the parameters should continue every 3-5 minutes.

- Purge water at a well will be containerized if a well has exceeded the MEG or MCL in previous sampling events. Any purge water that is collected will be treated at the groundwater treatment plant.

- Prior to sampling, disconnect the discharge tubing from the flow-through cell. If the water discharged by the pump is silty, wait for the water to clear before sampling. Ensure that bubbles are not observed in the discharge tubing. Record pertinent observations in field logbook and sampling records.

- Begin filling sample containers by allowing the pump discharge to flow gently down the inside of the container with as little agitation or aeration as possible. Collect the samples in the order below, as applicable:

  — VOCs
  — Inorganics.

- VOC samples requiring pH adjustment will have their pH checked to assure that the proper pH has been obtained. This will require that a test sample be collected to determine the amount of preservative that needs to be added to the sample containers prior to sampling. Details on sample preservation are discussed in Section 1.5.

- Label each sample as collected. Those samples (VOCs, etc.) requiring cooling will be placed into an ice cooler for delivery to the laboratory. Inorganic samples, after preservation, do not need to be cooled.

- After collection of the samples, restore the dedicated pumping assembly to the well by hanging the tube, electric line, and support cable inside the well by the specially-designed PVC well cap assembly. Lock well.

- Complete remaining portions of Field Record of Well Gauging, Purging, and Sampling form (Figure SOP048-1) after each well is sampled, including sample date and time, total quantity of water removed, well sampling sequence, types of sample bottles used, sample identification numbers, preservatives used, parameters requested for analysis, and field observations of sampling event.

1.5 SAMPLE PRESERVATION

The following preservation procedures are examples of typical preservation protocols specific to the indicated analyses. Pre-preserved bottles will be used if possible. Minimum sample preservation requirements for each parameter group are summarized below:

- **VOCs—**Aqueous VOC samples must be collected as specified below. Each VOC sample is taken in duplicate:

  — Uncap the sample bottle, taking care not to touch the Teflon-faced septum. If the septum is contaminated in any way, it should be replaced.

  — Fill a sample bottle, preserve with HC1, and check the pH. Adjust the volume of HC1 to assure pH<2.

— Add the amount of HC1 determined in the above step, and fill the sample vial slowly from the tubing, minimizing air entrainment, until the vial slightly overflows.

— Place the Teflon-faced silicon rubber septum on the convex meniscus, Teflon side (shiny side) down and screw cap on.

— Invert the bottle, tap lightly, and check for air bubbles.

— If air bubbles are present, open the bottle, add sample to eliminate air bubbles, and reseal. Repeat this procedure until the bottle is filled and no air bubbles are detected.

— Place samples on ice until shipment.

- **Inorganics—**Fill the sample bottle, preserve the sample to pH<2 with nitric acid (HNO_3), seal container, and place sample on ice for shipment.

Disposable pipettes should be used to introduce chemicals into the samples if necessary. Chemicals used for preserving should be poured into a 150-ml beaker. They should not be drawn directly from the preservative bottles because the bottle may become contaminated. Measurements for pH and temperature should not be taken from the sample containers. When preserving samples to a required pH, pH paper should be used to check the resultant pH. The sample should be poured across the pH paper. Never place pH paper directly into sample.

NOTE: Shipping regulations limit the amount of preservative which can be added. For a 1-L sample, this is generally 1.5 ml of acid preservative.

1.6 FIELD QUALITY CONTROL

Quality control samples are required to verify that the sample collection and handling process has not affected the quality of the groundwater samples. All field quality control samples must be prepared exactly as regular investigation samples with regard to sample volume, containers, and preservation. The following quality control samples will be collected for each sample delivery group (SDG) (an SDG may not exceed 20 samples) at the frequency noted:

- Field Duplicate—Required at a frequency of 10 percent per SDG.

- Matrix Spike/Matrix Spike Duplicate—Required at a frequency of 5 percent.

- Equipment Rinsate Blank—Required once prior to installation of dedicated pump systems.

- Source Water Blank—Required at a frequency of once per source per sampling event when equipment (rinsate) blank is required.

- Trip Blank—Required for VOC samples at a frequency of one per sample shipment.

1.7 DECONTAMINATION

Non-dedicated sampling equipment and field monitoring equipment will be decontaminated prior to use and following sampling of each well. This equipment will be decontaminated by the procedure listed below. Alternative procedures must be approved by the Project Manager prior to sampling event. Decontamination fluids will be collected in a 5-gal bucket and treated at the groundwater treatment plant.

The following decontamination procedure will be used:

- Flush the equipment with potable water
- Flush with non-phosphate detergent solution
- Flush with tap water to remove all of the detergent solution
- Flush with distilled/deionized water
- Flush with isopropyl alcohol
- Flush with distilled/deionized water.

It is recommended that the detergent and isopropyl alcohol used in the above sequence be used sparingly.

2. GROUNDWATER SAMPLING BY LOW-FLOW PURGE AND SAMPLING METHOD USING PERISTALTIC PUMPS

2.1 SCOPE OF APPLICATION

The purpose of this SOP is to establish the protocol for collecting groundwater samples using peristaltic pump systems. The procedure is designed to permit the collection of groundwater samples with minimum turbidity, and is intended to be used in conjunction with the analyses for the most common types of groundwater contaminants (VOCs and inorganic compounds).

2.2 EQUIPMENT/MATERIALS

- Work Plan.
- Well construction data, location map, field data from last sampling event.
- Field logbook and Field Record of Well Gauging, Purging, and Sampling forms (Figure SOP048-1).
- Water level measuring device, 0.01 ft accuracy (electronic preferred) for monitoring water level drawdown during pumping operations.
- Peristaltic pump.

- In-well tubing: Teflon or Teflon-lined polyethylene must be used to collect samples for organic analysis. For samples collected for inorganics analysis, Teflon or Teflon-lined polyethylene, PVC, Tygon, or polyethylene tubing may be used.

- Pump head tubing: Silicon tubing must be used to in the pump head assembly.

- Flow measurement supplies (e.g., graduated cylinder and stop watch).

- Power source (battery, etc.).

- Water quality indicator parameter monitoring instruments – pH, turbidity, specific conductance, and temperature. Optional indicators – Eh and dissolved oxygen.

- Flow-through cell (preferred) or clean container for water quality probe.

- Decontamination supplies (for monitoring instrumentation).

- Sample bottles and sample preservation supplies (as required by the analytical methods).

- Sample tags or labels.

- Cooler with bagged ice for sample bottles.

- Drum for purge water containment.

2.3 PRELIMINARY SITE ACTIVITIES

The following site activities are required prior to performing well purging and groundwater sampling. Field logbooks and sampling forms should be filled out as the procedure is being performed, as noted:

- Enter the following information in the field logbook and sampling form, as appropriate: site name, project number, field personnel, well identification, weather conditions, date and time, equipment used, and quality assurance/quality control data for field instrumentation.

- Check well for damage or evidence of tampering, record pertinent observations in field logbook and sampling form.

- Unlock well and remove well cap (if applicable).

- Measure VOCs with an ionization detector (photo or flame) instrument at the rim of the well and in the breathing zone and record the readings in the field logbook and the sampling form.

- Measure and record the height of protective casing above the concrete pad, or ground surface, as appropriate. This reading is compared to that recorded during well installation as an indication of possible well damage or settling that may have occurred.

- Measure and record the depth to water (to 0.01 ft) in the well to be sampled before purging begins. If the well casing does not have a reference point (usually a v-cut or indelible mark in the well casing), make one. If a reference point is made, it will be noted in the field logbook. Care should be taken to minimize disturbance of any particulate attached to the sides or at the bottom of the well. The depth to well bottom will not be measured following the completion of sampling because of the potential to stir up sediment at the bottom of the well.

- Position the intake of the sampling hose at the mid-point of the screened interval.

- Prepare the pump by checking electrical connections and discharge tubing. Locate the battery downwind of the well; connect the peristaltic pump to the battery.

2.4 WELL PURGING AND SAMPLING PROCEDURES

The following general procedure should be followed to obtain representative groundwater samples. Field logbooks and sampling forms should be filled out as the procedure is being performed, as noted:

- Enter the following information in the field logbook and sampling form, as appropriate, prior to purging: purge date and time, purge method, and total well depth.

- Measure the water level with the pump in well before starting the pump. Begin purging the well at 0.3-0.5 L/min, unless a different purge rate has been previously established for that well.

- If well diameter permits, establish that the water level has not dropped significantly such that the pump is dry (air in discharge) or tubing suction is broken. Ideally, the pump rate should cause little or no water level drawdown in the well (>0.5 ft and the water level should stabilize). The water level should be monitored every 3-5 minutes (or as appropriate) during pumping. Care should be taken not to cause pump suction to be broken, or entrainment of air in the pump system. Record pumping rate adjustments and depths to water. Pumping rates should, if needed, be reduced to the minimum capabilities of the pump (e.g., 0.3 L/min) to avoid pumping the well dry and/or to ensure stabilization of indicator parameters. If water levels continue to drop with the pump on the lowest flow rate, the pump will be shut off and the well will be allowed to recharge to prevent the well from going dry. **The well will not be purged to dryness prior to sampling to prevent erroneous field parameters and groundwater samples.** Sampling will commence as soon as the well has recharged to a sufficient level to collected the appropriate volume of samples with the pump.

- During purging of the well, monitor the field indicator parameters (turbidity, temperature, specific conductance, pH, etc.) every 3-5 minutes (or as appropriate). Purging of the standing well water is considered complete when three consecutive readings of the water quality indicator parameters agree within approximately 10 percent. Turbidity readings consistently below 10 NTU are considered to represent stabilization of discharge water for this parameter. If the parameters have stabilized, but the turbidity is not in the range of the 10 NTU goal, the pump flow rate should be decreased and measurement of the parameters should continue every 3-5 minutes.

- Purge water at a well will be containerized if a well has exceeded the MEG or MCL in previous sampling events. Any purge water that is collected will be treated at the groundwater treatment plant.

- Prior to sampling, disconnect the sample discharge tubing from the flow-through cell. If the water discharged by the pump is silty, wait for the water to clear before sampling. Ensure that bubbles are not observed in the discharge tubing.

- Collect groundwater samples directly from the silicon tubing into preserved (when appropriate) sample containers. Begin filling sample containers from the pump discharge, allowing the water to fill the containers by allowing the pump discharge to flow gently down the inside of the container with as little agitation or aeration as possible. Collect the samples in the order below, as applicable:

  — VOCs
  — Inorganics.

- VOC samples requiring pH adjustment will have their pH checked to assure that the proper pH has been obtained. This will require that a test sample be collected to determine the amount of preservative that needs to be added to the sample containers prior to sampling. Detail on sample preservation are discussed in Section 2.5.

- Label each sample as collected. Those samples (VOCs, etc.) requiring cooling will be placed into an ice cooler for delivery to the laboratory. Inorganic samples, after preservation, do not need to be cooled.

- After collection of the samples, restore the dedicated tubing assembly to the well by hanging the tube inside the well by the specially-designed PVC well cap assembly. Lock well.

- Complete remaining portions of Field Record of Well Gauging, Purging, and Sampling form (Figure SOP048-1) after each well is sampled, including: sample date and time, total quantity of water removed, well sampling sequence, types of sample bottles used, sample identification numbers, preservatives used, parameters requested for analysis, and field observations of sampling event.

- The silicon tubing used in the peristaltic pump will be changed after use at each well.

2.5 SAMPLE PRESERVATION

The following preservation procedures are examples of typical preservation protocols specific to the indicated analyses. Pre-preserved bottles will be used if possible. Minimum sample preservation requirements for each parameter group are summarized below:

- **VOCs—**Aqueous VOC samples must be collected as specified below. Each VOC sample is taken in duplicate:

  — Uncap the sample bottle, taking care not to touch the Teflon-faced septum. If the septum is contaminated in any way, it should be replaced.

  — Fill a sample bottle, preserve with HCL, and check the pH. Adjust the volume of HCL to assure pH<2.

  — Add the amount of HCL determined in the above step, and fill the sample vial slowly from the tubing, minimizing air entrainment, until the vial slightly overflows.

  — Place the Teflon-faced silicon rubber septum on the convex meniscus, Teflon side (shiny side) down, and screw cap on.

  — Invert the bottle, tap lightly, and check for air bubbles.

  — If air bubbles are present, open the bottle, add sample to eliminate air bubbles, and reseal. Repeat this procedure until the bottle is filled and no air bubbles are detected.

  — Place samples on ice until shipment.

- **Inorganics—**Fill the sample bottle, preserve the sample to pH<2 with nitric acid (HNO_3), seal container, and place sample on ice for shipment.

Disposable pipettes should be used to introduce chemicals into the samples if necessary. Chemicals used for preserving should be poured into a 150-ml beaker. They should not be drawn directly from the preservative bottles because the bottle may become contaminated. Measurements for pH and temperature should not be taken from the sample containers. When preserving samples to a required pH, pH paper should be used to check the resultant pH. The sample should be poured across the pH paper. Never place pH paper directly into sample.

NOTE: Shipping regulations limit the amount of preservative which can be added. For a 1-L sample, this is generally 1.5 ml of acid preservative.

2.6 FIELD QUALITY CONTROL

Quality control samples are required to verify that the sample collection and handling process has not affected the quality of the groundwater samples. All field quality control samples must be prepared exactly as regular investigation samples with regard to sample volume, containers, and preservation. The following quality control samples will be collected for each SDG (an SDG may not exceed 20 samples) at the frequency noted:

- Field Duplicate—Required at a frequency of 10 percent per SDG
- Matrix Spike/Matrix Spike Duplicate—Required at a frequency of 5 percent
- Equipment (Rinsate) Blank—Required once prior to installation of dedicated sample tubing
- Source Water Blank—Required at a frequency of one per source per sampling event
- Trip Blank—Required for VOC samples at a frequency of one per sample shipment.
- Temperature Blank—Required at a frequency of once per sample shipment container.

2.7 DECONTAMINATION

Non-dedicated sampling and field monitoring equipment will be decontaminated prior to use and following sampling of each well. This equipment will be decontaminated by the procedure listed below. Alternate procedures must be approved by the Project Manager prior to the sampling event. Decontamination fluids will be collected in a 5-gal bucket and treated at the groundwater treatment plant.

The following decontamination procedure will be used:

- Flush the equipment with potable water
- Flush with non-phosphate detergent solution
- Flush with tap water to remove all of the detergent solution
- Flush with distilled/deionized water
- Flush with isopropyl alcohol
- Flush with distilled/deionized water.

It is recommended that the detergent and isopropyl alcohol used in the above sequence be used sparingly.

3. SURFACE WATER AND LEACHATE SEEP SAMPLING PROCEDURE

3.1 SCOPE OF APPLICATION

The purpose of this SOP is to establish the protocol for collecting surface water and leachate seep samples. The procedure is designed to permit the collection of representative surface water and leachate seep samples, and has been adapted from the procedure outlined in the Work Plan. This SOP is suitable for collecting surface water and seep samples requiring analyses for the most common types of surface water contaminants (VOCs and inorganic compounds).

3.2 EQUIPMENT/MATERIALS

- Work Plan.
- Location map, field data from last sampling event.
- Field logbook and Field Record of Surface Water and Sediment Sampling forms (Figure SOP048-2).
- Water quality indicator parameter monitoring instruments – pH, turbidity, specific conductance, and temperature. Optional indicators – Eh and dissolved oxygen.
- Decontamination supplies (for monitoring instrumentation).
- Dedicated, pre-cleaned 1-L wide-mouth or volatile organic analyte sample container (for sample collection).
- Sample bottles and sample preservation supplies (as required by the analytical methods).
- Sample tags or labels.
- Cooler with bagged ice for sample bottles.

3.3 PRELIMINARY SITE ACTIVITIES

The following site activities are required prior to performing surface water or leachate seep sampling. Field logbooks and sampling forms should be filled out as the procedure is being performed, as noted:

- Enter the following information in the field logbook and sampling form, as appropriate: site name, project number, field personnel, sample station identification, weather conditions, date and time, equipment used, and quality assurance/quality control data for field instrumentation.

- Visually inspect sample station for evidence of changes in physical condition; record pertinent observations in field logbook and sampling form.

- Measure VOCs with a flame ionization detector instrument in the breathing zone and record the reading in the field logbook and sampling form.

3.4 SAMPLING PROCEDURE

The technique for surface water and leachate seep sampling must be selected after addressing such items as:

- Depth of waterbody
- Depth of sample
- Stratification
- Seasonal variations
- Analytical parameters of interest.

The following general procedure should be used to obtain representative surface water and leachate seep samples. Field logbooks and sampling forms should be filled out as the procedure is being performed, as noted:

- Enter the following information in the field logbook and sampling form, as appropriate, prior to sampling: date and time, sample method, and sample depth.

- Collect the sample from the surface water, within several tenths of a foot of the streambed, by immersing a new, dedicated 1-L glass or volatile organic analyte sample container into the waterbody. If a stream is being sampled, collect the sample upstream of the sampler with the opening of the sampling device oriented upstream but avoiding floating debris.

- Directly fill the appropriate sample containers from the 1-L or volatile organic analyte sampling device.

- Collect the samples in the order below, as applicable:

  — VOCs
  — Inorganics.

- Water sample containers are generally filled directly from the source or sampler without special considerations. The exception is the collection of aqueous VOC samples requiring pH adjustment. VOC samples will have their pH checked to assure that the proper pH has been obtained. This will require that a test sample be collected to determine the amount of preservative that needs to be added to the sample containers prior to sampling. Details on sample preservation methods are discussed in Section 3.6.

- Label each sample as collected. Those samples (VOCs, etc.) requiring cooling will be placed into an ice cooler for delivery to the laboratory. Inorganic samples, after preservation, do not need to be cooled.

- Measure water quality indicator parameters, if possible, by direct immersion of instrument probes into the waterbody immediately following sample collection. If direct measurement is not possible, measure these parameters from water remaining in the sampling device or another sample bottle. Record this information in the field logbook and sample data record.

- Complete remaining portions of the Field Record of Surface Water and Sediment Sampling form (Figure SOP048-2) after each station is sampled, including: time of sample collection, types of sample bottles used, sample identification numbers, preservatives used, parameters requested for analysis, and field observations of sampling event.

3.5 SAMPLE PRESERVATION

The following preservation procedures are examples of typical preservation protocols specific to the indicated analyses. Minimum sample preservation requirements for each parameter group are summarized below:

- **VOCs—**Aqueous VOC samples must be collected as specified below. Each sample is taken in duplicate:

  — Uncap the sample bottle, taking care not to touch the Teflon-faced septum. If the septum is contaminated in any way, it should be replaced.

  — Fill a sample bottle, preserve with HC1, and check the pH. Adjust the volume of HC1 to assure pH<2.

  — Add the amount of HC1 determined in the above step, and fill the sample vial slowly from the 1-L container, minimizing air entrainment, until the vial slightly overflows.

  — Place the Teflon-faced silicon rubber septum on the convex meniscus, Teflon side (shiny side) down and screw cap on.

  — Invert the bottle, tap lightly, and check for air bubbles.

  — If air bubbles are present, open the bottle, add sample to eliminate air bubbles, and reseal. Repeat this procedure until the bottle is filled and no air bubbles are detected.

  — Place samples on ice until shipment.

- **Inorganics—**Fill the sample bottle, preserve the sample to pH<2 with nitric acid (HNO_3), seal container, and place sample on ice for shipment.

Disposable pipettes should be used to introduce chemicals into the samples. Chemicals used for preserving should be poured into a 150-ml beaker. They should not be drawn directly from the preservative bottles because the bottle may become contaminated. Measurements for pH and temperature should not be taken from the sample containers. When preserving samples to a required pH, pH paper should be used to check the resultant pH. The sample should be poured across the pH paper. Never place pH paper directly into sample.

NOTE: Shipping regulations limit the amount of preservative which can be added. For a 1-L sample, this is generally 1.5 ml of acid preservative.

3.6 FIELD QUALITY CONTROL

Quality control samples are required to verify that the sample collection and handling process has not affected the quality of the surface water and leachate seep samples. All field quality control samples must be prepared exactly as regular investigation samples with regard to sample volume, containers, and preservation. The following quality control samples will be collected for each SDG (an SDG may not exceed 20 samples) at the frequency noted:

- Field Duplicate—Required at a frequency of 10 percent per SDG.
- Matrix Spike/Matrix Spike Duplicate—Required at a frequency of 5 percent.
- Equipment (Rinsate) Blank—Required at a frequency of once per day per media sampled.
- Source Water Blank—Required at a frequency of once per source per sampling event when equipment (rinsate) blank is required.
- Trip Blank—Required for VOC samples at a frequency of one per sample shipment.

3.7 DECONTAMINATION

Field monitoring equipment will be decontaminated prior to use and following sampling of each station by the procedure listed below. Laboratory pre-cleaned, dedicated 1-L glass sample collection containers are used once and discarded and, therefore, do not undergo any decontamination. Decontamination fluids will be collected in a 5-gal bucket and treated at the groundwater treatment plant.

The following decontamination procedure will be used:

- Flush the equipment with potable water
- Flush with non-phosphate detergent solution
- Flush with tap water to remove all of the detergent solution
- Flush with distilled/deionized water
- Flush with isopropyl alcohol
- Flush with distilled/deionized water.

It is recommended that the detergent and isopropyl alcohol used in the above sequence be used sparingly.

4. REFERENCES

U.S. Environmental Protection Agency. 1996. Groundwater Issue-Low Flow Sampling (Minimal Drawdown) Groundwater Sampling Procedures. April.

FIELD RECORD OF WELL GAUGING, PURGING, AND SAMPLING

Site Name:		Project Number:	
Well ID:		Well Lock Status:	
Well Condition:		Weather:	

Gauge Date:		Gauge Time:	
Sounding Method:		Measurement Ref:	
Stick Up/Down (ft):		Well Diameter (in.):	

Purge Date:		Purge Time:	
Purge Method:		Field Personnel:	
Ambient Air VOCs (ppm):		Well Mouth VOCs (ppm):	

WELL VOLUME

A. Well Depth (ft):		D. Well Volume/ft (L):	
B. Depth to Water (ft):		E. Well Volume (L) (C*D):	
C. Liquid Depth (ft) (A-B)		F. Three Well Volumes (L) (E*3):	

G. Measurable LNAPL? Yes _____ /ft No _____

Parameter	Beginning	1	2	3	4	5
Time (min.)						
Depth to Water (ft)						
Purge Rate (L/min)						
Volume Purged (L)						
pH						
Temperature (°C)						
Conductivity (µmhos/cm)						
Dissolved Oxygen (mg/L)						
Turbidity (NTU)						
eH (mV)						

Total Quantity of Water Removed (L):			
Samplers:		Sampling Time (Start/End):	
Sampling Date:		Decontamination Fluids Used:	
Sample Type:		Sample Preservatives:	
Sample Bottle IDs:			
Sample Parameters:			

Figure SOP048-1.

FIELD RECORD OF WELL GAUGING, PURGING, AND SAMPLING

Site Name:	Project Number:	Date:
Well ID:	Field Personnel:	

Parameter	6	7	8	9	10	11
Time (min.)						
Depth to Water (ft)						
Purge Rate (L/min)						
Volume Purged (L)						
pH						
Temperature (°C)						
Conductivity (µmhos/cm)						
Dissolved Oxygen (mg/L)						
Turbidity (NTU)						
eH (mV)						

Parameter	12	13	14	15	16	17
Time (min.)						
Depth to Water (ft)						
Purge Rate (L/min)						
Volume Purged (L)						
pH						
Temperature (°C)						
Conductivity (µmhos/cm)						
Dissolved Oxygen (mg/L)						
Turbidity (NTU)						
eH (mV)						

Comments and Observations:

Figure SOP048-1.

FIELD RECORD OF SURFACE WATER AND SEDIMENT SAMPLING

Site Name:			Project Number:
Sample Location ID:			Date:
Time:	Start:	End:	Sample Team Members:

SURFACE WATER INFORMATION

Type of Surface Water:
( ) Stream ( ) River
( ) Pond/Lake ( ) Seep

Water Depth and Sample Location _____ (ft)

Depth of Sample from Top of Water _____ (ft)

Equipment Used for Collection:
( ) None, Grab into Bottle
( ) Bomb Sampler
( ) Pump _____

Decontamination Fluids Used:
( ) Isopropyl Alcohol
( ) ASTM Type II Water
( ) Deionized Water
( ) Liquinox Solution
( ) Hexane
( ) HNO_3 Solution
( ) Potable Water
( ) None

Water Quality Parameters
( ) Temperature ____
( ) Conductivity ___ µmhs/cm
( ) pH ____ units
( ) Dissolved oxygen ____ mg/L
( ) Turbidity ____ NTU
( ) Eh ____ mv

Velocity Measurements Obtained? ( ) No ( ) Yes, See Flow Measurement Data Record

Field QC Data Used: ( )
Duplicate ID _____
( ) MS/MSD

Field Duplicate Collected
( ) Yes
( ) No

Sample Location Sketch: Method
( ) Winkler
( ) Probe

SEDIMENT INFORMATION

Type of Sample Collected:
( ) Discrete
( ) Composite

Sediment Type:
( ) Clay
( ) Sand
( ) Organic
( ) Gravel

Equipment Used for Collection:
( ) Gravity Corer
( ) Stainless Steel Split Spoon
( ) Dredge
( ) Hand Spoon/Trowel
( ) Aluminum Pans
( ) Stainless Steel Bucket
( ) _____

Decontamination Fluids Used:
( ) Isopropyl Alcohol
( ) ASTM Type II Water
( ) Deionized Water
( ) Liquinox Solution
( ) Hexane
( ) HNO_3 Solution
( ) Potable Water
( ) None

Sample Observations:
( ) Odor _____
( ) Color

Field QC Data: ( ) Field Duplicate Collected ( ) MS/MSD
Duplicate ID _____

SAMPLES COLLECTED

Check if Required at this Location	Matrix: Surface Water	Matrix: Sediment	Check if Preserved with Acid/Base	Volume Required	Check if Sample Collected	Sample Bottle IDs			

NOTES/SKETCH

Figure SOP048-2.

Standard Operating Procedure No. 059
for
Field Logbook

Prepared by

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Revision: 1
November 2012

CONTENTS

1. SCOPE AND APPLICATION

The purpose of this standard operating procedure (SOP) is to delineate protocols for recording field survey and sampling information in the Field Logbook.

2. MATERIALS

The following materials may be required:

- Field Logbook (Teledyne 415 Level Book, or equivalent)[1]
- Indelible ink pen (e.g., Sharpie®).

3. PROCEDURE

All information pertinent to a field survey or sampling effort will be recorded in a bound logbook. Each page/form will be consecutively numbered, dated, and signed. All entries will be made in indelible ink, and all corrections will consist of line-out deletions that are initialed and dated. The person making the correction will provide a brief explanation for the change. Entries are factual only. No personal opinions should be entered.

There should be no blank lines on a page. A single blank line or a partial blank line (i.e., at the end of a paragraph) should be lined to the end of the page. If only part of a page is used, the remainder of the page should have an "X" drawn across it. The bottom of each page must be signed and dated by the field personnel entering the information.

At a minimum, entries in the Field Logbook will include but not be limited to the following:

- Date.
- Project number and project name.
- Name and address of field contact.
- Identification of sample crew members.
- Documentation should include model numbers of equipment used (e.g., drilling rigs) and calibration (if applicable). Each day's entry should begin with time onsite, who is onsite (including observers other than the sampling crew), brief description of what work will be performed that day and how, and the weather.

[1] Pre-printed, bound forms are approved as well. See SOP No. 016 for recommended content and format.

- If samples are being taken in or near tidal waters, the time of high and low tide for the site should be determined from local gauges or tables and recorded.

- References such as maps of the sampling site.

- Times of key daily milestones should be entered (e.g., time borings began, times personnel arrived and left site, times subcontractors arrived and left site, etc.). Time should be recorded in the left-hand margin on the page in military time.

- Sample-specific information:

  — Unique, sequential field sample number

  — Purpose of sampling

  — Location, description, and log of photographs of each sampling point

  — Details of the sample site (e.g., elevation of the casing, casing diameter and depth, integrity of the casing, etc.)

  — Documentation of procedures for preparation of reagents or supplies which become an integral part of the sample (e.g., filters and absorbing reagents)

  — Type of media of sample (e.g., groundwater, surface water, soil, sediment, and product)

  — Suspected waste composition

  — Number and volume of sample taken

  — Sampling methodology, including distinction between grab and composite sample

  — Sample preservation

  — Date and time of collection

  — Collector's sample identification number(s)

  — Sample shipment (e.g., name of the laboratory and cartage agent: Federal Express, United Parcel Service, etc.)

  — Field observations (e.g., oily sheen on groundwater sample, incidental odors, soil color, grain size, plasticity, moisture content, layering, Unified Soil Classification System classification, etc.)

— Any field measurements made (e.g., pH, conductivity, explosivity, water depth, organic vapor analyzer readings, etc.)

— Signature and date by the personnel responsible for observations

— Decontamination procedures.

Sampling situations vary widely. No general rules can specify the extent of information that must be entered in a Field Logbook. However, records should contain sufficient information so that someone can reconstruct the sampling activity without relying on the sampler's memory. Further, the project work plan or field sampling plan should be reviewed to identify additional specific information or requirements that should be included in the Field Logbook.

The Project Manager will keep a master list of all Field Logbooks assigned to the Sampling Team Leaders. One Field Logbook kept by the Project Manager will be a master site log of daily activities and will contain the list of Field Logbooks assigned to Sampling Team Leaders.

Project name and number should be clearly marked on the outside cover using indelible ink. If more than one Field Logbook exists for the project, then the number of the Field Logbook should also be clearly marked on the outside cover.

4. MAINTENANCE

At the end of the field sampling effort, the Field Logbook should be scanned and filed in the electronic file for the project and maintained according to the EA Records Retention Policy or contract requirements.

5. PRECAUTIONS

None.

6. REFERENCES

EA Engineering, Science, and Technology, Inc. 2007. Standard Operating Procedure No. 016 for Surface Water, Groundwater, and Soil/Sediment Field Logbooks. August.

U.S. Environmental Protection Agency. 1980. *Interim Guidelines and Specifications for Preparing Quality Assurance Project Plans*, QAMS-005/80.

———. 1990. *Sampler's Guide to the Contract Laboratory Program*. EPA/540/P-90/006, Directive 9240.0-06, Office of Emergency and Remedial Response, Washington, D.C. December.

———. 1991. *User's Guide to the Contract Laboratory Program*. EPA/540/O-91/002, Directive 9240.0-01D. Office of Emergency and Remedial Response. January.

Standard Operating Procedure No. 061
for
Geographic Information System
Data Management and Deliverables

Prepared by

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Revision: 0
April 2013

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Page

1. INTRODUCTION

1.1 PURPOSE

The purpose of this standard operating procedure is to provide EA personnel responsible for Geographic Information System (GIS) activities within the company the guidance necessary to maintain company-wide uniformity of GIS setup, data storage, file naming conventions, products, and deliverables.

1.2 SCOPE

This standard operating procedure covers EA's basic requirements for creating, managing, and deliverables for a GIS project or GIS subtask within a project.

1.3 DEFINITIONS

- FGDC = Federal Geographic Data Commission
- ESRI = Environmental Systems Research Institute
- MXD = ESRI GIS project file extension.

1.4 RESPONSIBILITY

EA personnel assigned GIS tasks will be responsible for adhering to the standards included in this standard operating procedure and for ensuring that all GIS deliverables meet any and all contract specifications. In accordance with EA Quality Control procedures and published Senior Technical Review guidance, GIS deliverables should be reviewed by an appropriate Senior Technical Reviewer.

1.5 RELATED STANDARD OPERATING PROCEDURES

None.

2. EQUIPMENT

2.1 SOFTWARE

ESRI software (ArcInfo, ArcDesktop, ArcMap, and ArcEditor) Version 9.3 or newer is EA's standard GIS software. This also includes the ESRI suite of extensions (spatial analyst, 3D analyst, Business analyst, Network analyst, etc.) and any client-specific software requirements for projects. It is EA's policy to wait to update to a new version of GIS software until the first service pack or major revision of that software is released. However, this policy is superseded when a client and/or project requires deliverables in a newer version of the software.

2.2 HARDWARE

Standard computer provided by EA.

3. PROCEDURE

There are multiple elements that must be considered when setting up an ESRI GIS project or GIS subtask within a project that will maintain uniformity and consistency for all projects company-wide. These elements look at how the project is stored, how it is set up, storage of data, what data are allowed in the GIS, and reliability of the data. Explanation of the above elements is provided in this section.

At the end of each section, there will be a "Required" and "Guidelines" entry. The "Required" entry will describe what shall be followed and the "Guidelines" entry will describe points to be considered but do not have to be followed.

3.1 GEOGRAPHIC INFORMATION SYSTEM DIRECTORY STRUCTURE

GIS project files reference the physical location of data (spatial and attribute) to correctly manage the information. Because of this, it is important that all spatial and attribute data be stored in a standardized manner. This will not only help ensure that GIS projects function years after they have been completed but also will help any individual who is new to the project.

The following examples illustrate the recommended directory tree and directory descriptions that should be used when setting up GIS projects. Not all directories and subdirectories included in the example need to be created if there will be no data of the type utilized by the project. Additionally, some clients may have their own requirements that may override the standard directory tree. This is the only scenario in which deviating from the standard is acceptable.

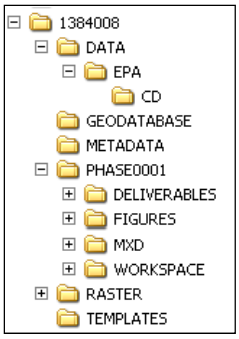
Required:

The example below represents EA's standard directory tree that shall be used to organize and create GIS projects.

GIS—This directory may be renamed according to how departments and offices store their projects.

GIS*PROJECT #*, e.g., GIS\1384008
GIS*PROJECT #*\DATA, e.g., GIS\1384008\DATA
GIS*PROJECT #*\DATA\CD, e.g., GIS\1384008\\DATA\CD
GIS*PROJECT #*\DATA\EMAIL, e.g., GIS\1384008\\DATA\EMAIL
GIS*PROJECT #*\DATA\WEB, e.g., GIS\1384008\\DATA\WEB
GIS*PROJECT #*\GEODATABASE, e.g., GIS\1384008\GEODATABASE
GIS*PROJECT #*\METADATA, e.g., GIS\1384008\METADATA
GIS*PROJECT #*\RASTER, e.g., GIS\1384008\RASTER
GIS*PROJECT #*\TEMPLATES, e.g., GIS\1384008\TEMPLATES
GIS*PROJECT #*\PHASE*NUMBER*, e.g., GIS\1384008\PHASE0001
GIS*PROJECT #*\PHASE*NUMBER*\DELIVERABLES, e.g., GIS\1384008\PHASE0001\DELIVERABLES

GIS*PROJECT #*\PHASE*NUMBER*\DELIVERABLES\DRAFT
– ex. GIS\1384008\PHASE0001\DELIVERABLES\DRAFT
GIS*PROJECT #*\PHASE*NUMBER*\DELIVERABLES\FINAL
– ex. GIS\1384008\PHASE0001\DELIVERABLES\FINAL
GIS*PROJECT #*\PHASE*NUMBER*\FIGURES
– ex. GIS\1384008\PHASE0001\FIGURES
GIS*PROJECT #*\PHASE*NUMBER*\FIGURES\PDF
– ex. GIS\1384008\PHASE0001\FIGURES\PDF
GIS*PROJECT #*\PHASE*NUMBER*\MXD
– ex. GIS\1384008\PHASE0001\MXD
GIS*PROJECT #*\PHASE*NUMBER*\WORKSPACE
– ex. GIS\1384008\PHASE0001\WORKSPACE.



A snapshot of what the directory tree would look like in Windows Explorer is displayed at right.

The following table describes the usage of the above directories.

Directory	Description	Note
GIS	GIS parent directory or directory where all projects are stored.	This directory may be renamed according to how departments and offices store their projects.
GIS**PROJECT#**	Subdirectory for a specific project.	This is the parent directory of the GIS project.
GIS\PROJECT#\ **GEODATABASE**	Geospatial database subdirectory. All features comprising project MXDs should be stored here.	This directory contains personal databases as well as file databases. This directory should only include final databases that are acceptable to be given to the client. This directory should not include any "working" files or data.
GIS\PROJECT#\ **RASTER**	Raster subdirectory. All raster types as well as geo-referenced imagery should be stored here.	This directory may contain subdirectories as needed. An example would be imagery broken out by year. NOTE: All imagery should be compressed.
GIS\PROJECT#\ **DATA**	DATA subdirectory. Incoming raw data, emails, etc. should be stored here.	This directory contains the original source files from clients or other sources. The directory should be organized in subdirectories based on source name, i.e., EPA. Subdirectories under source name should be organized by media to help in locating original files. An example would be EPA\CD\2010Wetland.zip.
GIS\PROJECT#\ **TEMPLATES**	Templates subdirectory. Stores project templates.	Templates stored here are used for figure creation.
GIS\PROJECT#\ **PHASE *number***	The PHASE *number* subdirectory. Organizes the project into its respective phases.	This is the parent directory for each phase.
GIS\PROJECT#\ PHASE *number***MXD**	The MXD subdirectory stores all MXDs used for DRAFT and FINAL products.	This directory stores the draft and final MXDs.
GIS\PROJECT#\ PHASE	Figures subdirectory. Organizes figures.	This is the parent directory for all figures.

Directory	Description	Note
*number***FIGURES**		
GIS\PROJECT#\ PHASE *number* \FIGURES**PDF**	PDF subdirectory. Contains PDF figures.	Contains PDF figures
GIS\PROJECT#\ PHASE *number*\ **DELIVERABLES**	Deliverables subdirectory. Is used for various stage deliverables to a client.	This directory contains the DRAFT and FINAL subdirectories for client deliverables.
GIS\PROJECT#\ PHASE *number*\ DELIVERABLES\ **DRAFT**	Draft subdirectory. Is used to compile draft deliverables.	This directory contains draft submittals to the client. Multiple drafts will have the submittal date incorporated in the zip file title.
GIS\PROJECT#\ PHASE *number*\ DELIVERABLES\ **FINAL**	Final subdirectory. Is used to compile final deliverables.	This directory contains final submittals to the client. Multiple finals will have the submittal date incorporated in the zip file title.
GIS\PROJECT#\ PHASE *number*\ **WORKSPACE**	The working files subdirectory. Working files, shape files, etc. to be deleted after incorporated into geospatial database should be stored here.	This directory is used for creating temporary files that are used to create final layers and data for the project draft and finals. Once files have been moved to MXDs and DATA storage areas they should be deleted from this directory.

Guidelines:

None.

3.2 GEOGRAPHIC INFORMATION SYSTEM PROJECT SETUP

There are three items required for every GIS project (MXD) creation before adding any data.

Required:

1. The MXD shall be set for storing the relative path to data sources rather than the full path. To store the relative path, go to the FILE pull down menu and click on "Document Properties." Click on "Data Source Option" and check the relative path box.

2. Data frames shall be set to the proper coordinate system dictated by the project. This ensures that data created or stored in the GIS will always have the correct coordinate system.

3. Third party extensions are not allowed if the final GIS deliverable includes the GIS MXD project file and requires the client to obtain/purchase the extension in order to recreate functionality and deliverables unless authorized by the client. An example would be the use of "Mapbooks," a third party extension, in the GIS project which automates the production of a tiled map series. Without Mapbooks, a client would not be able to reproduce a map series. This is only permitted when the client approves the extension and EA assists the client with obtaining the extension.

Guidelines:

- The use of multiple data frames and group layers is recommended for maintaining a logical easy-to-follow format in the MXD Table of Contents.

3.3 TEMPLATES

Templates are used to keep a consistent look across all maps and figures. EA has a set of standard templates that should be used on all projects company-wide (Appendix A). EA's standard templates should be used for all deliverables unless the client requires a specific layout. In cases where templates are customized for a specific client or projects, the template must be used for all deliverables to the project or client.

When creating templates for a project, the following is required.

Required:

- All templates should be stored in the TEMPLATES directory of the project.
- Each figure should include a North arrow, scale bar, file path, and a legend. The style and size of each should remain consistent throughout all figures.
- Source of data shall be included on all figures when the client requires.
- The scale will be set at an industry standard (i.e., 1 in. = 200 ft, 1 in. = 5 miles) and not an arbitrary scale (i.e., 1 in.:430 ft).
- Scale shall be consistent among figures in a report that depict geographical areas of the same size on paper of the same size, i.e., all figures in a report for the same geographical area presented on 8.5- × 11-in. paper should have the same scale.
- Label font size should be consistent within feature types regardless of the scale, i.e., all road names have the same font properties, and all stream names have the same font properties which may be different from the road layer. This means the font will be the same height per feature type on small and large scale figures when printed out.

Guidelines:

- The exterior neat line should allow for a minimum 0.5-in. margin for all sides if map is not intended to be bound in a report.
- A 0.75-in. margin is preferred on the binding edge.

3.4 GEOGRAPHIC INFORMATION SYSTEM DATA STORAGE

Regardless of the format that data are created or received in, it is required that a coordinate system be defined for all features whether they reside in a database, shape file, GRID, coverage, etc. Additionally, features, databases, and files shall be named in a fashion that is self describing to allow for easier identification of files.

All data, spatial or tabular, used in a GIS project for a final figure, map, or electronic deliverable are required to be stored in a geospatial database. The types of geospatial databases that can be used are Personal, File, or SDE.

Required:

- All data included in a final deliverable must be stored in one of the following geospatial database formats:

  — Personal (Microsoft Access)
  — File geospatial database
  — SDE geospatial database.

Guidelines:

- "Working" files can be in any format. A "working" file is any file that you create or are given to produce a new layer which is used in the final project.

- "Working" files will be discarded on a regular basis. Original or source files should not be altered but copied for revision.

- Many projects require a database schema specified by a client. An example would be the Spatial Data Standards, Facilities, Infrastructure, and Environment (SDSFIE). A client specification for data storage supersedes EA's standards.

3.5 ACQUIRING GEOGRAPHIC INFORMATION SYSTEM DATA

Large amounts of "acceptable" data can be found on the web at federal, state, and local websites. "Acceptable" data are data that include metadata describing the source, time of data collection, and how they were created. If metadata are not provided for a dataset, then the dataset should not be used. All data must be downloaded to the project directory for use in a project. Web links of GIS data through the web are not acceptable for any project that is final. Web-linked data can be used for working figures that will not be used for a deliverable.

Required:

- Data cannot be used if copyrighted, i.e., Google aerials.
- Data that are linked via the web have to be downloaded to the project directory.
- Data must have similar accuracy with overall GIS project.

Guidelines:

None.

3.6 METADATA

Metadata are an important aspect of any GIS project. Metadata become crucial in situations where a project's findings and/or data have been contested or questioned. It is required that all files used in a project will have metadata. If the file does not have an existing metadata file, then the GIS specialist will create one. The information to include is as follows.

Required:

- Source of data
- Coordinate and projection
- Description of data
- Time of data collection
- How it was created
- Who manipulated if applicable.

Guidelines:

This information is superseded when the client dictates the use of a metadata standard like the Federal Geographic Data Committee (FGDC) FGDC-STD-001-1998 adopted by all agencies. Additionally, this requirement can be disregarded for general maps, such as vicinity or location maps, that are created for ad-hoc purposes.

The exception to this is for projects that have general maps like "vicinity" or "location" maps and a basic site map. All EA generated layers must have metadata. Examples would be Global Positioning System collected data from the field, floodplain, or contour generation. All projects where GIS data are delivered to a client must have metadata

3.7 QUALITY ASSURANCE AND QUALITY CONTROL

Quality assurance and quality control should be integrated at all levels of the project. Specifics of EA's Quality Assurance/Quality Control Program can be found in the Corporate Quality Assurance Plan located on *Inside EA*. At a minimum, review by an appropriate Senior Technical Reviewer is required for all client deliverables. Additionally, the checks listed below should be followed:

Required:

CHECK	PDF	GIS FILES
Check directory structure	X	X
Check file naming conventions	X	X
Check files for projection	X	X
Check final files in data base	X	X
Check metadata	X	X
Check consistency between figures	X	X
Check source data are not streamed from external website		X

Guidelines:

With larger projects that span a large time period or have large and numerous datasets, PLTS should be considered for quality assurance/quality control reporting. PLTS can check data topology, data duplicates, polygon overlap, and database structure, to list a few. Other tools such as SDSFIE analyze tools can be used for quality assurance/quality control on SDSFIE data deliverables.

4. REFERENCES

Link to the Federal Geographic Data Committee standards
http://www.fgdc.gov/standards/projects/FGDC-standards-projects/metadata/base-metadata/index_html.

Appendix A

Engineering Style Templates

Engineering Style Portrait Template

Includes the following:

- EA Logo
- Project Title
- Figure Title
- Project Manager Initials
- Designed by Initials
- Drawn by Initials
- Checked by Initials Scale
- Date
- Project Number
- Figure Number
- File Path

EA Engineering, Science, and Technology, Inc.	PROJECT TITLE GOES HERE LOCATION	FIGURE TITLE GOES HERE

PROJECT MGR.	DESIGNED BY	DRAWN BY	CHECKED BY	SCALE	DATE	PROJECT NO.	FIGURE
-----	-----	-----	-----	NOT TO SCALE	-----	-----	-----

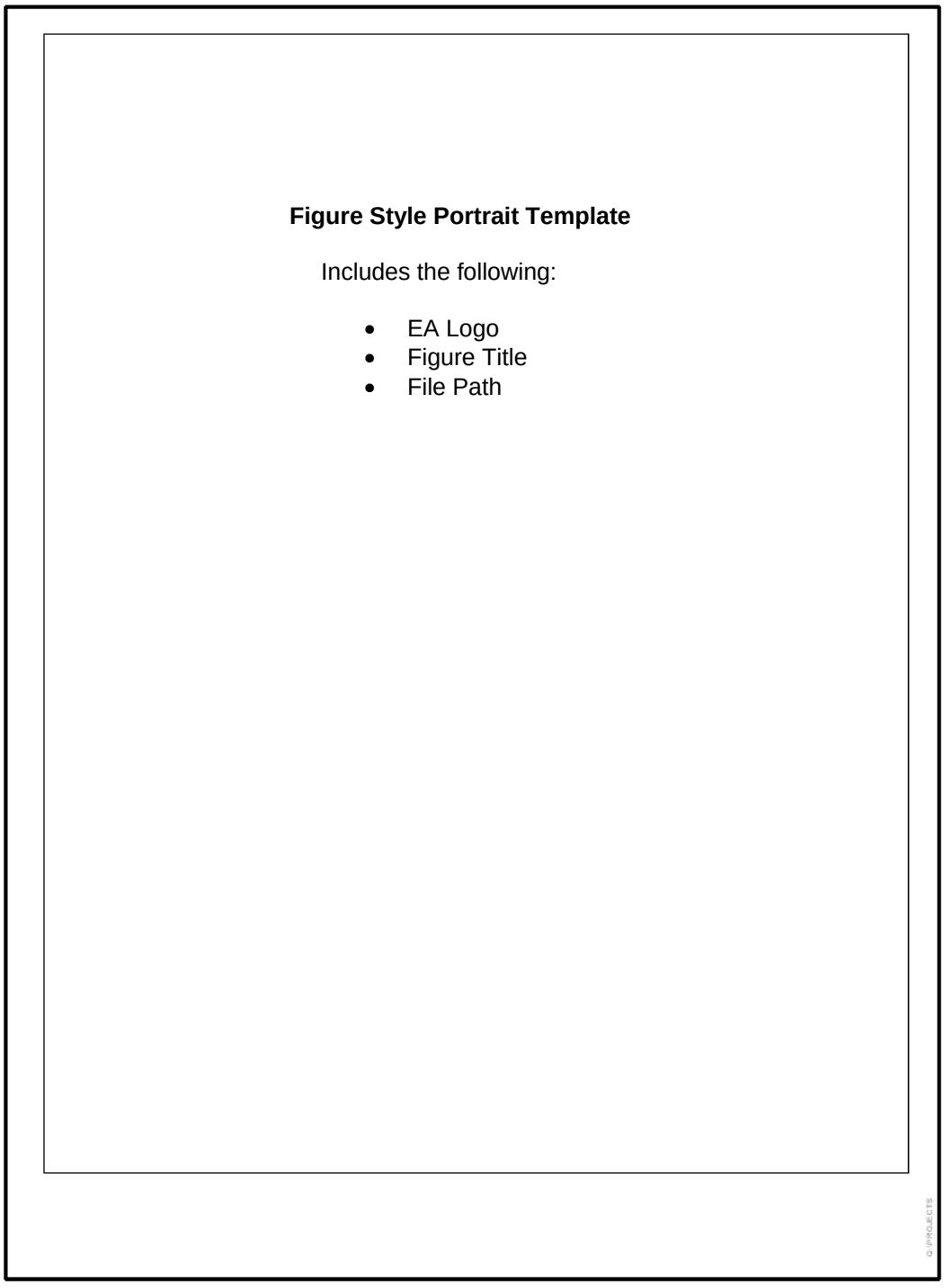


Figure Title

Engineering Style Landscape Template

Includes the following:

- EA Logo
- Project Title
- Figure Title
- Project Manager Initials
- Designed by Initials
- Drawn by Initials
- Checked by Initials Scale
- Date
- Project Number
- Figure Number
- File Path

EA Engineering, Science, and Technology, Inc.	PROJECT TITLES GOES HERE LOCATION	FIGURE TITLE GOES HERE	DESIGNED BY ----	DRAWN BY ----	DATE NOT TO SCALE	PROJECT NO. ----
			CHECKED BY ----	PROJECT MGR. ----	SCALE NOT TO SCALE	FIGURE ----

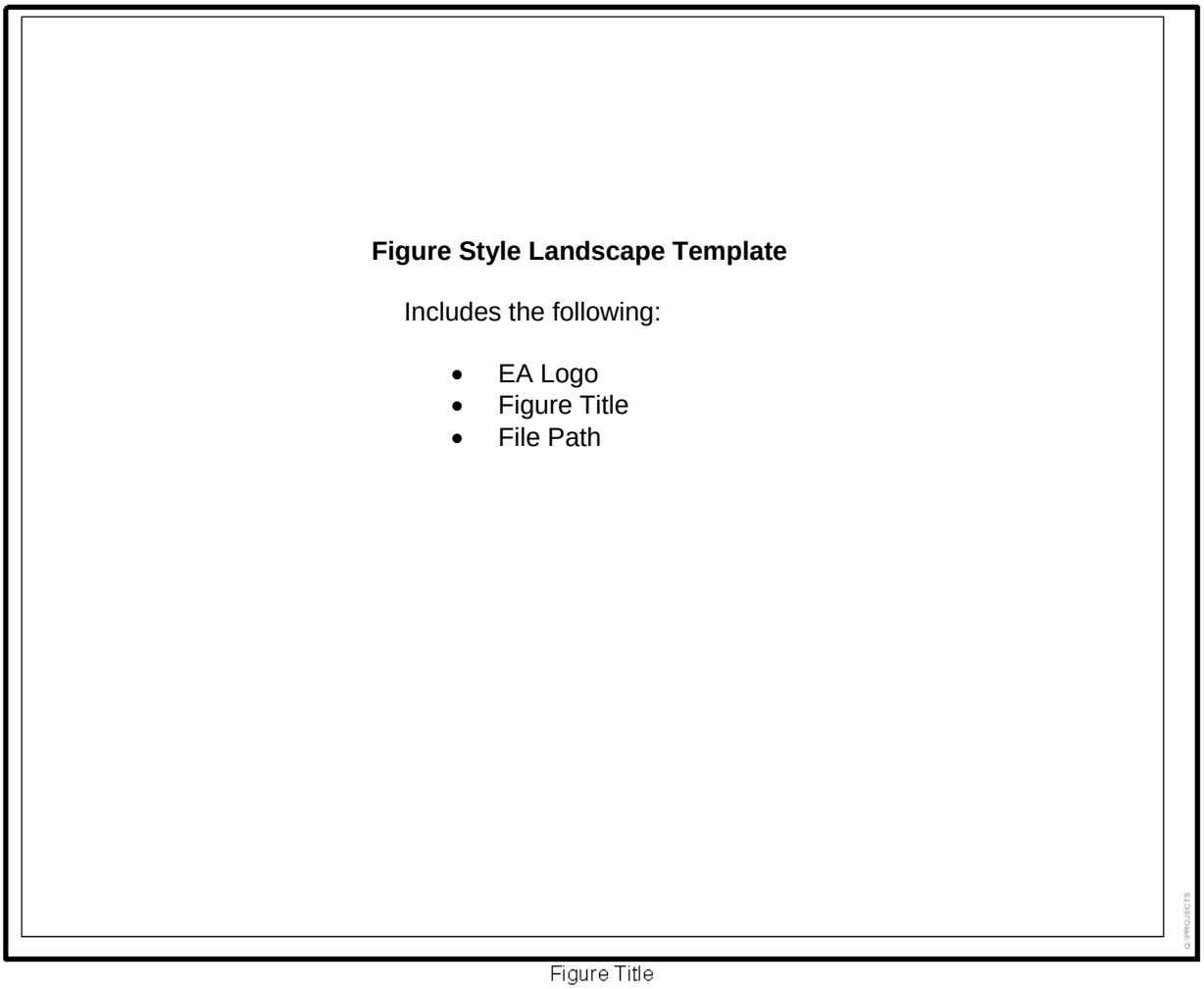
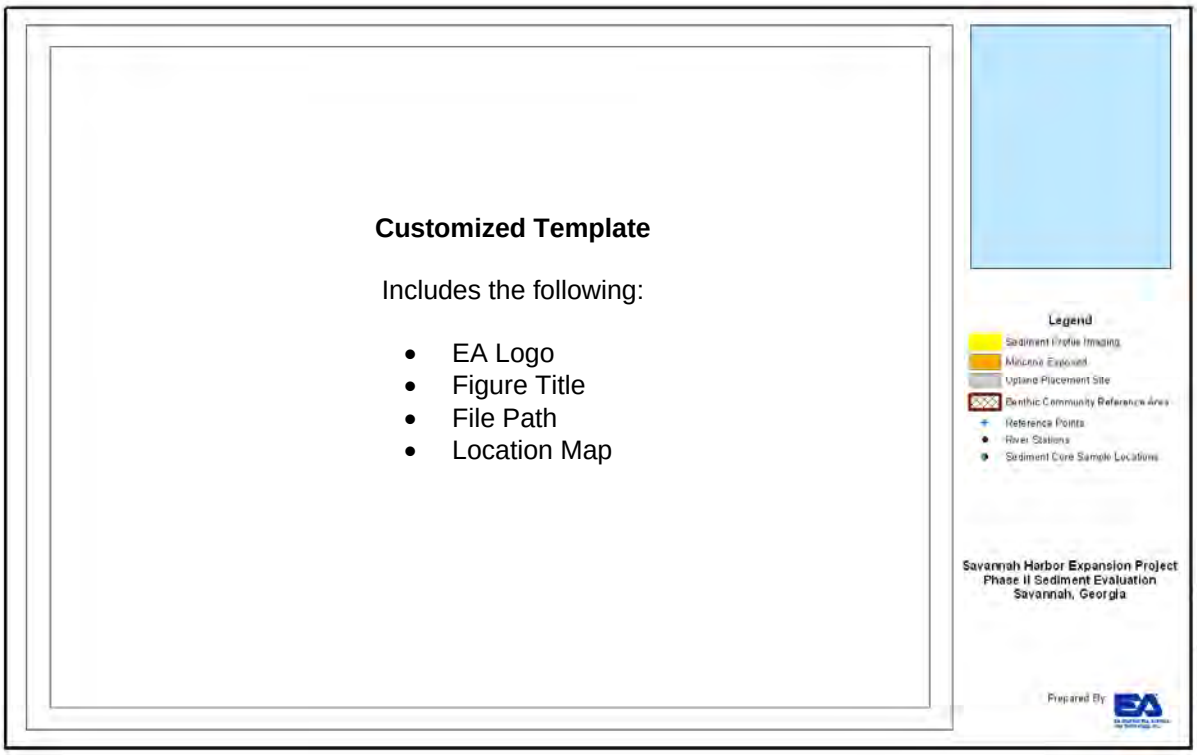


Figure Title


Customized Template
Includes the following:
EA Logo
Figure Title
File Path
Location Map
Legend
Sediment Profile Imaging
Material Exposed
Upland Placement Site
Benthic Community Reference Area
Reference Points
River Stations
Sediment Core Sample Locations
Savannah Harbor Expansion Project
Phase II Sediment Evaluation
Savannah, Georgia
Prepared By:

Standard Operating Procedure No. 100

Assessment of Bedrock Coreholes Using Downhole Geophysics

Prepared by

EA Engineering, Science, and Technology, Inc.
11019 McCormick Road
Hunt Valley, Maryland 21031

Revision 0
December 2013

CONTENTS

1. SCOPE AND APPLICATION

The purpose of this Standard Operating Procedure (SOP) is to establish the protocol for using downhole geophysical surveys to determine the depth and orientation of bedding planes and fractures, groundwater flow patterns in bedrock fracture zones, and bedrock contaminant migration pathways, as well as to identify zones for aquifer testing and groundwater sampling using packer assemblies. The geophysical logging will include caliper, fluid resistivity, heat-pulse flow meter, and acoustic televiewer. The caliper logging will be used to identify fractures and lithologic changes within the boring. Fluid resistivity logging will provide information on groundwater flow within the open boreholes, between the borehole and the fractures, and will provide water quality data for each location. The heat-pulse flow meter logging will identify permeable zones, apparent vertical hydraulic conductivity, and groundwater flow direction. Acoustic televiewer logging will provide data on the bedrock structures and orientation of fractures in the bedrock.

2. MATERIALS

The following materials may be required:

Multi-parameter tool and/or sonde(s) for caliper logging and fluid resistivity, heat-pulse flow meter, and acoustic televiewer	Field notebook
Associated hoisting mechanism(s) and centralizers	Decontamination supplies (SOP-05)
Data logger(s)	Personnel protective equipment as required by the Accident Prevention Plan

3. PROCEDURE

Bedrock coreholes will be installed using the methods described in SOP-19A. The bedrock coreholes will be left open to complete the downhole geophysical activities.

The static water level and depth of the borehole will be measured prior to initiation of geophysical logging using an electronic water level indicator graduated to 0.01 ft. The probe of the instrument will be lowered slowly until the indicator light illuminates and/or the alarm sounds. The probe will be pulled above the water surface and the measurement repeated. Borehole depth will be measured by lowering the probe of the instrument through the water column and noting when the probe reaches the bottom borehole. The depth to water and borehole depth will be noted ground surface.

Geophysical data will be collected digitally using a downhole logging system, with data logging at a rate of 5 to 10 feet per minute. A system wireline will be attached to a multi-parameter tool to be used to collect geophysical data, and the instrument(s) will be lowered into the borehole. Centralizers will be used as needed to ensure the instrument(s) is/are centered, and run smoothly in the hole. Geophysical testing will be conducted as the instrument is lowered to the bottom of the borehole. Multiple runs may be required to optimize data quality. Geophysical data will be recorded digitally in the field and reviewed by the equipment operator to determine if refinement of logging is required.

4. DECONTAMINATION

Following collection of geophysical data, a monitoring well will be installed per SOP-19 or the corehole will be abandoned per SOP-28 as needed. Non-dedicated downhole logging equipment will be decontaminated prior to use, between coring locations, and following completion of logging as per SOP 5.

5. PRECAUTIONS

Prior to collecting any samples, consult the Accident Prevention Plan for personal protective equipment required for sampling activities.

Decontaminate the sampling equipment and change gloves between samples to minimize the risk of cross-contamination.

APPENDIX B-2
Accutest Laboratories
Standard Operating Procedures

Lab Manager: Douglas Yargeau

QA Officer: Robert Treggiari

TITLE: LABORATORY ANALYSIS OF DISSOLVED GASES IN AQUEOUS SAMPLES

TEST METHOD REFERENCES: RSK-175, 8/11/94; RSK 174, 8/11/94, US EPA Region I, 2/21/02

REVISED SECTIONS: 11.4

1.0 SCOPE & APPLICATION

1.1 The analysis of dissolved gases in aqueous samples, primarily groundwater is used to monitor natural attenuation. Natural attenuation is a remedial approach to groundwater contamination that depends upon natural processes to degrade and dissipate contamination constituents. The monitoring of certain dissolved gases can determine if a contaminated site is conducive to aerobic or anaerobic bioremediation. The activity of bioremediation can be determined by certain dissolved gas byproducts as in methane.

1.2 Methane, ethane, ethene, and CO2 are the target compounds for this SOP. The applicable matrix is water (primarily groundwater).

2.0 SUMMARY

2.1 This method is for the determination of trace levels of dissolved gases in aqueous samples, primarily groundwater. A sealed volatile organic vial with teflon septa is prepared at ambient temperature by displacing a certain amount of the water with zero grade helium through the septa. The sample is shaken to disperse the dissolved gases into the headspace which is injected into a calibrated gas chromatograph coupled with a flame ionization detector (FID) and Thermal conductivity detector (TCD). The calibration is performed by external standard technique using a multipoint gas standard containing the compounds of interest in ppmv. The sample is quantitated based on the concentration of the helium headspace in ppmv and is carried through a series of calculations involving Henry's Law constant, temperature of the sample, volume of sample vial, headspace volume, dilution factor, and the molecular weight of the gas. Results are reported in ug/l of each dissolved gas.

3.0 METHOD REPORTING AND DETECTION LIMITS

3.1 The reporting limit (RL) is based on the lowest calibration standard. RL's may vary depending on matrix difficulties, sample volumes or weights, and percent moisture. Detected concentrations below this concentration cannot be reported without qualification. Reporting limits are:

3.1.1	Methane:	10 ug/l
3.1.2	Ethane:	10 ug/l
3.1.3	Ethene:	10 ug/l
3.1.4	CO2:	100 ug/l

3.2 The Method Detection Limit (MDL) represents the lowest reportable concentration of an individual analyte that meets the method qualitative identification criteria.

3.3 Method Detection limits (MDLs) are experimentally determined using the procedures described in 40 CFR, Part 136, Appendix B. Actual reported MDLs incorporate the sample volume analyzed and sample dilutions if needed, which may cause MDL variations from sample to sample.

3.4 In general, MDLs are determined through the analysis of at least 7 replicate blank spikes (using the same procedures for sample analysis). The MDL is calculated by multiplying the standard deviation of the replicate concentrations by the appropriate Student's t value (3.143 for 7 replicates). If more than 7 replicates are analyzed refer to 40 CFR, Part 136, Appendix B for the appropriate student's t value. MDLs are determined initially (prior to analysis), on an annual basis, and after major maintenance to equipment. MDL data is archived with Quality Assurance. Refer to the most recent study for current MDLs.

3.5 Current MDLs are entered into the LIMS and may be reported upon request. Additionally, current MDLs are also located in the MDL file on the QA server (LINUXMA1). These summary tables are saved under the applicable year. MDL hardcopy summary tables are also filed with QA and are available for review upon request. Obsolete hardcopy MDL summaries are filed with QA.

3.6 Refer to the Procedure for Developing Detection Limits SOP (MQA245) for additional procedural detail.

4.0 DEFINITIONS

4.1 ALIQUOT - a measured portion of a sample, or solution, taken for sample preparation and/or analysis.

4.2 BATCH – A group of samples which behave similarly with respect to the sampling or the testing procedures being employed and which are processed as a unit. For QC purposes, if the number of samples in a group is greater than 20 (or 10 for certain methods), then each group of 20 samples (or 10 samples for certain methods) or less will all be handled as a separate batch. Batch size for this test is 20 samples or less.

4.3 BLANK SPIKE – a sample matrix, free from the analytes of interest, spiked with verified known amounts of analytes from a source independent of the calibration standards or a material containing known and verified amounts of analytes. It is generally used to establish intra-laboratory or analyst specific precision and bias or to assess the performance of all or a portion of the measurement system.

4.4 CALIBRATION – the establishment of an analytical curve based on the absorbance, emission intensity, or other measured characteristic of known standards. The calibration standards must be prepared using the same type of acid or concentration of acids as used in the sample preparation.

4.5 CALIBRATION FACTOR (CF) - a measure of the gas chromatographic response of a target analyte to the mass injected. The calibration factor is analogous to the Relative Response Factor (RRF) used in the Volatile and Semivolatile fractions.

4.6 CALIBRATION STANDARDS – a series of known solutions used by the analyst for calibration of the instrument (i.e., preparation of the analytical curve).

4.7 CONTINUING CALIBRATION – analytical standard run every 12 hours to verify the initial calibration of the analytical system.

4.8 DRY WEIGHT – the weight of a sample based on percent solids. The weight after drying in an oven.

4.9 CONTAMINATION - a component of a sample or an extract that is not representative of the environmental source of the sample. Contamination may stem from other samples, sampling equipment, while in transit, from laboratory reagents, laboratory environment, or analytical instruments.

4.10 FIELD SAMPLE - a portion of material to be analyzed that is contained in single or multiple containers and identified by a unique sample number.

4.11 FIELD BLANK – this is any sample that is submitted from the field and is identified as a blank. This includes trip blanks, rinsates, equipment blanks, etc.

4.12 HOLDING TIME – the elapsed time expressed in days from the date of sampling until the date of its analysis.

4.13 INTERFERENTS – substances which affect the analysis for the analyte of interest.

4.14 GAS CHROMATOGRAPH (GC) - the instrument used to separate analytes on a stationary phase within a chromatographic column. The analytes are volatized directly from the sample (VOA water and low-soil) volatized from the sample extract (VOA medium soil), or injected as extracts (SVOA and PEST). In VOA and SVOA analysis, the compounds are detected by a Mass Spectrometer (MS). In PEST analysis, the compounds are detected by an Electron Capture (EC) detector. In the screening procedure (all fractions), the Flame Ionization Detector (FID) is used as the detector.

4.15 INITIAL CALIBRATION - analysis of analytical standards for a series of different specified concentrations; used to define the linearity and dynamic range of the response of the mass spectrometer or electron capture detector to the target compounds.

4.16 INITIAL CALIBRATION VERIFICATION – analysis of a check standard from a second source (either vendor or lot) from the initial calibration standards to verify the initial calibration.

4.17 INTEGRATION TIME RANGE - the retention time at the beginning of the area of integration to the retention time at the end of the area of integration.

4.18 INSUFFICIENT QUANTITY - when there is not enough volume (water sample) or weight (soil/sediment) to perform any of the required operations: sample analysis or extraction, percent moisture, MS/MSD, etc.

4.19 MATRIX - the predominant material of which the sample to be analyzed is composed. For the purpose of this SOP, a sample matrix is either water or soil/sediment. Matrix is not synonymous with phase (liquid or solid).

4.20 MATRIX EFFECT - in general, the effect of a particular matrix (water or soil/sediment) on the constituents with which it contacts. This is particularly pronounced for clay particles which may adsorb chemicals and catalyze reactions. Matrix effects may prevent extraction of target analytes, and may affect surrogate recoveries. In addition, non-target analytes may be extracted from the matrix causing interferences.

4.21 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.

4.22 MATRIX SPIKE DUPLICATE - a second aliquot of the same matrix as the matrix spike (above) that is spiked in order to determine the precision of the method.

4.23 METHOD BLANK - an analytical control consisting of all reagents, internal standards, and surrogate standards (or SMCs for VOA), that is carried throughout the entire analytical procedure. The method blank is used to define the level of laboratory, background, and reagent contamination.

4.24 PERCENT DIFFERENCE (%D) - To compare two values, the percent difference indicates both the direction and the magnitude of the comparison, i.e., the percent difference may be either negative, positive, or zero. (In contrast, see relative percent difference.)

4.25 PURGE AND TRAP (DEVICE) - analytical technique (device) used to isolate volatile (purgeable) organics by stripping the compounds from water or soil by a stream of inert gas, trapping the compounds on an adsorbent such as a porous polymer trap, and thermally desorbing the trapped compounds onto the gas chromatographic column.

4.26 PURGEABLES - volatile compounds.

4.27 REAGENT WATER - water in which an interferant is not observed at or above the minimum quantitation limit of the parameters of interest. Accutest uses deionized water (municipal water which passes through Accutest's DI treatment system).

4.28 RELATIVE PERCENT DIFFERENCE (RPD) - As used in this SOP to compare two values, the relative percent difference is based on the mean of the two values, and is reported as an absolute value, i.e., always expressed as a positive number or zero. In contrast, see percent difference.

4.29 RELATIVE RESPONSE FACTOR (RRF) - a measure of the relative mass spectral response of an analyte compared to its internal standard. Relative Response Factors are determined by analysis of standards and are used in the calculation of concentrations of analytes in samples. RRF is determined by the following equation:

$$RRF = \frac{A_x}{A_{is}} x \frac{C_{is}}{C_x}$$

Where,

A = area of the characteristic ion measured
C = concentration, or amount (mass)
is = internal standard
x = analyte of interest

4.30 RELATIVE RETENTION TIME (RRT) - the ratio of the retention time of a compound to that of a standard (such as an internal standard).

$$RRT = \frac{RT_c}{RT_{is}}$$

Where,

RT_c = Retention time for the volatile target or surrogate compound in continuing calibration.
RT_{is} = Retention time for the internal standard in calibration standard or in a sample.

4.31 RESPONSE - or Instrumental Response: a measurement of the output of the GC detector (MS, EC, or FID) in which the intensity of the signal is proportionate to the amount (or concentration) detected. Measured by peak area or peak height.

4.32 SOIL - used herein synonymously with soil/sediment and sediment.

4.33 SURROGATES (Surrogate Standard) – for volatiles, semivolatiles and pesticides/Aroclors, compounds added to every blank, sample, matrix spike, matrix spike duplicate, and standard; used to evaluate analytical efficiency by measuring recovery. Surrogates are brominated, fluorinated, or isotopically labeled compounds not expected to be detected in environmental media.

4.34 TWELVE-HOUR TIME PERIOD - The twelve (12) hour time period for GC/MS system instrument performance check, standards calibration (initial or continuing calibration), and method blank analysis begins at the moment of injection of the DFTPP or BFB analysis that the laboratory submits as documentation of instrument performance. The time period ends after 12 hours have elapsed according to the system clock. For pesticide/Aroclor analyses performed by GC/EC, the twelve hour time period in the analytical sequence begins at the moment of injection of the instrument blank that precedes sample analyses, and ends after twelve hours have elapsed according to the system clock.

4.35 VOLATILE COMPOUNDS - compounds amenable to analysis by the purge and trap technique. Used synonymously with purgeable compounds.

4.36 RETENTION TIME (RT) - the time a target analyte is retained on a GC column before elution. The identification of a target analyte is dependent on a target compound's retention time falling within the specified retention time window established for that compound. Retention time is dependent on the nature of the column's stationary phase, column diameter, temperature, flow rate, and other parameters.

4.37 DEIONIZED WATER (DI water) - water that has passed through Accutest's deionization system. Used as reagent water (water that an interferant is not observed at or above the minimum quantitation limit of the parameters of interest).

4.38 SPIKE BLANK OR LABORATORY CONTROL SAMPLE (LCS) – A blank spiked with a known concentration of analyte (from a second source from the calibration standard) or an external quality control standard with a known concentration of analyte used to determine accuracy of the method.

5.0 HEALTH & SAFETY

5.1 All safety practices must be followed as outlined in the Accutest Laboratories Employee Safety Handbook and Chemical Hygiene Plan. Safety glasses, gloves, and lab coats must be worn. All samples, solutions, and extracts must be treated as unknown and potentially hazardous.

5.1 The toxicity or carcinogenicity of each reagent used in this method has not been precisely determined; however, each chemical should be treated as a potential health hazard. Exposure to these reagents should be reduced to the lowest possible level.

5.2 Gas cylinders must be secured at all times.

6.0 COLLECTION, PRESERVATION, & HOLDING TIMES

6.1 Samples should be collected in a 40ml VOA vial with Teflon septa in duplicate. Approximately 0.25ml of a 1:1 hydrochloric acid is used to preserve samples to ph<2 to fix the gases unless CO2 is to be determined. HCl can convert inorganic carbon to CO2.

6.2 Vials must be filled to zero headspace to eliminate loss of gases and stored at 4°C ± 2°C.

6.3 Preserved samples have a 14 day holding time from time of collection. Un-preserved should be analyzed within 7 days from the time of collection.

7.0 APPARATUS AND MATERIALS

7.1 Gas Chromatograph System

7.1.1 Gas Chromatograph - Agilent 6890 series gas chromatograph with flame ionization detector (FID) and thermal conductivity detector (TCD) in series. Injection port is equipped with Electronic Pressure Control (EPC). **See table 1 for conditions.**

7.1.2 Column – Supleco Carboxen 1006, 30m x 0.53mm

7.2 Data System

7.2.1 Hewlett Packard Enviroquant PC based chemstation software, capable of multi-point calibration technique and full instrument control.

7.2.2 Hewlett Packard "Custom Reports" using a calculation spreadsheet to convert the headspace ppmv concentrations into ug/l final results using Henry's Law constant, temperature of the sample, volume of sample vial, headspace volume, and the molecular weight of the gas.

7.3 Automatic Shaker.

7.4 Gas tight syringes

7.4.1 0.01cc, 0.05cc, 0.10cc, 0.50cc, and 1.0cc hamilton gas tight syringe with style point #5 tip for sample and standard injections.

7.4.2 5cc syringe with luer lock tip and piercing needle to measure displaced sample out of vial.

7.5 Helium delivery system with adjustable regulator and piercing needle set at a flow of approximately 5cc/min for helium delivery to displace sample out of vial and create helium headspace.

7.6 40 ml I-chem pre-certified vials with teflon septa for sampling and method blank preparation.

7.7 Thermometer (verified against a NIST thermometer) for ambient temperature measurement (inserted through the septa of a VOA vial filled with DI water).

8.0 STANDARDS & REAGENTS

8.1 Commercially Available Gas Standards

8.1.1 Scott Specialty Gases Calibration Standard containing acetylene, n-butane, ethane, ethylene, methane, propane, propylene, propene, and nitrogen (all at 15 ppmv) – balance nitrogen.

8.1.2 Scott Specialty Gases Calibration Standard containing acetylene, carbon dioxide, carbon monoxide, ethane, ethylene, methane (all at 10,000 ppmv) – balance nitrogen.

8.1.3 Scott Specialty Gas Calibration Standard for Laboratory Control Sample containing carbon dioxide, carbon monoxide, hydrogen, methane, oxygen (10,000 ppmv) – balance nitrogen. This is a second source (different lot number from calibration standards).

8.2 Carrier, Detector Gas, and Headspace Gas

8.2.1 Helium

8.2.2 Hydrogen
8.2.3 Zero Air

8.3 De-ionized, reagent grade water

9.0 INTERFERENCES

9.1 Background levels of methane and CO2 can appear from helium.

9.2 Exposure of sample to atmosphere should be avoided due to background levels of target compounds that can contaminate samples and losses of light gases to atmosphere. **Keep vials sealed at all times.**

9.3 Gas Chromatographic system needs to baked out over night after batch analysis to eliminate the buildup of water vapor on the column that can distort chromatography.

10.0 PROCEDURE

10.1 **Calibration** – Calibration is performed by external standard technique. The calibration range encompasses 15 ppmv to 10,000 ppmv for methane, ethane, ethene and CO2. At least 4 concentrations must be used in the calibration curve.

10.1.1 Standards are drawn directly from gas cylinders, which are fitted with a regulator and syringe adapter. The gas cylinder outlet pressure should be set at approximately 10psig. Insert syringe without plunger into syringe adapter and let purge for 5-10 seconds to eliminate all dead volume. Insert plunger, turn off cylinder, and adjust syringe to the injection volume needed.

10.1.2 When GC is ready, manually inject standard and immediately push the “start” button on the GC to begin acquisition.

10.1.3 Low and Medium level Calibration Points - Syringe volumes of 0.001cc (2ppmv), 0.002cc (4ppmv), 0.010cc (20ppmv), 0.050cc (100ppmv), 0.25cc (500ppmv) and 0.5cc (1000ppmv) are injected from the 1000ppmv stock standard. Select the appropriate syringe for the most accurate measurement.

10.1.4 High Level Calibration Points - Syringe volumes of 0.10cc(8000ppmv methane,10000ppmv CO,CO2), 0.25cc(20000ppmv methane, 25000ppmv CO,CO2), and 0.5cc(40000ppmv methane and 50000 CO,CO2).

Vol. (ml)	10,000 ppmv stock	Conc. (ppmv)	Low Level Stock	Conc. (ppmv)
0.2	“	10000	0.20 ml 15 ppmv	15
0.05	“	2500		
0.02	“	1000		
0.005	“	250		
0.001	“	100		

10.1.5 Calibration factor is defined as peak divided by standard concentration in ppmv.

10.1.6 Initial Calibration – The linear regression coefficient must be equal to or greater than 0.995 (must be forced through zero). The low standard must have a signal/noise ratio > 5.

10.1.7 Initial Calibration Verification (ICV) – The initial calibration must be verified using a source independent from the calibration standards (if available). Results must be within 20% (% D) of the true value. If %D criteria are not met corrective action must be taken to determine the problem and the initial calibration must be re-analyzed. The concentration of the ICV should vary from the concentration of the calibration check (section 10.1.9).

10.1.8 Calibration Check – The check level (100ppbv) is acquired every 4 hours (or 25 samples – whichever is more often) and at the end of the run (the next day's calibration check may be used for this purpose. Inject 0.05cc of the 1000ppmv stock standard.

10.1.8.1 The percent difference (%D) must be <20.

10.1.8.2 If %D criteria fail, a new minimum 4 point calibration must be performed (see section 10.1.10).

10.1.9 A calibration check may only be repeated once. If the second analysis fails, a new initial calibration must be performed (an exception to this rule is if corrective action after the first 2 calibration checks resolves the problem and 2 passing sequential calibration checks are run). In situations where the first check fails to meet criteria, the instrument logbook should have clear documented notations as to what the problem was and what the corrective actions was performed to enable the second analysis to meet criteria.

10.2 **Retention Time Windows**

10.2.1 Retention Time Windows Retention time windows must be calculated for each analyte on each GC column and whenever a new GC column is installed. The data must be retained in the laboratory.

10.2.2 Make three injections of all standard mixtures at approximately equal intervals, during the 72-hr period.

10.2.3 Calculate the standard deviation of the three absolute retention times for each single component.

10.2.4 Apply plus or minus three times the standard deviations to retention time of each standard continuing calibration. This will be used to define the retention time window for the sample.

10.2.5 The above-described procedures may be performed automatically through the LIMS. See supervisor for procedure.

10.2.6 The retention time windows must be updated daily based on the calibration check standard (ie. The RT windows produced from the above-described study are applied to the actual RTs of the compounds in the first calibration check of the day).

10.2.7 If default standard deviation of 0.01 minutes is employed, the width of the window will be 0.03 minutes.

10.3 **Sample preparation**

10.3.1 Remove samples from storage and allow samples to reach ambient temperature.

10.3.2 Select a duplicate vial for a sample duplicate at a minimum of one per twenty.

10.3.3 Fill a blank vial with deionized, organic free water to zero headspace for a batch method blank.

10.3.4 Insert the purge needle from the helium delivery system, with a flow of approximately 5cc/min, into the septa of the sample vial.

10.3.5 Immediately insert the 5ml syringe with needle attached to collect and measure the 5ml displaced sample volume. Make sure needle tip is below the level of the purging helium needle tip. The pressure of the helium flow will force the water sample up into the collection syringe.

10.3.6 Collect 5ml of sample into the measuring syringe and simultaneously remove both needles. You have now displaced 5ml of sample with 5cc of inert helium.

10.3.7 Securely place samples, blanks, and QC on the shaker in organic prep and set timer for 5 minutes. When finished remove and place upright in vial rack for injection.

10.3.8 Place the NIST verified thermometer in the same rack for ambient temperature measurement.

10.4 **Analysis.**

10.4.1 The nominal sample injection volume is 0.2 cc

10.4.2 The daily sequence is as follows:

- Inject 100ppmv(0.05cc of 1000ppmv standard) daily calibration check standard
- Inject 100ppmv(0.05cc of 1000ppmv alternate standard) Laboratory Control Sample
- Inject 0.2cc of the helium blank used to prepare samples (Not required).
- Inject 0.2cc of prepared method blank, which is DI water prepared identical to samples.
- Inject 0.2cc of any samples and duplicates.

10.4.3 Quant the helium blank to determine background area of target compounds. Typically methane and CO2 have trace level hits resulting in peak areas that may be used for background peak area correction. Background correction is NOT used in this method.

10.4.4 Quant the method blank and run the custom report template to ensure all compounds are below the detection limits.

10.4.5 Quant each sample following analysis to determine if a smaller volume for dilution needs to be injected. Diluted injections should be performed next after the undiluted run to minimize any losses through the septa.

10.4.6 Measure and record the ambient temperature in degrees C (to nearest whole degree) from the NIST verified thermometer in the sample rack for each CCV analysis.

10.4.7 Measure and record the amount of water sample (ml) that remains in each sample, duplicate, and blank. The actual vial volume is then the remaining sample plus the displaced sample volume (5ml). Most VO vials are actually 40-44cc volume depending on the lot #s and vendors.

10.5 **Calculations.**

10.5.1 Calibration Factor (CF)

$$CF = \frac{As}{Cs}$$

Where: As = Area of the peak for the compound being measured.
Cs = Concentration of the compound being measured (μg/l).

10.5.2 Percent Relative Standard Deviation (% RSD).

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

Where: SD = Standard Deviation.
CFav = Average calibration factor from initial calibration.

10.5.3 Percent Difference (% D).

$$\%\ D = \frac{| CFav - Cfc |}{CFav} \times 100$$

Where: CFc = CF from continuing calibration (CBCHK).

10.5.4 **Sample Concentrations**

10.5.4.1 **PPMV concentration by external standard technique from calibration curve.**

$$\text{Conc. } (\mu g/l) = \frac{Ac \times D}{CFav}$$

Where: Ac = Area of peak for compound being measured.
D = Dilution factor based on volume of headspace injected = 0.5/cc inj
CFav = Average response factor from initial calibration.

10.5.4.2 **UG/L concentration conversion using Custom Reports**

10.5.4.2.1 Open the custom reports file “natatt” under the “Custom Reports” menu. See attachment of template.

10.5.4.3 Enter headspace created from helium displacement (typically 5cc)

10.5.4.3.1 Enter volume of sample (ml) remaining in vial. (vial volume – sample volume remaining)

10.5.4.4 The correct ug/l concentration will automatically be calculated based on Henry’s Law constant, temperature of the sample, volume of sample vial, headspace volume, Dilution Factor, and the molecular weight of the gas.

10.5.4.5 The attached template describes how all calculations are derived along with a table of Henry’s Constants for each gas.

10.5.5 Percent Recovery (% R) for LCS

$$\%\ R = \frac{\text{Concentration found}}{\text{Concentration spiked}} \times 100$$

10.5.6 Relative Percent Difference (RPD) for sample duplicates.

$$RPD = \frac{|SR1 - SR2|}{(1/2)\,(SR1+SR2)} \times 100$$

Where: SR1 = First sample replicate concentration (ug/l).
SR2 = Duplicate sample concentration (ug/l)

11.0 QUALITY ASSURANCE

QC Requirements Summary:

Calibration Check STD.	Every 4 hrs or 20 samples (whichever is more) at end of run (bracket samples)
ICV	Every initial calibration
LCS	Every 24 hrs
Helium Blank	Every 24 hrs
Batch blank	one per batch [a]
Matrix Duplicate	one per batch [a]

(a) The maximum number of samples per analytical batch is twenty.

11.1 ICV - refer to section 10.1.8.

11.2 Calibration Check – refer to section 10.1.9.

11.3 Method blank.

11.3.1 The method blank is carried through all stages of the sample preparation and measurement.

11.3.2 An acceptable method blank must be analyzed once for each analytical batch.

11.3.3 Evaluate the method blank to the MDL. No compound can be present above ½ the reporting level which is determined after running custom reports.

11.3.4 If the method blank does not meet contamination criteria, the entire batch must be re-prepped and reanalyzed.

11.4 Blank Spike and MS/MSD.

Create the headspace as outlined in section 10.3. Keeping the vial inverted prepare BS, MS and/or MSD by adding 0.500 ml of 10000 ppmv standards to the aqueous portion allowing the standards to bubble through the sample and collect in the 5 ml headspace of the sample. The QC is then analyzed exactly as a field sample (Section 10.3). This results in an aqueous concentration of 110 ug/L for methane, 223 ug/L for ethane, and 298 ug/L for ethene. The aqueous concentration of CO2 should be 100 ug/L.

11.4.1 The blank spike and MS/MSD % recoveries must be between 80-120%. The MSD agreement must be within 25% RPD.

11.4.2 If the recovery of blank spike is outside the QC limits check the calculation. Recalculate if an error is detected. If blank spike criteria are still not met, corrective action must be performed to determine the problem and a satisfactory LCS must be analyzed prior to sample analysis.

11.4.3 If the matrix spike recoveries do not meet acceptance criteria, check the blank spike recovery to verify that the method is in control. If the blank spike did not meet criteria, the method is out of control for the parameter in question and should be reanalyzed or qualified with an estimate

of potential bias. Otherwise, matrix interference is assumed and the data is reportable. No further corrective action is required.

11.5 A Precision and accuracy (P&A) study is performed as an initial determination of capability, on an annual basis (continued demonstration of capability – a successful PT result may be used in place of a P&A for continued DOC), and if any significant changes have been made to the instrument. In general, 4 replicates or blank spikes are analyzed using the same procedures and conditions for sample analysis. The percent recovery and standard deviation (of the 4 replicate percent recoveries) are compared to 80-120% (Rec) and 30 (SD). If percent recovery or standard deviation criteria are not met, corrective action must be taken to bring the system back into control. The P&A study replicates must be prepared from a source independent from the calibration standards .

11.6 Data assessment and acceptance criteria for quality control. Quality control limits are generated at least on an annual basis by QA using an in-house program. Blank spike and MS/MSD QC data are pooled for the previous year (or other specified time frame) and the data is processed and evaluated by QA. The new limits are entered into the LIMS where they can be viewed directly or by printing out a compound list (QC limits must be requested). The annual QC limit data is filed with QA.

12.0 DOCUMENTATION

12.1 The analytical logbook is a record of the analysis sequence; the logbook must be completed daily. Each instrument will have a separate logbook.

12.2 If samples require reanalysis, a brief explanation of the reason should be documented in this log.

12.3 The standard preparation logbook must be completed for all standard preparations. All information requested must be completed; the page must be signed and dated by the respective person.

12.4 The Accutest lot number must be cross-referenced on the gas standard cylinder.

12.5 The instrument Maintenance logbook must be completed when any type of maintenance is performed on the instrument. Each instrument will have a separate log.

12.6 All laboratory logbooks must be routinely reviewed and initialed or signed by the lab manager.

12.7 Any corrections to laboratory data must be done using a single line through the error. The initials of the person and date of correction must appear next to the correction.

13.0 DATA REVIEW

13.1 The analyst conducts the primary review of all data. This review begins with a check of all Instrument and method quality control and progresses through sample quality control concluding with a check to assure that the client's requirements have been executed. The analyst has the authority and responsibility to perform corrective action for any out-of-control parameter of non-conformance.

13.2 A secondary review is performed by department managers, and it includes review of the data produced by their department. All manual calculations, QC criteria, and a comparison of the data package to client specified requirements are checked. The department manager may reject data, initiate reanalysis, take additional corrective action, or reprocess data.

13.3 The laboratory director performs a full tertiary review of the data package following its assembly. This review includes an evaluation of QC data against acceptance criteria and a check of the data package contents to assure that all analytical requirements and specifications were executed.

13.4 Spot-check reviews are performed by the Quality Assurance Officer focusing on all elements of the deliverable including the client's specifications and requirements, analytical quality control, sample custody documentation and sample identification.

14.0 DATA REPORTING

14.1 A results page including positive results and/or RLs, units, methodology, preparation and/or analysis dates, and data qualifiers are reported. Additional quality control data including calibration summaries, MS/duplicate percent recoveries and RPDs, surrogat recoveries, blank spike recoveries, and method blank results may be reported upon request of the client. Additionally, raw data including any instrument printouts (quantitation reports, chromatograms) , laboratory logbooks, etc. may be reported to the client.

14.1.1 Data may be submitted to the client in a specified electronic format (EDD).

14.1.2 Electronic hardcopy reports may be submitted to the client on request (e-hardcopy).

14.1.3 Once the data is approved by the laboratory manager, it may be accessed by clients via LabLink™.

14.2 All data qualifiers used within the result page (sample or QC results) are defined at the bottom of the page. Refer to the Accutest Laboratories of New England , Inc. Qualifier Definitions form (QA108) for definition of all qualifiers used in Accutest Laboratories of New England result reports. This form is located on the QA server under QA forms. It may be provided to the client upon request.

14.3 Procedures for handling non-conforming data.

14.3.1 If quality control data does not meet criteria the non-conformance must be discussed in a case narrative and footnoted on the applicable quality control report summary.

14.3.2 If preservation or holding time criteria is not met and the samples are analyzed the result page must be footnoted with this information, and the non-conformance must be discussed in a case narrative or other suitable communication (telephone conversation log or email). Client notification documentation should be included with the data (telephone conversation log, fax, or email).

15.0 POLLUTION PREVENTION & WASTE MANAGEMENT

15.1 Pollution Prevention. Users of this method must perform all procedural steps that control the creation and/or escape of wastes of hazardous materials to the environment. The amounts of standards, reagents, and solvents must be limited to the amounts specified in this SOP. All safety practices designed to limit the escape of vapors, liquids, or solids to the environment must be followed. All method users must be familiar with the waste management practices described in section 15.2.

15.2 Waste Management. Individuals performing this method must follow established waste management procedures as described in the Sample and Waste Disposal SOP. This document describes the proper disposal of all waste materials generated during the testing of samples as follows:

15.2.1 Non-hazardous aqueous wastes
15.2.2 Hazardous aqueous wastes
15.2.3 Chlorinated organic solvents
15.2.4 Non-chlorinated organic solvents
15.2.5 Hazardous solid wastes
15.2.6 Non-hazardous solid wastes
15.2.7 Microbiological wastes

16.0 ADDITIONAL REFERENCES

16.1 “Analysis of Dissolved Methane, Ethane, and Ethylene in Ground Water by a Standard Gas Chromatographic Technique”. Published in Journal of Chromatographic Science, vol. 36 May 1998. Authored by Don H. Kampbell and Steve A. Vandegrif; RSKSOP-147, 1/14/93; RSKSOP-114, 1/15/91; US EPA Region I Technical Guidance for the Natural Attenuation Indicators: Methane, Ethane, and Ethene.

Table 1
Recommended Operating Conditions
(may change to optimize)

Carrier Gas	Helium
Detector Gas	Hydrogen, Zero Air
Injection Type	Splitless
Purge off time	0
Column Flow	EPC constant flow at 15psig @ 30c
Initial temperature	35c for 1minute
Oven Ramp	35c/min to 220c with no hold
Final Time	2 min
Injection temp	250c
FID temp	300c
TCD temp	200c

Lab Manager: Brad Madadian
QA Officer: Robert Treggiari

TITLE: AUTOMATED CYANIDE ANALYSIS. (Lachat)

TEST METHOD REFERENCE: EPA 335.4, SW846 9012B (Rev 2, November 2004) Mod.

REVISED SECTIONS: added section 8.0 notation; added 11.4; reordered section 11

1.0 SCOPE AND APPLICATION

1.1 These methods are applicable to the determination of cyanide in drinking water, waste and surface water, domestic and industrial wastes, and solids.

1.2 Test code: Cn

1.3 The Reporting (RL) is based on the lowest calibration standard. RL'S may vary depending on matrix difficulties and sample volumes or weight and percent moisture.

2.0 SUMMARY

2.1 Cyanide is released from Cyanide complexes through distillation (refer to distillation SOPS). It then reacts with Chloramine-T to form Cyanogen Chloride which combines with Pyridine and Barbituric Acid which is measured at 570 nm.

3.0 REPORTING AND METHOD DETECTION LIMIT

3.1 The reporting limit (RL) is based on the lowest calibration standard. RL's may vary depending on matrix difficulties, sample volumes or weights, and percent moisture. Detected concentrations below this concentration cannot be reported without qualification.

3.2 The Method Detection Limit (MDL) represents the lowest reportable concentration of an individual analyte that meets the method qualitative identification criteria.

3.3 Method Detection limits (MDLs) are experimentally determined using the procedures described in 40 CFR, Part 136, Appendix B. Actual reported MDLs incorporate the sample volume analyzed and sample dilutions if needed, which may cause MDL variations from sample to sample.

3.4 In general, MDLs are determined through the analysis of at least 7 replicate blank spikes (using the same procedures for sample analysis). The MDL is calculated by multiplying the standard deviation of the replicate concentrations by the appropriate Student's t value (3.143 for 7 replicates). If more than 7 replicates are analyzed refer to 40 CFR, Part 136, Appendix B for the appropriate student's t value. MDLs are determined initially (prior to analysis), on an annual basis, and after major maintenance to equipment. MDL data is archived with Quality Assurance. Refer to the most recent study for current MDLs. Refer to the SOP for MDLs (MQA245) for additional detail regarding MDL study procedures.

3.5 Current MDLs may be entered into the LIMS, and may be viewed by printing out the compound list from the LIMS. Additionally, MDLs are reported on the result page upon client request. Current MDL studies are filed

with Quality Assurance. Obsolete MDL studies are archived with the QA files. Electronic MDL data is found in the annual "MDL" folder on the QA server (LINUXMA1).

4.0 DEFINITION

4.1 ALIQUOT - a measured portion of a sample, or solution, taken for sample preparation and/or analysis.

4.2 BATCH – A group of samples which behave similarly with respect to the sampling or the testing procedures being employed and which are processed as a unit. For QC purposes, if the number of samples in a group is greater than 20 (or 10 for certain methods such as 335.4), then each group of 20 samples (or 10 samples for certain methods such as 335.4) or less will all be handled as a separate batch.

4.3 CALIBRATION CHECK STANDARD/CONTINUING CALIBRATION VERIFICATION (CCV). The calibration check standard is a mid-range calibration standard. The calibration check standard or CCV must be run at a frequency of 10 percent. The mid-level calibration check standard criteria is ± 10 percent of the true value. Refer to the specific quality control section of the SOP. The exception to this rule is if the recovery on the calibration check standard is high and the samples to be reported are less than the reporting limit.

4.4 CALIBRATION – the establishment of an analytical curve based on the absorbance, emission intensity, or other measured characteristic of known standards. The calibration standards must be prepared using the same reagents as used in the sample preparation.

4.5 CALIBRATION STANDARDS – a series of known solutions used by the analyst for calibration of the instrument (i.e., preparation of the analytical curve).

4.6 CONTAMINATION - a component of a sample or an extract that is not representative of the environmental source of the sample. Contamination may stem from other samples, sampling equipment, while in transit, from laboratory reagents, laboratory environment, or analytical instruments.

4.7 FIELD SAMPLE - a portion of material to be analyzed that is contained in single or multiple containers and identified by a unique sample number.

4.8 HOLDING TIME – the elapsed time expressed in days from the date of sampling until the date of its analysis.

4.9 IN-HOUSE - at the Contractor's facility.

4.10 INSUFFICIENT QUANTITY - when there is not enough volume (water sample) or weight (soil/sediment) to perform any of the required operations: sample analysis or extraction, percent moisture, MS/MSD.

4.11 INITIAL CALIBRATION VERIFICATION – analysis of a check standard from a second source (either vendor or lot) from the initial calibration standards to verify the initial calibration.

4.12 MATRIX - the predominant material of which the sample to be analyzed is composed. For the purpose of this SOP, a sample matrix is either water or soil/sediment. Matrix is not synonymous with phase (liquid or solid).

4.13 MATRIX DUPLICATE: A duplicate sample is distilled/analyzed at a minimum of 1 in 20 samples (or 10 samples for EPA 335.4). The relative percent difference (RPD) between the duplicate and the sample should be assessed. The duplicate RPD is calculated as shown below. Assess laboratory performance

against the control limits. In house limits are generated once sufficient duplicate data is available to generate limits (usually a minimum of 20 to 30 analyses). If a duplicate is out of control, flag the results with the appropriate footnote. If the sample and the duplicate are less than 5 times the reporting limits and are within a range of ± the reporting limit, then the duplicate is considered to be in control. Note: If control limits are not specified, use default limits of ± 20% RPD.

$$\frac{(|\text{Sample Result} - \text{Duplicate Result}|) \times 100}{(\text{Sample Result} + \text{Duplicate Result})/2} = \text{Duplicate RPD}$$

4.14 MATRIX SPIKE: The laboratory must add a known amount of each analyte to a minimum of 1 in 20 samples (1 in 10 for EPA335.4). In house limits are generated once sufficient matrix spike data is available to generate limits (usually a minimum of 20 to 30 analyses). If a matrix spike is out of control, then the results should be flagged with the appropriate footnote. If the matrix spike amount is less than one fourth of the sample amount, then the sample cannot be assessed against the control limits and should be footnoted to that effect.

$$\frac{(\text{Spiked Sample Result} - \text{Sample Result})}{(\text{Amount Spiked})} \times 100 = \text{Matrix Spike Recovery}$$

4.15 METHOD BLANK. The laboratory must distill and analyze a method blank with each set of samples. A minimum of one method blank is required for every 20 (1 in 10 for EPA335.4) samples. If no digestion step is required, then the method blank is equivalent to the reagent blank. The method blank must contain the parameter of interest at levels of less than ½ the reporting limit for that parameter. If the method blank contains levels over the reporting limits, the samples must be redigested or redistilled and reanalyzed. The exception to this rule is when the samples to be reported contain greater than 10 times the method blank level. In addition, if all the samples are less than a client required limit and the method blank is also less than that limit, then the results can be reported as less than that limit.

4.16 PERCENT MOISTURE - an approximation of the amount of water in a soil/sediment sample made by drying an aliquot of the sample at 105 °C. The percent moisture determined in this manner also includes contributions from all compounds that may volatilize at or below 105 °C, including water. Percent moisture may be determined from decanted samples and from samples that are not decanted.

4.17 REAGENT WATER – distilled or deionized water in which an interferant is not observed at or above the minimum quantitation limit of the parameters of interest. ASTM type II or equivalent. Accutest uses deionized water (municipal water which passes through Accutest's DI treatment system).

4.18 RELATIVE PERCENT DIFFERENCE (RPD) - As used in this SOP and elsewhere to compare two values, the relative percent difference is based on the mean of the two values, and is reported as an absolute value, i.e., always expressed as a positive number or zero.

5.0 HEALTH AND SAFETY

5.1 All safety practices must be followed as outlined in the Accutest Laboratories Employee Safety Handbook and Chemical Hygiene Plan. Safety glasses, gloves, and lab coats must be worn. All samples, solutions, and extracts must be treated as unknown and potentially hazardous.

5.2 The toxicity or carcinogenicity of each reagent used in this method has not been precisely determined; however, each chemical should be treated as a potential health hazard. Exposure to these reagents should be reduced to the lowest possible level.

5.3 Extra caution should be used when handling cyanide. All procedures must be performed within an operating fume hood. Generation of toxic hydrocyanic gas occurs with the mixing of cyanide and acid.

6.0 COLLECTION, PRESERVATION & HOLDING TIMES

6.1 The sample should be distilled and the distillate kept in 4° C ± 2° C. (for appropriate preservatives prior to distillation please refer to distillation sop)

6.2 All samples must be prepared and analyzed within 14 days from the time sampled.

7.0 APPARATUS

7.1 Automated continuous flow analyzer designed to deliver and react sample and reagents in the required order and ratios. Currently, the Lachat 8000 Automated Ion Analyzer is being used.

7.2 Autosampler XYZ

7.3 Multichannel pump

7.4 Reaction manifold

7.5 Colorimetric detector

7.6 Real time data acquisition device

8.0 REAGENTS

Note: All chemicals listed are reagent grade unless otherwise specified. Distilled, deionized water should be used whenever water is required. All applicable standard/reagent preparation information, including vendor, lot number, date of preparation, date of expiration, calculations, and initials must be entered in the appropriate standard/reagent preparation logbook. Vendors typically used by Accutest include Fisher Scientific, Absolute Standards, VWR, Accustandard, Supelco, Chemservices, Ultra, and ERA. Additional vendors may be utilized as necessary.

8.1 Phosphate buffer (0.71 M) Dissolve 97 g Potassium Dihydrogen phosphate (potassium phosphate, Monobasic, KH2PO4) in 800ml of deionized water. Dilute to volume.

8.2 Chloramine-T dissolve 2.0g Chloramine-T in 300ml of deionized water in a 500ml volumetric flask. Dilute to volume.

8.3 Pyridine Barbituric Acid: PREPARE UNDER HOOD place 15 g Barbituric Acid in a 1000 ml volumetric flask..Add 100ml deionized water, rinsing down the sides of the flask. Add 75 ml Pyridine and mix. Add 15 ml Hydrochloric Acid and continue to mix. Add 500ml deionized water and mix until all the Barbituric

Acid is dissolved. Dilute to volume with deionized water.

8.4 Carrier, 0.25M NaOH: Weigh out 10g of NaOH pellets into 700ml of DI water. Dilute to 1000ml of DI water.

8.5 Stock standard (1000 mg /L): In a 1000 ml volumetric flask dissolve 2.51 g KCN and 2 g KOH in about 800 ml of DI water. Dilute to the mark with DI water and mix.

8.6 Working Stock #1 (10 mg/L): Dilute 1 ml of 1000 mg/L stock standard (8.5) to 100 ml with DI water in volumetric flask and mix.

8.7 Working Stock #2 (1.0 mg/L): Dilute 10 ml of 10 mg/L working stock #1 (8.6) to 100 ml with DI water in a volumetric flask and mix.

8.8 Calibration Standard. Calibration standards should be made from the above standard solutions as shown below. Class A volumetric pipets and flasks should be used. All standards should be brought to a final volume of 100 ml with carrier solution (8.4).

Note: Refer to Distillation SOP. For Micro distillation standards are already distilled with samples.

ml of working stock #1	ml of working stock #2	final conc. (mg/L)
0.00	0.00	0.00 (0.25 M NaOH only)
0.00	1.00	0.010
0.00	3.00	0.030
0.00	5.00	0.050
1.00	0.00	0.100
2.00	0.00	0.200
3.00	0.00	0.300
4.00	0.00	0.400
5.00	0.00	0.500

9.0 INTERFERENCES

9.1 Refer to Distillation SOP.

10.0 PROCEDURE

10.1 Below is a step by step procedure for the analysis of samples for the determination of total cyanide.

10.2 Install the CN reaction manifold. Check all tubing and change any tubing that is flat, dirty, etc. Install the appropriate sample loop and the appropriate filter. Place the tubing in the reagents. Also make sure that the waste container is in place. Refer to the manual for additional information.

10.3 On the desk top open the lachat software by clicking on the "**Omni"** icon, and then click on "**OPEN**" on top of the tool bar. The "**OPEN"** window will now be on the screen. The templates, specific to each methods, are stored in the data file. Open the appropriate data file (ie., **CN**). Click on the template.

10.4 Three windows will open on screen, ("**the run worksheet", "run properties", and "channel one**). **"Run**

Worksheet" will contain the appropriate standards. To extend the worksheet (adding samples and QC, etc), right click on the bottom line of the worksheet (CalBlk), and click on "**append many".** The appended rows window will now be open and the appropriate number of rows needed should be entered, click "**ok**". The work sheet must now be sequenced to direct the auto-sampler to the correct sample locations.

10.5 Click and drag by starting in the gray sample No. column down to the last sample to highlight the rows (not including STD's). Right click, then go to columns, then to "**Auto Number Cups**" and click. The run should now be in numerical sequence.

10.6 In the "**Run Properties**" window, click on the Run tab and check "**Export Data as CSV file**" There are other areas of the "**Run Properties**" window that contain method specific information such as timing. These settings must not be adjusted without consulting the area lab supervisor, or an experienced analyst.

10.7 Allow the instrument to warm up for 30 minutes.

10.8 Start pumping reagents through the system.

10.9 To begin the analysis, click on "**START"** on toolbar. The instrument will begin to calibrate.The acceptance calibration criteria for correlation coefficient of 0.995 is set within the software. If the criteria is met, the instrument will proceed with sample analysis.

10.10 Observe the peaks in "**Channel One"** window. The baseline should be smooth and peaks must be well shaped and smooth. If peaks look abnormal, that may indicate the chemistry problems, such as pH differences. Small spikes are indicative of air bubbles in the system.

10.11 The run maybe stopped at anytime by clicking "**Stop**" in the top toolbar. It should be noted that every time the run has been stopped and started, a new file is created. If dilutions are required, the dilution factor needs to be entered in the "**Run Worksheet"** window. On MDF column, click on the box and enter the dilution factor.

10.12 When the run is completed the file is automatically exported to " **Lachat CSV files**" locally under "**My Document**" directory. Click on **"Lachat CSV Files".** All generated files are listed based on the following format: OM_date_time Am or PM (OM_4-11-2006_12-10-34PM).
Drag the generated files for that run/day to the current Month/Year directory. For ease of search, at the beginning of each month, create a directory to save the runs for the entire month, and name it based on the month, and the year (**ie**, **OCT 2006**). Once you have moved your file to the month/year directory, copy this file to" **Lachat**" directory located on the serve MAFILE1**WC_DATA** , rename the file to the format which is acceptable to LIMS. This naming scheme will be based on date, the matrix, no. of the run for that day, and finally the test code extension, (ie, **050206W1.CN**). Once the files have been renamed, drag the file to Server **WC**. Server **WC** is the processing branch of the LIMS for Wet Chemistry. Once the Run is processed in LIMS, go to GNAPP, and review the run. Package all raw data, and logbook copies in a folder, and turn the package to the area manager for data review, and quality control check..

10.13 To obtain a print out of the sample run, click on Tools in the top toolbar and then click on custom report. To format the report to contain the calibration curve and dilutions, click on report, then format in the custom report window.

10.14 At the end of the run, make sure to rinse out the system/manifold with DI water.

10.15 Results exceeding the upper range of the calibration curve must be diluted and re-analyzed. The diluted result should be within the upper range of the calibration curve.

11.0 QUALITY ASSURANCE

11.1 Below is a summary of the quality control requirements for this method. Make sure to check with laboratory supervisor or manager for any additional client specific quality control requirements.

11.2 **Initial Calibration.** A minimum of six calibration points must be analyzed. See section 10.9 for acceptance criteria. A calibration blank must be analyzed prior to running samples. See section 11.10 for acceptance criteria.

11.3 **ICV - Initial Calibration Verification.** A standard prepared from a source independent from the initial calibration standards (second source – either lot or manufacturer) must be analyzed to immediately after calibration to verify the initial calibration. The acceptance criteria for the ICV are ± 10%. If the ICV does not meet criteria, the problem must be resolved and a new initial calibration must be run before samples can be analyzed.

11.4 **ICB – Initial Calibration Blank.** A blank that does not contain the analyte of interest (Cyanide) and is analyzed to verify the calibration of the analytical system. ICB must be less than the reporting limit and must be analyzed after the ICV. If the ICB is greater than the reporting limit, all samples bracketed with this QC must be re-analyzed.

11.5 **CCV. Continuing Calibration Verification.** Analytical standard run every 10 samples to verify the calibration of the analytical system. The acceptance criteria for CCV is ± 10 % of the true value. If CCV fails, all samples bracketed with this QC must be re-analyzed.

11.6 **CCB. Continuing Calibration Blank.** A blank that does not contain the analyte of interest (CN) and is analyzed to verify the calibration of the analytical system. CCB must be less than the reporting detection limit and must be analyzed after the CCV. If CCB is greater than the reporting detection limit, all samples bracketed with this QC must be re-analyzed.

11.7 **Method Blank**. The laboratory must analyze a method blank with each set of samples. A minimum of one method blank is required for every 20 samples (10 samples for EPA 335.4). The method blank must not contain the analyte at greater than the reporting limit. If the method blank contains an analyte level over that limit, the samples must be reprepped and reanalyzed.

11.8 **Spike Blank.** A Minimum of two spike blank is required for every 20 samples (10 samples for EPA 335.4)). The spike blank recovery should be assessed using limits of 90-110 %. In house limits can be generated. These limits must be equal or better than the required limits. If the spike blank is outside of this range, the samples must be reanalyzed (if the spike blank recovery is biased high and the samples are non-detected they may be reported without qualification).

11.9 **Matrix Spike.** The laboratory must add a known amount of each analyte to a minimum of 1 in 20 samples (1in 10 for EPA335.4). The matrix spike recovery is calculated as shown below. Assess laboratory performance against the control limits of 75-125% (90-110% for EPA335.4). In house limits are generated once sufficient matrix spike data is available to generate limits (usually a minimum of 20 to 30 analyses). If a matrix spike is out of control, then the results should be flagged with the appropriate footnote. If the matrix spike amount is less than one fourth of the sample amount, then the sample cannot be assessed

against the control limits and should be footnoted to that effect.

$$\frac{(\text{Matrix Spike Result} - \text{Original Result}) \times 100}{\text{Amount of spike}}$$

11.10 **Matrix Duplicate.** The laboratory must prepare a duplicate sample for a minimum of 1 in 20 samples. (1 to 10 for EPA 335.4). The relative percent difference (rpd) between the duplicate sample and the original should be assessed. The Duplicate RPD should be calculated as shown below

$$\frac{(\text{Original Sample Result} - \text{Duplicate Result}) \times 100}{(\text{Original Sample Result} + \text{Duplicate Result}) \times 0.5} = \% \text{ RPD}$$

The Duplicate RPD should be assessed using in house limits. Until these limits can be generated, then the default limit of 20 percent RPD should be applied. If a duplicate RPD is out of control, then the

results should be flagged with the appropriate footnote. If the sample and the duplicate are less than 5 times the reporting limits and are within a range of ± the reporting limit, then the duplicate is considered to be in control.

11.11 External Check Sample. It is recommended that the laboratory analyze an external check standard on a quarterly basis. The limits supplied by the manufacturer should be applied. If the results for the external QC check are outside of the range, do not continue analysis. Consult the laboratory supervisor. Do not analyze samples until the problem is resolved.

11.12 A Precision and accuracy (P&A) study is performed as an initial determination of capability, on an annual basis (continued demonstration of capability – a successful PT result may be used in place of a P&A for continued DOC), and if any significant changes have been made to the instrument. In general, 4 replicates or blank spikes are analyzed using the same procedures and conditions for sample analysis. The percent recoveries are compared to either default limits of 90-110% or in-house control limits once established. The standard deviation of the 4 replicate percent recoveries are compared to either ±20 or to in-house limits once established. If percent recovery or standard deviation criteria are not met, corrective action must be taken to bring the system back into control. The P&A study must be performed using a source independent from the calibration standards (second source).

11.13 Quality Control data is generated (control charts) and reviewed on an annual basis by Quality Assurance (blank spike/ matrix spike recoveries and matrix duplicate RPDs).

12.0DOCUMENTATION

12.1 The analytical logbook is a record of the analysis sequence; the logbook must be completed daily. Items to include are:

12.1.1 Which method was used.
12.1.2 The sample ID, including MS or duplicate as necessary.
12.1.3 The initial volume aliquoted for distillation.
12.1.4 The final volume of the distillate.
12.1.5 Any comments or observations concerning the sample that may influence the analytical procedure.

12.1.6 All QC spikes must have documented the lot number of the spike solution used, the volume added and the concentration of the spiking solution.

12.2 If samples require reanalysis, a brief explanation of the reason should be documented in this log.

12.3 The standard preparation logbook must be completed for all standard preparations. All information requested must be completed; the page must be signed and dated by the respective person.

12.4 The Accutest lot number must be cross-referenced on the standard vial/container. The expiration date must be noted on the standard vial/container.

12.5 The instrument Maintenance logbook must be completed when any type of maintenance is performed on the instrument. Each instrument will have a separate log.

12.6 All laboratory logbooks must be routinely reviewed and initialed or signed by the lab manager.

12.7 Any corrections to laboratory data must be done using a single line through the error. The initials of the person and date of correction must appear next to the correction.

13.0 DATA REVIEW

13.1 The analyst conducts the primary review of all data. This review begins with a check of all method quality control and progresses through sample quality control concluding with a check to assure that the client's requirements have been executed.

13.2 A secondary review is performed by department managers, and it includes review of the data produced by their department. Manual calculations, QC criteria, and a comparison of the data package to client specified requirements are checked. The department manger may reject data, initiate reanalysis, take additional corrective action, or process data.

13.3 The laboratory director performs a full tertiary review of the data package following its assembly. This review includes an evaluation of QC data against acceptance criteria and a check of the data package contents to assure that all analytical requirements and specifications were executed.

13.4 Spot-check reviews are performed by the Quality Assurance Officer focusing on all elements of the deliverable including the client's specifications and requirements, analytical quality control, sample custody documentation and sample identification.

14.0 DATA REPORTING

14.1 A results page including positive results and/or RLs, units, methodology, analysis dates, and data qualifiers are reported. Additional quality control data including raw data, calibration summaries, MS/Dup percent recoveries and RPDs, spike blank recoveries, and method blank results may be reported upon request of the client.

14.2 Data may be submitted to the client in a specified electronic format (EDD).

14.3 Data may be submitted electronically to the client in PDF using the e-hardcopy system.

14.4 Once the data is approved by the laboratory manager, it may be accessed by clients via LabLink™.

14.5 Procedures for handling non-conforming data.

14.5.1 If quality control data does not meet criteria the non-conformance must be discussed in a case narrative and footnoted on the applicable quality control report summary.

14.5.2 If preservation or holding time criteria is not met and the samples are analyzed the result page must be footnoted with this information, and the non-conformance must be discussed in a case narrative or other suitable communication (telephone conversation log or email). Client notification documentation should be included with the data (telephone conversation log, fax, or email).

15.0 POLLUTION PREVENTION & WASTE MANAGEMENT

15.1 Pollution Prevention. Users of this method must perform all procedural steps that controls the creation and/or escape of wastes of hazardous materials to the environment. The amounts of standards, reagents, and solvents must be limited to the amounts specified in this SOP. All safety practices designed to limit the escape of vapors, liquids, or solids to the environment must be followed. All method users must be familiar with the waste management practices described in section 15.2.

15.2 Waste Management. Individuals performing this method must follow established waste management procedures as described in the Sample and Waste Disposal SOP. This document describes the proper disposal of all waste materials generated during the testing of samples as follows:

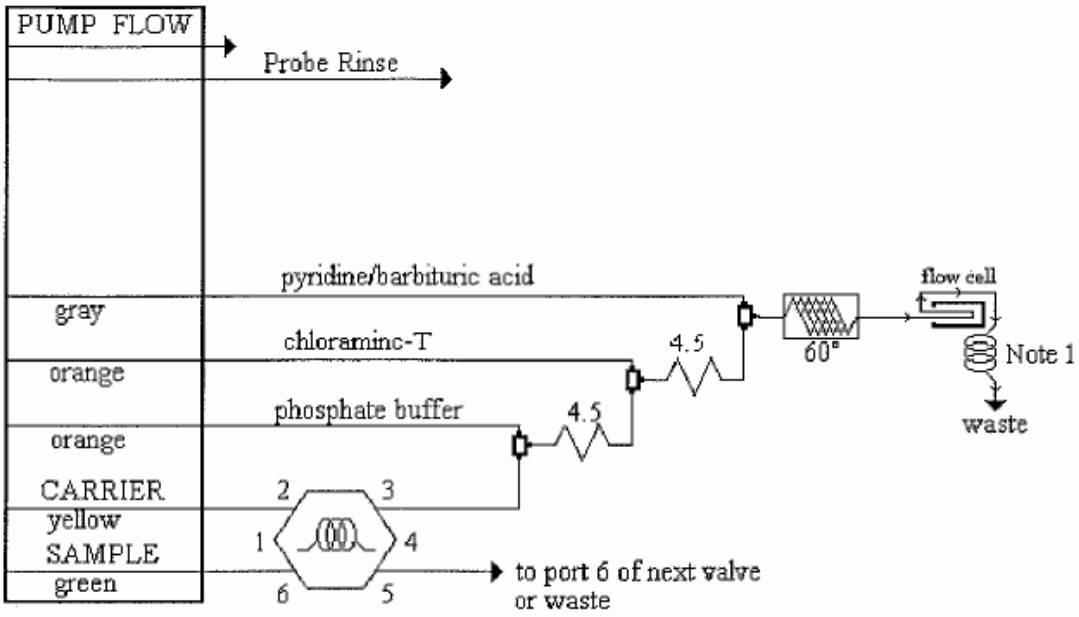
15.2.1 Non-Hazardous aqueous wastes
15.2.2 Hazardous aqueous wastes
15.2.3 Chlorinated organic solvents
15.2.4 Non-chlorinated organic solvents
15.2.5 Hazardous solid wastes
15.2.6 Non-hazardous solid wastes

16.0 METHOD PERFORMANCE

16.1 Method performance is evaluated by the annual QC limits (control charts) generated by QA, and the annual MDL study results. Refer to section 3.0 for MDLs, and section 11.12 for QC limits.

17.0 ADDITIONAL REFERENCES

17.1 QuickChem Method 10-204-00-1-A



Carrier: 0.25M sodium hydroxide solution (Reagent 1).
Manifold Tubing: 0.8 mm (0.032 in) i.d. This is 5.2 µL/cm.
AE Sample Loop: 150 cm x 0.8 mm i.d.
QC8000 Sample Loop: 150 cm x 0.8 mm i.d.
Interference Filter: 570 nm

Apparatus: An injection valve, a 10 mm path length flow cell , and a colorimetric detector module is required. The  shows 650 cm of tubing wrapped around the heater block at the specified temperature.

4.5: 70 cm of tubing on a 4.5 cm coil support

Note 1: 200 cm backpressure loop, 0.52 mm i.d.

Lab Manager: Brad Madadian
QA Officer: Robert Treggiari

TITLE: ALKALINITY, TOTAL (pH 4.5)

TEST METHOD REFERENCE: 2320 B. Standard Methods for the Examination of Water and Wastewater 21th Edition, 2005

REVISED SECTIONS: Section 8.0 notation

1.0 SCOPE AND APPLICATION

1.1 Alkalinity is the measure of the acid-neutralizing capacity of a water sample, as the sum of all titratable bases. Since the alkalinity of surface waters is primarily a function of carbonate, bicarbonate, and hydroxide content, it may be used as an indicator for the concentration of these constituents. The measured value will also include some borates, phosphates, silicates and other bases if they are present. The sample is not to be diluted, filtered, or altered in any way.

1.2 For samples of low alkalinity (<20 mg $CaCO_3$/l) an extrapolation technique is used. The amount of acid to lower the pH exactly 0.3 pH units is measured, after the initial endpoint has been attained. Because this corresponds to an exact doubling of the hydrogen ion concentration, an extrapolation may be made to the equivalence point.

1.3 This method is applicable to surface water, and saline waters, domestic and industrial waste.

1.4 Test code: ALK

2.0 SUMMARY

2.1 An unaltered sample is titrated to an electrometrically determined end point of PH 4.5. The sample must not be filtered, diluted, concentrated, or altered in any way.

3.0 METHOD REPORTING AND DETECTION LIMIT

3.1 The reporting limit for this analysis is 5.0 Mg/L.

3.2 The Method Detection Limit (MDL) represents the lowest reportable concentration of an individual analyte that meets the method qualitative identification criteria.

3.3 Method Detection limits (MDLs) are experimentally determined using the procedures described in 40 CFR, Part 136, Appendix B. Actual reported MDLs incorporate the sample volume analyzed and sample dilutions if needed, which may cause MDL variations from sample to sample.

3.4 In general, MDLs are determined through the analysis of at least 7 replicate blank spikes (using the same procedures for sample analysis). The MDL is calculated by multiplying the standard deviation of the replicate concentrations by the appropriate Student's t value (3.143 for 7 replicates). If more than 7 replicates are analyzed refer to 40 CFR, Part 136, Appendix B for the appropriate student's t value. MDLs are determined initially (prior to analysis), on an annual basis, and after major maintenance to equipment. MDL data is archived with Quality Assurance. Refer to the most recent study for current MDLs. Refer to the SOP for MDLs

(MQA245) for additional detail regarding MDL study procedures. The MDL represents the lowest reportable concentration of an individual analyte that meets the method qualitative identification criteria.

3.5 Current MDL studies are filed with Quality Assurance. Obsolete MDL studies are archived with the QA files. Electronic MDL data is found in the annual "MDL" folder on the QA server (LINUXMA1).

4.0 DEFINITION

4.1 ALIQUOT - a measured portion of a sample, or solution, taken for sample preparation and/or analysis.

4.2 BATCH – A group of samples which behave similarly with respect to the sampling or the testing procedures being employed and which are processed as a unit. For QC purposes, if the number of samples in a group is greater than 10, then each group of 10 samples or less will all be handled as a separate batch.

4.3 CONTAMINATION - a component of a sample or an extract that is not representative of the environmental source of the sample. Contamination may stem from other samples, sampling equipment, while in transit, from laboratory reagents, laboratory environment, or analytical instruments.

4.4 EXTERNAL CHECK STANDARD - The external check standard that is used to verify the accuracy of the calibration standards. An external check must be run with each analytical batch. The laboratory should initially assess laboratory performance of a check standard using the control limits generated by the external check supplier. Refer to the quality control section for each SOP. If the external check is outside of the control limits for a given parameter, all samples must be reanalyzed for that parameter after the problem has been resolved.

4.5 FIELD SAMPLE - a portion of material to be analyzed that is contained in single or multiple containers and identified by a unique sample number.

4.6 HOLDING TIME – the elapsed time expressed in days from the date of sampling until the date of its analysis.

4.7 INTERFERENTS – substances which affect the analysis for the element of interest.

4.8 MATRIX - the predominant material of which the sample to be analyzed is composed. For the purpose of this SOP, a sample matrix is either water or soil/sediment. Matrix is not synonymous with phase (liquid or solid).

4.9 MATRIX DUPLICATE - a second aliquot of the original sample prepared and analyzed in order to determine the precision of the method.

4.10 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.

4.11 RELATIVE PERCENT DIFFERENCE (RPD) - As used in this SOP to compare two values, the relative percent difference is based on the mean of the two values, and is reported as an absolute value, i.e., always expressed as a positive number or zero.

4.12 REAGENT WATER - water in which an interferant is not observed at or above the minimum quantitation limit of the parameters of interest. Accutest uses deionized water (municipal water, which passes through Accutest's DI treatment system).

5.0 HEALTH AND SAFETY

5.1 The analyst should follow normal safety procedures as outlined in the Accutest Laboratories Chemical Hygiene Plan, which includes the use of lab coat and safety glasses. In addition, all acids are corrosive and should be handled with care. Flush spills with plenty of water. If acids contact any part of the body, flush with water and contact the supervisor.

6.0 COLLECTION, PRESERVATION & HOLDING TIMES

6.1 The sample must be stored at 4 C.

6.2 All samples must be analyzed within 14 days from sampling date.

7.0 APPARATUS

7.1 pH meter with glass electrode capable of reading 0.05 pH unit.
7.2 Titration vessel, 100 ml or 200 ml beaker.
7.3 Magnetic stirrer; stirbars.
7.4 Pipettes, class A.
7.5 Volumetric flasks, class A.
7.6 Burets, 50 ml and 10 ml micro.

8.0 STANDARDS AND REAGENTS

NOTE: All chemicals listed below are reagent grade unless otherwise specified. Distilled, deionized water should be used whenever water is required. All applicable standard/reagent preparation information, including vendor, lot number, date of preparation, calculations, and initials must be entered in the appropriate standard/reagent preparation logbook. Vendors typically used by Accutest include Fisher Scientific, VWR, Accustandard, Absolute Standards, Supelco, Chemservices, Ultra, and ERA. Additional vendors may be utilized as necessary.

8.1 Sodium Carbonate solution, approximately 0.05 N:Place 2.5 + or - 0.2 g (weigh to nearest 0.1 mg) of sodium carbonate (Na_2CO_3, dried at 250^{o} C for 4 hours and cooled in a desiccator) into a 1 liter flask and dilute to the mark. Do not store for more than 1 week.

8.2 Standard Acid, 0.1 N:

8.2.1 Sulfuric acid: Add 3.0 ml of concentrated H_2SO_4 to 500 ml of DI water in a 1 liter volumetric flask. Dilute to 1 liter with water.

or

8.2.2 Hydrochloric Acid: Add 8.3 ml of concentrated HCl to 500 ml DI water in a 1 liter volumetric Flask. Dilute to 1 liter with water.

8.3 Standard Acid 0.02 N: Dilute 200.0 ml of 0.1 N Standard Acid (use 8.2.1 or 8.2.2) to 1 liter with distilled water.

8.4 Sodium Phosphate, dibasic: 0.10 M Weigh 26.81 g of sodium phosphate dibasic heptahydrate ($Na_2HPO_4 \cdot 7H_2O$). Dissolve in approximately 600 ml of DI water in a 1000 ml volumetric flask. Dilute to 1

liter. Alkalinity value is 5000 mg/l as $CaCO_3$. Add 5 ml of this solution to 100 ml of DI water for blank spike and also 5 ml to 100 ml of sample for matrix spike. This solution must be discarded after 6 months.

8.5 All applicable standard/reagent preparation information, including vendor, lot number, date of preparation, expiration date, calculations, and initials must be entered in the appropriate standard/reagent preparation logbook. Vendors typically used by Accutest include Fisher Scientific, VWR, Accustandard, Supelco, Chemservices, Ultra, and ERA. Additional vendors may be utilized as necessary.

9.0 INTERFERENCES

9.1 Samples that contain oil, soaps or suspended solids may coat the electrode and cause a sluggish electrode response. Allow additional time between measurements to reach equilibrium at the electrode.

10.0 PROCEDURE

Below is a step by step procedure for the analysis of samples for ALK. The automated spreadsheet is used for Documentation, calculations of Standardization, and the analysis. This application can be found on the server.

10.1 **Standardizing acid solutions: 0.10 N**

10.1.1 Calibrate the pH meter according to manufacture's instructions, and document the calibration in the pH meter log book.

10.1.2 Rinse the 50 ml buret with approximately 20 ml of standard acid, then fill it.

10.1.3 Add 40.0 ml of 0.05N Sodium Carbonate Solution to approximately 60 ml of distilled water in a 250 ml beaker.

10.1.4 Add a stir bar and place on a magnetic stirrer. Mix gently.

10.1.5 Place pH probe in the sodium carbonate solution and titrate to a pH of approximately 5. Record the pH after every 0.50 to 2 ml acid addition. Remove electrode and rinse with DI water into the beaker.

10.1.6 Cover the beaker with a watch glass and boil the solution for 3- 5 minutes. Allow to cool. Rinse watch glass with DI water into beaker.

10.1.7 Continue the titration to the pH inflection point and record the pH after every 0.050 ml addition . Continue the titration to pH 3.0.

10.1.8 Plot the pH of the sodium carbonate vs. ml of acid added around the inflection point, starting a few mls prior to boiling the solution, and continuing to pH 3.

10.1.9 Calculate the normality of the acid as follows;

$$\text{Normality N} = \frac{A \times B}{53.00 \times C}$$

Where: A = g Na_2CO_3 weighed
B = ml Na_2CO_3 solution
C = ml Acid used at inflection point.

10.2 **Standardizing Acid solution, 0.02 N**: Repeat steps 10.1.1 through 10.1.9 using 15 ml of Sodium Carbonate Solution and 0.020N standard acid.

10.3 **Sample analysis**

An automated Alkalinity spreadsheet is located on MAFILE!\APPS server. Use this application to record all measurments.

10.3.1 Samples above 20 mg/l and less than 1000 mg/l

10.3.2 Place 100 ml of the sample in the titration vessel (beaker or flask), with minimal agitation. Place a stir bar in the titration vessel.

10.3.3 While stirring gently but thoroughly, measure the pH of the sample. Record the pH on the analysis log.

10.3.4 Slowly add 0.02 N standard acid allowing the pH meter to equilibrium between additions.

10.3.5 Titrate the sample to pH 4.5. Record the volume of titration on the analysis log.

10.4 **Samples below 20 mg/l**

10.4.1 Titrate the sample as described in 10.3.1 through 10.3.4, using a 10 ml microburette and 0.02 N titrant. Stop titration at a pH in range of 4.3-4.7. Record the volume of titration and exact pH.

10.4.2 Very carefully add titrant to lower the pH 0.3 pH units and again record the volume.
Note: Samples above 1000 mg/l follow steps 10.3.1 to 10.3.5 using 0.10N titrant

10.5 **Calculations**.

10.5.1 Samples above 20 mg/l:

Alkalinity , mg/l $CaCO_3$ = $\frac{A \times N \times 50000}{\text{ml sample}}$

Where: A = ml of standard acid
N = Normality of standard acid

10.5.2 Samples below 20 mg/l

Alkalinity, mg.l $CaCO_3$ = $\frac{(2A-C) \times N \times 50000}{\text{ml sample}}$

Where: A = ml titrant to first pH
C = total ml titrant to reach pH 0.3 units lower
N = Normality of Acid.

11.0 QUALITY ASSURANCE

11.1 Below is a summary of the quality control requirements for this method. Make sure to check with laboratory supervisor or manager for any additional client specific quality control requirements.

11.2 **Method Blank**. The laboratory must analyze a method blank with each set of samples. A minimum of one method blank is required for every 10 samples. The method blank must contain the analyte at less than the reporting limit (1/2 the RL for some clients). If the method blank contains an analyte level over that limit the problem must be identified and corrected prior to sample analysis.

11.3 **Matrix Duplicate.** The laboratory must prepare a duplicate sample for a minimum of 1 in 10 samples. The relative percent difference (RPD) between the duplicate sample and the original should be assessed. The Duplicate RPD should be calculated as shown below

$$\frac{\text{(Original Sample Result – Duplicate Result) x 100}}{\text{(Original Sample Result + Duplicate Result) x 0.5}} = \%\ \text{RPD}$$

The Duplicate RPD should be assessed using in house limits. Until these limits can be generated, then the default limit of 20 percent RPD should be applied. If a duplicate RPD is out of control, then the results should be flagged with the appropriate footnote. If the sample and the duplicate are less than 5 times the reporting limits and are within a range of ± the reporting limit, then the duplicate is considered to be in control.

11.4 **Matrix spike.** The laboratory must add a known amount of each analyte to a minimum of 1 in 10 samples. The spike recovery should be assessed using in house limits. Until these limits can be generated, default limits of 75-125 % recovery should be applied. If a matrix recovery is out of control, then the recovery should be flagged with the appropriate footnote. If a matrix spike amount is less than one fourth of the sample amount, then the sample can be assessed against the control limits and should be footnoted to that effect.

$$\frac{\text{(Matrix Spike Result – Original Result) x 100}}{\text{Amount of Spike}}$$

11.5 **Spike Blank.** The laboratory must analyze a spike blank with each set of samples. A minimum of one spike blank is required for every 10 samples. The net recovery should be within 20 percent of the true value. If the spike blank is outside of this range, the problem must be identified and corrected before sample analysis can proceed.

11.6 **External Standard.** An external standard is analyzed with each analytical batch. The net recovery should be within 10% of the true value (if the external is prepared in house) or within the manufacturer's acceptance criteria if purchased from an outside vendor. If the external is outside this range, the problem must be identified and corrected before sample analysis can proceed.

11.7 A Precision and accuracy (P&A) study is performed as an initial determination of capability, on an annual basis (continued demonstration of capability – a successful PT result may be used in place of a P&A for continued DOC), and if any significant changes have been made to the instrument. In general, 4 replicates or blank spikes are analyzed using the same procedures and conditions for sample analysis. The mean percent recovery is compared to the spike blank control limits of 20%. The standard deviation (of the percent recovery of the 4 spike blanks) is compared to the control limit of ±20. If percent recovery or standard deviation criteria are not met, corrective action must be taken to bring the system back into control.

11.8 Quality Control data is generated (control charts) and reviewed on an annual basis by Quality Assurance (blank spike/ matrix spike recoveries and matrix duplicate RPDs).

12.0 DOCUMENTATION

12.1 The Standard preparation log application must be completed for all standard preparations. All information requested must be completed.

12.2 The Accutest lot number must be cross-referenced on the standard vial/container.

12.3 Any comments or observations concerning the sample that may influence the analytical procedure.

12.4 Any corrections to laboratory data must be done using a single line through the error. The initials of the person and date of corrections must appear next to the correction.

12.5 All laboratory logs must be reviewed and initialed or signed by the lab manager.

13.0 DATA REVIEW

13.1 The analyst conducts the primary review of all data. This review begins with a check of all method quality control and progresses through sample quality control concluding with a check to assure that the client's requirements have been executed.

13.2 A secondary review is performed by department managers, and it includes review of the data produced by their department. Manual calculations, QC criteria, and a comparison of the data package to client specified requirements are checked. The department manger may reject data, initiate reanalysis, and take additional corrective action, or process data.

13.3 The laboratory director performs a full tertiary review of the data package following its assembly. This review includes an evaluation of QC data against acceptance criteria and a check of the data package contents to assure that all analytical requirements and specifications were executed.

13.4 Spot-check reviews are performed by the Quality Assurance Officer focusing on all elements of the deliverable including the client's specifications and requirements, analytical quality control, sample custody documentation and sample identification.

14.0 DATA REPORTING

14.1 A results page including positive results and/or RLs, units, methodology, analysis dates, and data qualifiers are reported. Additional quality control data including matrix duplicate RPDs, matrix spike recovery, blank spike and method blank results may be reported upon request of the client.

14.2 Data may be submitted to the client in a specified electronic format (EDD).

14.3 Once the data is approved by the laboratory manager, it may be accessed by clients via LabLink™.

14.4 Procedures for handling non-conforming data.

14.4.1 If quality control data does not meet criteria the non-conformance must be discussed in a case narrative and footnoted on the applicable quality control report summary.

14.4.2 If preservation or holding time criteria is not met and the samples are analyzed the result page must be footnoted with this information, and the non-conformance must be discussed in a case narrative or other suitable communication (telephone conversation log or email). Client notification documentation should be included with the data (telephone conversation log, fax, or email).

15.0 POLLUTION PREVENTION & WASTE MANAGEMENT

15.1 Pollution Prevention. Users of this method must perform all procedural steps that control the creation and/or escape of wastes of hazardous materials to the environment. The amounts of standards, reagents, and solvents must be limited to the amounts specified in this SOP. All safety practices designed to limit the escape of vapors, liquids, or solids to the environment must be followed. All method users must be familiar with the waste management practices described in section 15.2.

15.2 Waste Management. Individuals performing this method must follow established waste management procedures as described in the Sample and Waste Disposal SOP. This document describes the proper disposal of all waste materials generated during the testing of samples as follows:

15.2.1 Non-Hazardous aqueous wastes
15.2.2 Hazardous aqueous wastes
15.2.3 Chlorinated organic solvents
15.2.4 Non-chlorinated organic solvents
15.2.5 Hazardous solid wastes
15.2.6 Non-hazardous solid wastes
15.2.7 Microbiological waste

16.0 METHOD PERFORMANCE

16.1 Method performance is evaluated by the annual QC limits (control charts) generated by QA, and the annual MDL study results. Refer to section 3.0 for MDLs, and section 11.8 for QC limits.

17.0 ADDITIONAL REFERENCES

17.1 No additional references are required for this method.

TITLE: TOTAL ORGANIC CARBON IN AQUEOUS SAMPLES

TEST METHOD REFERENCES: - SW846 9060A Modified,
-5310 B, Standard Methods for the Examination of Water and Wastewater 21st Edition, 2005

REVISED SECTIONS: 4.7; 10.1

1.0 SCOPE AND APPLICATION

1.1 This method can be used to determine total organic carbon or dissolved organic carbon in any aqueous matrix. The total organic carbon is actually being determined as non-purgable organic carbon. Volatile compounds are lost during the sparging process to remove inorganic carbon.

1.2 The product for total organic carbon is TOC and the product for dissolved organic carbon is DOC. TOC work groups require prep and analytical work groups. DOC product requires analytical work group only.

1.3 Samples containing high levels of particulates may need to be run using the boat sampler normally used for soil samples. When analyzing these samples using the boat sampler, all steps of the soil SOP, including acidification and heating to remove inorganic carbonates, must be followed. (Acidification and sparging may also be used to remove inorganic carbonates.)

1.4 The modification to method 9060A is that water samples are not homogenzied in a blender.

2.0 SUMMARY

2.1 Total organic carbon is determined by combusting an acidified sample and quantitating the carbon dioxide released using infrared analysis. The quantitation is done by comparison to a linear calibration curve. Dissolved organic carbon is determined following the same method, but the sample is filtered through a 0.45 filter before analysis.

3.0 REPORTING LIMIT AND METHOD DETECTION LIMIT

3.1 The reporting limit (RL) is based on the lowest calibration standard. RL's may vary depending on matrix difficulties, sample volumes or weights, and percent moisture. Detected concentrations below this concentration cannot be reported without qualification. The normal reporting limit for TOC and DOC in aqueous samples is 1.0 mg/l.

3.2 The Method Detection Limit (MDL) represents the lowest reportable concentration of an individual analyte that meets the method qualitative identification criteria.

3.3 Method Detection limits (MDLs) are experimentally determined using the procedures described in 40 CFR, Part 136, Appendix B. Actual reported MDLs incorporate the sample volume analyzed and sample dilutions if needed, which may cause MDL variations from sample to sample.

3.4 In general, MDLs are determined through the analysis of at least 7 replicate blank spikes (using the same procedures for sample analysis). The MDL is calculated by multiplying the standard deviation of the replicate concentrations by the appropriate Student's t value (3.143 for 7 replicates). If more than 7 replicates are analyzed refer to 40 CFR, Part 136, Appendix B for the appropriate student's t value. MDLs are determined initially (prior to analysis), on an annual basis, and after major maintenance to equipment. MDL data is archived with Quality Assurance. Refer to the most recent study for current MDLs. For additional detail regarding MDL studies, refer to the MDL SOP MQA245.

3.5 Current MDL studies are filed with Quality Assurance. Obsolete MDL studies are archived with the QA files. Electronic MDL data is found in the annual "MDL" folder on the QA server (LINUXMA1).

4.0 DEFINITIONS

4.1 ALIQUOT - a measured portion of a sample, or solution, taken for sample preparation and/or analysis.

4.2 BATCH: A group of samples which behave similarly with respect to the sampling or the testing procedures being employed and which are processed as a unit. For QC purposes, if the number of samples in a group is greater than 20, then each group of 20 samples or less will all be handled as a separate batch.

4.3 CALIBRATION CHECK STANDARD (CCV). The calibration check standard is a mid-range calibration standard. It is recommended that the calibration check standard be run at a frequency of approximately 10 percent. (For some methods this is mandatory and for some it is a recommendation only. Refer to individual method SOP's) For most methods, the mid-level calibration check standard criteria is $\pm$ 10 percent of the true value. The exception to this rule is if the recovery on the calibration check standard is high and the samples to be reported are less than the detection limit.

4.4 EXTERNAL CHECK STANDARD (ICV). The external check standard is a standard from a separate source than the calibration curve that is used to verify the accuracy of the calibration standards (also called an initial calibration verification – ICV). An external check must be run a minimum of once per quarter for all analyses where a check is commercially available. The laboratory should initially assess laboratory performance of a check standard using the control limits generated by the external check supplier. In house limits should also be generated once sufficient external check standard data is available to generate limits (usually a minimum of 20 to 30

analyses). If the external check is outside of the control limits for a given parameter, all samples must be reanalyzed for that parameter after the problem has been resolved.

4.5 SPIKE BLANK OR LAB CONTROL SAMPLE. Digest and analyze a laboratory control sample or spike blank with each set of samples. A minimum of one lab control sample or spike blank is required for every 10 or 20 samples (refer to quality assurance section). Assess laboratory performance against the control limits specified in the SOP. In house limits should also be generated once sufficient external check standard data is available to generate limits (usually a minimum of 20 to 30 analyses). If the lab control is outside of the control limits for a parameter, all samples must be reanalyzed for that parameter. The exception is if the lab control recovery is high and the results of the samples to be reported are less than the reporting limit. In that case, the sample results can be reported with no flag. Note: If control limits are not specified in the SOP, then default limits of 80 to 120 percent should be used.

4.6 MATRIX: The component or substrate (e.g., water, soil) which contains the analyte of interest.

4.7 MATRIX DUPLICATE: A duplicate sample is analyzed at a minimum of 1 in 10 or 20 samples (refer to quality assurance section). The relative percent difference (RPD) between the duplicate and the sample should be assessed. The duplicate RPD is calculated as shown below. Assess laboratory performance against the control limits that are specified in the SOP. In house limits are generated once sufficient duplicate data is available to generate limits (usually a minimum of 20 to 30 analyses). If a duplicate is out of control, flag the results with the appropriate footnote. If the sample and the duplicate are less than 5 times the reporting limits and are within a range of ± the reporting limit, then the duplicate is considered to be in control. Note: If control limits are not specified in the SOP, use default limits of ± 20% RPD. For method SM5310, RPD must be within 10%.

$$\frac{(|\text{Sample Result} - \text{Duplicate Result}|) \times 100}{(\text{Sample Result} + \text{Duplicate Result})/2} = \text{Duplicate RPD}$$

4.8 MATRIX SPIKE: The laboratory must add a known amount of each analyte to a minimum of 1 in 10 or 20 samples (refer to quality assurance section). The matrix spike recovery is calculated as shown below. Assess laboratory performance against the control limits that are specified in the SOP. In house limits are generated once sufficient matrix spike data is available to generate limits (usually a minimum of 20 to 30 analyses). If a matrix spike is out of control, then the results should be flagged with the appropriate footnote. If the matrix spike amount is less than one fourth of the sample amount, then the sample cannot be assessed against the control limits and should be footnoted to that effect. Note: If control limits are not specified in the SOP, then default limits of 75 to 125 percent should be used.

$$\frac{(\text{Spiked Sample Result} - \text{Sample Result})}{(\text{Amount Spiked})} \times 100 = \text{Matrix Spike Recovery}$$

4.9 MATRIX SPIKE DUPLICATES: Intra-laboratory split samples spiked with identical concentrations of target analyte(s). The spiking occurs prior to sample preparation and analysis. They are used to document the precision and bias of a method in a given sample matrix.

4.10 METHOD BLANK. The laboratory must digest and analyze a method blank with each set of samples. A minimum of one method blank is required for every 10 or 20 samples (refer to quality assurance section). The method blank must contain the parameter of interest at levels of less than the reporting limit for that parameter. If the method blank contains levels over the reporting limits, the samples must be reanalyzed. The exception to this rule is when the samples to be reported contain greater than 10 times the method blank level. In addition, if all the samples are less than a client required limit and the method blank is also less than that limit, then the results can be reported as less than that limit.

4.11 METHOD DETECTION LIMITS (MDLS). MDLs should be established for all appropriate methods, using a solution spiked at approximately 3 times the estimated detection limit. To determine the MDL values, take seven replicate aliquots of the spiked sample and process through the entire analytical method. The MDL is calculated by multiplying the standard deviation of three replicate analyses by 3.14, which is the student's t value for a 99% confidence level. MDLs should be determined approximately once per year for frequently analyzed parameters.

4.12 REAGENT BLANK: The reagent blank is a blank that has the same matrix as the samples, i.e., all added reagents, but did not go through sample preparation procedures. The reagent blank is an indicator for contamination introduced during the analytical procedure. The concentration of the analyte of interest in the reagent blank must be less than the reporting limit for that analyte. If the reagent blank contains levels over the reporting limits, the samples must be reanalyzed. The exception to this rule is when the samples to be reported contain greater than 10 times the reagent blank level. In addition, if all the samples are less than a client required limit and the reagent blank is also less than that limit, then the results can be reported as less than that limit.

4.13 REAGENT GRADE: Analytical reagent (AR) grade, ACS reagent grade, and reagent grade are synonymous terms for reagents which conform to the current specifications of the Committee on Analytical Reagents of the American Chemical Society.

4.14 REAGENT WATER: Water that has been generated by any method which would achieve the performance specifications for ASTM Type II water. For organic analyses, see the definition of organic-free reagent water. Accutest uses deionized water (municipal water which passes through Accutest's DI treatment system).

4.15 REFERENCE MATERIAL: A material containing known quantities of target analytes in solution or in a homogeneous matrix. It is used to document the bias of the analyti-cal process.

4.16 STANDARD CURVE: A plot of concentrations of known analyte standards versus the instrument response to the analyte. Calibration standards are prepared by successively diluting a standard solution to produce working standards which cover the working range of the instrument. Standards should be prepared at the frequency specified in the appropriate section. The calibration standards should be prepared using the same type of acid or solvent and at the same concentration as will result in the samples following sample preparation. This is applicable to organic and inorganic chemical analyses.

4.17 TRIP BLANK: A sample of analyte-free media taken from the laboratory to the sampling site and returned to the laboratory unopened. A trip blank is used to document contamination attributable to shipping and field handling procedures. This type of blank is useful in documenting contamination of volatile organics samples.

5.0 HEALTH & SAFETY

5.1 All safety practices must be followed as outlined in the Accutest Laboratories Employee Safety Handbook and Chemical Hygiene Plan. Safety glasses, gloves, and lab coats must be worn. All samples, solutions, and extracts must be treated as unknown and potentially hazardous.

5.2 The toxicity or carcinogenicity of each reagent used in this method has not been precisely determined; however, each chemical should be treated as a potential health hazard. Exposure to these reagents should be reduced to the lowest possible level.

6.0 COLLECTION, PRESERVATION, AND HOLDING TIME

6.1 Collection and Preservation

6.1.1 Containers: Samples should be collected in 40 ML VOA, zero headspace glass containers.

6.1.2 Preservation: Samples should be preserved with H_3PO_4 , H_2SO_4 or HCl to a pH of < 2. Samples should be stored at $4°C \pm 2°C$.

Note: Total carbon preservation is not directly addressed in the methods. Normally an unpreserved sample is used . Acid preserved sample is used for Total Organic Carbon.

6.2 Holding Time: Samples should be analyzed within 28 days from the date of collection.

7.0 APPARATUS

7.1 The following items are needed for the analysis of samples following the method outlined below:

7.2 Shimadzu TOC L analyzer with an auto-sampler.

7.2.1 Every day, the humidifier should be checked to ensure that the water level is within the two white lines on the side of the humidifier.

7.2.2 Whenever calibration check recoveries are low or blank results are high, the flow and the condition of the catalyst should be checked. Refer to the instrument manual for additional information. Never change the catalyst without first checking with the area supervisor or manager.

7.3 Analytical balance, capable or weighing to 0.1 mg. The calibration of the analytical balance should be verified each day before use.

7.4 Volumetric glassware, class A. For standards and reagent preparation.

7.5 Filter paper, 0.45 um pore size. (For DOC only)

7.6 40 ml VOC bottles with septum.

8.0 REAGENTS

8.1 All chemicals listed below are reagent grade unless otherwise specified. Deionized water should be used whenever water is required. Make sure to properly label all reagents and record the reagent preparation in the reagent log book.

8.2 Potassium hydrogen phthalate (KHP), Calibration stock solution, 1000 mg carbon/L. Dissolve 2.125 g of potassium hydrogen phthalate (primary standard grade, dried to a constant weight, approximately 1 hour at 105^0 C) in approximately 800 ml of DI water. Add concentrated HCl or H_3PO_4 to bring the pH to less than 2 and dilute to a final volume of 1000 ml with DI water. Prepare this solution quarterly.

8.3 Carbonate-Bicarbonate Stock solution, 1000 mg/l. Weigh 0.3500 g of sodium bicarbonate and 0.4418 g of sodium carbonate into a 100 ml volumetric flask. Dissolve in DI water and dilute to a final volume of 100 ml. Prepare this solution quarterly.

8.4 Sparger Check Solution (Carbonate-Bicarbonate Standard solution, 50 mg/l). Dilute 5.00 ml of the 1000 mg/l carbonate-bicarbonate stock solution to 100 ml with DI water.

Note: Do not use Sparger for TCAR determinations.

8.5 Potassium hydrogen phthalate (KHP) calibration stock solutions. All standards should be made up in volumetric flasks using volumetric pipets.

Note: The concentrations shown below are suggested levels. Alternate calibrations may be constructed in the TOC method file and used.

8.5.1 40 PPM Calibration. Std., Dilute 4.0 ml of solution 8.2, and 5 drops of 1:1 H_3PO_4 or HCl to 100 ml with DI water.

8.5.2 30 PPM Calibration. Std., Dilute 3.0 ml of solution 8.2, and 5 drops of 1:1 H_3PO_4 or HCl 100 ml with DI water.

8.5.3 10 PPM Calibration. Std., Dilute 1.0 ml of solution 8.2, and 5 drops of 1:1 H_3PO_4 or HCl to 100 ml with DI water.

8.5.4 1.0 PPM Calibration. Std., Dilute 10 ml of 10 PPM solution 8.5.3, and 5 drops of 1:1 H_3PO_4 or HCl 100 ml with DI water.

Place the reagent blank (DI water plus 5 drops of 1:1 HCl or H_3PO_4), and calibration stock standards 8.5.1, 8.5.2, 8.5.3 8.5.4 in position 1, 2, 3, 4 respectively, and the auto-sampler will dilute and make the following calibration standards: 0.0,1.0, 5.0, 10.0, 20.0, 30.0, 40.0 ppm.

Note: For pH adjustment of calibration stock standards and the reagent blanks, it is recommended to use the same type of acid as was used for sample preservation.

Note: Do not add acid to calibration standards for TCAR determinations.

8.6 Potassium hydrogen phthalate (KHP), Initial & Continuing Calibration Verification Standard, 1000 mg carbon/L. Dissolve 2.125 g of potassium hydrogen phthalate (primary standard grade, dried to a constant weight, approximately 1 hour at 105º C) in approximately 800 ml of DI water. Add concentrated HCl or H3PO4 to bring the pH to less than 2 and dilute to a final volume of 1000 ml with DI water. Prepare this solution quarterly and from a **different lot or manufacturer than the KPH calibration standards used in 8.5. Also this standard is available already made from Absolute Standards Inc.**.

8.6.1 Initial Calibration Verification Solution, 30 Mg C/L. Dilute 3 ml of solution 8.6 into 100 ml volumetric flask containing 80 ml of DI water. Add concentrated HCl to bring the pH to less than 2. Mix well and bring to final volume of 100 ml.

8.6.2 Continuing Calibration Verification (CCV) **SW846 9060A**, Dilute 2.0 ml of 8.6 solution and 5 drops of 1:1 HCl or H_3PO_4 to 100 ml with DI water.

8.6.3 Continuing Calibration Verification (CCV) **SM 5310B,** Dilute 2 ml of 8.2 solution and 5 drops of 1:1 HCl or H_3PO_4 to 100 ml with DI water

8.7 2 Molar HCL solutions. Add 36.5 ml of concentrated HCl in 300 ml of DI water, dilute to 500 ml with DI water. Mix well.

Note: Do not use for TCAR determinations.

8.8 1:1 Phosphoric Acid: Add 50.0 ml of Conc. H_3PO_4 to 50.0 ml of DI water. Mix and Cool.

8.9 1:1 Hydrochloric Acid: Add 50.0 ml of Conc. HCL to 50.0 ml of DI water. Mix and Cool.

9 INTERFERENCES

9.1 High results may be obtained if the inorganic carbon is not completely removed from the sample before analysis. The sample must be acidified to a pH of less than 2 and sparged for at least 1 minute to remove the inorganic carbon.

9.2 Large particulates in the sample may not be pulled into the needle during the sample injection and may result in a low bias.

9.3 Filtration can result in loss or gain of DOC, depending on the physical properties of the carbons containing compounds and the adsorption of carbonaceous material on the filter or its desorption from it.

10 PROCEDURE

10.1 Below is the procedure to be followed for the analysis of aqueous samples for total organic carbon using the Shimadzu TOC L analyzer. All standards and samples must be analyzed using a minimum of duplicate injections.

Note: If **SW846 9060** method is being used, all standards and samples must be analyzed in quadruple injections. Results of the average and the range must also be reported. The range will be reported as a footnote to the data report.

10.2 Turn on the Oxygen. The oxygen pressure at the tank regulator must be at 200 KPA to maintain pressure at the instrument. Check to make sure that the humidifier contains sufficient DI water. It should be filled to HI line on the side of the humidifier (Humidifier is located inside the water analyzer at the front right side of the instrument).

10.3 Turn on the instrument using the main power switch on the right backside towards the top of the TOC instrument and the left side of the autosampler. After a few seconds the TOC instrument will shut down which means the instrument ready condition is not established. Turn on the power switch on the left of the instrument front door. The lit power switch will change from orange to green when ready.

10.4 On the desktop, double click on **TOC-Control L** icon and double click on the sample table editor icon. User name and password window will appear. Type **Wetchem** in the user name space and **TOC2012** for the password. Please note that this software is case sensitive.

10.5 Go to **H/W Setting** on the sample table and double click on it. TOC AQ and TOC SO will appear below the **H/W Setting.** If AQ samples are to be analyzed choose TOC AQ. Using a right click on the mouse, choose the TOC AQ icon and a menu will appear that follows:

Line1: Connect
Line2: Background Monitor Setting
Line3: Shutdown
Line4: Maintenance

Line5: Instrument Setting

10.5.1 Click on Connect, a **Sequence** window will pop up. It indicates the sequence which is currently in progress. Also the open port in this window should indicate at 100% otherwise the Instrument and PC isn't communicating.

Note: If the open port is not at 100%, shutdown the software and instrument then boot up the PC. Restart the process 10.3 through 10.5 again.

10.5.2 Once the communication is established, click on the **Instrument Setting** in the 10.5 menu and **Instrument Properties** window will appear. Go to the **TOC** tab in **Instrument Properties** and click on the 680 Deg C option button. This will heat up the TC furnace for TOC AQ.

10.5.3 While the furnace is heating up, go to the 10.5 menu and click on **Background Monitor Setting.** A window will come into view that is called the **peak view window**. Set the view setting at 50x and monitor the baseline. Let the instrument warm up for 30 to 45 minutes. Once the instrument is ready the baseline will be flat or in a slight seesaw motion.

10.6 Verify the carrier gas flow rate displayed on the TOC tab in the Background Monitor window reads 150 ml/min. Never allow the carrier gas supply pressure to exceed 250 KPA.

10.7 Check the levels in the Rinse, Diluent and HCL solution bottles. Make sure the transfer lines are well below the liquid levels. If any of these are not, refill as necessary.

10.8 On the tool bar, go to the file tab, then go to open, and last go to sample table. Choose TOC AQ up to 40ppm.tlx file (TC AQ up to 40ppm.tlx for TCAR). Go to file in tool bar and save the file as a new file name. The new name scheme must be as follows: TOC, month, day, year, matrix, and the number of the run for that day. For example: TOC092812W1, which would be the TOC run on September 28, 2012 water run number 1 for that day. TOC092812W2 would be the second run for that day.

10.9 The template contains all the information regarding the calibration curve, sparge time, etc.

10.9.1 First line of the template contains the calibration curve.

10.9.2 Enter the sample sequence starting from second line as follows:

Line 2 CCV
Line 3 CCB
Line 4 Spargerchkconf
Line 5 ICV

Continue and enter the sample sequence including the quality control, etc.

10.9.3 Pour out the standards and the samples (section 10.12). Label each bottle and place on auto-sampler.

10.9.4 Remove any unused rows/lines by high lighting and cutting. Again, save the file under the proper name before starting the analysis.

10.9.5 On the tool bar, go to the **Instrument** tab and then go to **Maintenance** and finally choose **Replace Flow Line Content.** Select start in the flow content window. This will fill in the lines with the diluents, DI water and HCL solution. Once this is finished close the window. .

10.9.6 The vial ID's in the PC must be matched to the sample positions in the auto sampler. To do this, select the **View Vial Settings** icon in the left hand corner of the sample table window. The **View Vial dialog box** will open showing the standards and samples lined up in a row with the **Vial** column used for vial ID's. Check each position entered against the actual position on the auto sampler and verify the sequence. Once they match select OK.

10.9.7 Select the **Start** icon on the tool bar. Selecting **Instrument – Start Measurement** after connection is established can also start analysis. The **Measurement Start** window will pop up with three options. Select the appropriate process that will take place after analysis is complete.

10.9.8 After the calibration is completed, the instrument will check the coefficient of correlation to be 0.995 or greater. If these criteria are not established, the instrument will stop. A corrective action must be taken to correct any problems, and the calibration curve must be reanalyzed prior to sample analysis.

10.10 After the calibration is completed, Monitor CCV, CCB, Sparger chk, and ICV standard. ICV standard must agree within 10 percent of the true value. If it is not within this range, determine the source of the problem before proceeding.

10.11 After every 10 samples and at the end of the run, a continuing calibration check sample and a continuing calibration blank should be analyzed. The continuing calibration check should be a standard near the mid-range of the curve. It is recommended that a check standard at 20 mgC/l be used as the continuing calibration check standard. The continuing calibration check should agree within 10 percent of the true value. The result for the continuing calibration blank must be less than the reporting level. If either the CCV or the CCB do not meet criteria, then all samples bracketed by this QC must be reanalyzed.

10.12 Sample log and handling

10.12.1 All samples and check standards must be analyzed with duplicate injections (quadruple for **SW 846 9060**; see 10.1).

10.12.2 Shake the samples well to make sure they are completely homogenized.

10.12.2.1 If a sample contains a high level of particulates, check with the lab supervisor before proceeding. The sample may need to be analyzed using the soil module.

10.12.3 Using SM5310B with each batch of 20 samples or less, a matrix spike, matrix duplicate method blank, and spike blank must be analyzed. If SW846 9060A is used, then with each batch of 10 samples or less, a matrix spike, matrix spike duplicate, method blank and spike blank must be analyzed. All of these quality control points must be analyzed using duplicate injections (quadruple injections for SW846 9060A).

10.12.3.1 Prepare the matrix spike and matrix spike duplicate by adding 0.500 ml of 1000 mgC/l standard solution to 50 ml of acidified sample. (use matrix spike and matrix duplicate using SM5310B)

10.12.3.2 The spike blank may be prepared by adding 0.500 ml of 1000 mgC/l standard solution to 50 ml of DI blank. Alternatively, the external check may be used as the spike blank.

10.12.4 Analyze continuing calibration checks and continuing calibration blanks as outlined in 10.11 above.

10.12.4.1 The software may not accept large dilution factors (>100). In that case, enter in the dilution in the column to the right of the sample ID and correct the final result in the LIMS system for the sample dilution. The instrument will dilute samples automatically, although a sample with suspected high concentration should be

diluted prior to analysis to protect the instrument. The instrument will attempt a dilution by reducing the injection volume. This information could be accessed in the "Sample View Window" and on the sample report. If the TOC concentration is still too high, the sample will be diluted with dilution water. The "Auto – dilution factor" is only available in the sample view window and will not be included in the sample report.

10.12.5 Use sample window to view the peaks as the analysis is in process. With the cursor positioned on the autosampler window click on any row to be highlighted, the result for the completed samples will show in the view window.

10.12.6 Make sure the sparger check result is less than reporting detection limit of 1 PPM and preferably less than half the reporting detection limit.

10.12.7 After the analysis is complete, check through the data to make sure that all QC is within criteria, that samples are within the curve, and that good reproducibility is obtained for all injections. If the sample injections have a coefficient of variation (CV) of greater than 2 percent, then verify the repeated analysis with additional replicate injection (Parameters for CV already programmed in the template). If, on the repeated analysis, a high rpd is still obtained, then the sample results should be reported with a flag due to possible sample non-homogeneity.

$$CV = (\text{Std Dev}_{n-1} / \text{mean}) \times 100$$

Note: Measure and record in the logbook the pH of samples after analysis using pH paper strips. The pH of samples must be <2. If the sample is not properly preserved, this information must be communicated to the client.

10.12.8 Review the run for completeness and data and quality control problems. Go to "File" (in the tool bar of the sample table window) select "ASCII export." The "Save As" window will now open. Save the file in the location "C:/TOCL/Data" using the same naming procedure as seen in section 10.8. This file can be accessed through windows explorer and should be transferred to LIMS. Make any necessary corrections in GNAPPR such as entering the spike amounts. Approve in batch in GNAPP and provide the data to the supervisor for additional review. See the area supervisor or manager for further details.

11 QUALITY ASSURANCE

11.1 Below is a summary of the quality control requirements for this method. Make sure to check with the laboratory supervisor or manager for any additional client specific quality control requirements.

11.2 Method Blank. The laboratory must prepare and analyze a method blank with each analysis. When using method ***SW846 9060A***, a minimum of one method blank is required for every 10 samples (20 samples if ***SM 5310B*** is used). The method blank must contain the analyte at less that the reporting limit. If the method blank contains over that limit, the samples must be reanalyzed. The exception to this rule is when the samples to be reported contain greater than 10 times the method blank level. In addition, if all the samples are less than a client required limit and the method blank is also less than that limit, then the results can be reported as less than that limit.

11.3 Spike Blank. The laboratory must prepare and analyze a spike blank with each analysis. When using method ***SW846 9060A***, a minimum of one spike blank is required for every 10 samples (20 samples if ***SM 5310B*** is used). The laboratory should assess laboratory performance of the spike blank against recovery limits of 80 to 120 percent. If the lab control recovery is high and the results of the samples to be reported are less than the reporting limit, then the sample results can be reported with no flag. In all other situations, all samples associated with a spike blank outside of recovery limits must be reanalyzed.

11.4 Matrix Spike. The laboratory must add a known amount of analyte to a minimum of 1 in 10 samples when using method ***SW846 9060A*** (20 samples if ***SM 5310B*** is used).

11.4.1 The spike recovery should be assessed using in house limits. Until these limits can be generated, then default limits of 75 to 125 percent recovery should be applied. If a matrix spike is out of control, then the results should be flagged with the appropriate footnote. If the matrix spike amount is less than one fourth of the sample amount, then the sample cannot be assessed against the control limits and should be footnoted to that effect.

11.4.2 The matrix spike recovery should be calculated as shown below.

$$\frac{\text{(Spiked Sample Result - Sample Result)}}{\text{(Amount Spiked)}} \times 100 = \text{MS Recovery}$$

11.5 Matrix Duplicate/Matrix Spike Duplicate. The laboratory should analyze a Matrix Spike duplicate sample for a minimum of 1 in 10 samples when using method ***SW846 9060A***, and a Matrix duplicate sample for a minimum of 1 in 20 samples if ***SM 5310B*** is used. The relative percent difference (rpd) between the duplicate and the sample should be assessed. Matrix spike duplicates may be used in place of matrix duplicates. The duplicate rpd is calculated as shown below.

11.5.1 The duplicate RPD should be assessed using in house limits. Until these limits can be generated, then default limits of 20 percent RPD should be applied. If a duplicate is out of control, then the results should be flagged with the appropriate footnote. If the sample and

the duplicate are less than 5 times the reporting limits and are within a range of ± the reporting limit, then the duplicate is considered to be in control.

11.5.2 The duplicate RPD should be calculated as shown below.

$$\frac{\text{(Sample Result - Duplicate Result) x 100}}{\text{Result + Duplicate Result) x 0.5}} = \text{\% RPD}$$

11.6 Quality Control Sample (also referred to as Initial Calibration Verification Standard, (ICV). It is recommended that a standard from a separate source than the calibration should be run at the beginning of each run. This ICV should be within 10 percent of the true value. If it is not, the problem must be resolved before any samples can be analyzed.

11.7 Analyze the continuing calibration verification solution after every tenth sample and at the end of the sample run. If the CCV solution is not within 10 percent of the true value, then no samples can be reported in the area bracketed by that CCV. (Note: the exception is if the CCV is biased high and the samples are less than the detection limit. In that case, the samples can be reported with no flag.) The CCV concentration should be at or near the mid-range of the calibration curve.

11.8 Analyze the continuing calibration blank solution after every CCV check (every tenth sample and at the end of the sample run). The continuing calibration blank must be less than the reporting limit. If the CCB solution is not less than the reporting limit, then no samples can be reported in the area bracketed by that CCB. (Note: the exception is if the CCB is biased high and the samples are less than the detection limit. In that case, the samples can be reported with no flag.).

11.9 Minimum Reporting Limit (MRL) check. A blank spike at the concentration of the minimum reporting limit (MRL) must be prepared and analyzed with each sample batch for drinking water samples. The acceptance criteria are 50-150% recovery. If the acceptance criteria is not met then corrective action must be taken to correct the exceedence and all associated samples must be reanalyzed with a satisfactory MRL check.

11.10 A Precision and accuracy (P&A) study is performed as an initial determination of capability, on an annual basis (continued demonstration of capability – a successful PT result may be used in place of a P&A for continued DOC), and if any significant changes have been made to the instrument. In general, 4 replicates or blank spikes are analyzed using the same procedures and conditions for sample analysis. The percent recoveries are compared to either default limits of 90-110% or in-house control limits once established. The standard deviation of the 4 replicate percent recoveries are compared to either ±20 or to in-house limits once established. If percent recovery or standard deviation criteria are not met, corrective action must be taken to bring the system back into control.

11.11 Quality control data are generated at least on an annual basis by QA using an in-house program.

Blank spike and MS/Dup data are pooled for the previous year (or other specified time frame) and the data is processed and evaluated by QA. The annual QC data is filed with QA.

12 DOCUMENTATION

12.1 All sample ID's, dilution, weights and any other information should be recorded. Make sure to record any comments regarding unusual sample appearance or any other problems or observations.

12.2 The instrument Maintenance logbook must be completed when any type of maintenance is performed on the instrument. Each instrument will have a separate log.

12.3 All laboratory logbooks must be routinely reviewed and initialed or signed by the lab manager.

12.4 Any corrections to laboratory data must be done using a single line through the error. The initials of the person and date of correction must appear next to the correction.

13 DATA REVIEW

13.1 Primary Review. All samples should be updated to both prep (GP) and analysis (GN) batches in the LIMS system. The analyst should calculate all matrix spike, duplicate, external, and CCV recoveries and review the results of all blanks.

13.2 All documentation must be completed, including reagent references and spike amounts and spiking solution references.

13.3 A final report should be printed out from the TOC software. Make sure to check that all samples meet replication requirements (< 2% CV) and that the samples are within the range of the calibration curve.

13.4 A data file should be exported to the LIMS system and the spike amounts should be entered into the file at the GNAPP process step.

13.5 A final data package, consisting of the raw data (TOC printout), copy of the TOC analysis runlog and workgroup printout, should be turned into the area supervisor for review.

13.6 Department managers perform a secondary review, and it includes review of the data produced by their department. All manual calculations, QC criteria, and a comparison of the data package to client specified requirements are checked. The department manager may reject data, initiate reanalysis, take additional corrective action, or reprocess data.

13.7 The laboratory director performs a full tertiary review of the data package following its

assembly. This review includes an evaluation of QC data against acceptance criteria and a check of the data package contents to assure that all-analytical requirements and specifications were executed.

13.8 Spot-check reviews are performed by the Quality Assurance Officer focusing on all elements of the deliverable including the client's specifications and requirements, analytical quality control, sample custody documentation and sample identification.

14 DATA REPORTING

14.1 A results page including positive results and/or RLs, units, methodology, preparation and/or analysis dates, and data qualifiers are reported. Additional quality control data including calibration summaries, MS/duplicate percent recoveries and RPDs, blank spike recoveries, and method blank results may be reported upon request of the client. Additionally, raw data including any instrument printouts, laboratory logbooks, etc. may be reported to the client.

14.1 Data may be submitted to the client in a specified electronic format (EDD).

14.2 Data may be submitted to the client electronically (e-hardcopy) in PDF.

14.3 Once the data is approved by the laboratory manager, it may be accessed by clients via LabLink™.

14.2 Procedures for handling non-conforming data.

14.2.1 If quality control data does not meet criteria the non-conformance must be discussed in a case narrative and footnoted on the applicable quality control report summary.

14.2.2 If preservation or holding time criteria is not met and the samples are analyzed the result page must be footnoted with this information, and the non-conformance must be discussed in a case narrative or other suitable communication (telephone conversation log or email). Client notification documentation should be included with the data (telephone conversation log, fax, or email).

15 POLLUTION PREVENTION & WASTE MANAGEMENT.

15.1 Users of this method must perform all procedural steps in a manner that controls the creation and/or escape of wastes or hazardous materials to the environment. The amounts of standards, reagents, and solvents must be limited to the amounts specified in this SOP. All safety practices designed to limit the escape of vapors, liquids or solids to the environment must be followed. All method users must be familiar with the waste management practices described in section 15.2.

15.2 Waste Management. Individuals performing this method must follow established waste management procedures as described in the waste management SOP. This document describes the proper disposal of all waste materials generated during the testing of samples as follows:

15.2.1 Non hazardous aqueous wastes.
15.2.2 Hazardous aqueous wastes
15.2.3 Chlorinated organic solvents
15.2.4 Non-chlorinated organic solvents
15.2.5 Hazardous solid wastes
15.2.6 Non-hazardous solid wastes
15.2.7 Microbiological wastes

16 ADDITIONAL REFERENCES

16.1 Shimadzu TOC L instrument manual.

ACCUTEST LABORATORIES
STANDARD OPERATING PROCEDURE

Lab Manager: Brad Madadian
QA Officer: Robert Treggiari

TITLE: CHEMICAL OXYGEN DEMAND

TEST METHOD REFERENCE: Method 5220C, Standard Methods for the Examination of Water and Wastewater, 21th Edition 2005,

REVISED SECTIONS: Section 8.0 notation

1.0 SCOPE AND APPLICATION

1.1 This method is used as a measure of the organic matter content of a sample that is susceptible to oxidation by a strong chemical oxidant. This procedure is written to incorporate the use of both the low level and mid- level Hach COD kits.

1.1 This method is applicable to surface water, and saline waters, domestic and industrial waste. Modified version of this test could be utilized for measuring COD in solids.

1.2 Test code: COD

1.0 SUMMARY

1.2 A sample is refluxed for 2 hours in a strong acid solution with a known excess of potassium dichromate(K2Cr2O7). After digestion, the remaining unreduced K2Cr2O7 is titrated with ferrous ammonium sulfate to determine the amount of K2Cr2O7 consumed and the oxidizable organic matter is calculated in terms of oxygen equivalent.

10.0 METHOD REPORTING AND DETECTION LIMIT

10.2 The reporting limit for this analysis is 20 Mg/L for waters, and 100 Mg/Kg for soils.

10.3 The Method Detection Limit (MDL) represents the lowest reportable concentration of an individual analyte that meets the method qualitative identification criteria.

10.4 Method Detection limits (MDLs) are experimentally determined using the procedures described in 40 CFR, Part 136, Appendix B. Actual reported MDLs incorporate the sample volume analyzed and sample dilutions if needed, which may cause MDL variations from sample to sample.

10.5 In general, MDLs are determined through the analysis of at least 7 replicate blank spikes (using the same procedures for sample analysis). The MDL is calculated by multiplying the standard deviation of the replicate concentrations by the appropriate Student's t value (3.143 for 7 replicates). If more than 7 replicates are analyzed refer to 40 CFR, Part 136, Appendix B for the appropriate student's t value. MDLs are determined initially (prior to analysis), on an annual basis, and after major maintenance to equipment. MDL data is archived with Quality Assurance. Refer to the most recent study for current MDLs. Refer to the SOP for MDLs (MQA245) for additional detail regarding MDL study procedures.

11.0 DEFINITION

11.2 ALIQUOT - a measured portion of a sample, or solution, taken for sample preparation and/or analysis.

11.3 BATCH – A group of samples which behave similarly with respect to the sampling or the testing procedures being employed and which are processed as a unit. For QC purposes, if the number of samples in a group is greater than 20 (or 10 for certain methods), then each group of 20 samples (or 10 samples for certain methods) or less will all be handled as a separate batch.

11.4 CONTAMINATION - a component of a sample or an extract that is not representative of the environmental source of the sample. Contamination may stem from other samples, sampling equipment, while in transit, from laboratory reagents, laboratory environment, or analytical instruments.

11.5 EXTERNAL CHECK STANDARD - The external check standard that is used to verify the accuracy of the calibration standards. An external check must be run with each analytical batch. The laboratory should initially assess laboratory performance of a check standard using the control limits generated by the external check supplier. Refer to the quality control section for each SOP. If the external check is outside of the control limits for a given parameter, all samples must be reanalyzed for that parameter after the problem has been resolved.

11.6 FIELD SAMPLE - a portion of material to be analyzed that is contained in single or multiple containers and identified by a unique sample number.

11.7 HOLDING TIME – the elapsed time expressed in days from the date of sampling until the date of its analysis.

11.8 INTERFERENTS – substances which affect the analysis for the element of interest.

11.9 MATRIX - the predominant material of which the sample to be analyzed is composed. For the purpose of this SOP, a sample matrix is either water or soil/sediment. Matrix is not synonymous with phase (liquid or solid).

11.10 MATRIX DUPLICATE - a second aliquot of the original sample prepared and analyzed in order to determine the precision of the method.

11.11 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.

11.12 RELATIVE PERCENT DIFFERENCE (RPD) - As used in this SOP to compare two values, the relative percent difference is based on the mean of the two values, and is reported as an absolute value, i.e., always expressed as a positive number or zero.

11.13 REAGENT WATER - water in which an interferant is not observed at or above the minimum quantitation limit of the parameters of interest. Accutest uses deionized water (municipal water which passes through Accutest's DI treatment system).

5.0 HEALTH AND SAFETY

5.1 The analyst should follow normal safety procedures as outlined in the Accutest Laboratories Chemical Hygiene Plan, which includes the use of lab coat and safety glasses. In addition, all acids are corrosive and should be handled with care. Flush spills with plenty of water. If acids contact any part of the body, flush with water and contact the supervisor.

10.0 COLLECTION, PRESERVATION & HOLDING TIMES

10.4 Aqueous samples must be preserved with H_2SO_4 to pH less than 2, and stored at 4 Deg. C.

10.5 Soil samples must be stored at 4°C ± 2°C.

10.6 All samples must be analyzed within 28 days from sampling date.

11.0 APPARATUS AND MATERIALS

11.4 Heating block
11.5 Microburette with a teflon stopcock
11.6 Magnetic stirrer; stirbars.
11.7 Pipettes, class A.
11.8 Volumetric flasks, class A.

12.0 STANDARDS AND REAGENTS

NOTE: All chemicals listed below are reagent grade unless otherwise specified. Distilled, deionized water should be used whenever water is required. All applicable standard/reagent preparation information, including vendor, lot number, date of preparation, calculations, and initials must be entered in the appropriate standard/reagent preparation logbook. Vendors typically used by Accutest include Fisher Scientific, VWR, Accustandard, Absolute Standards, Supelco, Chemservices, Ultra, and ERA. Additional vendors may be utilized as necessary.

8.1 Hach Test Kit Reagents, Low Level (0.00 - 150 mg/L):

8.1.1 Standard Potassium Dichromate Digestion Solution, 0.025 N (0.00417M): Add to about 500 ml water 1.2259 g of potassium dichromate, primary standard grade, previously dried at 103□C for 2 hr.. Dissolve, cool to room temperature, and dilute to 1000 ml. Note: This solution is to be used for standardization of the FAS only and should not be used as a digestion reagent

8.1.2 Sulfuric Acid, Concentrated

8.1.3 Ferroin Indicator Solution: Dissolve 1.485 g of 1,10 phenanthroline monohydrate and 695 mg of $FeSO_4 \cdot 7H_2O$ in water and dilute to 100 ml. This indicator solution may also be purchased already prepared.

8.1.4 Hach prepared digestion tubes containing pre-made and pre-measured reagents. The pre-made reagents include the catalysts and chloride compensator.

8.1.5 Standard Ferrous Ammonium Sulfate Titrant (FAS), approximately 0.0125 N: Dissolve 4.90 g $Fe(NH_4)_2(SO_4)_2 \cdot 6H_2O$ in water. Add 20 ml concentrated sulfuric acid, cool, and dilute to 1000 ml. Standardize solution daily against standard potassium dichromate solution.

1.1.1 Potassium Hydrogen Phthalate (KHP) Stock Standard: Weigh 4.250 g (previously dried at 120□C to a constant weight) of potassium hydrogen phthalate and dilute to 500 ml with DI water. This solution has a theoretical COD of 10,000 mg of oxygen per l of solution. This solution is stable when refrigerated for up to 3 months in the absence of visible biological growth.

1.1.2 1000ppm KHP solution, Dilute 10ml of 10000ppm solution (8.1.6) to 100ml of DI water.

8.1.8 Potassium Hydrogen Phthalate Calibration Standards. With each batch of low level samples that are digested, check standards at the levels of 20 and 50 mg/l should be prepared. The calibration standards can be made as shown below.

ml of 1000 mg/l	Final Volume (ml)	Final Conc. Mg/l
5.0	100	50.0
1.0	100	20.0

8.2 Hach Test Kit Reagents, Mid Level (0.00 - 1500 mg/L):

8.2.1 Standard Potassium Dichromate Digestion Solution, 0.25 N (0.0417M): Add to about 500 ml water 12.259 g of potassium dichromate, primary standard grade, previously dried at 103 Deg C for 2 hr.. Dissolve, cool to room temperature, and dilute to 1000 ml. Note: This solution is to be used for standardization of the FAS only and should not be used as a digestion reagent

8.2.2 Sulfuric Acid, Concentrated

8.2.3 Ferroin Indicator Solution: Dissolve 1.485 g of 1,10 phenanthroline monohydrate and 695 mg of $FeSO_4 \cdot 7H_2O$ in water and dilute to 100 ml. This indicator solution may also be purchased already prepared.

8.2.4 Hach prepared digestion tubes containing pre-made and pre-measured reagents. The pre-made reagents include the catalysts and chloride compensator.

8.2.5 Standard Ferrous Ammonium Sulfate Titrant (FAS), approximately 0.125 N: Dissolve 49.0 g $Fe(NH_4)_2(SO_4)_2 \cdot 6H_2O$ in water. Add 20 ml concentrated sulfuric acid, cool, and dilute to 1000 ml. Standardize solution daily against standard potassium dichromate solution.

8.2.6 Potassium Hydrogen Phthalate (KPH) Stock Standard: Weigh 4.250 g (previously dried at 120°C to a constant weight) of potassium hydrogen phthalate and dilute to 500 ml with DI water. This solution has a theoretical COD of 10,000 mg of oxygen per l of solution. This solution is stable when refrigerated for up to 3 months in the absence of visible biological growth.

1.1.1 With each batch of high level samples that are digested, check standards at the levels of 150 and 750 mg/l must be prepared.

ml of 10000 Mg/l	Final Vol.(ml)	Final Conc. Mg/l
1.50	100	150
7.50	100	750

13.0 INTERFERENCES

13.4 Positive interferences can be caused by the oxidation of reduced inorganic species such as ferrous iron, sulfide, and manganous manganese. Nitrite can also cause interferences, but nitrite levels in samples are generally low and this is normally an insignificant interference. Negative interferences can be caused by volatile straight-chain aliphatic compounds that are not oxidized to any appreciable extent. The use of mercuric sulfate and silver sulfate can improve the oxidation of the straight chain aliphatics. In addition, this method should not be used for samples containing more than 20000 mg/l of chloride.

14.0 PROCEDURE

Below is a step by step procedure for the analysis of samples for COD. Use the automated spreadsheet for documentation, and calculations of Standardization, and the analysis. This application can be found on server.

10.1 COD DIGESTION PROCEDURE

Below is a step-by-step procedure to digest the samples before titrating them for determination of COD.

The low level Hach kit should initially be used for most sample analysis, unless the sample has a history of a higher COD level. If you are preparing a dilution for the mid-level kit, make sure that the dilution will yield a result above the 50 mg/L level. For emergency or rush samples, it is suggested that digestions be done with both the high and the low level kits.

10.2 Low Level Hach kit digestion procedure:

CAUTION: When adding sample aliquots to the reagent vials, always add them along the side of the tube, such it forms a layer on top of the reagent mix. This must be performed in a hood with appropriate protective gear. After the sample aliquot has been added, cap the tube, verify the cap is tight, and invert the sample. This must be performed behind the hood sash. Verify the cap seal is not leaking. Invert the sample two more times and place in the digestion block. Do not use any tubes that do not seal or are cracked. **CAUTION:** The sample/reagent mix will be very hot.

1.1.1 Pipette 2.0 mL of liquid sample into the pre-prepared Hach digestion tubes (0 - 150 mg/L). If the sample is solid, stir the sample well and then add 0.40 g of sample to the digestion tube, along with 2.0 ml of water. Prepare one duplicate and one matrix spike for each set of 20 samples. Prepare two blanks with 2.0 ml of water each. An external check standard should also be prepared with each batch. A 20 and a 50 mg/L standard should also be prepared. Make sure that each of the tubes is clearly labeled.

Note: Larger dilutions will be required for most solid samples and in most cases they will be run using the mid-level Hach kit. Water samples that require dilution should also be analyzed using the mid-level kit.

1.1.2 Place capped tubes in a block digester preheated to 150 Deg. C and reflux for 2 hours. Cool to room temperature and place the vessels in a test tube rack.

Note: If samples turn green either prior to digestion or within the first 15 minutes of digestion a dilution (using DI water) should be prepared.

10.3 Mid Level Hach kit digestion procedure:

1.1.1 Pipette 2.0 mL of liquid sample into the pre-prepared Hach digestion tubes (50 - 1500 mg/L). If the sample is solid, stir the sample well and then add 0.40 g of sample to the digestion tube, along with 2.0 ml of water. Prepare one duplicate and one matrix spike for each set of 20 samples. Prepare two blanks with 2.0 ml of water each. An external check standard should also be prepared with each batch. A 150 and a 750 mg/L standard should also be prepared. Make sure that each of the tubes is clearly labeled.

Note: Larger dilutions will be required for most solid samples.

1.1.2 Place capped tubes in a block digester preheated to 150□C and reflux for 2 hours. Cool to room temperature and place the vessels in a test tube rack.

10.4 COD TITRATION PROCEDURE

Below is a step-by-step procedure for the titration of samples for the determination of COD. This procedure is shown for both the low and the mid-level Hach COD kits.

1.1.1 Low Level Hach Kit. If you are using the low level Hach kit (0 - 150 mg/L), pipette 2.00 mL of Potassium Dichromate solution, 0.025 N (0.00417 M), into two clean empty vials. Add 3 mL of sulfuric acid to each of the vials and swirl gently to mix, cool. Add a drop of Ferrion Indicator Solution and titrate with the FAS standard solution, 0.0125 N, until the color changes from greenish-blue to orange-brown. Make sure that the sample is continuously stirred during the titration. Record the number of ml used. Calculate the molarity of the FAS solution using the following equation:

$$\text{Molarity of FAS solution} = \frac{\text{Volume of 0.00417 M dichromate digestion solution, ml}}{\text{Volume of FAS solution,ml}} \times 0.025$$

The molarity to be used for the final calculations should be the average of the molarity calculated for each of the undigested blanks. Record the standardization data on the top of the COD analysis form as indicated.

10.4.2 Titrate each of the digested samples and blanks as follows: Take the digested sample or blank and add a small Teflon coated stir bar. Add 1 to 2 drops of ferrion indicator and stir rapidly on a magnetic stirrer while titrating with the standardized FAS solution from a burette. As in the FAS standardization, the end point is reached when there is a sharp color change from blue-green to reddish brown. Again, this color change is not permanent and the blue-green color may quickly reappear. Record the starting and ending volumes of FAS solution on the COD analysis form as indicated.

Note: The molarity of the FAS solution decreases over time and more FAS will be required to titrate the calibration blanks as the FAS solution ages.

10.5 Mid Level Hach Kit

1.1.1 If you are using the mid level Hach kit (0.00 -1500 mg/L), pipette 2.00 mL of Potassium Dichromate solution, 0.25 N (0.0417 M), into two clean empty vials. Add 3 mL of sulfuric acid to each of the vials and swirl gently to mix. Add a drop of Ferrion Indicator Solution and titrate with the FAS standard solution, 0.125 N, until the color changes from greenish-blue to orange-brown. Make sure that the sample is continuously stirred during the titration. Record the number of ml used. Calculate the molarity of the FAS solution using the following equation:

Note: The calculation used is dependent upon the molarity of the dichromate solution used. The factor applied should be equal to 6 times the molarity of the dichromate solution.

$$\text{Molarity of FAS Solution} = \frac{\text{Volume of 0.0417 M dichromate digestion solution, ml} \times 0.25}{\text{Volume of FAS solution, ml}}$$

The molarity to be used for the final calculations should be the average of the molarity calculated for each of the undigested blanks. Record the standardization data on the top of the COD analysis form as indicated.

10.5.2 Titrate each of the digested samples and blanks as follows: Take the digested sample or blank and add a small Teflon coated stir bar. Add 1 drop of ferrion indicator and stir rapidly on a magnetic stirrer while titrating with the standardized FAS solution from a burette. As in the FAS standardization, the end point is reached when there is a sharp color change from blue-green to reddish brown. Again, this color change is not permanent and the blue-green color may quickly reappear. Record the starting and ending volumes of FAS solution on the COD analysis form as indicated.

Note: The molarity of the FAS solution decreases over time and more FAS will be required to titrate the calibration blanks as the FAS solution ages.

11.0 QUALITY ASSURANCE

1.1 Below is a summary of the quality control requirements for this method. Make sure to check with laboratory supervisor or manager for any additional client specific quality control requirements.

1.2 **Calibration Standards.** The laboratory must digest and analyze a 20 ppm and 50 ppm calibration standards for low level kits or (150 ppm and 750 ppm standards for mid level kits, when used) with each batch of 20 or less samples. The 20 ppm standard results must be within ± 30% recovery. The 50 ppm , 150 ppm,and 750 ppm standard results must be within ± 10% recovery. If this criteria is not met corrective action must be taken and the problem resolved (as indicated by passing calibration standards) prior to sample analysis.

1.3 **Method Blank**. The laboratory must analyze a method blank with each set of samples. A minimum of one method blank is required for every 20 samples. The method blank must contain the analyte at less than the reporting limit (1/2 the RL for some clients). If the method blank contains an analyte level over that limit the problem must be identified and corrected prior to sample analysis.

1.4 **Matrix Duplicate.** The laboratory must prepare a duplicate sample for a minimum of 1 in 20 samples. The relative percent difference (RPD) between the duplicate sample and the original should be assessed. The Duplicate RPD should be calculated as shown below

$$\frac{\text{(Original Sample Result – Duplicate Result)} \times 100}{\text{(Original Sample Result + Duplicate Result)} \times 0.5} = \%\ \text{RPD}$$

The Duplicate RPD should be assessed using in house limits. Until these limits can be generated, then the default limit of 20 percent RPD should be applied. If a duplicate RPD is out of control, then the results should be flagged with the appropriate footnote. If the sample and the duplicate are less than 5 times the reporting limits and are within a range of ± the reporting limit, then the duplicate is considered to be in control.

1.5 **Matrix spike.** The laboratory must add a known amount of each analyte to a minimum of 1 in 20 samples. The spike recovery should be assessed using in house limits. Until these limits can be generated, default limits of 75-125 % recovery should be applied. If a matrix recovery is out of control, then the recovery should be flagged with the appropriate footnote. If a matrix spike amount is less than one fourth of the sample amount, then the sample can be assessed against the control limits and should be footnoted to that effect.

$$\frac{(\text{Matrix Spike Result} - \text{Original Result}) \times 100}{\text{Amount of Spike}}$$

1.6 **Spike Blank.** The laboratory must analyze a spike blank with each set of samples. A minimum of one spike blank is required for every 20 samples. The net recovery should be within 20 percent of the true value. If the spike blank is outside of this range, the problem must be identified and corrected before sample analysis can proceed.

1.7 **External Standard.** An external standard is analyzed with each analytical batch. The net recovery should be within 10% of the true value (if the external is prepared in house) or within the manufacturer's acceptance criteria if purchased from an outside vendor. If the external is outside this range, the problem must be identified and corrected before sample analysis can proceed.

1.8 A Precision and accuracy (P&A) study is performed as an initial determination of capability, on an annual basis (continued demonstration of capability – a successful PT result may be used in place of a P&A for continued DOC), and if any significant changes have been made to the instrument. In general, 4 replicates or blank spikes are analyzed using the same procedures and conditions for sample analysis. The mean percent recovery is compared to the spike blank control limits of 20%. The standard deviation (of the percent recovery of the 4 spike blanks) is compared to the control limit of 20. If percent recovery or standard deviation criteria are not met, corrective action must be taken to bring the system back into control. The P&A study replicates must be prepared from a source independent from the calibration standards.

1.9 Quality Control data is generated (control charts) and reviewed on an annual basis by Quality Assurance (blank spike/ matrix spike recoveries and matrix duplicate RPDs).

12.0 DOCUMENTATION

1.1 The Standard preparation log application must be completed for all standard preparations. All information requested must be completed.

1.2 The Accutest lot number must be cross-referenced on the standard vial/container.

1.3 Any comments or observations concerning the sample that may influence the analytical procedure.

1.4 Any corrections to laboratory data must be done using a single line through the error. The initials of the person and date of corrections must appear next to the correction.

1.5 All laboratory logs must be reviewed and initialed or signed by the lab manager.

13.0 DATA REVIEW

13.1 The analyst conducts the primary review of all data. This review begins with a check of all method quality control and progresses through sample quality control concluding with a check to assure that the client's requirements have been executed.

1.1 A secondary review is performed by department managers, and it includes review of the data produced by their department. Manual calculations, QC criteria, and a comparison of the data package to client specified requirements are checked. The department manger may reject data, initiate reanalysis, take additional corrective action, or process data.

1.2 The laboratory director performs a full tertiary review of the data package following its assembly. This review includes an evaluation of QC data against acceptance criteria and a check of the data package contents to assure that all analytical requirements and specifications were executed.

1.3 Spot-check reviews are performed by the Quality Assurance Officer focusing on all elements of the deliverable including the client's specifications and requirements, analytical quality control, sample custody documentation and sample identification.

1.0 DATA REPORTING

1.1 A results page including positive results and/or RLs, units, methodology, analysis dates, and data qualifiers are reported. Additional quality control data including matrix duplicate RPDs, matrix spike recovery, blank spike and method blank results may be reported upon request of the client.

1.2 Data may be submitted to the client in a specified electronic format (EDD).

1.3 Once the data is approved by the laboratory manager, it may be accessed by clients via LabLink™.

2.0 POLLUTION PREVENTION & WASTE MANAGEMENT

1.3 Pollution Prevention. Users of this method must perform all procedural steps that control the creation and/or escape of wastes of hazardous materials to the environment. The amounts of standards, reagents, and solvents must be limited to the amounts specified in this SOP. All safety practices designed to limit the escape of vapors, liquids, or solids to the environment must be followed. All method users must be familiar with the waste management practices described in section 15.2.

1.4 Waste Management. Individuals performing this method must follow established waste management procedures as described in the Sample and Waste Disposal SOP. This document describes the proper disposal of all waste materials generated during the testing of samples as follows:

1.3.1 Non-Hazardous aqueous wastes
1.3.2 Hazardous aqueous wastes
1.3.3 Chlorinated organic solvents
1.3.4 Non-chlorinated organic solvents
1.3.5 Hazardous solid wastes
1.3.6 Non-hazardous solid wastes
1.3.7 Microbiological waste

3.0 METHOD PERFORMANCE

1.3 Method performance is evaluated by the annual QC limits (control charts) generated by QA, and the annual MDL study results. Refer to section 3.5 for MDLs, and section 11.8 for QC limits.

4.0 ADDITIONAL REFERENCES

14.1 No additional references are required for this method.

Lab Manager: Brad Madadian
QA Officer: Robert Treggiari

TITLE: HARDNESS AS CaCO3

TEST METHOD REFERENCE: 2340 C. Standard Methods for the Examination of Water and Wastewater 21th Edition, 2005

REVISED SECTIONS: Section 8.0 notation

1.0 SCOPE AND APPLICATION

1.1 This method is applicable to drinking, surface, and saline waters, domestic and industrial wastes.

1.2 The method is suitable for all concentration ranges of hardness

1.3 Test code: HRD

2.0 SUMMARY

2.1 Calcium and magnesium ions in the sample are sequestered upon the addition of disodium ethylenediamine tetraacetate (Na2EDTA). The end point of the reaction is detected by means of Calmagite or Eriochrome Black indicator, which has a red color in the presence of calcium and magnesium and a blue color when the cations are sequestered.

3.0 METHOD REPORTING AND DETECTION LIMIT

3.1 The reporting limit for this analysis is 4.0 Mg/L.

3.2 The Method Detection Limit (MDL) represents the lowest reportable concentration of an individual analyte that meets the method qualitative identification criteria.

3.3 Method Detection limits (MDLs) are experimentally determined using the procedures described in 40 CFR, Part 136, Appendix B. Actual reported MDLs incorporate the sample volume analyzed and sample dilutions if needed, which may cause MDL variations from sample to sample.

3.4 In general, MDLs are determined through the analysis of at least 7 replicate blank spikes (using the same procedures for sample analysis). The MDL is calculated by multiplying the standard deviation of the replicate concentrations by the appropriate Student's t value (3.143 for 7 replicates). If more than 7 replicates are analyzed refer to 40 CFR, Part 136, Appendix B for the appropriate student's t value. MDLs are determined initially (prior to analysis), on an annual basis, and after major maintenance to equipment. MDL data is archived with Quality Assurance. Refer to the most recent study for current MDLs. Refer to the SOP for MDLs (MQA245) for additional detail regarding MDL study procedures. For additional detail regarding MDL studies, refer to the MDL SOP MQA245.

3.5 Current MDL studies are filed with Quality Assurance. Obsolete MDL studies are archived with the QA files. Electronic MDL data is found in the annual "MDL" folder on the QA server (LINUXMA1).

4.0 DEFINITION

4.1 ALIQUOT - a measured portion of a sample, or solution, taken for sample preparation and/or analysis.

4.2 BATCH – A group of samples which behave similarly with respect to the sampling or the testing procedures being employed and which are processed as a unit. For QC purposes, if the number of samples in a group is greater than 10, then each group of 10 samples or less will all be handled as a separate batch.

4.3 CONTAMINATION - a component of a sample or an extract that is not representative of the environmental source of the sample. Contamination may stem from other samples, sampling equipment, while in transit, from laboratory reagents, laboratory environment, or analytical instruments.

4.4 EXTERNAL CHECK STANDARD - The external check standard that is used to verify the accuracy of the calibration standards. An external check must be run with each analytical batch. The laboratory should initially assess laboratory performance of a check standard using the control limits generated by the external check supplier. Refer to the quality control section for each SOP. If the external check is outside of the control limits for a given parameter, all samples must be reanalyzed for that parameter after the problem has been resolved.

4.5 FIELD SAMPLE - a portion of material to be analyzed that is contained in single or multiple containers and identified by a unique sample number.

4.6 HOLDING TIME – the elapsed time expressed in days from the date of sampling until the date of its analysis.

4.7 INTERFERENTS – substances which affect the analysis for the element of interest.

4.8 MATRIX - the predominant material of which the sample to be analyzed is composed. For the purpose of this SOP, a sample matrix is either water or soil/sediment. Matrix is <u>not</u> synonymous with phase (liquid or solid).

4.9 MATRIX DUPLICATE - a second aliquot of the original sample prepared and analyzed in order to determine the precision of the method.

4.10 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.

4.11 RELATIVE PERCENT DIFFERENCE (RPD) - As used in this SOP to compare two values, the relative percent difference is based on the mean of the two values, and is reported as an absolute value, i.e., always expressed as a positive number or zero.

4.12 REAGENT WATER - water in which an interferant is not observed at or above the minimum quantitation limit of the parameters of interest. Accutest uses deionized water (municipal water which passes through Accutest's DI treatment system).

5.0 HEALTH AND SAFETY

5.1 The analyst should follow normal safety procedures as outlined in the Accutest Laboratories Chemical Hygiene Plan, which includes the use of lab coat and safety glasses. In addition, all acids are corrosive and should be handled with care. Flush spills with plenty of water. If acids contact any part of the body, flush with water and contact the supervisor.

5.2 The toxicity or carcinogenicity of each reagent used in this method has not been precisely determined; however, each chemical should be treated as a potential health hazard. Exposure to these reagents should be reduced to the lowest possible level.

6.0 COLLECTION, PRESERVATION & HOLDING TIMES

6.1 The sample must be acidified to a pH of less than 2 by addition of 1:1 Nitric acid, and kept under refrigeration at 4 Deg C.

6.2 All samples must be analyzed within 180 days from sampling date.

7.0 APPARATUS AND MATERIALS

7.1 Titration vessel, 50 ml or 100 ml beaker.
7.2 Magnetic stirrer; stirbars.
7.3 Pipettes, class A.
7.4 Volumetric flasks, class A.
7.5 Burets, 50 ml and 10 ml micro.

8.0 REAGENTS AND STANDARDS

NOTE: All chemicals listed below are reagent grade unless otherwise specified. Distilled, deionized water should be used whenever water is required. All applicable standard/reagent preparation information, including vendor, lot number, date of preparation, calculations, and initials must be entered in the appropriate standard/reagent preparation logbook. Vendors typically used by Accutest include Fisher Scientific, VWR, Accustandard, Absolute Standards, Supelco, Chemservices, Ultra, and ERA. Additional vendors may be utilized as necessary.

8.1 Buffer solution. Dissolve 1.179 g of disodium EDTA (analytical grade) and 780 mg of $MgSO4 \cdot 7H_2O$ (or 644 mg $MgCl_2 \cdot 6H_2O$) in 50 ml of DI water. Add this solution to a 250 ml volumetric flask containing 16.9 g of ammonium chloride (NH_4Cl) and 143 ml concentrated ammonium hydroxide (NH_4OH) with mixing and dilute to the mark with DI water. Store in a plastic bottle for no longer than 1 month.

8.2 Inhibitor solution. These are to be used only if interferences are evident during the titration (Please check with lab supervisor or lab manager first).

8.2.1 Inhibitor II: Dissolve 5.0 g of $Na_2S\ 9H_2O$ in 100 ml of DI water. Cover with tightly fitted rubber stopper. This inhibitor deteriorates through air oxidation. It produces a sulfide precipitate that obscures the end point when appreciable concentration of heavy metals are present.

8.3 Indicator solutions. A Calgamite indicator solution can be purchased commercially or by dissolving 0.10 g calgamite in 100 ml DI water. Use 4 to 5 drops per 50 ml solution to be titrated. Adjust the number of drops if necessary.

8.4 Standard EDTA titrant, 0.01M. Place 3.723 g of analytical reagent grade disodium ethylenediamine tetraacetate dihydrate, $Na_2H_2C_{10}H_{12}O_8N2 \cdot 2H_2O$ in a 1 liter volumetric flask and dilute to the mark with DI

water. Check with standard calcium solution by titration. (See section 1 under procedure.) Store in polyethylene.

8.5 Standard calcium solution. Place 1.000 g of anhydrous calcium carbonate in a 500 ml flask. Slowly add 1:1 HCl (<10 ml) until all of the $CaCO_3$ has dissolved. Add 200 ml distilled water to the flask and mix. Boil this solution for a few minutes to expel CO_2. Cool. Add a few drops of methyl red indicator and adjust to intermediate orange color by adding dropwise, 3N NH_4OH or 1+1 HCl as required. Quantitatively transfer to a 1 Liter volumetric flask and dilute to mark with DI water.

8.6 Hydrochloric acid solution, 1:1. Add 100 mls of concentrated HCl to 100 mls of DI. Mix and cool.

8.7 Methyl red indicator. Dissolve 0.10 g methyl red in DI water in a 100 ml volumetric flask and dilute to the mark.

8.8 Ammonium hydroxide solution, 3 N. Dilute 210 ml of concentrated ammonium hydroxide (NH_4OH) to 1 liter with DI water.

8.9 Ammonium hydroxide solution, 1 N. Dilute 70 ml of concentrated ammonium hydroxide to 1 liter with DI water.

8.10 NaOH, 0.1 N. In a 1000 ml volumetric flask, dissolve 4 grams of NaOH in 750 ml of DI water. Cool, and bring to volume with DI water.

9.0 INTERFERENCES

9.1 Some metal ions interfere by causing fading or indistinct endpoints or by stoichiometric consumption of EDTA. These interferences can be reduced by adding certain inhibitors before titration.

9.2 Conduct titrations at or near normal room temperature. The color change will be impractically slow as the The sample temperature approaches freezing temperatures.

10.0 PROCEDURE

Below is a step by step procedure for the analysis of samples for HRD. Use the automated spreadsheet for documentation, and calculations of Standardization, and the analysis. This application can be found on server. Before starting on the samples, standardize the EDTA solution following the procedure outlined below.

10.1 Place 10.0 ml of standard calcium solution in a vessel containing about 50 ml of DI water. Add 1 to 2 ml of buffer solution. Usually 1 ml will be sufficient to give a pH of 10.0 $\pm$ 0.1. Record the pH. Add 4 to 5 drops of calgamite indicator. Titrate slowly with continuous stirring with the EDTA until the last reddish tinge disappears. Add the last few drops at 3 to 5 second intervals. At the end point the color is blue. Total titration duration should be 5 minutes from the time of the buffer addition. Calculate the normality of the EDTA as shown below, and document the result in the analysis log book.

N of EDTA = (0.20)/(ml of EDTA added)

10.2 Start the titration of samples by measuring 25 ml of sample into a 50 ml titration vessel. Add 25 ml of DI water, and mix. Note: Select a sample size that requires less than 15 ml of EDTA titrant. For quality control sample, measure 3 aliquots. One will be the duplicate sample, one will be the original sample

analysis, and one will be the matrix spike sample. Set up a preparation blank and a spike blank by placing 50 mls of DI water into the titration vessels. Add 1 ml of standard calcium carb. solution (8.5) to spike blank and matrix spike. The final concentration of the spike will be 40 mg/l.

NOTE: Highly polluted samples (industrial waste, organic contaminants, etc.) should first go through a metal digestion step before analysis. Please check with lab supervisor or manager.

10.3 Neutralize the samples with 1N ammonium hydroxide and dilute to a final volume of approximately 50 ml.

10.4 Add 1 to 2 ml of buffer solution to each sample.

10.5 Add 4 to 5 drops of calgamite indicator solution to each sample.

10.6 Titrate the sample slowly with continuous stirring with the standard EDTA titrant until the last reddish tint disappears. The solution is normally blue at the end point. Total titration duration should be 5 minutes from the time of buffer addition.

NOTE: Completion of the titration within 5 minutes minimizes the tendency for CaCO3 to precipitate.

NOTE: If it appears that interferences are present, repeat the titration as above, but add inhibitor immediately after step 10.4.

10.7 Calculations.

10.7.1

$$\text{Hardness , mg/l } CaCO_3 = \frac{(A - B) \times N \times 50000}{\text{ml sample}}$$

Where: A = ml of EDTA titrant.
B= ml of EDTA used for Method Blank
N = Normality of EDTA titrant.

11.0 QUALITY ASSURANCE

11.1 Below is a summary of the quality control requirements for this method. Make sure to check with laboratory supervisor or manager for any additional client specific quality control requirements.

11.2 **Method Blank**. The laboratory must analyze a method blank with each set of samples. A minimum of one method blank is required for every 10 samples. The method blank must contain the analyte at less than the reporting limit (1/2 the RL for some clients). If the method blank contains an analyte level over that limit the problem must be identified and corrected prior to sample analysis.

11.3 **Matrix Duplicate.** The laboratory must prepare a duplicate sample for a minimum of 1 in 10 samples. The relative percent difference (rpd) between the duplicate sample and the original should be assessed. The Duplicate RPD should be calculated as shown below

$$\frac{\text{(Original Sample Result – Duplicate Result) x 100}}{\text{(Original Sample Result + Duplicate Result) x 0.5}} = \%\ \text{RPD}$$

The Duplicate RPD should be assessed using in house limits. Until these limits can be generated, then the default limit of 20 percent RPD should be applied. If a duplicate RPD is out of control, then the results should be flagged with the appropriate footnote. If the sample and the duplicate are less than 5 times the reporting limits and are within a range of ± the reporting limit, then the duplicate is considered to be in control.

11.4 **Matrix spike.** The laboratory must add a known amount of each analyte to a minimum of 1 in 10 samples. The spike recovery should be assessed using in house limits. Until these limits can be generated, default limits of 75-125 % recovery should be applied. If a matrix recovery is out of control, then the recovery should be flagged with the appropriate footnote. If a matrix spike amount is less than one fourth of the sample amount, then the sample can be assessed against the control limits and should be footnoted to that effect.

$$\frac{\text{(Matrix Spike Result – Original Result) x 100}}{\text{Amount of Spike}}$$

11.5 **Spike Blank.** The laboratory must analyze a spike blank with each set of samples. A minimum of one spike blank is required for every 10 samples. The net recovery should be within 20 percent of the true value. If the spike blank is outside of this range, the problem must be identified and corrected before sample analysis can proceed.

11.6 **External Standard.** An external standard is analyzed with each analytical batch. The net recovery should be within 10% of the true value (if the external is prepared in house) or within the manufacturer's acceptance criteira if purchased from an outside vendor. If the external is outside this range, the problem must be identified and corrected before sample analysis can proceed.

11.7 A Precision and accuracy (P&A) study is performed as an initial determination of capability, on an annual basis (continued demonstration of capability – a successful PT result may be used in place of a P&A for continued DOC), and if any significant changes have been made to the instrument. In general, 4 replicates or blank spikes are analyzed using the same procedures and conditions for sample analysis. The mean percent recovery is compared to the spike blank control limits of 20%. The standard deviation (of the percent recovery of the 4 spike blanks) is compared to the control limit of 20. If percent recovery or standard deviation criteria are not met, corrective action must be taken to bring the system back into control. The P&A study replicates must be prepared from a source independent from the calibration standards (as applicable).

11.8 Quality Control data is generated (control charts) and reviewed on an annual basis by Quality Assurance (blank spike/ matrix spike recoveries and matrix duplicate RPDs).

12.0 DOCUMENTATION

12.1 Which method was used.

12.2 The sample ID, duplicate as necessary.

12.3 The initial volume aliquoted.

12.4 Any comments or observations concerning the sample that may influence the analytical procedure.

12.5 The date the analysis performed.

12.6 Any corrections to laboratory data must be done using a single line through the error. The initials of the person and date of corrections must appear next to the correction.

12.7 All laboratory logbooks must be reviewed and initialed or signed by the lab manager.

13.0 DATA REVIEW

13.1 The analyst conducts the primary review of all data. This review begins with a check of all method quality control and progresses through sample quality control concluding with a check to assure that the client's requirements have been executed.

13.2 A secondary review is performed by department managers, and it includes review of the data produced by their department. Manual calculations, QC criteria, and a comparison of the data package to client specified requirements are checked. The department manger may reject data, initiate reanalysis, take additional corrective action, or process data.

13.3 The laboratory director performs a full tertiary review of the data package following its assembly. This review includes an evaluation of QC data against acceptance criteria and a check of the data package contents to assure that all analytical requirements and specifications were executed.

13.4 Spot-check reviews are performed by the Quality Assurance Officer focusing on all elements of the deliverable including the client's specifications and requirements, analytical quality control, sample custody documentation and sample identification.

14.0 DATA REPORTING

14.1 A results page including positive results and/or RLs, units, methodology, analysis dates, and data qualifiers are reported. Additional quality control data including matrix duplicate RPDs, matrix spike recovery, blank spike and method blank results may be reported upon request of the client.

14.2 Data may be submitted to the client in a specified electronic format (EDD).

14.3 Once the data is approved by the laboratory manager, it may be accessed by clients via LabLink™.

14.4 Procedures for handling non-conforming data.

14.4.1 If quality control data does not meet criteria the non-conformance must be discussed in a case narrative and footnoted on the applicable quality control report summary.

14.4.2 If preservation or holding time criteria is not met and the samples are analyzed the result page must be footnoted with this information, and the non-conformance must be

discussed in a case narrative or other suitable communication (telephone conversation log or email). Client notification documentation should be included with the data (telephone conversation log, fax, or email).

15.0 POLLUTION PREVENTION & WASTE MANAGEMENT

15.1 Pollution Prevention. Users of this method must perform all procedural steps that control the creation and/or escape of wastes of hazardous materials to the environment. The amounts of standards, reagents, and solvents must be limited to the amounts specified in this SOP. All safety practices designed to limit the escape of vapors, liquids, or solids to the environment must be followed. All method users must be familiar with the waste management practices described in section 15.2.

15.2 Waste Management. Individuals performing this method must follow established waste management procedures as described in the Sample and Waste Disposal SOP. This document describes the proper disposal of all waste materials generated during the testing of samples as follows:

15.2.1 Non-Hazardous aqueous wastes
15.2.2 Hazardous aqueous wastes
15.2.3 Chlorinated organic solvents
15.2.4 Non-chlorinated organic solvents
15.2.5 Hazardous solid wastes
15.2.6 Non-hazardous solid wastes
15.2.7 Microbiological waste

16.0 METHOD PERFORMANCE

16.1 Method performance is evaluated by the annual QC limits (control charts) generated by QA, and the annual MDL study results. Refer to section 3.5 for MDLs, and section 11.8 for QC limits.

17.0 ADDITIONAL REFERENCES

17.1 None.

Lab Manager: Brad Madadian

QA Officer: Robert Treggiari

TITLE: SULFIDE

TEST METHOD REFERENCE: 4500-S F . Standard Methods for the Examination of Water and Wastewater 21th Edition, 2005

REVISED SECTION: Section 8.0 notation

1.0 SCOPE & APPLICATION

1.1 This method is applicable to total and dissolved sulfides in drinking water and surface waters, sewage and industrial wastes. Acid insoluble sulfides are not measured by the use of this test.

1.2 A modification of this method is used to determine water- soluble sulfides in soil samples.

2.0 SUMMARY

2.1 Excess iodine is added to sample which may or may not have been treated with zinc acetate to produce zinc sulfide. The iodine oxidizes the sulfide to sulfur upon addition of HCL. The excess iodine is back titrated with sodium thiosulfate.

3.0 METHOD REPORTING LIMIT AND DETECTION LIMIT

3.1 The normal reporting limit for sulfide in waters is 2 mg/l and 4 mg/kg for soils.

3.2 The Method Detection Limit (MDL) represents the lowest reportable concentration of an individual analyte that meets the method qualitative identification criteria.

3.3 Method Detection limits (MDLs) are experimentally determined using the procedures described in 40 CFR, Part 136, Appendix B. Actual reported MDLs incorporate the sample volume analyzed and sample dilutions if needed, which may cause MDL variations from sample to sample.

3.4 In general, MDLs are determined through the analysis of at least 7 replicate blank spikes (using the same procedures for sample analysis). The MDL is calculated by multiplying the standard deviation of the replicate concentrations by the appropriate Student's t value (3.143 for 7 replicates). If more than 7 replicates are analyzed refer to 40 CFR, Part 136, Appendix B for the appropriate student's t value. MDLs are determined initially (prior to analysis), on an annual basis, and after major maintenance to equipment. MDL data is archived with Quality Assurance. Refer to the most recent study for current MDLs. For additional detail regarding MDL studies, refer to the MDL SOP MQA245.

3.5 The MDL represents the lowest reportable concentration of an individual analyte that meets the method qualitative identification criteria.

3.6 Current MDL studies are filed with Quality Assurance. Obsolete MDL studies are archived with the QA files. Electronic MDL data is found in the annual "MDL" folder on the QA server (LINUXMA1).

4.0 DEFINITIONS

4.1 ALIQUOT - a measured portion of a sample, or solution, taken for sample preparation and/or analysis.

4.2 BATCH – A group of samples which behave similarly with respect to the sampling or the testing procedures being employed and which are processed as a unit. For QC purposes, if the number of samples in a group is greater than 20 (or 10 for certain methods), then each group of 20 samples (or 10 samples for certain methods) or less will all be handled as a separate batch.

4.3 DRY WEIGHT – the weight of a sample based on percent solids. The weight after drying. See Percent Moisture.

4.4 CONTAMINATION - a component of a sample or an extract that is not representative of the environmental source of the sample. Contamination may stem from other samples, sampling equipment, while in transit, from laboratory reagents, laboratory environment, or analytical instruments.

4.5 FIELD SAMPLE - a portion of material to be analyzed that is contained in single or multiple containers and identified by a unique sample number.

4.6 FIELD BLANK – this is any sample that is submitted from the field and is identified as a blank. This includes trip blanks, rinsates, equipment blanks, etc.

4.7 HOLDING TIME – the elapsed time expressed most commonly in days from the date of sampling until the date of its analysis.

4.8 INSUFFICIENT QUANTITY - when there is not enough volume (water sample) or weight (soil/sediment) to perform any of the required operations: sample analysis or extraction, percent moisture, MS/DUP, etc.

4.9 MATRIX: The component or substrate (e.g., water, soil) which contains the analyte of interest.

4.10 Matrix Spike: The laboratory must add a known amount of each analyte to a minimum of 1 in 10 samples. The matrix spike recovery is calculated and assessed against the control limits that are generated in house. If control limits are not available, then a default limits of 75 to 125 percent should be applied.

4.11 MATRIX DUPLICATE: A duplicate sample is analyzed at a minimum of 1 in 10 samples. The relative percent difference (RPD) between the duplicate and the sample should be assessed. The duplicate

RPD is calculated as shown below. Assess laboratory performance against the control limits. In house limits are generated once sufficient duplicate data is available to generate limits (usually a minimum of 20 to 30 analyses). If a duplicate is out of control, flag the results with the appropriate footnote. If the sample and the duplicate are less than 5 times the reporting limits and are within a range of ± the reporting limit, then the duplicate is considered to be in control. Note: If control limits are not specified, use default limits of ± 20% RPD.

$$\frac{(|\text{Sample Result} - \text{Duplicate Result}|) \times 100}{(\text{Sample Result} + \text{Duplicate Result})/2} = \text{Duplicate RPD}$$

4.12 METHOD BLANK. The laboratory must analyze a method blank with each set of samples. A minimum of one method blank is required for every 10 samples. The method blank must contain the parameter of interest at levels of less that the reporting limit for that parameter. If the method blank contains levels over the reporting limits, the samples must be reanalyzed. The exception to this rule is when the samples to be reported contain greater than 10 times the method blank level. In addition, if all the samples are less than a client required limit and the method blank is also less than that limit, then the results can be reported as less than that limit.

4.13 PERCENT MOISTURE - an approximation of the amount of water in a soil/sediment sample made by drying an aliquot of the sample at 105 °C. The percent moisture determined in this manner also includes contributions from all compounds that may volatilize at or below 105 °C, including water. Percent moisture may be determined from decanted samples and from samples that are not decanted.

4.14 RELATIVE PERCENT DIFFERENCE (RPD) - To compare two values, the relative percent difference is based on the mean of the two values, and is reported as an absolute value, i.e., always expressed as a positive number or zero. In contrast, see percent difference.

4.15 REAGENT GRADE: Analytical reagent (AR) grade, ACS reagent grade, and reagent grade are synonymous terms for reagents that conform to the current specifications of the Committee on Analytical Reagents of the American Chemical Society.

4.16 REAGENT WATER: Water that has been generated by any method which would achieve the performance specifications for ASTM Type II water. Water in which an interferant is not observed at or above the minimum quantitation limit of the parameters of interest. Accutest uses deionized water (municipal water which passes through Accutest's DI treatment system).

4.17 REFERENCE MATERIAL: A material containing known quantities of target analytes in solution or in a homogeneous matrix. It is used to document the bias of the analytical process.

4.18 SPIKE BLANK OR LAB CONTROL SAMPLE. Analyze a laboratory control sample or spike blank with each set of samples. A minimum of one lab control sample or spike blank is required for every 10 samples. Assess laboratory performance against the control limits. In house limits should also be generated once sufficient data is available to generate limits (usually a minimum of 20 to 30 analyses). If the lab control is outside of the control limits for a parameter, all samples must be reanalyzed for that parameter. The exception is if the lab control recovery is high and the results of

the samples to be reported are less than the reporting limit. In that case, the sample results can be reported with no flag. Note: If control limits are not specified, then default limits of 80 to 120 percent should be used.

5.0 HEALTH & SAFETY

5.1 All safety practices must be followed as outlined in the Accutest Laboratories Employee Safety Handbook and Chemical Hygiene Plan. Safety glasses, gloves, and lab coats must be worn. All samples, solutions, and extracts must be treated as unknown and potentially hazardous.

5.2 The toxicity or carcinogenicity of each reagent used in this method has not been precisely determined; however, each chemical should be treated as a potential health hazard. Exposure to these reagents should be reduced to the lowest possible level.

6.0 COLLECTION, PRESERVATION, AND HOLDING TIMES

6.1 Water samples should be collected in two 250-300 ml containers. Soil samples should be collected in one 250-300ml glass container.

6.2 Water samples should be preserved with zinc acetate and sodium hydroxide. Water and soil samples should be kept under refrigeration at 4° ± 2° C until they are analyzed.

6.3 All samples should be analyzed within 7 days of the date of collection.

7.0 APPARATUS & MATERIALS

The following items are needed for the analysis of samples following the method outlined below:

7.1 Burette.
7.2 Graduated glass or plastic beakers.
7.3 Stir bars.
7.4 Stir plates.
7.5 Class A Volumetric pipettes.
7.6 Filtering Apparatus with 934 AH Whatman Glass Fibers or equivalent.

8.0 STANDARDS & REAGENTS

NOTE: All chemicals listed below are reagent grade unless otherwise specified. Distilled, deionized water should be used whenever water is required. All applicable standard/reagent preparation information, including vendor, lot number, date of preparation, calculations, and initials must be entered in the appropriate standard/reagent preparation logbook. Vendors typically used by Accutest include Fisher Scientific, VWR, Accustandard, Absolute Standards, Supelco, Chemservices, Ultra, and ERA. Additional vendors may be utilized as necessary.

8.1 Hydrochloric Acid, HCL, (6 N). Add 100 ml of concentrated HCL to 100 ml of DI water. Cool, mix.

8.2 Standard Iodine Solution (0.025 N). Dissolve 20 to 25g of anhydrous potassium iodide (KI) and 3.2 g of iodine in 400 ml of DI water and dilute to 1000 ml in a volumetric flask. Standardize this solution against 0.025 N sodium thiosulfate.

8.3 Sodium Thiosulfate solution (0.025N). Dissolve 6.205 g of Na2S2O3.5H2O in approximately 500 ml of DI water. Add 9 ml of 1 N NaOH and dilute to a final volume of 1000 ml in a volumetric flask. Note: this solution is commercially available. Request the certificate of the analysis from the vendor and keep in file.

8.4 Starch Indicator solution. This solution is also commercially available.

8.5 Zinc acetate solution. Dissolve 220 g of zinc acetate (Zn(C2H3O2)2)2H2O in 870 ml of DI water.

8.6 Sodium Hydroxide, (6N). Dissolve 24 g of sodium hydroxide in 50 ml of DI water. Dilute to 100 ml with DI water.

8.7 Sodium Hydroxide, (1N). Dissolve 40 g of sodium hydroxide in 500 ml of DI water. Dilute to 1000 ml with DI water.

8.8 Sulfide 537 mg/L Stock Solution. Dissolve 4.02 g of sodium sulfide nonahydrate, Na2S.9H2O, in approximately 980 ml of DI water. Adjust the PH of this solution to >9 and < 11 with 1 N sodium hydroxide solution (8.7). Dilute to 1000 ml with DI water in a volumetric flask. Note: Sodium sulfide nonahydrate is extremely hydroscopic. Make sure that the compound is dry before weighing (Excess moisture can be removed by rinsing the solid with a small amount of ether). Prepare weekly.

9.0 INTERFERENCES

9.1 Reduced sulfur compounds, which decompose in acid, such as sulfite and thiosulfate, may yield erratic results. Volatile iodine consuming substances will give high results. Oxidation may also affect sample results and samples should be taken with a minimum of aeration.

10.0 PROCEDURE

10.1 Below is a step by step procedure for analysis of samples for sulfide. The automated spreadsheet is used for documentation, calculation of standardization, and the analysis. The application can be found on the server. Before starting on the samples, standardize the iodine solution daily, using the following procedure.

10.1.1 Volumetrically pipet 5.00 ml of the Iodine solution into the beaker. Place the beaker on stir plate and add 2 to 4 mls of 6 N HCL to bring the PH< 2. Measure out 200 ml of DI water into beaker with a stir bar. This solution should now be an amber color.

10.1.2 Fill a burette with 0.025 N Sodium thiosulfate solution. Titrate the iodine solution with sodium thiosulfate until the amber color fades to yellow, then add enough starch solution to

obtain a blue color. Slowly continue to titrate until blue color disappears. Record the volume used. Repeat this procedure in duplicate.

10.1.3 Calculate the normality of the iodine as shown below. The iodine normality used should be the average of the normalities for the two standardizations of the DI water blanks.

$$\text{N of iodine sloution} = \frac{\text{(ml of Na thiosulfate) x (0.025)}}{\text{(5.00 ml,of iodine)}}$$

10.2 For soil samples, weigh out 25 g and dilute to 250 ml with DI water. Mix well. Let settle and filter through Whatman 934 AH or equivalent filter paper. Measure out 200 ml to use for analysis. Make sure to prepare a matrix spike, duplicate sample, a method blank, and a spike blank.

Note: The soil sample should be prepared immediately before the titration is to be done.

10.3 For water samples that **are not preserved with NaOH/zinc acetate,** mix and, measure out 200 ml of sample to use for analysis. Follow step 10.5.

10.4 For water samples that **are preserved with NaOH/zinc acetate,** mark the side of the original sample bottle at the level of the sample meniscus.

10.5 Pipet 5 ml of the iodine solution into a 400 ml beaker and add 2 to 4 mls of 6 N HCl (to bring the PH less than 2) for most soils and water samples that are not preserved with NaOH/Zinc Acetate, and 20 ml of 6N HCl for samples preserved with NaOH/Zinc Acetate. Swirl to mix. Gently transfer the contents of the original sample bottle to the beaker (or the 200 ml from step 10.3) with minimum agitation under the iodine surface. Rinse the sample bottle with adequate DI water to the beaker to make sure all zinc sulfides have been transferred to the beaker. **If the Iodine color disappears, add more Iodine immediately to the sample**. Add a stir bar and start stirring slowly, check the pH to be less than 2. Titrate the sample slowly with sodium thiosulfate solution until the solution changes to a lighter yellow (straw) color. Then add a small amount of starch solution and the solution should turn blue. Continue to titrate until the last bluish tint disappears and the solution appears clear.

10.6 Set up quality control samples for each batch, including a method blank, a spike blank, matrix spike and a duplicate sample. The spike blank and matrix spike should be spiked with 5 ml of 537 mg/l sulfide spiking solution (8.8). Follow step 10.5.

Note: For MS and SB use 20 ml of Iodine

10.7 For samples that are preserved with NaOH/ zinc acetate, measure sample volume used by filling the sample bottle with water to the line marked at the meniscus and then measuring the amount of water used in a class A graduated cylinder.

10.8 If interferences are suspected, the sample may be taken through a pretreatment step as described below:

10.8.1 If the sample was not treated with zinc acetate and sodium hydroxide, then shake the sample well and measure out 200 ml of sample. Add 0.30 ml of zinc acetate solution and 4 to 5 drops of 6N NaOH solution to bring the PH to above 9. Mix gently. Let the precipitate settle until the sample can be readily filtered. Follow 10.8.3.

10.8.2 If the sample was treated with zinc acetate and sodium hydroxide, verify the PH is above 9. mark the side of the original sample bottle at the level of the sample meniscus. Then follow 10.8.3. Additional zinc acetate may be added to ensure complete precipitation.

10.8.3 Filter the sample (200 ml from step 10.8.1 or the entire sample from step 10.8.2) through glass filter paper(Whatman 934AH or equivalent). Place the filter paper in the original sample container (i.e. the container to which the zinc acetate and NaOH were added), and add approximately 200 ml of DI water to the container and proceed with the titration starting at step 10.5. If a lower detection limit of 0.2 mg/l is requested, concentrate the sample by a factor of 10 to 1 (filter 1000 ml of sample and return to original battle with addition of 100 ml and titrate).

10.9 Calculations The calculations to be used are shown below

WATER $$\frac{\{ (Vi)\ (Ni) - (Vt)\ (Nt) \} \times 16000}{Vti} = \text{Mg Sulfide/L}$$

SOIL $$\frac{\{ (Vi)\ (Ni) - (Vt)\ (Nt) \} \times 16000 \times Vf}{Vti \times \text{sample weight in g} \times \%sol/100} = \text{Mg Sulfide/Kg}$$

Where Vi= Volume of iodine solution in Ml
Ni = Normality of iodine solution
Vt = Volume of sodium thiosulfate in Ml

Nt = Normality of sodium thiosulfate
Vti= Volume of sample titrated in Ml
Vf = Final volume of sample after preparation in Ml

11.0 QUALITY ASSURANCE

11.1 Below is a summary of the quality control requirements for this method. Make sure to check with the laboratory supervisor or manager for any additional client specific quality control requirements.

11.2 Method Blank. The laboratory must analyze method blank with each set of samples. A minimum of one method blank is required for every 10 samples. The method blank must not contain the analyte at more

than the reporting limit. If the method blank contains an analyte level over that limit, the samples must be reanalyzed.

11.3 Laboratory control sample/Spike Blank. The laboratory must analyze a spike blank with each set of samples. A minimum of one LCS (SB) is required for every 10 samples. In house limits should be generated once, sufficient data is available (usually a minimum of 20 to 30 analysis). If the lab control is outside of the control limits for a parameter, all samples must be reanalyzed. The exception is if the lab control recovery is high and the results of the samples to be reported are less than the reporting limits. In that case, the sample results can be reported with no flag. Note; If control limits are not available, then a default limits of 80 to 120 percent should be used.

11.4 An external QC sample (ICV) is analyzed every 10 samples. The acceptance criteria are 90-110% recovery. If the ICV is outside of the control limits, all samples must be reanalyzed. The exception is if the ICV recovery is high and the results for the samples are non-detected. In that case the sample results can be reported with no flag.

11.5 Matrix Spike. The Laboratory must add a known amount of each analyte to a minimum of 1 in 10 samples. The spike recovery should be assessed using in house limits. Until these limits can be generated, then default limits of 75 to 125 percent recovery should be applied. If insufficient sample is available to prepare a matrix spike, then blank spike may be substituted.

$$\frac{(\text{spiked sample Result} - \text{sample Result}) \times 100}{(\text{Amount spiked})} = \text{Ms Recovery}$$

11.6 Matrix duplicate. The laboratory must prepare a duplicate sample for a minimum of 1 in 10 samples. If insufficient sample is available to prepare a duplicate, then a duplicate blank spike maybe substituted. The RPD between the duplicate and the sample should be assessed. The duplicate RPD is calculated as shown below.

11.6.1 The duplicate RPD should be assessed using in house limits. Until these limits can be generated, then default limits of 20 percent should be applied. If a duplicates out of control, then the results should be flagged with the appropriate footnote. If the sample and the duplicate are less than 5 times the reporting limits and are within a range of ± the reporting limit, then the duplicate is considered to be in control.

11.6.2 The duplicate RPD should be calculated as shown below.

$$\frac{(\text{I Sample results} - \text{Duplicate results I})}{(\text{Sample results} + \text{Duplicate results})} = \%\text{RPD}$$

11.7 A Precision and accuracy (P&A) study is performed as an initial determination of capability, on an annual basis (continued demonstration of capability – a successful PT result may be used in place of a P&A for continued DOC), and if any significant changes have been made to the instrument. In general, 4 replicates or blank spikes are analyzed using the same procedures and conditions for sample analysis. The percent recoveries are compared to either default limits of 80-120% or in-house control limits once established. The standard deviation of the 4 replicate percent recoveries

are compared to either ±20 or to in-house limits once established. If percent recovery or standard deviation criteria are not met, corrective action must be taken to bring the system back into control.

11.8 Quality control data are generated at least on an annual basis by QA using an in-house program. Blank spike and MS/Dup data are pooled for the previous year (or other specified time frame) and the data is processed and evaluated by QA. The annual QC data is filed with QA.

12.0 DOCUMENTATION

12.1 If samples require reanalysis, a brief explanation of the reason should be documented.

12.2 The standard preparation logbook application must be completed for all standard preparations. All information requested must be completed; the page must be signed and dated by the respective person.

12.3 The Accutest lot number must be cross-referenced on the standard vial.

12.4 All laboratory logbooks must be reviewed and initialed or signed by the lab manager.

12.5 Any corrections to laboratory data must be done using a single line through the error. The initials of the person and date of correction must appear next to the correction.

13.0 DATA REVIEW

13.1 The analyst conducts the primary review of all data. This review begins with a check of all method quality control and progresses through sample quality control concluding with a check to assure that the client's requirements have been executed. The analyst has the authority and responsibility to perform corrective action for any out-of-control parameter of non-conformance.

13.2 A secondary review is performed by department managers, and it includes review of the data produced by their department. All manual calculations, QC criteria, and a comparison of the data package to client specified requirements are checked. The department manager may reject data, initiate reanalysis, take additional corrective action, or reprocess data.

13.3 The laboratory director performs a full tertiary review of the data package following its assembly. This review includes an evaluation of QC data against acceptance criteria and a check of the data package contents to assure that all analytical requirements and specifications were executed.

13.4 Spot-check reviews are performed by the Quality Assurance Officer focusing on all elements of the deliverable including the client's specifications and requirements, analytical quality control, sample custody documentation and sample identification.

14.0 DATA REPORTING

14.1 A results page including positive results and/or RLs, units, methodology, preparation and/or analysis dates, and data qualifiers are reported. Additional quality control data duplicate RPDs, blank spike recoveries, and method blank results may be reported upon request of the client. Additionally, raw data including any instrument printouts, laboratory logbooks, etc. may be reported to the client.

14.2 Data may be submitted to the client in a specified electronic format (EDD).

14.2 Once the data is approved by the laboratory manager, it may be accessed by clients via LabLink™.

14.3 Procedures for handling non-conforming data.

14.3.1 If quality control data does not meet criteria the non-conformance must be discussed in a case narrative and footnoted on the applicable quality control report summary.

14.3.2 If preservation or holding time criteria is not met and the samples are analyzed the result page must be footnoted with this information, and the non-conformance must be discussed in a case narrative or other suitable communication (telephone conversation log or email). Client notification documentation should be included with the data (telephone conversation log, fax, or email).

15.0 POLLUTION PREVENTION & WASTE MANAGEMENT

15.1 Pollution Prevention. Users of this method must perform all procedural steps that control the creation and/or escape of wastes of hazardous materials to the environment. The amounts of standards, reagents, and solvents must be limited to the amounts specified in this SOP. All safety practices designed to limit the escape of vapors, liquids, or solids to the environment must be followed. All method users must be familiar with the waste management practices described in section 15.2.

15.2 Waste Management. Individuals performing this method must follow established waste management procedures as described in the Sample and Waste Disposal SOP. This document describes the proper disposal of all waste materials generated during the testing of samples as follows:

15.2.1 Non-hazardous aqueous wastes
15.2.2 Hazardous aqueous wastes
15.2.3 Chlorinated organic solvents
15.2.4 Non-chlorinated organic solvents
15.2.5 Hazardous solid wastes
15.2.6 Non-hazardous solid wastes
15.2.7 Microbiological wastes

16.0 METHOD PERFORMANCE

16.1 Method performance is evaluated by the annual QC limits (control charts) generated by QA, and the annual MDL study results. Refer to section 3.0 for MDLs, and section 11 for QC limits.

17.0 ADDITIONAL REFERENCES

17.1 No additional references are required for this method.

Lab Manager: Brad Madadian

QA Officer: Robert Treggiari

TITLE: SULFIDE

TEST METHOD REFERENCE: 4500-S F . Standard Methods for the Examination of Water and Wastewater 21th Edition, 2005

REVISED SECTION: Section 8.0 notation

1.0 SCOPE & APPLICATION

1.1 This method is applicable to total and dissolved sulfides in drinking water and surface waters, sewage and industrial wastes. Acid insoluble sulfides are not measured by the use of this test.

1.2 A modification of this method is used to determine water- soluble sulfides in soil samples.

2.0 SUMMARY

2.1 Excess iodine is added to sample which may or may not have been treated with zinc acetate to produce zinc sulfide. The iodine oxidizes the sulfide to sulfur upon addition of HCL. The excess iodine is back titrated with sodium thiosulfate.

3.0 METHOD REPORTING LIMIT AND DETECTION LIMIT

3.1 The normal reporting limit for sulfide in waters is 2 mg/l and 4 mg/kg for soils.

3.2 The Method Detection Limit (MDL) represents the lowest reportable concentration of an individual analyte that meets the method qualitative identification criteria.

3.3 Method Detection limits (MDLs) are experimentally determined using the procedures described in 40 CFR, Part 136, Appendix B. Actual reported MDLs incorporate the sample volume analyzed and sample dilutions if needed, which may cause MDL variations from sample to sample.

3.4 In general, MDLs are determined through the analysis of at least 7 replicate blank spikes (using the same procedures for sample analysis). The MDL is calculated by multiplying the standard deviation of the replicate concentrations by the appropriate Student's t value (3.143 for 7 replicates). If more than 7 replicates are analyzed refer to 40 CFR, Part 136, Appendix B for the appropriate student's t value. MDLs are determined initially (prior to analysis), on an annual basis, and after major maintenance to equipment. MDL data is archived with Quality Assurance. Refer to the most recent study for current MDLs. For additional detail regarding MDL studies, refer to the MDL SOP MQA245.

3.5 The MDL represents the lowest reportable concentration of an individual analyte that meets the method qualitative identification criteria.

3.6 Current MDL studies are filed with Quality Assurance. Obsolete MDL studies are archived with the QA files. Electronic MDL data is found in the annual "MDL" folder on the QA server (LINUXMA1).

4.0 DEFINITIONS

4.1 ALIQUOT - a measured portion of a sample, or solution, taken for sample preparation and/or analysis.

4.2 BATCH – A group of samples which behave similarly with respect to the sampling or the testing procedures being employed and which are processed as a unit. For QC purposes, if the number of samples in a group is greater than 20 (or 10 for certain methods), then each group of 20 samples (or 10 samples for certain methods) or less will all be handled as a separate batch.

4.3 DRY WEIGHT – the weight of a sample based on percent solids. The weight after drying. See Percent Moisture.

4.4 CONTAMINATION - a component of a sample or an extract that is not representative of the environmental source of the sample. Contamination may stem from other samples, sampling equipment, while in transit, from laboratory reagents, laboratory environment, or analytical instruments.

4.5 FIELD SAMPLE - a portion of material to be analyzed that is contained in single or multiple containers and identified by a unique sample number.

4.6 FIELD BLANK – this is any sample that is submitted from the field and is identified as a blank. This includes trip blanks, rinsates, equipment blanks, etc.

4.7 HOLDING TIME – the elapsed time expressed most commonly in days from the date of sampling until the date of its analysis.

4.8 INSUFFICIENT QUANTITY - when there is not enough volume (water sample) or weight (soil/sediment) to perform any of the required operations: sample analysis or extraction, percent moisture, MS/DUP, etc.

4.9 MATRIX: The component or substrate (e.g., water, soil) which contains the analyte of interest.

4.10 Matrix Spike: The laboratory must add a known amount of each analyte to a minimum of 1 in 10 samples. The matrix spike recovery is calculated and assessed against the control limits that are generated in house. If control limits are not available, then a default limits of 75 to 125 percent should be applied.

4.11 MATRIX DUPLICATE: A duplicate sample is analyzed at a minimum of 1 in 10 samples. The relative percent difference (RPD) between the duplicate and the sample should be assessed. The duplicate

RPD is calculated as shown below. Assess laboratory performance against the control limits. In house limits are generated once sufficient duplicate data is available to generate limits (usually a minimum of 20 to 30 analyses). If a duplicate is out of control, flag the results with the appropriate footnote. If the sample and the duplicate are less than 5 times the reporting limits and are within a range of ± the reporting limit, then the duplicate is considered to be in control. Note: If control limits are not specified, use default limits of ± 20% RPD.

$$\frac{(|\text{Sample Result} - \text{Duplicate Result}|) \times 100}{(\text{Sample Result} + \text{Duplicate Result})/2} = \text{Duplicate RPD}$$

4.12 METHOD BLANK. The laboratory must analyze a method blank with each set of samples. A minimum of one method blank is required for every 10 samples. The method blank must contain the parameter of interest at levels of less that the reporting limit for that parameter. If the method blank contains levels over the reporting limits, the samples must be reanalyzed. The exception to this rule is when the samples to be reported contain greater than 10 times the method blank level. In addition, if all the samples are less than a client required limit and the method blank is also less than that limit, then the results can be reported as less than that limit.

4.13 PERCENT MOISTURE - an approximation of the amount of water in a soil/sediment sample made by drying an aliquot of the sample at 105 °C. The percent moisture determined in this manner also includes contributions from all compounds that may volatilize at or below 105 °C, including water. Percent moisture may be determined from decanted samples and from samples that are not decanted.

4.14 RELATIVE PERCENT DIFFERENCE (RPD) - To compare two values, the relative percent difference is based on the mean of the two values, and is reported as an absolute value, i.e., always expressed as a positive number or zero. In contrast, see percent difference.

4.15 REAGENT GRADE: Analytical reagent (AR) grade, ACS reagent grade, and reagent grade are synonymous terms for reagents that conform to the current specifications of the Committee on Analytical Reagents of the American Chemical Society.

4.16 REAGENT WATER: Water that has been generated by any method which would achieve the performance specifications for ASTM Type II water. Water in which an interferant is not observed at or above the minimum quantitation limit of the parameters of interest. Accutest uses deionized water (municipal water which passes through Accutest's DI treatment system).

4.17 REFERENCE MATERIAL: A material containing known quantities of target analytes in solution or in a homogeneous matrix. It is used to document the bias of the analytical process.

4.18 SPIKE BLANK OR LAB CONTROL SAMPLE. Analyze a laboratory control sample or spike blank with each set of samples. A minimum of one lab control sample or spike blank is required for every 10 samples. Assess laboratory performance against the control limits. In house limits should also be generated once sufficient data is available to generate limits (usually a minimum of 20 to 30 analyses). If the lab control is outside of the control limits for a parameter, all samples must be reanalyzed for that parameter. The exception is if the lab control recovery is high and the results of

the samples to be reported are less than the reporting limit. In that case, the sample results can be reported with no flag. Note: If control limits are not specified, then default limits of 80 to 120 percent should be used.

5.0 HEALTH & SAFETY

5.1 All safety practices must be followed as outlined in the Accutest Laboratories Employee Safety Handbook and Chemical Hygiene Plan. Safety glasses, gloves, and lab coats must be worn. All samples, solutions, and extracts must be treated as unknown and potentially hazardous.

5.2 The toxicity or carcinogenicity of each reagent used in this method has not been precisely determined; however, each chemical should be treated as a potential health hazard. Exposure to these reagents should be reduced to the lowest possible level.

6.0 COLLECTION, PRESERVATION, AND HOLDING TIMES

6.1 Water samples should be collected in two 250-300 ml containers. Soil samples should be collected in one 250-300ml glass container.

6.2 Water samples should be preserved with zinc acetate and sodium hydroxide. Water and soil samples should be kept under refrigeration at 4° ± 2° C until they are analyzed.

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NOTE: All chemicals listed below are reagent grade unless otherwise specified. Distilled, deionized water should be used whenever water is required. All applicable standard/reagent preparation information, including vendor, lot number, date of preparation, calculations, and initials must be entered in the appropriate standard/reagent preparation logbook. Vendors typically used by Accutest include Fisher Scientific, VWR, Accustandard, Absolute Standards, Supelco, Chemservices, Ultra, and ERA. Additional vendors may be utilized as necessary.

8.1 Hydrochloric Acid, HCL, (6 N). Add 100 ml of concentrated HCL to 100 ml of DI water. Cool, mix.

8.2 Standard Iodine Solution (0.025 N). Dissolve 20 to 25g of anhydrous potassium iodide (KI) and 3.2 g of iodine in 400 ml of DI water and dilute to 1000 ml in a volumetric flask. Standardize this solution against 0.025 N sodium thiosulfate.

8.3 Sodium Thiosulfate solution (0.025N). Dissolve 6.205 g of Na2S2O3.5H2O in approximately 500 ml of DI water. Add 9 ml of 1 N NaOH and dilute to a final volume of 1000 ml in a volumetric flask. Note: this solution is commercially available. Request the certificate of the analysis from the vendor and keep in file.

8.4 Starch Indicator solution. This solution is also commercially available.

8.5 Zinc acetate solution. Dissolve 220 g of zinc acetate (Zn(C2H3O2)2)2H2O in 870 ml of DI water.

8.6 Sodium Hydroxide, (6N). Dissolve 24 g of sodium hydroxide in 50 ml of DI water. Dilute to 100 ml with DI water.

8.7 Sodium Hydroxide, (1N). Dissolve 40 g of sodium hydroxide in 500 ml of DI water. Dilute to 1000 ml with DI water.

8.8 Sulfide 537 mg/L Stock Solution. Dissolve 4.02 g of sodium sulfide nonahydrate, Na2S.9H2O, in approximately 980 ml of DI water. Adjust the PH of this solution to >9 and < 11 with 1 N sodium hydroxide solution (8.7). Dilute to 1000 ml with DI water in a volumetric flask. Note: Sodium sulfide nonahydrate is extremely hydroscopic. Make sure that the compound is dry before weighing (Excess moisture can be removed by rinsing the solid with a small amount of ether). Prepare weekly.

9.0 INTERFERENCES

9.1 Reduced sulfur compounds, which decompose in acid, such as sulfite and thiosulfate, may yield erratic results. Volatile iodine consuming substances will give high results. Oxidation may also affect sample results and samples should be taken with a minimum of aeration.

10.0 PROCEDURE

10.1 Below is a step by step procedure for analysis of samples for sulfide. The automated spreadsheet is used for documentation, calculation of standardization, and the analysis. The application can be found on the server. Before starting on the samples, standardize the iodine solution daily, using the following procedure.

10.1.1 Volumetrically pipet 5.00 ml of the Iodine solution into the beaker. Place the beaker on stir plate and add 2 to 4 mls of 6 N HCL to bring the PH< 2. Measure out 200 ml of DI water into beaker with a stir bar. This solution should now be an amber color.

10.1.2 Fill a burette with 0.025 N Sodium thiosulfate solution. Titrate the iodine solution with sodium thiosulfate until the amber color fades to yellow, then add enough starch solution to

obtain a blue color. Slowly continue to titrate until blue color disappears. Record the volume used. Repeat this procedure in duplicate.

10.1.3 Calculate the normality of the iodine as shown below. The iodine normality used should be the average of the normalities for the two standardizations of the DI water blanks.

$$\text{N of iodine sloution} = \frac{\text{(ml of Na thiosulfate) x (0.025)}}{\text{(5.00 ml,of iodine)}}$$

10.2 For soil samples, weigh out 25 g and dilute to 250 ml with DI water. Mix well. Let settle and filter through Whatman 934 AH or equivalent filter paper. Measure out 200 ml to use for analysis. Make sure to prepare a matrix spike, duplicate sample, a method blank, and a spike blank.

Note: The soil sample should be prepared immediately before the titration is to be done.

10.3 For water samples that **are not preserved with NaOH/zinc acetate,** mix and, measure out 200 ml of sample to use for analysis. Follow step 10.5.

10.4 For water samples that **are preserved with NaOH/zinc acetate,** mark the side of the original sample bottle at the level of the sample meniscus.

10.5 Pipet 5 ml of the iodine solution into a 400 ml beaker and add 2 to 4 mls of 6 N HCl (to bring the PH less than 2) for most soils and water samples that are not preserved with NaOH/Zinc Acetate, and 20 ml of 6N HCl for samples preserved with NaOH/Zinc Acetate. Swirl to mix. Gently transfer the contents of the original sample bottle to the beaker (or the 200 ml from step 10.3) with minimum agitation under the iodine surface. Rinse the sample bottle with adequate DI water to the beaker to make sure all zinc sulfides have been transferred to the beaker. **If the Iodine color disappears, add more Iodine immediately to the sample**. Add a stir bar and start stirring slowly, check the pH to be less than 2. Titrate the sample slowly with sodium thiosulfate solution until the solution changes to a lighter yellow (straw) color. Then add a small amount of starch solution and the solution should turn blue. Continue to titrate until the last bluish tint disappears and the solution appears clear.

10.6 Set up quality control samples for each batch, including a method blank, a spike blank, matrix spike and a duplicate sample. The spike blank and matrix spike should be spiked with 5 ml of 537 mg/l sulfide spiking solution (8.8). Follow step 10.5.

Note: For MS and SB use 20 ml of Iodine

10.7 For samples that are preserved with NaOH/ zinc acetate, measure sample volume used by filling the sample bottle with water to the line marked at the meniscus and then measuring the amount of water used in a class A graduated cylinder.

10.8 If interferences are suspected, the sample may be taken through a pretreatment step as described below:

10.8.1 If the sample was not treated with zinc acetate and sodium hydroxide, then shake the sample well and measure out 200 ml of sample. Add 0.30 ml of zinc acetate solution and 4 to 5 drops of 6N NaOH solution to bring the PH to above 9. Mix gently. Let the precipitate settle until the sample can be readily filtered. Follow 10.8.3.

10.8.2 If the sample was treated with zinc acetate and sodium hydroxide, verify the PH is above 9. mark the side of the original sample bottle at the level of the sample meniscus. Then follow 10.8.3. Additional zinc acetate may be added to ensure complete precipitation.

10.8.3 Filter the sample (200 ml from step 10.8.1 or the entire sample from step 10.8.2) through glass filter paper(Whatman 934AH or equivalent). Place the filter paper in the original sample container (i.e. the container to which the zinc acetate and NaOH were added), and add approximately 200 ml of DI water to the container and proceed with the titration starting at step 10.5. If a lower detection limit of 0.2 mg/l is requested, concentrate the sample by a factor of 10 to 1 (filter 1000 ml of sample and return to original battle with addition of 100 ml and titrate).

10.9 Calculations The calculations to be used are shown below

WATER $$\frac{\{ (Vi)\ (Ni) - (Vt)\ (Nt) \} \times 16000}{Vti} = \text{Mg Sulfide/L}$$

SOIL $$\frac{\{ (Vi)\ (Ni) - (Vt)\ (Nt) \} \times 16000 \times Vf}{Vti \times \text{sample weight in g} \times \%sol/100} = \text{Mg Sulfide/Kg}$$

Where Vi= Volume of iodine solution in Ml
Ni = Normality of iodine solution
Vt = Volume of sodium thiosulfate in Ml

Nt = Normality of sodium thiosulfate
Vti= Volume of sample titrated in Ml
Vf = Final volume of sample after preparation in Ml

11.0 QUALITY ASSURANCE

11.1 Below is a summary of the quality control requirements for this method. Make sure to check with the laboratory supervisor or manager for any additional client specific quality control requirements.

11.2 Method Blank. The laboratory must analyze method blank with each set of samples. A minimum of one method blank is required for every 10 samples. The method blank must not contain the analyte at more

than the reporting limit. If the method blank contains an analyte level over that limit, the samples must be reanalyzed.

11.3 Laboratory control sample/Spike Blank. The laboratory must analyze a spike blank with each set of samples. A minimum of one LCS (SB) is required for every 10 samples. In house limits should be generated once, sufficient data is available (usually a minimum of 20 to 30 analysis). If the lab control is outside of the control limits for a parameter, all samples must be reanalyzed. The exception is if the lab control recovery is high and the results of the samples to be reported are less than the reporting limits. In that case, the sample results can be reported with no flag. Note; If control limits are not available, then a default limits of 80 to 120 percent should be used.

11.4 An external QC sample (ICV) is analyzed every 10 samples. The acceptance criteria are 90-110% recovery. If the ICV is outside of the control limits, all samples must be reanalyzed. The exception is if the ICV recovery is high and the results for the samples are non-detected. In that case the sample results can be reported with no flag.

11.5 Matrix Spike. The Laboratory must add a known amount of each analyte to a minimum of 1 in 10 samples. The spike recovery should be assessed using in house limits. Until these limits can be generated, then default limits of 75 to 125 percent recovery should be applied. If insufficient sample is available to prepare a matrix spike, then blank spike may be substituted.

$$\frac{(\text{spiked sample Result} - \text{sample Result}) \times 100}{(\text{Amount spiked})} = \text{Ms Recovery}$$

11.6 Matrix duplicate. The laboratory must prepare a duplicate sample for a minimum of 1 in 10 samples. If insufficient sample is available to prepare a duplicate, then a duplicate blank spike maybe substituted. The RPD between the duplicate and the sample should be assessed. The duplicate RPD is calculated as shown below.

11.6.1 The duplicate RPD should be assessed using in house limits. Until these limits can be generated, then default limits of 20 percent should be applied. If a duplicates out of control, then the results should be flagged with the appropriate footnote. If the sample and the duplicate are less than 5 times the reporting limits and are within a range of ± the reporting limit, then the duplicate is considered to be in control.

11.6.2 The duplicate RPD should be calculated as shown below.

$$\frac{(\text{I Sample results} - \text{Duplicate results I})}{(\text{Sample results} + \text{Duplicate results})} = \%\text{RPD}$$

11.7 A Precision and accuracy (P&A) study is performed as an initial determination of capability, on an annual basis (continued demonstration of capability – a successful PT result may be used in place of a P&A for continued DOC), and if any significant changes have been made to the instrument. In general, 4 replicates or blank spikes are analyzed using the same procedures and conditions for sample analysis. The percent recoveries are compared to either default limits of 80-120% or in-house control limits once established. The standard deviation of the 4 replicate percent recoveries

are compared to either ±20 or to in-house limits once established. If percent recovery or standard deviation criteria are not met, corrective action must be taken to bring the system back into control.

11.8 Quality control data are generated at least on an annual basis by QA using an in-house program. Blank spike and MS/Dup data are pooled for the previous year (or other specified time frame) and the data is processed and evaluated by QA. The annual QC data is filed with QA.

12.0 DOCUMENTATION

12.1 If samples require reanalysis, a brief explanation of the reason should be documented.

12.2 The standard preparation logbook application must be completed for all standard preparations. All information requested must be completed; the page must be signed and dated by the respective person.

12.3 The Accutest lot number must be cross-referenced on the standard vial.

12.4 All laboratory logbooks must be reviewed and initialed or signed by the lab manager.

12.5 Any corrections to laboratory data must be done using a single line through the error. The initials of the person and date of correction must appear next to the correction.

13.0 DATA REVIEW

13.1 The analyst conducts the primary review of all data. This review begins with a check of all method quality control and progresses through sample quality control concluding with a check to assure that the client's requirements have been executed. The analyst has the authority and responsibility to perform corrective action for any out-of-control parameter of non-conformance.

13.2 A secondary review is performed by department managers, and it includes review of the data produced by their department. All manual calculations, QC criteria, and a comparison of the data package to client specified requirements are checked. The department manager may reject data, initiate reanalysis, take additional corrective action, or reprocess data.

13.3 The laboratory director performs a full tertiary review of the data package following its assembly. This review includes an evaluation of QC data against acceptance criteria and a check of the data package contents to assure that all analytical requirements and specifications were executed.

13.4 Spot-check reviews are performed by the Quality Assurance Officer focusing on all elements of the deliverable including the client's specifications and requirements, analytical quality control, sample custody documentation and sample identification.

14.0 DATA REPORTING

14.1 A results page including positive results and/or RLs, units, methodology, preparation and/or analysis dates, and data qualifiers are reported. Additional quality control data duplicate RPDs, blank spike recoveries, and method blank results may be reported upon request of the client. Additionally, raw data including any instrument printouts, laboratory logbooks, etc. may be reported to the client.

14.2 Data may be submitted to the client in a specified electronic format (EDD).

14.2 Once the data is approved by the laboratory manager, it may be accessed by clients via LabLink™.

14.3 Procedures for handling non-conforming data.

14.3.1 If quality control data does not meet criteria the non-conformance must be discussed in a case narrative and footnoted on the applicable quality control report summary.

14.3.2 If preservation or holding time criteria is not met and the samples are analyzed the result page must be footnoted with this information, and the non-conformance must be discussed in a case narrative or other suitable communication (telephone conversation log or email). Client notification documentation should be included with the data (telephone conversation log, fax, or email).

15.0 POLLUTION PREVENTION & WASTE MANAGEMENT

15.1 Pollution Prevention. Users of this method must perform all procedural steps that control the creation and/or escape of wastes of hazardous materials to the environment. The amounts of standards, reagents, and solvents must be limited to the amounts specified in this SOP. All safety practices designed to limit the escape of vapors, liquids, or solids to the environment must be followed. All method users must be familiar with the waste management practices described in section 15.2.

15.2 Waste Management. Individuals performing this method must follow established waste management procedures as described in the Sample and Waste Disposal SOP. This document describes the proper disposal of all waste materials generated during the testing of samples as follows:

15.2.1 Non-hazardous aqueous wastes
15.2.2 Hazardous aqueous wastes
15.2.3 Chlorinated organic solvents
15.2.4 Non-chlorinated organic solvents
15.2.5 Hazardous solid wastes
15.2.6 Non-hazardous solid wastes
15.2.7 Microbiological wastes

16.0 METHOD PERFORMANCE

16.1 Method performance is evaluated by the annual QC limits (control charts) generated by QA, and the annual MDL study results. Refer to section 3.0 for MDLs, and section 11 for QC limits.

17.0 ADDITIONAL REFERENCES

17.1 No additional references are required for this method.

ACCUTEST LABS
STANDARD OPERATING PROCEDURE

Lab Manager: Brad Madadian

QA Officer: Robert Treggiari

TITLE: CHLORIDE

TEST METHOD REFERENCE: 4500 Cl C. Standard Methods for the Examination of Water and Wastewater 21th Edition, 2005

TEST CODES: CHL

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Revised Sections: ***Test Method Ref., 10.1; 10.2***

1.0 SCOPE AND APPLICATION

1.1 This method is applicable to the determination of chloride in surface waters, saline waters, and domestic and industrial wastes. A modification of this method can be used to determine soluble chloride in soil samples.
Note: This method should not be used for drinking waters.

2.0 SUMMARY OF METHOD

2.1 Sample is titrated with mercuric nitrate in the presence of mixed diphenylcarbazone-xylene cyanol FF indicator with sample pH adjusted to 2.5+/-0.1 by HNO3. The end point of the titration is the formation of the blue-violet mercury diphenylcarbazone complex.

3.0 REPORTING LIMIT AND METHOD DETECTION LIMIT

3.1 Reporting Limit. The Reporting Limit (RL) is based on the lowest calibration standard. RL'S may vary depending on matrix difficulties and sample volumes or weight and percent moisture.

3.2 The reporting limit for this method has been established at 1.0 mg/l for waters and 10 mg/l for soils.

3.3 Method Detection Limits

3.3.1 Detection limits are experimentally determined using the procedures described in 40 CFR, Part 136, Appendix B. Actual reported MDLs incorporate the sample weight or volume analyzed and sample dilutions if needed, which may cause MDL variations from sample to sample.

3.3.2 In general, MDLs are determined through the analysis of at least 7 replicate blank spikes (using the same procedures for sample analysis). The MDL is calculated by multiplying the standard deviation of the replicate concentrations by the appropriate Student's t value (3.143 for 7 replicates). If more than 7 replicates are analyzed refer to 40 CFR, Part 136, Appendix B for the appropriate student's t value. MDL studies are performed on an annual basis or after any major changes to the

instrumentation. For additional detail regarding MDL studies, refer to the MDL SOP MQA245.

3.3.3 The MDL represents the lowest reportable concentration of an individual analyte that meets the method qualitative identification criteria.

3.3.4 Current MDL studies are filed with Quality Assurance. Obsolete MDL studies are archived with the QA files. Electronic MDL data is found in the annual "MDL" folder on the QA server (LINUXMA1).

4.0 DEFINITIONS

BATCH: A group of samples which behave similarly with respect to the sampling or the testing procedures being employed and which are processed as a unit. For QC purposes, if the number of samples in a group is greater than 20, then each group of 20 samples or less will all be handled as a separate batch.

EXTERNAL CHECK STANDARD. The external check standard is a standard from a separate source to verify the accuracy of the analysis. An external check must be run a minimum of once per quarter for all analyses where a check is commercially available. The laboratory should assess laboratory performance of a external check standard using the control limits generated by the external check supplier. If the external check is outside of the control limits for a given parameter, all samples must be reanalyzed for that parameter after the problem has been resolved.

SPIKE BLANK OR LAB CONTROL SAMPLE. Prepare and analyze a laboratory control sample or spike blank with each set of samples. A minimum of one lab control sample or spike blank is required for every 20 samples. For a running batch, a new spike blank is required for each different analysis day. In house limits should also be generated once sufficient data is available to generate limits (usually a minimum of 20 to 30 analyses). If the lab control is outside of the control limits for a parameter, all samples must be reanalyzed for that parameter. The exception is if the lab control recovery is high and the results of the samples to be reported are less than the reporting limit. In that case, the sample results can be reported with no flag. Note: If control limits are not available, then default limits of 80 to 120 percent should be used.

MATRIX: The component or substrate (e.g., water, soil) which contains the analyte of interest.

MATRIX SPIKE: Aliquot of matrix (water or solid) 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 the recovery. A matrix spike sample is analyzed at a minimum of 1 in 20 samples. The percent recovery of matrix spike should be assessed. In house limits are generated once sufficient data is available to generate limits (usually a minimum of 20 to 30 analysis).

MATRIX DUPLICATE: A duplicate sample is digested at a minimum of 1 in 20 samples. The relative percent difference (RPD) between the duplicate and the sample should be assessed. The duplicate RPD is calculated as shown below. In house limits are generated once sufficient duplicate data is available to generate limits (usually a minimum of 20 to 30 analyses). If a duplicate is out of control, flag the results with the appropriate footnote. If the sample and the duplicate are less than

5 times the reporting limits and are within a range of ± the reporting limit, then the duplicate is considered to be in control. Note: If control limits are not available, use default limits of ± 20% RPD.

$$\frac{(\,|\,\text{Sample Result} - \text{Duplicate Result}\,|\,) \times 100}{(\text{Sample Result} + \text{Duplicate Result})/2} = \text{Duplicate RPD}$$

METHOD BLANK. The laboratory must prepare and analyze a method blank with each set of samples. A minimum of one method blank is required for every 20 samples. For a running batch, a new method blank is required for each different analysis day. If no digestion/extraction step is required, then the method blank is equivalent to the reagent blank. The method blank must contain the parameter of interest at levels of less that the reporting limit for that parameter. If the method blank contains levels over the reporting limits, the samples must be re-prepped and reanalyzed. The exception to this rule is when the samples to be reported contain greater than 10 times the method blank level. In addition, if all the samples are less than a client required limit and the method blank is also less than that limit, then the results can be reported as less than that limit.

METHOD DETECTION LIMITS (MDLS). MDLs should be established for all appropriate methods, using a solution spiked at approximately 2-5 times the estimated detection limit. To determine the MDL values, take seven replicate aliquots of the spiked sample and process through the entire analytical method. The MDL is calculated by multiplying the standard deviation of three replicate analyses by 3.14, which is the student's t value for a 99% confidence level. MDLs should be determined approximately once per year for frequently analyzed parameters.

REAGENT GRADE: Analytical reagent (AR) grade, ACS reagent grade, and reagent grade are synonymous terms for reagents which conform to the current specifications of the Committee on Analytical Reagents of the American Chemical Society.

REAGENT WATER: Water that has been generated by any method which would achieve the performance specifications for ASTM Type II water. - water in which an interferant is not observed at or above the minimum quantitation limit of the parameters of interest. Accutest uses deionized water (municipal water which passes through Accutest's DI treatment system).

REFERENCE MATERIAL: A material containing known quantities of target analytes in solution or in a homogeneous matrix. It is used to document the bias of the analytical process.

5.0 HEALTH & SAFETY

5.1 The analyst should follow normal safety procedures as outlined in the Accutest Laboratory Employee Safety Manual and Chemical Hygiene Plan which includes the use of safety glasses and lab coats. In addition, all acids are corrosive and should be handled with care. Flush spills with plenty of water. If acids contact any part of the body, flush with water and contact the supervisor

5.2 The toxicity or carcinogenicity of each reagent used in this method has not been precisely determined; however, each chemical should be treated as a potential health hazard. Exposure to these reagents should be reduced to the lowest possible level. The laboratory is

responsible for maintaining a current awareness file of OSHA regulations regarding the safe handling of the chemicals specified in this method. A reference file of data handling sheets should be made available to all personnel involved in these analyses.

6.0 PRESERVATION & HOLDING TIME

6.1 The samples should be stored at 4^0 C $\pm$ 2°C.

6.2 The samples should be analyzed within 28 days of the date of collection.

7.0 INTERFERENCES

7.1 Anions and cations at concentrations normally found in surface waters do not interfere, although bromide and iodide are titrated with mercuric nitrate in the same manner as chloride. Chromate, ferric, and sulfite ions interfere when present in concentrations greater than 10 mg/l. The sulfite interference can be eliminated by oxidizing 50 ml of the sample solution with hydrogen peroxide

8.0 APPARATUS

8.1 Micro-burette
8.2 Erlenmeyer flasks or beakers.
8.3 Stirring plate.
8.4 Stir bars.
8.5 pH Paper

9.0 REAGENTS

9.1 Standard Sodium chloride, 0.0141 N: Dissolve 824.0 mg of sodium chloride that has been dried at 140^0 C for 1 hour in DI water in a 1 liter volumetric flask and dilute to a final volume of 1 liter with DI water. **Note:** this solution can also be purchased.

9.2 0.1 N, Nitric acid solution. Add 6.4 ml of concentrated nitric acid to DI water in a 1000 ml volumetric flask and dilute to the final volume with DI water.

9.3 0.1 N, Sodium hydroxide solution. Dissolve 4.0 g of sodium hydroxide in approximately 800 ml of DI water and dilute to a final volume of 1000 ml with DI water.

9.4 Mercuric nitrate titrant (0.0141 N): Dissolve 2.42 g of mercuric nitrate ($Hg(NO_3)2.H_2O$) in 25 ml of DI water acidified with 0.25 ml of concentrated nitric acid. Dilute to a final volume of 1 liter with DI water. Filter if necessary. Standardize against standard sodium chloride solution (9.1) using the same procedure outlined below to determine chloride concentrations. Adjust the titrant to 0.0141 N. This should be stored in a dark bottle. **Note;** This solution can be purchased. Make sure if purchased, to receive and file the certificate of tracability since this standard has been standardized against a primary standard. Use the normality provided with this certificate for calculation.

9.5 Acidic mixed Indicator reagent: Dissolve, in the order named, 0.250 g of s-diphenylcarbazone, 4 ml conc HNO3, and 30 mg xylene cyanol FF in 100 ml of 95% ethyl alcohol or isopropyl alcohol. Store in a dark bottle in the refrigerator for a maximum of 1 months.

Note: Deterioration causes a slow end point and high results.

9.6 Non-acidic mixed Indicator reagent: Dissolve, in the order named, 0.250 g of s-diphenylcarbazone, and 30 mg xylene cyanol FF in 100 ml of 95% ethyl alcohol or isopropyl alcohol. Store in a dark bottle in the refrigerator for a maximum of 1 months.

9.7 Sodium Chloride Spiking Solution A (2000 mg/L): Dissolve 3.296 g of sodium chloride that has been dried at 600^0 C for 1 hour in DI water in a 1 liter volumetric flask and dilute to a final volume of 1 liter with DI water.

9.8 Sodium Chloride spiking solution B (250 mg/L): Dilute 25 ml of solution 9.7 in 200 ml of DI water.

9.9 All applicable standard/reagent preparation information, including vendor, lot number, date of preparation, calculations, and initials must be entered in the appropriate standard/reagent preparation logbook. Vendors typically used by Accutest include Fisher Scientific, VWR, Accustandard, Supelco, Chemservices, Ultra, and ERA. Additional vendors may be utilized as necessary.

10.0 PROCEDURE

Below is a step by step procedure for the analysis of samples for CHL. Use the automated spreadsheet for documentation, calculations of Standardization, and the analysis. This application can be found on server.

Note: Make sure that the mercuric nitrate titration solution has been standardized before starting this procedure. If the certificate of analysis is not provided, standardize in replicates containing 5 ml of of standard NaCl (9.1) and 10 mg sodium bicarbonate(NaHCO3) diluted to 100 ml with DI water. Titrate following the procedures outlined in steps 10.2 through 10.5 below using the 100 ml of standard.

10.1 For soil samples, homogenize sample and do not include any large rocks or debris in sample aliquot. Proceed to weigh out 15.0 g of sample and add 150 ml of DI water. Stir or tumble samples for 1 hour. Filter the sample through GFF filter paper and continue with steps 10.2 through 10.5 below.

10.2 Measure 50 ml aliquots of sample or sample filtrate for soils, and two more aliquots for sample duplicate and matrix spike. For the method blank and spike blank 50 ml of DI water. Spike the matrix spike sample and the blank spike with 2.00 ml of 250 mg/L chloride spiking solution. For soil samples; spike the matrix spike sample and the blank spike with 6.00ml of 250mg/L chloride solution (B).

Note: **Samples containing greater than 100 mg/l of chloride should be analyzed with dilutions.**

10.3 Add 1 ml of acidic mixed indicator reagent (9.5) and a stirring bar to the sample and mix well. The color of the solution must turn to green-blue at this point. For most potable waters, the pH will be 2.5 +/- 0.1 upon addition of this indicator.

10.3.1 A light green indicates pH less than 2.0,

10.3.2 A pure blue indicates pH more than 3.8

10.4 Because the pH control is critical, adjust the pH of highly alkaline or acid samples to 2.5+/- 0.1 with 0.1 N HNO3, or 0.1 N NaOH. Determine the amount of acid or alkali required to obtain the above pH and discard this sample portion. Treat a separate sample portion with the determined amount of acid or alkali and continue analysis using the non acidic mixed indicator (9.6).

10.5 Titrate the sample with 0.0141N mercuric nitrate while stirring. The end point is reached when a blue-violet color persists throughout the solution.

10.6 If the sample is above 100 mg/l of chloride, dilute the sample and analyze the dilution as described in 10.2 through 10.5 above.

11.0 CALCULATION

11.1 Water samples should be calculated using the equation shown below.

$$\text{chloride in mg/l} = \frac{(B - A) \times N \times 35450}{\text{ml of sample}}$$

11.2 Soil samples should be calculated using the equation shown below.

$$\text{chloride in mg/kg} = \frac{(B - A) \times N \times 35450 \times \text{final volume}}{(\text{g of sample})(\%\text{sol}/100)}$$

12.0QC REQUIREMENTS

12.1 Below is a summary of the quality control requirements for this method. Make sure to check with the laboratory supervisor or manager for any additional client specific quality control requirements.

12.2 Method Detection Limits (MDLs). MDLs should be established using a solution spiked at approximately 3-5 times the estimated detection limit. To determine the MDL values, take seven replicate aliquots of the spiked sample and process through the entire analytical method. The MDL is calculated by multiplying the standard deviation of the replicate

analyses by 3.14, which is the student's t value for a 99% confidence level . MDLs should be determined approximately once per year.

12.3 Method Blank. The laboratory must analyze a method blank with each set of samples. A minimum of one method blank is required for every 20 samples. For a running batch, a new method blank is required for each different analysis day. The method blank must contain the analyte at less that the reporting limit. If the method blank contains an analyte level over that limit, the samples must be reanalyzed.

12.4 Spike Blank. The laboratory must analyze a spike blank with each set of samples. A minimum of one spike blank is required for every 20 samples. The net recovery should be within 20 percent of the true value.

12.5 External Check Sample. The laboratory must analyze an external check standard at least once per month. It is recommended that this be analyzed with each batch or when available. The recovery should be assessed, the limits supplied by the external check manufacturer should be applied.

12.6 Matrix Duplicate. The laboratory must prepare a duplicate sample for a minimum of 1 in 20 samples. The relative percent difference (rpd) between the duplicate and the sample should be assessed. The duplicate rpd is calculated as shown below.

12.6.1 The duplicate RPD should be assessed using in house limits. Until these limits can be generated, then default limits of 20 percent RPD should be applied. If a duplicate is out of control, then the results should be flagged with the appropriate footnote. If the sample and the duplicate are less than 5 times the reporting limits and are within a range of $\pm$ the reporting limit, then the duplicate is considered to be in control.

12.6.2 The duplicate RPD should be calculated as shown below.

$$\frac{(\text{Sample Result} - \text{Duplicate Result}) \times 100}{(\text{Sample Result} + \text{Duplicate Result}) \times 0.5} = \% \text{ RPD}$$

12.7 Matrix Spike. The laboratory must add a known amount of each analyte to a minimum of 1 in 20 samples. The spike recovery should be assessed using in house limits. Until these limits can be generated, default limits of 75-125% recovery should be applied. If a matrix spike recovery is out of control, then the recovery should be flagged with the appropriate footnotes. If the matrix spike amount is less than one fourth of the sample amount, then the sample can be assessed against the control limits and should be footnoted to that effect.

$$\frac{(\text{Matrix Spike Result} - \text{Original Sample Result}) \times 100}{\text{Amount of Spike}}$$

12.8 Prior to running samples, the laboratory must demonstrate initial proficiency by generating data of acceptable accuracy and precision (P&A study) for target analyte in a clean matrix. This procedure must be repeated on an annual basis, whenever new staff are trained, or when significant changes in instrumentation are made.

12.8.1 Four blank spikes are prepared and analyzed using the same procedures and conditions as samples. Calculate the average recovery and standard deviation of the recoveries of the analytes in each of the four QC samples. Until in-house limits are established for initial and annual demonstration of capabilities, use the recoveries of 80-120% as guidance for evaluating the results.

12.9 Quality control data are generated at least on an annual basis by QA using an in-house program. Blank spike and MS/Dup data are pooled for the previous year (or other specified time frame) and the data is processed and evaluated by QA. The annual QC data is filed with QA.

13.0 DOCUMENTATION

13.1 The Standard preparation log application must be completed for all standard preparations. All information requested must be completed.

13.2 The Accutest lot number must be cross-referenced on the standard vial/container.

13.3 Any comments or observations concerning the sample that may influence the analytical procedure.

13.4 Any corrections to laboratory data must be done using a single line through the error. The initials of the person and date of corrections must appear next to the correction.

13.5 All laboratory logs must be reviewed and initialed or signed by the lab manager.

14.0 DATA REVIEW

14.1 The analyst conducts the primary review of all data. This review begins with a check of all Instrument and method quality control and progresses through sample quality control concluding with a check to assure that the client's requirements have been executed. The analyst has the authority and responsibility to perform corrective action for any out-of-control parameter of non-conformance.

14.1 A secondary review is performed by department managers, and it includes review of the data produced by their department. All manual calculations, QC criteria, and a department manager may reject data, initiate reanalysis, take additional corrective action, or reprocess data.

14.2 The laboratory director performs a full tertiary review of the data package following its assembly. This review includes an evaluation of QC data against acceptance criteria and a check of the data package contents to assure that all analytical requirements and specifications were executed.

14.3 Spot-check reviews are performed by the Quality Assurance Officer focusing on all elements of the deliverable including the client's specifications and requirements, analytical quality control, sample custody documentation and sample identification.

15.0REPORTING

15.1 A results page including positive results and/or RLs, units, methodology, analysis dates, and data qualifiers are reported. Additional quality control data including matrix duplicate RPDs, matrix spike recovery, blank spike and method blank results may be reported upon request of the client.

15.2 Data may be submitted to the client in a specified electronic format (EDD).

15.3 Once the data is approved by the laboratory manager, it may be accessed by clients via LabLink™.

15.4 Procedures for handling non-conforming data.

15.4.1 If quality control data does not meet criteria the non-conformance must be discussed in a case narrative and footnoted on the applicable quality control report summary.

15.4.2 If preservation or holding time criteria is not met and the samples are analyzed the result page must be footnoted with this information, and the non-conformance must be discussed in a case narrative or other suitable communication (telephone conversation log or email). Client notification documentation should be included with the data (telephone conversation log, fax, or email).

16.0 POLLUTION PREVENTION & WASTE MANAGEMENT

16.1 Pollution Prevention. Users of this method must perform all procedural steps that controls the creation and/or escape of wastes of hazardous materials to the environment. The amounts of standards, reagents, and solvents must be limited to the amounts specified in this SOP. All safety practices designed to limit the escape of vapors, liquids, or solids to the environment must be followed. All method users must be familiar with the waste management practices described in section 17.2

16.2 Waste Management. Individuals performing this method must follow established waste management procedures as described in the Sample and Waste Disposal SOP. This document describes the proper disposal of all waste materials generated during the testing of samples as follows:

16.2.1 Non-hazardous aqueous wastes
16.2.2 Hazardous aqueous wastes
16.2.3 Chlorinated organic solvents
16.2.4 Non-chlorinated organic solvents
16.2.5 Hazardous solid wastes
16.2.6 Non-hazardous solid wastes
16.2.7 Microbiological wastes

17.0 ADDITIONAL REFERENCES

17.1 No additional references are required for this method.

Lab Manager: Brad Madadian

QA Officer: Robert Treggiari

TITLE: ***SULFATE, TURBIDIMETRIC ANALYSIS***

TEST METHOD REFERENCE: ASTM D 516-90 -02

REVISED SECTIONS: 7.1.3; 10.2; 10.3; 10.6

1.0 SCOPE & APPLICATION

1.1 This method is applicable to surface and saline water, and domestic and industrial wastes and solids.

2.0 SUMMARY

2.1 In this method, sulfate ion is converted to a barium sulfate suspension under controlled conditions. The resulting turbidity is determined by a spectrophotometer and compared to a curve prepared from standard sulfate solutions.

3.0 METHOD REPORTING LIMIT AND DETECTION LIMIT

3.1 The reporting limit (RL) is based on the lowest calibration standard. RL's may vary depending on matrix difficulties, sample volumes or weights, and percent moisture. Detected concentrations below this concentration cannot be reported without qualification.

3.2 The Method Detection Limit (MDL) represents the lowest reportable concentration of an individual analyte that meets the method qualitative identification criteria.

3.3 Method Detection limits (MDLs) are experimentally determined using the procedures described in 40 CFR, Part 136, Appendix B. Actual reported MDLs incorporate the sample volume analyzed and sample dilutions if needed, which may cause MDL variations from sample to sample.

3.4 In general, MDLs are determined through the analysis of at least 7 replicate blank spikes (using the same procedures for sample analysis). The MDL is calculated by multiplying the standard deviation of the replicate concentrations by the appropriate Student's t value (3.143 for 7 replicates). If more than 7 replicates are analyzed refer to 40 CFR, Part 136, Appendix B for the appropriate student's t value. MDLs are determined initially (prior to analysis), on an annual basis, and after major maintenance to equipment. MDL data is archived with Quality Assurance. Refer to the most recent study for current MDLs. For additional detail regarding MDL studies, refer to the MDL SOP MQA245.

3.5 Current MDL studies are filed with Quality Assurance. Obsolete MDL studies are archived with the QA files. Electronic MDL data is found in the annual "MDL" folder on the QA server (LINUXMA1).

4.0 DEFINITIONS

4.1 ALIQUOT - a measured portion of a sample, or solution, taken for sample preparation and/or analysis.

4.2 BATCH – A group of samples which behave similarly with respect to the sampling or the testing procedures being employed and which are processed as a unit. For QC purposes, if the number of samples in a group is greater than 20 (or 10 for certain methods), then each group of 20 samples (or 10 samples for certain methods) or less will all be handled as a separate batch.

4.3 CALIBRATION CHECK STANDARD/CONTINUING CALIBRATION VERIFICATION (CCV). The calibration check standard is a mid-range calibration standard. It is recommended that the calibration check standard or CCV must be run at a frequency of approximately 10 percent. For most methods, the mid-level calibration check standard criteria is ± 10 percent of the true value. Refer to the specific quality control section for each SOP. The exception to this rule is if the recovery on the calibration check standard is high and the samples to be reported are less than the detection limit.

4.4 CALIBRATION – the establishment of an analytical curve based on the absorbance, emission intensity, or other measured characteristic of known standards. The calibration standards must be prepared using the same type of acid or concentration of acids as used in the sample preparation.

4.5 CALIBRATION STANDARDS – a series of known solutions used by the analyst for calibration of the instrument (i.e., preparation of the analytical curve).

4.6 DRY WEIGHT – the weight of a sample based on percent solids. The weight after drying. See Percent Moisture.

4.7 CONTAMINATION - a component of a sample or an extract that is not representative of the environmental source of the sample. Contamination may stem from other samples, sampling equipment, while in transit, from laboratory reagents, laboratory environment, or analytical instruments.

4.8 EXTERNAL CHECK STANDARD/INITIAL CALIBRATION VERIFICATION (ICV). The external check standard or ICV is a standard from a separate source than the calibration curve that is used to verify the accuracy of the calibration standards. An external check must be run a minimum of once per quarter for most analyses where a check is commercially available. The laboratory should initially assess laboratory performance of a check standard using the control limits generated by the external check supplier. Refer to the quality control section for each SOP. If the external check is outside of the control limits for a given parameter, all samples must be reanalyzed for that parameter after the problem has been resolved.

4.9 FIELD SAMPLE - a portion of material to be analyzed that is contained in single or multiple containers and identified by a unique sample number.

4.10 FIELD BLANK – this is any sample that is submitted from the field and is identified as a blank. This includes trip blanks, rinsates, equipment blanks, etc.

4.11 HOLDING TIME – the elapsed time expressed most commonly in days from the date of sampling until the date of its analysis.

4.12 INTERFERENTS – substances which affect the analysis for the analyte of interest.

4.13 INITIAL CALIBRATION - analysis of analytical standards for a series of different specified concentrations; used to define the linearity and dynamic range of the response of the mass spectrometer or electron capture detector to the target compounds.

4.14 INSUFFICIENT QUANTITY - when there is not enough volume (water sample) or weight (soil/sediment) to perform any of the required operations: sample analysis or extraction, percent moisture, MS/MSD, etc.

4.15 MATRIX: The component or substrate (e.g., water, soil) which contains the analyte of interest.

4.16 MATRIX DUPLICATE: A duplicate sample is digested/distilled/analyzed at a minimum of 1 in 10 samples. The relative percent difference (RPD) between the duplicate and the sample should be assessed. The duplicate RPD is calculated as shown below. Assess laboratory performance against the control limits. In house limits are generated once sufficient duplicate data is available to generate limits (usually a minimum of 20 to 30 analyses). If a duplicate is out of control, flag the results with the appropriate footnote. If the sample and the duplicate are less than 5 times the reporting limits and are within a range of ± the reporting limit, then the duplicate is considered to be in control. Note: If control limits are not specified, use default limits of ± 20% RPD.

$$\frac{(|\text{Sample Result} - \text{Duplicate Result}|) \times 100}{(\text{Sample Result} + \text{Duplicate Result})/2} = \text{Duplicate RPD}$$

4.17 MATRIX SPIKE: The laboratory must add a known amount of each analyte to a minimum of 1 in 10 samples. The matrix spike recovery is calculated as shown below. Assess laboratory performance against the control limits. In house limits are generated once sufficient matrix spike data is available to generate limits (usually a minimum of 20 to 30 analyses). If a matrix spike is out of control, then the results should be flagged with the appropriate footnote. If the matrix spike amount is less than one fourth of the sample amount, then the sample cannot be assessed against the control limits and should be footnoted to that effect. Note:

$$\frac{(\text{Spiked Sample Result} - \text{Sample Result})}{(\text{Amount Spiked})} \times 100 = \text{Matrix Spike Recovery}$$

4.18 METHOD BLANK. The laboratory must digest or distill (as appropriate to the method) and/or analyze a method blank with each set of samples. A minimum of one method blank is required for every 10 samples. If no digestion step is required, then the method blank is equivalent to the reagent blank. The method blank must contain the parameter of interest at levels of less that the reporting limit for that parameter. If the method blank contains levels over the reporting limits, the samples must be redigested or redistilled and reanalyzed. The exception to this rule is when the samples to be reported contain greater than 10 times the method blank level. In addition, if all the samples are less than a client required limit and the method blank is also less than that limit, then the results can be reported as less than that limit.

4.19 PERCENT DIFFERENCE (%D) - To compare two values, the percent difference indicates both the direction and the magnitude of the comparison, i.e., the percent difference may be either negative, positive, or zero. (In contrast, see relative percent difference.)

4.20 PERCENT MOISTURE - an approximation of the amount of water in a soil/sediment sample made by drying an aliquot of the sample at 105 °C. The percent moisture determined in this manner also includes contributions from all compounds that may volatilize at or below 105 °C, including water. Percent moisture may be determined from decanted samples and from samples that are not decanted.

4.21 RELATIVE PERCENT DIFFERENCE (RPD) - To compare two values, the relative percent difference is based on the mean of the two values, and is reported as an absolute value, i.e., always expressed as a positive number or zero. In contrast, see percent difference.

4.22 REAGENT BLANK: The reagent blank is a blank that has the same matrix as the samples, i.e., all added reagents, but did not go through sample preparation procedures. The reagent blank is an indicator for contamination introduced during the analytical procedure. (Note: for methods requiring no preparation step, the reagent blank is equivalent to the method blank.) Either a reagent blank or a method blank must be analyzed with each batch of 20 samples or less. The concentration of the analyte of interest in the reagent blank must be less than the reporting limit for that analyte. If the reagent blank contains levels over the reporting limits, the samples must be reanalyzed. The exception to this rule is when the samples to be reported contain greater than 10 times the reagent blank level. In addition, if all the samples are less than a client required limit and the reagent blank is also less than that limit, then the results can be reported as less than that limit.

4.23 REAGENT GRADE: Analytical reagent (AR) grade, ACS reagent grade, and reagent grade are synonymous terms for reagents that conform to the current specifications of the Committee on Analytical Reagents of the American Chemical Society.

4.24 REAGENT WATER: Water that has been generated by any method which would achieve the performance specifications for ASTM Type II water. Water in which an interferant is not observed at or above the minimum quantitation limit of the parameters of interest. Accutest uses deionized water (municipal water which passes through Accutest's DI treatment system).

4.25 REFERENCE MATERIAL: A material containing known quantities of target analytes in solution or in a homogeneous matrix. It is used to document the bias of the analytical process.

4.26 SPIKE BLANK OR LAB CONTROL SAMPLE. Digest and analyze a laboratory control sample or spike blank with each set of samples. A minimum of one lab control sample or spike blank is required for every 10 samples. Assess laboratory performance against the control limits. In house limits should also be generated once sufficient external check standard data is available to generate limits (usually a minimum of 20 to 30 analyses). If the lab control is outside of the control limits for a parameter, all samples must be reanalyzed for that parameter. The exception is if the lab control recovery is high and the results of the samples to be reported are less than the reporting limit. In that case, the sample results can be reported with no flag. Note: If control limits are not specified, then default limits of 80 to 120 percent should be used.

4.27 STANDARD CURVE: A plot of concentrations of known analyte standards versus the instrument response to the analyte. Calibration standards are prepared by successively diluting a standard solution to produce working standards that cover the working range of the instrument. Standards should be prepared at the frequency specified in the appropriate section. The calibration standards should be prepared using the same type of acid or solvent and at the same concentration as will result in the samples following sample preparation.

5.0 HEALTH & SAFETY

5.1 All safety practices must be followed as outlined in the Accutest Laboratories Employee Safety Handbook and Chemical Hygiene Plan. Safety glasses, gloves, and lab coats must be worn. All samples, solutions, and extracts must be treated as unknown and potentially hazardous.

5.2 The toxicity or carcinogenicity of each reagent used in this method has not been precisely determined; however, each chemical should be treated as a potential health hazard. Exposure to these reagents should be reduced to the lowest possible level.

6.0 COLLECTION, PRESERVATION, AND HOLDING TIMES

6.1 Collection and Preservation

6.1.1 Containers: 500 ml plastic bottle

6.1.2 Preservation: Cool to 4°C ± 2°C.

6.2 Holding Time: 28 days from the date of collection.

7.0 APPARATUS & MATERIALS

7.1 The items needed for the analysis of sulfate are listed below.

7.1.1 Magnetic stirrer.
7.1.2 Stir bars.
7.1.3 Turbidity meter
7.1.4 Stopwatch for measuring reaction time.
7.1.5 Measuring spoon, 0.2 to 0.3 ml volume.

8.0 STANDARDS & REAGENTS

Note: All applicable standard/reagent preparation information, including vendor, lot number, date of preparation, date of expiration, calculations, and initials must be entered in the appropriate standard/reagent preparation logbook. Vendors typically used by Accutest include Fisher Scientific, VWR, Accustandard, Supelco, Chemservices, Ultra, and ERA. Additional vendors may be utilized as necessary.

8.1 Conditioning reagent. Place 300 ml of DI water, 30 ml of concentrated hydrochloric acid, 100 ml of 95% ethanol or isopropanol, and 75 g of NaCl in solution in a container. Add 50 ml of glycerol and mix well.

8.2 Barium chloride crystals, 20 to 30 mesh.

8.3 Standard stock sulfate solution, 1000 mg/L: Dissolve 1.479 g of sodium sulfate (dried at 105 C°) in 1 liter of DI water. This solution is stable for 3 months.

8.4 Calibration Standards. A calibration curve containing 5 points and a blank should be analyzed with each analytical run.

8.4.1 Calibration blank. The calibration blank should be made up from DI water.

8.4.2 5.0 mg/L standard. Volumetrically pipet 0.50 ml of 1000 mg/L sulfate solution into a 100 ml volumetric flask containing approximately 80 ml of DI water. Dilute to volume with DI water and mix well.

8.4.3 10 mg/L standard. Volumetrically pipet 1.00 ml of 1000 mg/L sulfate solution into a 100 ml volumetric flask containing approximately 80 ml of DI water. Dilute to volume with DI water and mix well.

8.4.4 20.0 mg/L standard. Volumetrically pipet 2.00 ml of 1000 mg/L sulfate solution into a 100 ml volumetric flask containing approximately 80 ml of DI water. Dilute to volume with DI water and mix well.

8.4.5 30 mg/L standard. Volumetrically pipet 3.00 ml of 1000 mg/L sulfate solution into a 100 ml volumetric flask containing approximately 80 ml of DI water. Dilute to volume with DI water and mix well.

8.4.6 40.0 mg/L standard. Volumetrically pipet 4.00 ml of 1000 mg/L sulfate solution into a 100 ml volumetric flask containing approximately 80 ml of DI water. Dilute to volume with DI water and mix well.

8.4.7 Calibration Check Standard (20 mg/L). Volumetrically pipet 2.0 ml of 1000 mg/L sulfate solution into a 100 ml volumetric flask containing approximately 80 ml of DI water. Dilute to volume with DI water and mix well.

8.4.8 External Check Solution. This must be from a different lot than the stock used for the calibration curve. The external should be analyzed with each analysis, if available. If not available, a spike blank can be substituted. This can also be made in house by dissolving 1.812 g of Potassium Sulfate (dried at 105 C°) in a 1 liter of DI water. This solution is stable for 3 month.

9.0 INTERFERENCES

9.1 High results may be obtained for samples containing silica. Suspended matter and color can also be interferences in this method. Either background correction or dilutions can be used to minimize interferences.

9.2 Polyphosphates and high concentration of chloride will inhibit barium sulfate precipitation.

9.3 If interferences suspected, dilute sample 1:1 with DI water, and analyze. If results agree, it is assumed that the interferences are absent.

10.0 PROCEDURE

Below is the procedure to be followed for the analysis of water samples for sulfate using the turbidimetric analysis method. Turn on the instrument and let it warm up for 30 minutes. Open the sulfate electronic logbook.

NOTE: All standards and samples should be at room temperature.

10.1 Prepare a calibration curve and analyze the curve as outlined below.

10.1.1 Place 100 ml of standard into a 250 ml beaker.

10.1.2 Add exactly 5.0 ml of conditioning reagent and mix with a stir bar on a magnetic stirring plate.

10.1.3 While the solution is being stirred, add a measuring spoonful of barium chloride crystals (0.3 g) and begin timing immediately.

10.1.4 Stir for 58 to 62 seconds. Timing is critical and the stirring period must be in this time range.

10.1.5 Immediately pour the sample into the sample cell.

10.1.6 Measure the turbidity at 30 second intervals for 4 minutes. Record all readings.

10.1.7 Calculate the calibration curve by linear regression using the maximum reading obtained in the 4 minute interval for each standard as the absorbance for that standard.

10.1.8 Make sure that the calibration curve meets all quality control limits (correlation coefficient greater than 0.995, intercept less than the detection limit, and a slope comparable to the slope normally obtained with this method) before continuing with the analysis.

10.2 Once a calibration curve is obtained, filter sample using 0.45 um filter paper, then analyze the samples following the steps outlined in 10.1.1 through 10.1.6. A calibration check standard should be analyzed after every 10 samples or less. Analyze a method blank using 100 ml of DI water. Analyze a blank spike by adding 2 ml of the 1000 mg/L stock standard solution to 100 ml of DI water. Analyze a matrix

spike by adding 2 ml of 1000 mg/L stock standard solution to 100 ml of the sample. Analyze a duplicate sample.

10.3 For samples with significant background color read a background correction point as follows:

10.3.1 Place 20 ml of sample in a small beaker.

10.3.2 Add exactly 1.0 ml of conditioning reagent and mix with a stir bar, stirring for 1 minute ± 2 seconds.

10.3.3 Pour the sample into the sample cell. Read the absorbance.

10.3.4 Subtract this absorbance from the sample absorbance.

10.4 For samples greater than the highest standard, reanalyze the sample at an appropriate dilution. The diluted result should fall within the mid to upper portion of the calibration curve.

10.5 The concentration in a sample can be calculated from the sample absorbance (or background corrected absorbance) using linear regression from the calibration curve.

10.6 For soil samples, homogenize sample and do not include any large rocks or debris in sample aliquot. Proceed to mix 15 g of sample with 150 ml of DI water, mix for 1 hr, let settle and filter with 0.45 um filter paper. Analyze a method blank using 15 g of sand to 150 ml of DI water. Analyze a spike blank by spiking 3.0 ml of 1000 mg/L stock standard solution into 50 ml of DI water. Bring this up to 150 ml using DI water. Add to 15 g of sand. Analyze a matrix spike by taking 15 g of sample, add 150 ml of DI water and spike this with 3.0 ml of 1000 mg/L stock standard solution. Analyze a matrix duplicate by taking 15 g of sample and add 150 ml of DI water. Make sure to mix the method blank, spike blank, matrix spike, and matrix duplicate for 1 hr, let settle and filter using 0.45 um filter paper.

10.7 CALCULATIONS

10.7.1 Calculate the sulfate concentration in water samples as follows:

SO4 (in mg/L) = Conc. in mg/L x Dilution factor

11.0 QUALITY ASSURANCE

11.1 Quality Control Sample (also referred to as Initial Calibration Verification Standard, (ICV). It is recommended that a standard from a separate source than the calibration should be run at the beginning of each run to verify the calibration curve. This ICV should be within 10 percent of the true value (or within the manufacturer's specified limits – as applicable). If it is not, the problem must be resolved before any samples can be analyzed.

11.2 Calibration Check Standard (CCV). Analyze the calibration check standard after the ICV and after every 10 samples. This standard should be within 10 percent of the true value. If it is not, the problem

must be resolved before sample analysis can continue. Samples analyzed prior to the failing check standard must be reanalyzed.

11.3 Calibration Blank Standard (CCB). Analyze the calibration blank after each CCV or at the minimum after the last CCV, at the end of the run to ensure contamination was not a problem during the batch analyiss.

11.4 Method blank. A method blank must be prepared with each analytical batch of 10 samples using the same reagents to prepare samples. The method blank must be taken through all steps used to process samples. The method blank must not contain the analyte at greater than the reporting limit. If the method blank contains over that limit, the samples must be reanalyzed.

11.5 Spike blank. The laboratory must analyze a spike blank with each set of samples. A minimum of one spike blank is required for every 10 samples. Until sufficient lab control data becomes available, the laboratory should assess the laboratory performance of the spike blank for samples against recovery limits of 80-120%. If the lab control recovery is high and the results of the samples to be reported are less than the reporting limit, then the sample results can be reported with no flag.

11.6 Matrix spike. The laboratory must add a known amount of each analyte to a minimum of 1 in 10 samples. The spike recovery should be assessed using in house limits. Until these limits can be generated, then default limits of 75-125 % recovery should be applied.

11.7 Matrix duplicate. The laboratory must analyze a duplicate sample for a minimum of 1 in 10 samples. Until in-house limits can be generated, the relative percent difference (RPD) between the duplicate and the sample should be assessed against an RPD limit of 20.

11.8 A Precision and accuracy (P&A) study is performed as an initial determination of capability, on an annual basis (continued demonstration of capability – a successful PT result may be used in place of a P&A for continued DOC), and if any significant changes have been made to the instrument. In general, 4 replicates or blank spikes are analyzed using the same procedures and conditions for sample analysis. The percent recoveries are compared to either default limits of 80-120% or in-house control limits once established. The standard deviation of the 4 replicate percent recoveries are compared to either ±20 or to in-house limits once established. If percent recovery or standard deviation criteria are not met, corrective action must be taken to bring the system back into control.

11.9 Quality Control data is generated (control charts) and reviewed on an annual basis by Quality Assurance (blank spike/ matrix spike recoveries and matrix duplicate RPDs).

12.0 DOCUMENTATION

12.1 Use the electronic Sulfate analysis logbook application.

12.2 If samples require reanalysis, a brief explanation of the reason should be documented in this log.

12.3 The standard preparation electronic logbook must be completed for all standard preparations. All information requested must be completed; the page must be signed and dated by the respective person.

12.4 The Accutest lot number must be cross-referenced on the standard vial/container.

12.5 The instrument Maintenance logbook must be completed when any type of maintenance is performed on the instrument. Each instrument will have a separate log.

12.6 Any corrections to laboratory data must be done using a single line through the error. The initials of the person and date of correction must appear next to the correction.

13.0 DATA REVIEW

13.1 The analyst conducts the primary review of all data. This review begins with a check of all Instrument and method quality control and progresses through sample quality control concluding with a check to assure that the client's requirements have been executed. The analyst has the authority and responsibility to perform corrective action for any out-of-control parameter of non-conformance.

13.2 A secondary review is performed by department managers, and it includes review of the data produced by their department. All manual calculations, QC criteria, and a comparison of the data package to client specified requirements are checked. The department manager may reject data, initiate reanalysis, take additional corrective action, or reprocess data.

13.3 The laboratory director performs a full tertiary review of the data package following its assembly. This review includes an evaluation of QC data against acceptance criteria and a check of the data package contents to assure that all analytical requirements and specifications were executed.

13.4 Spot-check reviews are performed by the Quality Assurance Officer focusing on all elements of the deliverable including the client's specifications and requirements, analytical quality control, sample custody documentation and sample identification.

14.0 DATA REPORTING

14.1 A results page including positive results and/or RLs, units, methodology, preparation and/or analysis dates, and data qualifiers are reported. Additional quality control data including calibration summaries, MS/duplicate percent recoveries and RPDs, blank spike recoveries, and method blank results may be reported upon request of the client. Additionally, raw data including any instrument printouts, laboratory logbooks, etc. may be reported to the client.

14.2 Data may be submitted to the client in a specified electronic format (EDD).

14.3 Data may be submitted to the client electronically as a PDF (e-hardcopy).

14.4 Once the data is approved by the laboratory manager, it may be accessed by clients via LabLink™.

14.5 Procedures for handling non-conforming data.

14.5.1 If quality control data does not meet criteria the non-conformance must be discussed in a case narrative and footnoted on the applicable quality control report summary.

14.5.2 If preservation or holding time criteria is not met and the samples are analyzed the result page must be footnoted with this information, and the non-conformance must be discussed in a case narrative or other suitable communication (telephone conversation log or email). Client notification documentation should be included with the data (telephone conversation log, fax, or email).

15.0 POLLUTION PREVENTION & WASTE MANAGEMENT

15.1 Pollution Prevention. Users of this method must perform all procedural steps that control the creation and/or escape of wastes of hazardous materials to the environment. The amounts of standards, reagents, and solvents must be limited to the amounts specified in this SOP. All safety practices designed to limit the escape of vapors, liquids, or solids to the environment must be followed. All method users must be familiar with the waste management practices described in section 15.2.

15.2 Waste Management. Individuals performing this method must follow established waste management procedures as described in the Sample and Waste Disposal SOP. This document describes the proper disposal of all waste materials generated during the testing of samples as follows:

15.2.1 Non-hazardous aqueous wastes
15.2.2 Hazardous aqueous wastes
15.2.3 Chlorinated organic solvents
15.2.4 Non-chlorinated organic solvents
15.2.5 Hazardous solid wastes
15.2.6 Non-hazardous solid wastes
15.2.7 Microbiological wastes

16.0 METHOD PERFORMANCE

16.1 Method performance is evaluated by the annual QC limits (control charts) generated by QA, and the annual MDL study results. Refer to section 3.5 for MDLs, and section 11.9 for QC limits.

17.0 ADDITIONAL REFERENCES

17.1 No additional references are required for this method.

Lab Manager: Brad Madadian

QA Officer: Robert Treggiari

TITLE: NITRATE/NITRITE AND NITRATE ONLY BY CADMIUM REDUCTION ANALYSIS (LACHAT AUTOANALYZER)

TEST METHOD Reference: EPA 353.2 (water) Rev.2.0 1993; EPA 353.2 Mod. (soil)

REVISED SECTIONS*:* Section 8.0 notation

1.0 SCOPE & APPLICATION

1.1 This method is based on EPA method 353.2 and used as a measure of the nitrate/nitrite or nitrate only in drinkingwater and wastewater samples. A modification of the method can be used as a measure of the nitrate/nitrite in soil samples.

2.0 SUMMARY

2.1 The nitrate is reduced to nitrite by a cadmium reduction column. The nitrite is then determined by diazotizing with sulfanilamide and coupling with N-(1-napthyl)-ethylenediamine dihydrochloride to form a high colored azo dye which is measured colorimetrically.

3.0 METHOD REPORTING LIMIT AND DETECTION LIMIT

3.1 The reporting limit (RL) is based on the lowest calibration standard. RL's may vary depending on matrix difficulties, sample volumes or weights, and percent moisture. Detected concentrations below this concentration cannot be reported without qualification.

3.2 The Method Detection Limit (MDL) represents the lowest reportable concentration of an individual analyte that meets the method qualitative identification criteria.

3.3 Method Detection limits (MDLs) are experimentally determined using the procedures described in 40 CFR, Part 136, Appendix B. Actual reported MDLs incorporate the sample volume analyzed and sample dilutions if needed, which may cause MDL variations from sample to sample.

3.4 In general, MDLs are determined through the analysis of at least 7 replicate blank spikes (using the same procedures for sample analysis). The MDL is calculated by multiplying the standard deviation of the replicate concentrations by the appropriate Student's t value (3.143 for 7 replicates). If more than 7 replicates are analyzed refer to 40 CFR, Part 136, Appendix B for the appropriate student's t value. MDLs are determined initially (prior to analysis), on an annual basis, and after major maintenance to equipment. MDL data is archived with Quality Assurance. Refer to the most recent study for current MDLs. Refer to the SOP for MDLs (MQA245) for additional detail regarding MDL study procedures.

3.5 Current MDLs may be entered into the LIMS, and may be viewed by printing out the compound list from the LIMS. Additionally, MDLs are reported on the result page upon client request. Current MDL studies are filed with Quality Assurance. Obsolete MDL studies are archived with the QA files. Electronic MDL data is found in the annual "MDL" folder on the QA server (LINUXMA1).

4.0 DEFINITIONS

4.1 ALIQUOT - a measured portion of a sample, or solution, taken for sample preparation and/or analysis.

4.2 BATCH – A group of samples which behave similarly with respect to the sampling or the testing procedures being employed and which are processed as a unit. For QC purposes, if the number of samples in a group is greater than 20 (or 10 for certain methods), then each group of 20 samples (or 10 samples for certain methods) or less will all be handled as a separate batch.

4.3 CALIBRATION CHECK STANDARD/CONTINUING CALIBRATION VERIFICATION (CCV). The calibration check standard is a mid-range calibration standard. It is recommended that the calibration check standard or CCV must be run at a frequency of approximately 10 percent. For most methods, the mid-level calibration check standard criteria is ± 10 percent of the true value. Refer to the specific quality control section for each SOP. The exception to this rule is if the recovery on the calibration check standard is high and the samples to be reported are less than the detection limit.

4.4 CALIBRATION – the establishment of an analytical curve based on the absorbance, emission intensity, or other measured characteristic of known standards. The calibration standards must be prepared using the same type of acid or concentration of acids as used in the sample preparation.

4.5 CALIBRATION STANDARDS – a series of known solutions used by the analyst for calibration of the instrument (i.e., preparation of the analytical curve).

4.6 DRY WEIGHT – the weight of a sample based on percent solids. The weight after drying. See Percent Moisture.

4.7 CONTAMINATION - a component of a sample or an extract that is not representative of the environmental source of the sample. Contamination may stem from other samples, sampling equipment, while in transit, from laboratory reagents, laboratory environment, or analytical instruments.

4.8 EXTERNAL CHECK STANDARD/INITIAL CALIBRATION VERIFICATION(ICV). The external check standard or ICV is a standard from a separate source than the calibration curve that is used to verify the accuracy of the calibration standards. An external check must be run a minimum of once per quarter for most analyses where a check is commercially available. The laboratory should initially assess laboratory performance of a check standard using the control limits generated by the external check supplier. Refer to the quality control section for each SOP. If the external check is outside of the control limits for a given parameter, all samples must be reanalyzed for that parameter after the problem has been resolved.

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4.10 FIELD BLANK – this is any sample that is submitted from the field and is identified as a blank. This includes trip blanks, rinsates, equipment blanks, etc.

4.11 HOLDING TIME – the elapsed time expressed most commonly in days from the date of sampling until the date of its analysis.

4.12 INTERFERENTS – substances which affect the analysis for the analyte of interest.

4.13 INITIAL CALIBRATION - analysis of analytical standards for a series of different specified concentrations; used to define the linearity and dynamic range of the response of the mass spectrometer or electron capture detector to the target compounds.

4.14 INSUFFICIENT QUANTITY - when there is not enough volume (water sample) or weight (soil/sediment) to perform any of the required operations: sample analysis or extraction, percent moisture, MS/MSD, etc.

4.15 MATRIX: The component or substrate (e.g., water, soil) which contains the analyte of interest.

4.16 MATRIX DUPLICATE: A duplicate sample is digested/distilled/analyzed at a minimum of 1 in 10 samples (or 10 samples for certain methods). The relative percent difference (RPD) between the duplicate and the sample should be assessed. The duplicate RPD is calculated as shown below. Assess laboratory performance against the control limits. In house limits are generated once sufficient duplicate data is available to generate limits (usually a minimum of 20 to 30 analyses). If a duplicate is out of control, flag the results with the appropriate footnote. If the sample and the duplicate are less than 5 times the reporting limits and are within a range of ± the reporting limit, then the duplicate is considered to be in control. Note: If control limits are not specified, use default limits of ± 20% RPD.

$$\frac{(|\text{Sample Result} - \text{Duplicate Result}|) \times 100}{(\text{Sample Result} + \text{Duplicate Result})/2} = \text{Duplicate RPD}$$

4.17 MATRIX SPIKE: The laboratory must add a known amount of each analyte to a minimum of 1 in 10 samples. The matrix spike recovery is calculated as shown below. Assess laboratory performance against the control limits of 90-110%. In house limits are generated once sufficient matrix spike data is available to generate limits (usually a minimum of 20 to 30 analyses). If a matrix spike is out of control, then the results should be flagged with the appropriate footnote. If the matrix spike amount is less than one fourth of the sample amount, then the sample cannot be assessed against the control limits and should be footnoted to that effect. Note:

$$\frac{(\text{Spiked Sample Result} - \text{Sample Result})}{(\text{Amount Spiked})} \times 100 = \text{Matrix Spike Recovery}$$

4.18 METHOD BLANK. The laboratory must digest or distill (as appropriate to the method) and/or analyze a method blank with each set of samples. A minimum of one method blank is required for

every 10 samples. If no digestion step is required, then the method blank is equivalent to the reagent blank. The method blank must contain the parameter of interest at levels of less that the reporting limit for that parameter. If the method blank contains levels over the reporting limits, the samples must be reanalyzed. The exception to this rule is when the samples to be reported contain greater than 10 times the method blank level. In addition, if all the samples are less than a client required limit and the method blank is also less than that limit, then the results can be reported as less than that limit.

4.19 PERCENT MOISTURE - an approximation of the amount of water in a soil/sediment sample made by drying an aliquot of the sample at 105 °C. The percent moisture determined in this manner also includes contributions from all compounds that may volatilize at or below 105 °C, including water. Percent moisture may be determined from decanted samples and from samples that are not decanted.

4.20 RELATIVE PERCENT DIFFERENCE (RPD) - To compare two values, the relative percent difference is based on the mean of the two values, and is reported as an absolute value, i.e., always expressed as a positive number or zero. In contrast, see percent difference.

4.21 REAGENT BLANK: The reagent blank is a blank that has the same matrix as the samples, i.e., all added reagents, but did not go through sample preparation procedures. The reagent blank is an indicator for contamination introduced during the analytical procedure. (Note: for methods requiring no preparation step, the reagent blank is equivalent to the method blank.) Either a reagent blank or a method blank must be analyzed with each batch of 20 samples or less. The concentration of the analyte of interest in the reagent blank must be less than the reporting limit for that analyte. If the reagent blank contains levels over the reporting limits, the samples must be reanalyzed. The exception to this rule is when the samples to be reported contain greater than 10 times the reagent blank level. In addition, if all the samples are less than a client required limit and the reagent blank is also less than that limit, then the results can be reported as less than that limit.

4.22 REAGENT GRADE: Analytical reagent (AR) grade, ACS reagent grade, and reagent grade are synonymous terms for reagents that conform to the current specifications of the Committee on Analytical Reagents of the American Chemical Society.

4.23 REAGENT WATER: Water that has been generated by any method which would achieve the performance specifications for ASTM Type II water. Water in which an interferant is not observed at or above the minimum quantitation limit of the parameters of interest. Accutest uses deionized water (municipal water which passes through Accutest's DI treatment system).

4.24 REFERENCE MATERIAL: A material containing known quantities of target analytes in solution or in a homogeneous matrix. It is used to document the bias of the analytical process.

4.25 SPIKE BLANK OR LAB CONTROL SAMPLE. Digest and analyze a laboratory control sample or spike blank with each set of samples. A minimum of one lab control sample or spike blank is required for every 10 samples. Assess laboratory performance against the control limits of 90-110%. In house limits should also be generated once sufficient external check standard data is available to generate limits (usually a minimum of 20 to 30 analyses). In house limits must equal or better than the required limits. If the lab control is outside of the control limits for a parameter, all

samples must be redigested or redistilled and reanalyzed for that parameter. The exception is if the lab control recovery is high and the results of the samples to be reported are less than the reporting limit. In that case, the sample results can be reported with no flag.

4.26 STANDARD CURVE: A plot of concentrations of known analyte standards versus the instrument response to the analyte. Calibration standards are prepared by successively diluting a standard solution to produce working standards that cover the working range of the instrument. Standards should be prepared at the frequency specified in the appropriate section. The calibration standards should be prepared using the same type of acid or solvent and at the same concentration as will result in the samples following sample preparation.

5.0 HEALTH & SAFETY

5.1 All safety practices must be followed as outlined in the Accutest Laboratories Employee Safety Handbook and Chemical Hygiene Plan. Safety glasses, gloves, and lab coats must be worn. All samples, solutions, and extracts must be treated as unknown and potentially hazardous.

5.2 The toxicity or carcinogenicity of each reagent used in this method has not been precisely determined; however, each chemical should be treated as a potential health hazard. Exposure to these reagents should be reduced to the lowest possible level.

6.0 COLLECTION, PRESERVATION, AND HOLDING TIMES

6.1 Collection and Preservation

6.1.1 Containers 250 ml plastic container (water); 300 ml glass container (soil)

6.2 Preservation Both soils and water samples should be kept under refrigeration at 4°C until analysis. Water samples should be preserved with sulfuric acid to a pH of less than 2 if they are to be analyzed for nitrate + nitrite. If nitrate only is requested, then the sample can be separated into 2 aliquots. The nitrate + nitrite aliquot should be preserved with sulfuric acid and the nitrite aliquot should be unpreserved.

6.3 Holding Time: All preserved samples should be analyzed within 28 days of the date of collection. Unpreserved samples must be analyzed within 48 hours of the time of collection. Nitrite must be analyzed within 48 hours.

7.0 APPARATUS & MATERIALS

7.1 Automated continuous flow analyzer designed to deliver and react sample and reagents in the required order and ratios. Currently, the Lachat 8000 Automated Ion Analyzer is being used.

7.2 Autosampler XYZ

7.3 Multichannel pump

7.4 Reaction manifold, including cadmium-copper reduction column.

7.5 Colorimetric detector

7.6 Real time data acquisition device (either electronic or hard copies).

7.7 Balance. Analytical balance capable of accurately weighing to the nearest 0.0001 g.

7.8 Volumetric glassware. Class A volumetric pipettes and flasks as required.

8.0 STANDARDS & REAGENTS

NOTE: All chemicals listed below are reagent grade unless otherwise specified. Distilled, deionized water should be used whenever water is required. All applicable standard/reagent preparation information, including vendor, lot number, date of preparation, calculations, and initials must be entered in the appropriate standard/reagent preparation logbook. Vendors typically used by Accutest include Fisher Scientific, VWR, Accustandard, Absolute Standards, Supelco, Chemservices, Ultra, and ERA. Additional vendors may be utilized as necessary.

8.1 15 N Sodium Hydroxide. In a 1 liter beaker, slowly add 150 g of NaOH to 250 ml of DI water. Swirl until dissolved. **Caution - the solution will get very hot!!**

8.2 Ammonium Chloride Buffer Solution. In a 1000 ml beaker, dissolve 85.0 g ammonium chloride (NH_4Cl) and 1.0 g disodium ethylenediamine tetraacetic acid dihydrate (Na_2 EDTA $2H_2O$) in about 800 ml of DI water. Adjust the pH up to 8.5 with 15 M sodium hydroxide. Dilute to 1000ml with DI water in a 1000 ml volumetric flask and mix. Degas this solution with He at 20 psi for 2 min.

NOTE: Ammonium chloride has been found occasionally to contain significant nitrate contamination. If the zero cannot be set, this may be the problem. An alternative way for making ammonium chloride buffer is:
In the hood, to a 1000 ml volumetric flask add 500 ml of DI water, 105 ml concentrated Hcl, 95 ml ammonium hydroxide (NH_4OH), and 1.0 g disodium EDTA. Dissolve and dilute to the mark with DI water. Adjust the pH to 8.5 with concentrated ammonium hydroxide. Degas this solution with He at 20 psi for 2 min.

8.3 Sulfanilamide color reagent. Add 100 ml of concentrated phosphoric acid (H_3PO_4), 40.0 g of sulfanilamide, and 1.0 g of N-(1-napthyl)ethylenediamine dihydrochloride (NED) to a 1 liter volumetric containing approximately 600 ml of DI water. Stir to dissolve and dilute to a final volume of 1 liter with DI water. Store in a dark bottle. This solution is stable for approximately 1 month. Degas this solution with He at 20 psi for 2 min.

8.4 Hydrochloric Acid, 1 N. Add 8 ml of concentrated HCl to approximately 85 ml of DI water. Dilute to a final volume of 100 ml with DI water.

8.5 Carrier solution. Degassed DI water. Degas DI water with He. Use He at 20 psi for 2 min.

8.6 Two percent copper sulfate solution. Dissolve 20 g of copper sulfate (CuSO4.5H2O) in approximately 800 ml of DI water in a 1 liter volumetric flask. Dilute to a final volume of 1 liter and mix well.

8.7 Cadmium reduction column. The cadmium reduction column should be prepared as described below.

Note 1: It is recommended that extra cadmium be prepared through step 6.8.3 so that new columns may be packed as needed.

Note 2: Cadmium is toxic. Make sure to wear gloves for all procedures and collect any cadmium waste in a marked solid waste container.

8.7.1 Place 10 to 20 g of coarse cadmium granules in a beaker. Cadmium granules ranging from 0.3 to 1.5 mm in diameter are recommended. Wash the Cd first with acetone and then with DI water. Then wash the Cd with two 50 ml portions of 1 N HCl. Rinse the Cd granules well with DI water.

8.7.2 Add 100 ml of 2% copper sulfate solution to the cadmium granules. Swirl for about 5 minutes, then decant the liquid and repeat the process with a fresh portion of 2% copper sulfate solution. Continue this process until the blue color of the copper sulfate solution persists.

8.7.3 Decant off the copper sulfate solution and rinse the copper granules with at least 5 portions of ammonium chloride buffer to remove the colloidal copper. The cadmium should be black or dark gray at this point and can be stored in a capped bottle under ammonium chloride buffer solution.

8.7.4 Open one end of a column, removing the colored lead and the foam plug, and clamp the column so that the open end is pointing up. Fill the column with ammonium chloride buffer. Pour the prepared cadmium granules into the column and tap the column lightly so that the granules settle to the bottom of the column. Fill the column to about 5 mm from the open end. Make sure that there are no air bubbles. Push in the foam plug and screw on the cap. Rinse the outside of the column with DI water.

8.7.5 Insert the column into the manifold by first pumping all reagents into the manifold. Then turn the pump off. Connect the column to the appropriate tubing, making sure that no air is in the tubing. Then return the pump to its normal speed.

8.8 Stock nitrate solution, 1000 mg/l. Dissolve 7.218 g of KNO_3 and dilute to 1 liter with DI water in a 1 liter volumetric flask. Preserve with 2 ml of chloroform per liter. This solution is stable for 6 months.

8.9 Standard nitrate solution, 100 mg/l. Dilute 10.0 ml of 1000 mg/l nitrate solution to 100 ml with DI water in a volumetric flask.

8.10 Standard nitrate solution, 10.0 mg/l. Dilute 10.0 ml of 1000 mg/l nitrate solution to 1000 mg/l with DI water in a volumetric flask.

8.11 Nitrate Calibration Standards. Calibration standards should be made from the above standard solutions as shown below. Class A volumetric pipettes and flasks should be used for all dilutions. A calibration check standard should also be prepared that is at or near the mid-point of the calibration curve.

NOTE: Other calibration standards may be used if necessary. Bring all standards to 100 ml final volume with DI water.

Ml of 8.11	Ml of 8.10	Final Conc.(mg/l)
0.00		0.00
1.00		0.10
3.00		0.30
5.00		0.50
8.00		0.80
10.0		1.00
	2.00	2.00
	5.00	5.00

8.12 Stock nitrite solution, 1000 mg/l. Dissolve 6.072 g of KNO_2 in 500 ml of DI water and dilute to 1 liter in a 1 liter volumetric flask. Preserve with 2 ml of chloroform and keep under refrigeration.

8.13 Standard nitrite solution, 10.0 mg/l. Dilute 1.0 ml of 1000 mg/l nitrite solution (8.13) to 100 ml with DI water in a volumetric flask.

8.14 2.0 ppm Standard nitrite solutions. The nitrite solution is used to check the efficiency of the cadmium reduction column, by comparing a nitrite standard directly with a nitrite standard of the same concentration. Dilute 20 ml of 10 ppm nitrite solution (8.14) to 100 ml with DI water in a volumetric flask.

8.15 Spike solution 100 Mg/L. Dilute 10 ml of 1000 mg/l nitrate solution (8.9) to 100 ml with DI water in a volumetric flask.

9.0 INTERFERENCES

9.1 Build up of suspended matter in the reduction column will restrict sample flow. However, samples can be filtered through a 0.45 um membrane filter to avoid this interference.

9.2 High concentrations of iron, copper, or other heavy metals can cause low results, but this can be avoided by adding EDTA to the samples to complex the metals.

9.3 Residual chlorine can produce a negative interference. This can be eliminated by dechlorinating the sample with sodium thiosulfate.

9.4 Samples that contain large concentrations of oil and grease will coat the surface of the cadmium. This interference is eliminated by pre-extracting the sample.

10.0 PROCEDURE

10.1 Below is a step by step procedure for the analysis of samples for the determination of nitrate/nitrite and nitrate only. At the end of this SOP is a short summary outlining the overall procedure.

10.2 Install the nitrate reaction manifold, excluding the cadmium column. Check all tubing and change any tubing that is flat, dirty, etc. Install the appropriate sample loop and the appropriate filter. Place the tubing in the bottles for the sulfanilamide color reagent, the ammonia buffer solution, and the degassed DI water carrier. Also make sure that the waste container is in place. Refer to the manual for additional information.

10.3 For soil samples, follow the digestion procedure described below. The matrix spike should be prepared by adding 4.0 ml of 100 mg/l nitrate solution directly to the soil and mixing well before the acid digestion. The spike blank should also be prepared by digesting 4.00 ml of the 100 mg/l standard. Make sure to prepare a method blank with each batch of samples.

10.3.1 Weigh out 1.0 g of sample (dry weight) into a 200 ml Erlenmeyer flask. Add 50 ml of DI water and 4 drops of concentrated sulfuric acid. Add another 50 ml of DI water and then boil the sample on a hot plate for 15 minutes.

NOTE: This procedure is operationally defined, so make sure that the same heating time is used for all samples.

10.3.2 Transfer the sample to a centrifuge tube and centrifuge for 5 to 10 minutes. Decant the wash into a 200 ml volumetric flask.

10.3.3 Add 50 ml of DI water to the solids in the centrifuge tube and mix well. Then centrifuge the sample for 5 to 10 minutes and decant the wash into the volumetric flask.

10.3.4 Repeat the above step a second time and again decant the wash into the volumetric flask.

10.3.5 Bring the sample to a final volume of 200 ml with DI water. Filter the sample through a 0.45 um filter before analysis.

10.4 For water samples no preparation is necessary unless the sample is turbid. If the sample is turbid, filter before analysis through a 0.45 um membrane filter paper. If the pH of the sample is below 5 or above

9, adjust to between 5 and 9 with either con. HCl or conc NH_4OH. The matrix spike should be prepared by adding 0.2 ml of 100 mg/l nitrate solution to approximately 10 ml of sample. The spike blank should be prepared by diluting 0.2 ml of 100 mg/l nitrate solution to a final volume of 10 ml with DI water. Make sure to analyze a method blank for each batch of samples.

10.5 On the desk top open the lachat software by clicking on the "**Omni"** icon, and then click on "**OPEN"** on top of the tool bar. The "**OPEN"** window will now be on the screen. The templates, specific to each methods, are stored in the data file. Open the appropriate data file (ie., NO3). Click on the template.

10.6 Three windows will open on screen, **("the run worksheet", "run properties", and "channel one**). "**Run worksheet"** will contain the appropriate standards. To extend the worksheet (adding samples and QC, etc), right click on the bottom line of the worksheet (CalBlk), and click on "**append many".** The appended rows window will now be open and the appropriate number of rows needed should be entered, click ""**ok**". The work sheet must now be sequenced to direct the auto-sampler to the correct sample locations.

10.7 Click and drag by starting in the gray sample No. column down to the last sample to highlight the rows (not including STD's). Right click, then go to columns, then to "**Auto Number Cups"** and click. The run should now be in numerical sequence.

10.8 In the **"Run Properties"** window, click on the Run tab and check "**Export Data as CSV file"** There are other areas of the "**Run Properties"** window that contain method specific information such as timing. These settings must not be adjusted without consulting the area lab supervisor, or an experienced analyst.

10.9 Allow the instrument to warm up for 30 minutes.

10.10 Start pumping reagents through the system. When reagents are pumped through the manifold, then the cadmium column can be installed.

10.11 To begin the analysis, click on "START" on toolbar. The instrument will begin to calibrate. The acceptance calibration criteria for correlation coefficient of 0.995 are set within the software. If the criteria is met, the instrument will proceed with sample analysis.

10.12 Observe the peaks in "**Channel One"** window. The baseline should be smooth and peaks must be well shaped and smooth. If peaks look abnormal, that may indicate the chemistry problems, such as pH differences. Small spikes are indicative of air bubbles in the system.

10.13 The run maybe stopped at anytime by clicking **"Stop"** in the top toolbar. It should be noted that every time the run has been stopped and started, a new file is created. If dilutions are required, the dilution factor needs to be entered in the "**Run Worksheet"** window. On **MDF** column, click on the box and enter the dilution factor.

10.14 When the run is completed the file is automatically exported to " **Lachat CSV files**" locally under "My Document" directory. Click on **"Lachat CSV Files**". All generated files are listed based on the following format: **OM_date_time Am or PM** (**OM_4-11-2006_12-10-34PM**). Drag the generated files for that run/day to the current Month/Year directory. For the ease of search later on, at the

beginning of each month, create a directory to save the runs for the entire month, and name it based on the month, and the year (ie, **OCT 2006**). Once you have moved your file to the month/year directory, copy this file to" **Lacha**t" directory located on the server **Mafile1\WC_DATA** , rename the file to the format which is acceptable to LIMS. This naming scheme will be based on date, the matrix, no. of the run for that day, and finally the test code extension, (ie, **050206W1.NO3**). Once the files have been renamed, drag the file to Server **LIMS_WC**. Server **LIMS_WC** is the processing branch of the LIMS for Wet Chemistry. Once the Run is processed in LIMS, go to GNAPP, and review the run. Package all raw data, and logbook copies in a folder, and turn the package to the area manager for data review, and quality control check.

10.15 To obtain a print out of the sample run, click on Tools in the top toolbar and then click on custom report. To format the report to contain the calibration curve and dilutions, click on report, then format in the custom report window.

10.16 Make sure to check the cadmium reduction efficiency at the beginning of the run. If the efficiency is outside of the range of 85-115%, then the column should be reactivated.

10.17 At the end of the run, make sure to pump ammonia buffer into the cadmium column and cap off the column, making sure that no air is entrained. Rinse out the remainder of the system with DI water.

10.18 Results exceeding the upper range of the calibration curve must be diluted and re-analyzed. The diluted result should be within the upper range of the calibration curve.

11.0 QUALITY ASSURANCE

11.1 Calibration curve. The correlation coefficient of the calibration curve must be 0.995 or greater. The curve must be verified using a standard source independent from the calibration standards (second source). Only the low and high points of the curve may be removed to meet correlation coefficient criteria. If a middle point is removed, it must be approved by the supervisor and documented in the analysis logbook. Removing the low point raises the reporting limit, and removing the high point reduces the calibration range.

11.2 Quality Control Sample (also referred to as Initial Calibration Verification Standard (ICV) a standard from a different source than the calibration standard must be analyzed with each initial calibration. Normally this is analyzed at the beginning of the run after the CCV and CCB checks. For this method, the ICV should be within 10% of the true value.

NOTE: It is recommended that this standard be analyzed with each run.

11.3 Method Blank. The laboratory must digest and analyze a method blank with each set of samples. A minimum of one method blank is required for every 10 samples. The method blank must not contain the analyte greater than the reporting limit. If the method blank contains over that limit, the samples must be re-analyzed (if the samples are non-detected they may be reported without qualification).

11.4 Spike Blank. The laboratory must digest and analyze a spike blank with each set of samples. A minimum of one lab control sample or spike blank is required for every 10 samples. Assess laboratory performance against the control limits of 90-110%. In house limits should also be

generated once sufficient external check standard data is available to generate limits (usually a minimum of 20 to 30 analyses). In house limits must equal or better than the required limits. If the lab control is outside of the control limits for a parameter, all samples must be redigested or redistilled and reanalyzed for that parameter. The exception is if the lab control recovery is high and the results of the samples to be reported are less than the reporting limit. In that case, the sample results can be reported with no flag.

11.5 MATRIX SPIKE: The laboratory must add a known amount of each analyte to a minimum of 1 in 10 samples. The matrix spike recovery is calculated as shown below. Assess laboratory performance against the control limits of 90-110%. In house limits are generated once sufficient matrix spike data is available to generate limits (usually a minimum of 20 to 30 analyses). If a matrix spike is out of control, then the results should be flagged with the appropriate footnote. If the matrix spike amount is less than one fourth of the sample amount, then the sample cannot be assessed against the control limits and should be footnoted to that effect

$$\frac{(Spiked\ Sample\ Result\ -\ Sample\ Result)}{(Amount\ Spiked)}\ x\ 100\ =\ MS\ Recovery$$

11.6 Matrix Duplicate. The laboratory must analyze a duplicate sample for a minimum of 1 in 10 samples. The relative percent difference (RPD) between the duplicate and the sample should be assessed. The duplicate RPD is calculated as shown below. The duplicate RPD should be assessed using in house limits. Until these limits can be generated, then default limits of 20% RPD should be applied. If a duplicate is out of control, then the results should be flagged with the appropriate footnote. If the sample and the duplicate are less than 5 times the reporting limits and are within a range of ± the reporting limit, then the duplicate is considered to be in control.

11.8.1The duplicate RPD should be calculated as shown below:

$$\frac{(Sample\ Result\ -\ Duplicate\ Result)\ x\ 100}{(Sample\ Result\ +\ Duplicate\ Result)\ x\ 0.5}\ =\ \%RPD$$

11.9 Continuing Calibration Verification. (Also known as the instrument performance check solution). Analyze the continuing calibration verification solution and the continuing calibration blank after the initial calibration, after every tenth sample, and at the end of the sample run. If the CCV solution is not within 10% of the true value, then no samples can be reported in the area bracketed by that CCV.

NOTE: **The exception is if the CCV is biased high and the samples are less than the detection limit. In that case, the samples can be reported with no flag.**

The CCV concentration should be at or near the mid-range of the calibration curve.

11.10 Continuing Calibration Blank. Analyze the CCV solution and the CCB after the initial calibration, after every tenth sample, and at the end of the sample run. If the CCB is not less than the reporting limit, then no samples can be reported in the area bracketed by the failing CCB.

11.11 A Precision and accuracy (P&A) study is performed as an initial determination of capability, on an annual basis (continued demonstration of capability – a successful PT result may be used in place of a P&A for continued DOC), and if any significant changes have been made to the instrument. In general, 4 replicates or blank spikes are analyzed using the same procedures and conditions for sample analysis. The percent recoveries are compared to either default limits of 80-120% or in-house control limits once established. The standard deviation of the 4 replicate percent recoveries are compared to either ±20 or to in-house limits once established. If percent recovery or standard deviation criteria are not met, corrective action must be taken to bring the system back into control. The P&A study must be performed using a source independent from the calibration standards (second source).

11.12 Quality Control data is generated (control charts) and reviewed on an annual basis by Quality Assurance (blank spike/ matrix spike recoveries and matrix duplicate RPDs).

12.0 DOCUMENTATION

12.1 The analytical logbook is a record of the analysis sequence; the logbook must be completed daily. Each instrument will have a separate logbook.

12.2 If samples require reanalysis, a brief explanation of the reason should be documented in this log.

12.3 The standard preparation logbook application must be completed for all standard preparations. All information requested must be completed; the page must be signed and dated by the respective person.

12.4 The Accutest lot number must be cross-referenced on the standard vial/container.

12.5 The instrument Maintenance logbook must be completed when any type of maintenance is performed on the instrument. Each instrument will have a separate log.

12.6 All laboratory logbooks must be routinely reviewed and initialed or signed by the lab manager.

12.7 Any corrections to laboratory data must be done using a single line through the error. The initials of the person and date of correction must appear next to the correction.

13.0 DATA REVIEW

13.1 The analyst conducts the primary review of all data. This review begins with a check of all Instrument and method quality control and progresses through sample quality control concluding with a check to assure that the client's requirements have been executed. The analyst has the authority and responsibility to perform corrective action for any out-of-control parameter of non-conformance.

13.2 A secondary review is performed by department managers, and it includes review of the data produced by their department. All manual calculations, QC criteria, and a comparison of the data package to client specified requirements are checked. The department manager may reject data, initiate reanalysis, take additional corrective action, or reprocess data.

13.3 The laboratory director performs a full tertiary review of the data package following its assembly. This review includes an evaluation of QC data against acceptance criteria and a check of the data package contents to assure that all analytical requirements and specifications were executed.

13.4 Spot-check reviews are performed by the Quality Assurance Officer focusing on all elements of the deliverable including the client's specifications and requirements, analytical quality control, sample custody documentation and sample identification.

14.0 DATA REPORTING

14.1 A results page including positive results and/or RLs, units, methodology, preparation and/or analysis dates, and data qualifiers are reported. Additional quality control data including calibration summaries, MS/duplicate percent recoveries and RPDs, blank spike recoveries, and method blank results may be reported upon request of the client. Additionally, raw data including any instrument printouts, laboratory logbooks, etc. may be reported to the client.

14.2 Data may be submitted to the client in a specified electronic format (EDD).

14.3 Data may be submitted to the client electronically as a PDF (e-hardcopy).

14.4 Once the data is approved by the laboratory manager, it may be accessed by clients via LabLink™.

14.5 Procedures for handling non-conforming data.

14.5.1 If quality control data does not meet criteria the non-conformance must be discussed in a case narrative and footnoted on the applicable quality control report summary.

14.5.2 If preservation or holding time criteria is not met and the samples are analyzed the result page must be footnoted with this information, and the non-conformance must be discussed in a case narrative or other suitable communication (telephone conversation log or email). Client notification documentation should be included with the data (telephone conversation log, fax, or email).

15.0 POLLUTION PREVENTION & WASTE MANAGEMENT

15.1 Pollution Prevention. Users of this method must perform all procedural steps that controls the creation and/or escape of wastes of hazardous materials to the environment. The amounts of standards, reagents, and solvents must be limited to the amounts specified in this SOP. All safety practices designed to limit the escape of vapors, liquids, or solids to the environment must be followed. All method users must be familiar with the waste management practices described in section 15.2.

15.2 Waste Management. Individuals performing this method must follow established waste management procedures as described in the Sample and Waste Disposal SOP. This document describes the proper disposal of all waste materials generated during the testing of samples as follows:

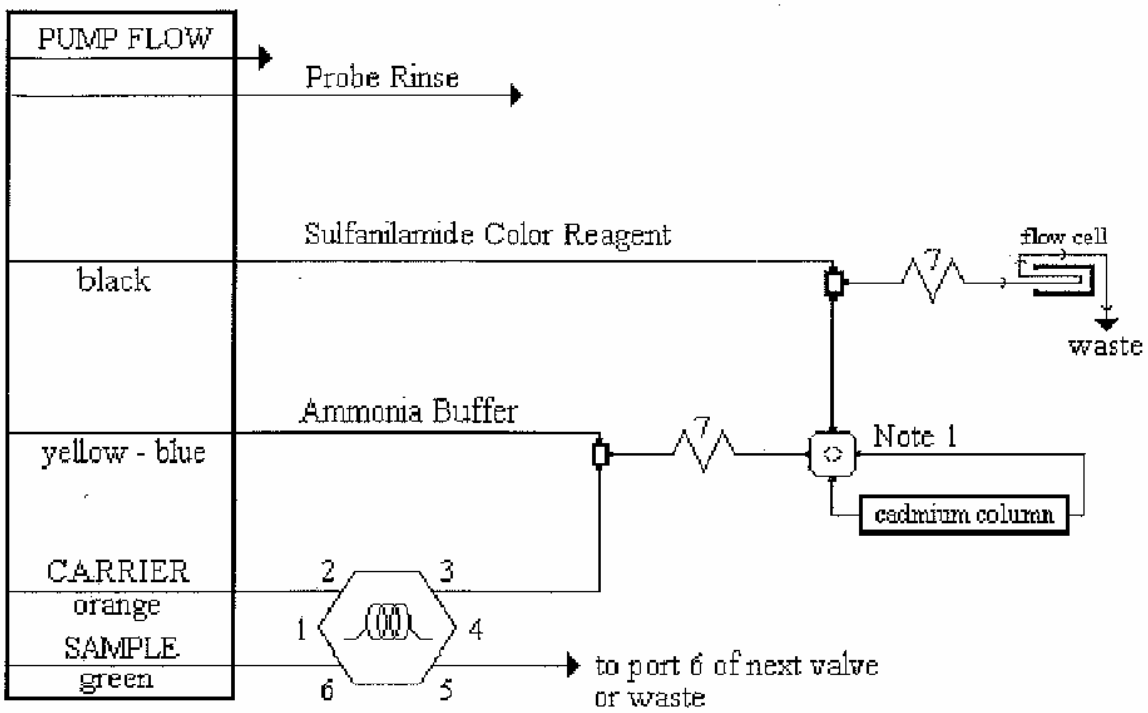
15.2.1 Non-hazardous aqueous wastes
15.2.2 Hazardous aqueous wastes
15.2.3 Chlorinated organic solvents
15.2.4 Non-chlorinated organic solvents
15.2.5 Hazardous solid wastes
15.2.6 Non-hazardous solid wastes
15.2.7 Microbiological wastes

16.0 METHOD PERFORMANCE

16.1 Method performance is evaluated by the annual QC limits (control charts) generated by QA, and the annual MDL study results. Refer to section 3.5 for MDLs, and section 11.12 for QC limits.

17.0 ADDITIONAL REFERENCES

17.1 Lachat QuickChem Method 10-107-04-1-C

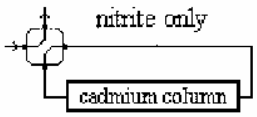


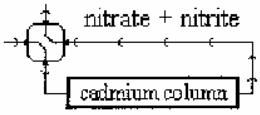
Carrier: Helium Degassed DI water
Manifold Tubing: 0.8 mm (0.032 in) i.d. This is 5.2 µL/cm.
AE Sample Loop: 17 cm x 0.8 mm i.d.
QC8000 Sample Loop: 22.5 cm x 0.8 mm i.d.
Interference Filter: 520 nm

Apparatus: An injection valve, a 10 mm path length flow cell , and a colorimetric detector module is required.

7: 135 cm of tubing on a 7 cm coil support

Note 1: This a 2 state switching valve used to place the cadmium column in-line with the manifold





Lab Manager: Brad Madadian

QA Officer: Robert Treggiari

TITLE: BIOCHEMICAL OXYGEN DEMAND (5 DAY BOD)

TEST METHOD REFERENCE: SM5210 B-11. Standard Methods for the Examination of Water and Wastewater; Online

REVISED SECTIONS: 6.2; 8.18; 10.4.2; 10.8.7; added 11.7

1.0 SCOPE & APPLICATION

1.1 This method is applicable to the analysis of water and waste water samples for the biochemical oxygen demand observed over a 5 day period of time. The oxygen demand is measured as a function of the reduction in dissolved oxygen.

2.0 SUMMARY

2.1 The sample is incubated for 5 days at 20°C in the dark. The reduction in dissolved oxygen concentration during the incubation period yields a measure of the biochemical oxygen demand.

3.0 METHOD REPORTING LIMIT AND DETECTION LIMIT

3.1 The reporting limit (RL) is based on the lowest calibration standard. RL's may vary depending on matrix difficulties, sample volumes or weights, and percent moisture. Detected concentrations below this concentration cannot be reported without qualification.

3.2 The Method Detection Limit (MDL) represents the lowest reportable concentration of an individual analyte that meets the method qualitative identification criteria.

3.3 Method Detection limits (MDLs) are experimentally determined using the procedures described in 40 CFR, Part 136, Appendix B. Actual reported MDLs incorporate the sample volume analyzed and sample dilutions if needed, which may cause MDL variations from sample to sample.

3.4 In general, MDLs are determined through the analysis of at least 7 replicate blank spikes (using the same procedures for sample analysis). The MDL is calculated by multiplying the standard deviation of the replicate concentrations by the appropriate Student's t value (3.143 for 7 replicates). If more than 7 replicates are analyzed refer to 40 CFR, Part 136, Appendix B for the appropriate student's t value. MDLs are determined initially (prior to analysis), on an annual basis, and after major maintenance to equipment. MDL data is archived with Quality Assurance. Refer to the most recent study for current MDLs.

4.0 DEFINITIONS

4.1 ALIQUOT - a measured portion of a sample, or solution, taken for sample preparation and/or analysis.

4.2 BATCH – A group of samples which behave similarly with respect to the sampling or the testing procedures being employed and which are processed as a unit. For QC purposes, if the number of samples in a group is greater than 20 (or 10 for certain methods), then each group of 20 samples (or 10 samples for certain methods) or less will all be handled as a separate batch.

4.3 CALIBRATION CHECK STANDARD/CONTINUING CALIBRATION VERIFICATION (CCV). The calibration check standard is a mid-range calibration standard. It is recommended that the calibration check standard or CCV must be run at a frequency of approximately 10 percent. For most methods, the mid-level calibration check standard criteria is ± 10 percent of the true value. Refer to the specific quality control section for each SOP. The exception to this rule is if the recovery on the calibration check standard is high and the samples to be reported are less than the detection limit.

4.4 CALIBRATION – the establishment of an analytical curve based on the absorbance, emission intensity, or other measured characteristic of known standards. The calibration standards must be prepared using the same type of acid or concentration of acids as used in the sample preparation.

4.5 CALIBRATION STANDARDS – a series of known solutions used by the analyst for calibration of the instrument (i.e., preparation of the analytical curve).

4.6 DRY WEIGHT – the weight of a sample based on percent solids. The weight after drying. See Percent Moisture.

4.7 CONTAMINATION - a component of a sample or an extract that is not representative of the environmental source of the sample. Contamination may stem from other samples, sampling equipment, while in transit, from laboratory reagents, laboratory environment, or analytical instruments.

4.8 EXTERNAL CHECK STANDARD/INITIAL CALIBRATION VERIFICATION (ICV). The external check standard or ICV is a standard from a separate source than the calibration curve that is used to verify the accuracy of the calibration standards. An external check must be run a minimum of once per quarter for most analyses where a check is commercially available. The laboratory should initially assess laboratory performance of a check standard using the control limits generated by the external check supplier. Refer to the quality control section for each SOP. If the external check is outside of the control limits for a given parameter, all samples must be reanalyzed for that parameter after the problem has been resolved.

4.9 FIELD SAMPLE - a portion of material to be analyzed that is contained in single or multiple containers and identified by a unique sample number.

4.10 FIELD BLANK – this is any sample that is submitted from the field and is identified as a blank. This includes trip blanks, rinsates, equipment blanks, etc.

4.11 HOLDING TIME – the elapsed time expressed most commonly in days from the date of sampling until the date of its analysis.

4.12 INTERFERENTS – substances which affect the analysis for the analyte of interest.

4.13 INITIAL CALIBRATION - analysis of analytical standards for a series of different specified concentrations; used to define the linearity and dynamic range of the response of the mass spectrometer or electron capture detector to the target compounds.

4.14 INSUFFICIENT QUANTITY - when there is not enough volume (water sample) or weight (soil/sediment) to perform any of the required operations: sample analysis or extraction, percent moisture, MS/MSD, etc.

4.15 MATRIX: The component or substrate (e.g., water, soil) which contains the analyte of interest.

4.16 MATRIX DUPLICATE: A duplicate sample is digested/distilled/analyzed at a minimum of 1 in 20 samples (or 10 samples for certain methods). The relative percent difference (RPD) between the duplicate and the sample should be assessed. The duplicate RPD is calculated as shown below. Assess laboratory performance against the control limits. In house limits are generated once sufficient duplicate data is available to generate limits (usually a minimum of 20 to 30 analyses). If a duplicate is out of control, flag the results with the appropriate footnote. If the sample and the duplicate are less than 5 times the reporting limits and are within a range of ± the reporting limit, then the duplicate is considered to be in control. Note: If control limits are not specified, use default limits of ± 20% RPD.

$$\frac{(|\text{Sample Result} - \text{Duplicate Result}|) \times 100}{(\text{Sample Result} + \text{Duplicate Result})/2} = \text{Duplicate RPD}$$

4.17 MATRIX SPIKE: The laboratory must add a known amount of each analyte to a minimum of 1 in 20 samples (or 10 samples for certain methods). The matrix spike recovery is calculated as shown below. Assess laboratory performance against the control limits. In house limits are generated once sufficient matrix spike data is available to generate limits (usually a minimum of 20 to 30 analyses). If a matrix spike is out of control, then the results should be flagged with the appropriate footnote. If the matrix spike amount is less than one fourth of the sample amount, then the sample cannot be assessed against the control limits and should be footnoted to that effect. Note:

$$\frac{(\text{Spiked Sample Result} - \text{Sample Result})}{(\text{Amount Spiked})} \times 100 = \text{Matrix Spike Recovery}$$

4.18 METHOD BLANK. The laboratory must digest or distill (as appropriate to the method) and/or analyze a method blank with each set of samples. A minimum of one method blank is required for every 20 samples. For a running batch, a new method blank is required for each different digestion day. If no digestion step is required, then the method blank is equivalent to the reagent blank. The method blank must contain the parameter of interest at levels of less that the reporting limits for that parameter. If the method blank contains levels over the reporting limits, the samples must be redigested or redistilled and reanalyzed. The exception to this rule is when the samples to be reported contain greater than 10 times the method blank level. In addition, if all the samples are less than a client required limit and the method blank is also less than that limit, then the results can be reported as less than that limit.

4.19 PERCENT DIFFERENCE (%D) - To compare two values, the percent difference indicates both the direction and the magnitude of the comparison, i.e., the percent difference may be either negative, positive, or zero. (In contrast, see relative percent difference.)

4.20 PERCENT MOISTURE - an approximation of the amount of water in a soil/sediment sample made by drying an aliquot of the sample at 105 °C. The percent moisture determined in this manner also includes contributions from all compounds that may volatilize at or below 105 °C, including water. Percent moisture may be determined from decanted samples and from samples that are not decanted.

4.21 RELATIVE PERCENT DIFFERENCE (RPD) - To compare two values, the relative percent difference is based on the mean of the two values, and is reported as an absolute value, i.e., always expressed as a positive number or zero. In contrast, see percent difference.

4.22 REAGENT BLANK: The reagent blank is a blank that has the same matrix as the samples, i.e., all added reagents, but did not go through sample preparation procedures. The reagent blank is an indicator for contamination introduced during the analytical procedure. (Note: for methods requiring no preparation step, the reagent blank is equivalent to the method blank.) Either a reagent blank or a method blank must be analyzed with each batch of 20 samples or less. The concentration of the analyte of interest in the reagent blank must be less than the reporting limit for that analyte. If the reagent blank contains levels over the reporting limits, the samples must be reanalyzed. The exception to this rule is when the samples to be reported contain greater than 10 times the reagent blank level. In addition, if all the samples are less than a client required limit and the reagent blank is also less than that limit, then the results can be reported as less than that limit.

4.23 REAGENT GRADE: Analytical reagent (AR) grade, ACS reagent grade, and reagent grade are synonymous terms for reagents that conform to the current specifications of the Committee on Analytical Reagents of the American Chemical Society.

4.24 REAGENT WATER: Water that has been generated by any method which would achieve the performance specifications for ASTM Type II water. Water in which an interferant is not observed at or above the minimum quantitation limit of the parameters of interest. Accutest uses deionized water (municipal water which passes through Accutest's DI treatment system).

4.25 REFERENCE MATERIAL: A material containing known quantities of target analytes in solution or in a homogeneous matrix. It is used to document the bias of the analytical process.

4.26 SPIKE BLANK OR LAB CONTROL SAMPLE. Digest and analyze a laboratory control sample or spike blank with each set of samples. A minimum of one lab control sample or spike blank is required for every 20 samples (or 10 samples for certain methods). Assess laboratory performance against the control limits. In house limits should also be generated once sufficient external check standard data is available to generate limits (usually a minimum of 20 to 30 analyses). If the lab control is outside of the control limits for a parameter, all samples must be redigested or redistilled and reanalyzed for that parameter. The exception is if the lab control recovery is high and the results of the samples to be reported are less than the reporting limit. In that case, the sample results can be reported with no flag. Note: If control limits are not specified, then default limits of 80 to 120 percent should be used.

4.27 STANDARD ADDITION (MSA): The practice of adding known amounts of an analyte to one or more aliquots of a sample immediately prior to analysis. It is typically used to evaluate interferences.

4.28 STANDARD CURVE: A plot of concentrations of known analyte standards versus the instrument response to the analyte. Calibration standards are prepared by successively diluting a standard

solution to produce working standards that cover the working range of the instrument. Standards should be prepared at the frequency specified in the appropriate section. The calibration standards should be prepared using the same type of acid or solvent and at the same concentration as will result in the samples following sample preparation.

5.0 HEALTH & SAFETY

5.1 All safety practices must be followed as outlined in the Accutest Laboratories Employee Safety Handbook and Chemical Hygiene Plan. Safety glasses, gloves, and lab coats must be worn. All samples, solutions, and extracts must be treated as unknown and potentially hazardous.

5.2 The toxicity or carcinogenicity of each reagent used in this method has not been precisely determined; however, each chemical should be treated as a potential health hazard. Exposure to these reagents should be reduced to the lowest possible level.

6.0 COLLECTION, PRESERVATION, AND HOLDING TIMES

6.1 Collection and Preservation

6.1.1 Containers - 500 ml or 1L (plastic or glass).

6.1.2 Preservation – the sample should be stored at 4°C ± 2°C. No preservatives should be added to the sample.

6.2 Holding Time – All samples should be analyzed within 48 hours. (40 CFR 136 tables)

7.0 APPARATUS & MATERIALS

7.1 Dissolved oxygen meter and probe, YSI Model 5000 meter with YSI Model 5905 probe or equivalent.

7.2 300 ml BOD bottles with ground glass stoppers and plastic caps.

7.3 Low temperature incubator, Fisher Model 146 or equivalent.

7.4 Plastic Carboy, 20 liter size.

7.5 Buret.

7.6 Stir plates and stir bars.

7.7 Chlorine Test paper, capable of quantitating the level of chlorine present. Waterworks Free Chlorine and Total Chlorine Water Quality Test Strips.

8.0 STANDARDS & REAGENTS

Note: All chemicals listed are reagent grade unless otherwise specified. Distilled, deionized water should be used whenever water is required. All applicable standard/reagent preparation information, including vendor, lot number, date of preparation, expiration date, calculations, and

initials must be entered in the appropriate standard/reagent preparation logbook. Vendors typically used by Accutest include Fisher Scientific, VWR, Absolute Standards, Accustandard, Supelco, Chemservices, Ultra, and ERA. Additional vendors may be utilized as necessary.

8.1 POLYSEED - EPA approved Lyophilized bacteria for BOD seed.

8.2 Glucose, reagent grade.

8.3 Glutamic acid, reagent grade.

8.4 Phosphate Buffer Solution. Dissolve 8.5 g of KH_2PO_4, 21.75 g of K_2HPO_4, 33.4 g of $Na_2HPO_4 \cdot 7H_2O$, and 1.7 g NH_4Cl in about 500 ml of DI water and dilute to 1 liter with DI water. The pH of this solution should be 7.2 without any further adjustment.

8.5 Magnesium Sulfate Solution. Dissolve 22.5 g of $MgSO_4 \cdot 7H_2O$ in DI water and dilute to a final volume of 1 liter.

8.6 Calcium Chloride Solution. Dissolve 27.5 g of $CaCl_2$ in DI water and dilute to a final volume of 1 liter.

8.7 Ferric Chloride Solution. Dissolve 0.25 g of $FeCl_3 \cdot 6H_2O$ in distilled water and dilute to 1 liter.

8.8 Manganous Sulfate Solution. In a 1000 ml volumetric flask, dissolve 480 g of $MnSO_4 \cdot 4H_2O$ in 500 ml DI water and dilute to 1000 ml with DI water. The $MnSO_4$ solution should not give a color when added to an acidified KI solution.

8.9 Dilution Water. See 10.4

8.10 Sodium Iodide Solution: Dissolve 500 g of NaOH and 135 g of NaI in 500 ml of DI water and dilute to 1000 ml.

8.11 Sodium Azide Solution: Dissolve 10 g of NaN3 in 40 ml DI water.

8.12 Alkali-Iodide-Azide reagent. Combine solutions 8.10 and 8.11 and mix.

8.13 Starch Solution.

8.14 Concentrated H2SO4.

8.15 Standard Sodium Thiosulfate Titrant. Dissolve 6.205 g of Na2S2O3·5H2O in DI water. Add 0.4 g of solid NaOH and dilute to 1 liter with DI water. Standardize with bi-Iodate solution.

8.16 Standard Potassium bi-Iodate Solution. Dissolve 0.8124 g KH(IO3)2 in DI water and dilute to 1000 ml.

8.17 Potassium Fluoride Solution. Dissolve 40 g of $KF \cdot 2H_2O$ in DI water and dilute to 100 ml with DI water.

8.18 Sodium Sulfite solution, Dissolve 1.575g of Na_2So_3 in 1000 ml of DI water. This solution is not stable and should be prepared as needed.

8.19 1.0 N H_2SO_4 Solution: Slowly and while stirring, add 28 ml of conc. Sulfuric acid to 500ml of DI water. Mix and cool. Dilute to 1000ml with DI water.

8.20 1.0N Sodium Hydroxide, Dissolve 40 g of Sodium Hydroxide in 500ml of DI water. Mix and cool. Dilute to 1000ml with DI water.

8.21 1+1 Acetic Acid, to 100ml of DI water add 100 ml of conc. Acetic acid. Mix.

8.22 Potassium Iodide (KI) Dissolve 10g of KI in 100 ml of DI water.

8.23 GGA Spike Solution: Dry glucose and glutamic acid at 103°C for 1 hour. Add 150 mg glucose and 150 mg glutamic acid to fresh DI water and dilute to 1 L. Prepare this solution fresh daily.

NOTE: Commercially prepared reagents equivalent to those discussed above may be used.

9.0 INTERFERENCES

9.1 See Section 10.9.1, and 10.9.2.

9.2 Samples supersaturated with DO- Samples containing DO concentration above saturation at 20°C may be encountered in cold waters or in water where photosynthesis occurs. To prevent loss of oxygen during incubation of such samples, reduce DO to saturation by bringing sample to 20+/- 3°C in partially filled bottle while agitating by vigorous shaking.

9.3 Samples containing Hydrogen peroxide-Hydrogen peroxide remaining in samples from some industrial bleaching processes such as those used at paper mills and textile plants can cause supersaturated oxygen levels. Mix such samples vigorously in open containers for sufficient time to allow the hydrogen peroxide to dissipate before setting up for the BOD tests. Check the H2O2 removal by observing dissolved oxygen conc. Over time during the mixing or by using the H2O2 specific test paper.

10.0 PROCEDURE

Below is a step by step procedure for the analysis of samples for BOD. Use the automated spreadsheet for documentation, and calculations of standardization, and the analysis. This application can be found on MAFILE1/APPS server.

10.1 It is important that the instrument be placed in the intended operating position, vertical, tilted, or on its back, before it is prepared for use and calibrated.

10.2 Below is the procedure to be followed for the analysis of water samples for 5 day BOD.

10.3 Auto Calibration of the Oxygen Meter.

10.3.1 Place the probe in a BOD bottle containing about 1” of fresh DI water.

10.3.2 Turn on the oxygen meter for 30 minutes and allow the probe to polarize and the temperature to stabilize. **Note: If calibration is performed prematurely the values will drift and may be out specification.**

10.3.3 Before calibration, Make sure there are no water droplets on the membrane. Water droplets will cause a low calibration reading. Excess water may be removed by shaking the probe downward. If droplets persist it may be necessary to carefully remove them using a clean cloth or paper towel.

10.3.4 From the main menu, press the **[CALIBRATE**] soft-key to change to calibration mode. Verify the instrument barometer reading against the barometer. If they do not agree, from the calibration menu press the **[DO CAL]** soft-key, then press the **[NEXT]** soft-key until the barometric pressure is flashing. Using the **[UP]** and **[DOWN]** soft-key, enter the true local barometric pressure. Press **[ENTER]** to confirm. The message "**PRESSURE CALIBRATION SAVED"** will be displayed.

10.3.5 Make sure the display readings are stable, and then press the **[AUTO CAL]** soft-key to calibrate dissolved oxygen. The message "**D.O. CALIBRATION SAVED"** will be display.

10.3.6 Press **[MODE]** to return to the main mode. The instrument is now calibrated and ready to measure dissolved oxygen and temperature. Ambient room temperature should also be recorded and documented on the BOD spreadsheet.

10.3.7 Verify the calibration against Winkler titration. Winkler titration is performed in duplicate; record both the individual and average results. If results do not agree, then a manual calibration must be performed. Refer to instrument manual, and area supervisor.

10.4 Preparation of Dilution Water.

10.4.1 Add 1 ml of the magnesium sulfate solution, 1 ml of the calcium chloride solution, 1 ml of the iron (III) chloride solution, and 1 ml of the phosphate buffer solution for every liter of aged (48 hours) DI water being prepared.

10.4.2 Aerate the dilution water by rapidly circulating the water or by bubbling compressed air into the water for 30-60 minutes. Ensure that the dissolved oxygen concentration is at least 7.5 mg/L, and the temperature to be 20+/- 3°C.

10.4.3 Use a siphon hose and establish the flow of the dilution water when ready to set up samples.

10.5 Preparation of Seed.

10.5.1 Put 500 ml of dilution water in a 1000 ml beaker.

10.5.2 Add one capsule of POLYSEED to the seed bottle. (That is, open the capsule and pour the contents into the bottle).

10.5.3 Aerate and stir the seed for one hour before use and use within 6 hours of preparation.

10.6 Winkler Titration. This procedure must be done weekly. Follow steps in Section 10.10.

10.7 Analysis of samples for BOD.

10.7.1 Prepare seven BOD bottles as follows:

10.7.2 Dilution Water Blank: Fill one BOD bottles with dilution water (10.4.1).

10.7.3 Seed Control: Set up five BOD bottles and prepare five samples at 2.0mls, 5mls, 7.5mls, 10mls and 12.5mls of POLYSEED and dilute to the neck of the BOD bottle with dilution water (10.4.1).

10.7.4 Seed Correction: Add 2.0 ml of POLYSEED solution to a BOD bottle and dilute to the neck of the BOD bottle with dilution water (10.4.1). The DO uptake attributed to the seed should be in the range of 0.6 and 1.0 mg/L.

10.7.5 Glucose/Glutamic Acid Check Solution:

10.7.5.1 Add 2.0 ml of POLYSEED solution and 6 ml of glucose/glutamic acid solution (8.23) to a BOD bottle. Dilute to the neck of the BOD bottle with dilution water.

10.8 Sample Prep

10.8.1 Adjust the sample temperature to 20+/- 3°C. Check the pH of samples. If it is not between 6.0 and 8.0, adjust the pH between 7.0 and 7.2 with either solution 8.19 or 8.20. Record the initial, adjusted pH, and sample temperature.

10.8.2 Check the samples for the presence and levels of residual chlorine using chlorine test papers. If chlorine is present, calculate the amount of sodium sulfite solution needed as follows.

10.8.2.1 Measure 100 ml of pH adjusted sample and add 1 ml of 1+1 acetic acid (8.21), 1.0 ml of potassium iodide solution (8.22) and titrate with Na_2SO_3 (8.18) to the starch-iodide endpoint for residual chlorine and record the volume.

10.8.2.2 Add to the sample the relative volume of Na_2SO_3 solution determined above, mix and after 10 to 20 minutes check sample for residual chlorine.

Note: Excess Na_2SO_3 exerts an oxygen demand and reacts slowly with certain organic chloramine compounds.

10.8.3 Prepare sample bottles by adding desired sample volume, and fill with dilution water approximately two-thirds full. Add 2 ml of seed to each bottle. Fill the bottle with dilution water.

10.8.4 There should also be a duplicate dilution series for every twenty samples.

10.8.5 Determine dilutions for samples from previous history or from type of sample. Dilutions should be planned so that a depletion of 2 mg/l and a residual DO of at least 1 mg/l are obtained. A range of dilutions will be required to meet the criteria. With well known samples, three dilutions may be used. For unknowns, use a minimum of six dilutions as follows:

Mls of Sample				**Dilution %**
3.0				1.0
5.0				1.7
10	+	Dilute to the neck of BOD bottle with Dilution Water	=	3.3
50				16.7
100				33.3
300				100

NOTE: For dilutions greater than 1:100 make a primary dilution before making final dilution to the bottle.

10.8.6 Read initial DO for each bottle immediately (within 30 minutes) after adding dilution water to each bottle (fill to neck so that stoppered bottle contains no air bubbles) and record in BOD log book.

10.8.7 Cap bottles with plastic cap over glass stopper. Place in incubator for 5 days at 20º +/- 1°C.

10.8.8 After 5 d ± 6h of incubation, read the final DO for each bottle and record results in the BOD logbook.

10.9 CALCULATIONS

10.9.1 Determine the seed correction by calculating the linear regression for the DO depletion VS. the ml of seed used for the seed control bottles. The slope of the line will be the DO depletion per ml. Since 2ml of seed are normally used, the seed correction should be 2 times the slope.

10.9.2 Calculate the BOD concentration in water samples as follows:

$$5\ Day\ BOD = \frac{(Initial\ DO - Final\ DO) - Seed\ Correction}{Percent\ of\ Sample\ Used}$$

10.9.3 Calculate the average of appropriate results for each sample.

NOTE: The difference between the initial and the final DO should be 2.0 or greater for an accurate calculation. In addition, the final DO should be 1.0 or greater. If the final DO is less than 1.0, the value must be reported as greater than the largest value that would be obtained for that dilution factor. The final result must be the average of all dilutions that meet the above criteria.

10.10 PROCEDURES FOR WINKLER TITRATION FOR DO STANDARDIZATION.

10.10.1 Winkler Titration is analyzed in duplicate. To two 300 ml aliquots of dilution water, add 1 ml of $MnSO_4$ solution followed by 1 ml of alkali-iodide-azide reagent. Stopper the bottle, making sure to exclude any air bubbles, and mix by inverting the bottle several times. Let settle until there is a clear supernatant in the top 2/3 of the bottle.

10.10.2 Add 1.0 ml conc. sulfuric acid. Restopper and mix until dissolution is complete.

10.10.3 Titrate a volume corresponding to 200 ml original after correction for sample loss by displacement with reagents with 0.025 M sodium thiosulfate solution to a light straw color.

10.10.4 Add a few drops of starch solution and continue titration to the first disappearance of the blue color.

10.10.5 If the end point is overrun, add a measured volume of the treated sample and again titrate to the first disappearance of the blue color.

10.10.6 Calculations-For titration of 200 ml sample,

1 ml of 0.025 M sodium thiosulfate = 1 mg DO/L.

Record the results in the BOD logbook and average results.

Compare the results of the Winkler titration with the results of the DO meter for dilution water blank. The results for each method should be within 10% of each other. If they are not, do not continue with any analyses until the problem has been corrected.

11.0 QUALITY ASSURANCE

11.1 Winkler titration is to be done at least once per month for calibration of the DO meter. This is recorded in the BOD logbook.

11.2 An independent source standard should be analyzed at least every 6 months. The results should be within the acceptance criteria provided by the manufacturer. If the independent source standard does not meet acceptance criteria, corrective action must be taken to determine the source of the problem before sample analysis can continue.

11.3 The seed correction standard must be analyzed with each batch and should have a result between 0.6 mg/l and 1.0 mg/l.

11.4 One dilution water blank must be analyzed with each batch and should have a result of 0.20 mg/l or less. If not- suspect a contamination. Analyze the samples and footnote the associated samples accordingly.

11.5 A glucose spike must be prepared and analyzed for every 20 samples or once per day, whichever is greater. The glucose spike results should be evaluated against in-house generated limits of 198

+/- 30.5 mg/L. If the glucose spike does not meet acceptance criteria corrective action must be taken to determine the source of the problem before sample analysis can continue. Associated samples must be re-analyzed or the data footnoted.

NOTE: If the quality control limits for any of the above points are not met, consult the laboratory manager before proceeding with the analysis.

11.6 The seed control standards discussed above must be analyzed. A plot of **DO** depletion, in milligrams per liter, versus milliliter of seed, should present a straight line for which the slope indicates **DO** depletion per milliliter of seed. The **DO** axis intercept is oxygen depletion caused by the dilution water and should be less than 0.20 mg/L.

11.7 Sample results showing large differences (>30%) between the high and low results should be monitored. If a repeated pattern exists the problem should be investigated to identify the cause.

11.8 A Precision and accuracy (P&A) study is performed as an initial determination of capability, on an annual basis (continued demonstration of capability – a successful PT result may be used in place of a P&A for continued DOC), and if any significant changes have been made to the instrument. In general, 4 replicates or blank spikes are analyzed using the same procedures and conditions for sample analysis. The percent recoveries are compared to either default limits or in-house control limits of 198 +/- 30.5 mg/L. The standard deviation of the 4 replicate percent recoveries are compared to either ± 20 or to in-house limits once established. If percent recovery or standard deviation criteria are not met, corrective action must be taken to bring the system back into control.

12.0 DOCUMENTATION

12.1 The analytical logbook is a record of the analysis sequence; the logbook must be completed daily. Each instrument will have a separate logbook.

12.2 If samples require reanalysis, a brief explanation of the reason should be documented in this log.

12.3 The standard preparation logbook application must be completed for all standard preparations. All information requested must be completed; the page must be signed and dated by the respective person.

12.4 The Accutest lot number must be cross-referenced on the standard vial/container.

12.5 The instrument Maintenance logbook must be completed when any type of maintenance is performed on the instrument. Each instrument will have a separate log.

12.6 All laboratory logbooks must be routinely reviewed and initialed or signed by the lab manager.

12.7 Any corrections to laboratory data must be done using a single line through the error. The initials of the person and date of correction must appear next to the correction.

13.0 DATA REVIEW

13.1 The analyst conducts the primary review of all data. This review begins with a check of all Instrument and method quality control and progresses through sample quality control concluding with a check to assure that the client's requirements have been executed. The analyst has the authority and responsibility to perform corrective action for any out-of-control parameter of non-conformance.

13.2 A secondary review is performed by department managers, and it includes review of the data produced by their department. All manual calculations, QC criteria, and a comparison of the data package to client specified requirements are checked. The department manager may reject data, initiate reanalysis, take additional corrective action, or reprocess data.

13.3 The laboratory director performs a full tertiary review of the data package following its assembly. This review includes an evaluation of QC data against acceptance criteria and a check of the data package contents to assure that all analytical requirements and specifications were executed.

13.4 Spot-check reviews are performed by the Quality Assurance Officer focusing on all elements of the deliverable including the client's specifications and requirements, analytical quality control, sample custody documentation and sample identification.

14.0 DATA REPORTING

14.1 A results page including positive results and/or RLs, units, methodology, preparation and/or analysis dates, and data qualifiers are reported. Additional quality control data including calibration summaries, MS/duplicate percent recoveries and RPDs, blank spike recoveries, and method blank results (as applicable to the method) may be reported upon request of the client. Additionally, raw data including any instrument printouts, laboratory logbooks, etc. may be reported to the client.

14.2 Data may be submitted to the client in a specified electronic format (EDD).

14.3 Data may be submitted to the client electronically as a PDF (e-hardcopy).

14.4 Once the data is approved by the laboratory manager, it may be accessed by clients via LabLink™.

15.0 POLLUTION PREVENTION & WASTE MANAGEMENT

15.1 Pollution Prevention. Users of this method must perform all procedural steps that control the creation and/or escape of wastes of hazardous materials to the environment. The amounts of standards, reagents, and solvents must be limited to the amounts specified in this SOP. All safety practices designed to limit the escape of vapors, liquids, or solids to the environment must be followed. All method users must be familiar with the waste management practices described in section 15.2.

15.2 Waste Management. Individuals performing this method must follow established waste management procedures as described in the Sample and Waste Disposal SOP. This document describes the proper disposal of all waste materials generated during the testing of samples as follows:

15.2.1 Non-hazardous aqueous wastes
15.2.2 Hazardous aqueous wastes
15.2.3 Chlorinated organic solvents

15.2.4 Non-chlorinated organic solvents
15.2.5 Hazardous solid wastes
15.2.6 Non-hazardous solid wastes

16.0 ADDITIONAL REFERENCES

16.1 No additional references are required for this method.

ACCUTEST LABORATORIES
STANDARD OPERATING PROCEDURE

Lab Manager: Brad Madadian

QA Officer: Robert Treggiari

TITLE: TOTAL ORGANIC CARBON IN SOILS SAMPLES USING SW846 METHOD 9060 (MODIFIED), AND US EPA REGION 2 LLOYD KAHN METHOD.

TEST METHOD REFERENCES: SW846 Method 9060, September 1986, modified, and EPA Region 2 Lloyd Kahn Method, July 1988.

REVISED SECTIONS: 8.1; 9.5; 10.1; 10.2; 10.3; 10.4; 10.4.1; 10.4.2; 10.4.3; 10.5; 10.6; 10.6.1; 10.6.2; 10.6.3; 10.6.4; 10.6.5; 10.6.6; removed 10.6.7; revised 10.7.3; 10.8.2; 10.8.3; 10.8.4; 10.8.5; 10.8.6; 10.8.7; 10.9; 10.9.3; 11.2; 11.3; 11.5

1.0 SCOPE AND APPLICATION

1.1 This method can be used to determine total organic carbon in any solid matrix. It may also be used for liquid matrices containing a high level of total organic carbon. Samples that are primarily aqueous may also be analyzed using this method, but sample sizes should be limited to $\leq$ 0.10 g.

1.2 The product code for total organic carbon is TOC for the Corp. of Engineers methods and for the modified SW846 9060 method. The product code is TOCLK for total organic carbon run by the Lloyd Kahn EPA Region 2 method.

2.0 SUMMARY

2.1 Total organic carbon is determined by combusting an acidified sample and quantitating the carbon dioxide released using infrared analysis. The quantitation is done by comparison to a linear calibration curve.

3.0 REPORTING LIMIT AND METHOD DETECTION LIMIT

3.1 The Reporting Limit (RL) is based on the lowest calibration standard. RL'S may vary depending on matrix difficulties, sample volumes or weights, and percent moisture. Detected concentrations below this concentration cannot be reported without qualification.

3.2 The normal reporting limit for TOC in soils is 1000 mg/kg. This is based on a 0.1 g sample size. A minimum reporting limit of 100 mg/kg can be obtained by using a 1.0 g sample size. A reporting limit of 100 mg/kg is required for samples being analyzed for Lloyd Kahn TOC. A low level calibration standard is run at the level of this reporting limit.

3.3 The Method Detection Limit (MDL) represents the lowest reportable concentration of an individual analyte that meets the method qualitative identification criteria.

3.4 Method Detection limits (MDLs) are experimentally determined using the procedures described in 40 CFR, Part 136, Appendix B. Actual reported MDLs incorporate the sample volume analyzed and sample dilutions if needed, which may cause MDL variations from sample to sample.

3.5 In general, MDLs are determined through the analysis of at least 7 replicate blank spikes (using the same procedures for sample analysis). The MDL is calculated by multiplying the standard deviation of the replicate concentrations by the appropriate Student's t value (3.143 for 7 replicates). If more than 7 replicates are analyzed refer to 40 CFR, Part 136, Appendix B for the appropriate student's t value. MDLs are determined initially (prior to analysis), on an annual basis, and after major maintenance to equipment. MDL data is archived with Quality Assurance. Refer to the most recent study for current MDLs.

3.6 Current MDL studies are filed with Quality Assurance. Obsolete MDL studies are archived with the QA files. Electronic MDL data is found in the annual "MDL" folder on the QA server (LINUXMA1).

4.0 DEFINITIONS

4.1 ALIQUOT - a measured portion of a sample, or solution, taken for sample preparation and/or analysis.

4.2 BATCH: A group of samples which behave similarly with respect to the sampling or the testing procedures being employed and which are processed as a unit. For QC purposes, if the number of samples in a group is greater than 20, then each group of 20 samples or less will all be handled as a separate batch.

4.3 CALIBRATION CHECK STANDARD. The calibration check standard is a mid-range calibration standard. The calibration check standard must be run at a frequency of 10 percent. The acceptance criteria for this standard is $\pm$ 10 percent of the true value.

4.4 EXTERNAL CHECK STANDARD. The external check standard is a standard from a separate source than the calibration curve that is used to verify the accuracy of the calibration standards. An external check must be run a minimum of once per quarter for all analyses where a check is commercially available. If the external check is outside of the control limits for a given parameter, all samples must be reanalyzed for that parameter after the problem has been resolved.

4.5 INITIAL CALIBRATION VERIFICATION – analysis of a check standard from a second source (either vendor or lot) from the initial calibration standards to verify the initial calibration.

4.6 SPIKE BLANK SAMPLE. Analyze a laboratory control sample or spike blank with each set of samples. A minimum of one lab control sample or spike blank is required for every 10 samples. Assess laboratory performance against the control limits specified in the SOP. In house limits should also be generated once sufficient data is available to generate limits (usually a minimum of 20 to 30 analyses). If the lab control is outside of control limits, all

samples must be reanalyzed. The exception is if the lab control recovery is high and the results of the samples to be reported are less than the reporting limit. In that case, the sample results can be reported with no flag. Note: If control limits are not specified in the SOP, then default limits of 80 to 120 percent should be used.

4.7 LAB CONTROL SAMPLE. A solid lab control sample from an external source may be analyzed with a batch, depending on individual client requirements. The solid lab control is evaluated using manufacturer's limits. If the lab control is outside of the control limits for a parameter, all samples must be re-analyzed. The exception is if the lab control recovery is high and the results of the samples to be reported are less than the reporting limit. In that case, the sample results can be reported with no flag.

4.8 MATRIX: The component or substrate (e.g., water, soil) which contains the analyte of interest.

4.9 MATRIX SPIKE DUPLICATE: A duplicate sample is spiked and analyzed at a minimum of 1 in 10 samples. The relative percent difference (RPD) between the MS and the MSD should be assessed. The duplicate RPD is calculated as shown below. Assess laboratory performance against the in house limits. In house limits are generated once sufficient duplicate data is available to generate limits (usually a minimum of 20 to 30 analyses). If a duplicate is out of control, flag the results with the appropriate footnote. If control limits are not specified in the SOP, use default limits of ± 20% RPD.

$$\frac{(|\text{MS Result} - \text{MSD Result}|) \times 100}{(\text{MS Result} + \text{MSD Result})/2} = \text{Duplicate RPD}$$

4.10 MATRIX SPIKE: The laboratory must add a known amount of analyte to a minimum of 1 in 10 samples. The matrix spike recovery is calculated as shown below. Assess laboratory performance against the in house control limits. If a matrix spike is out of control, then the results should be flagged with the appropriate footnote. If the matrix spike amount is less than one fourth of the sample amount, then the sample cannot be assessed against the control limits and should be footnoted to that effect. Note: If control limits are not specified in the SOP, then default limits of 75 to 125 percent should be used.

$$\frac{(\text{Spiked Sample Result} - \text{Sample Result})}{(\text{Amount Spiked})} \times 100 = \text{Matrix Spike Recovery}$$

4.11 METHOD BLANK. The laboratory must analyze a method blank with each set of samples. A minimum of one method blank is required for every 10 samples. The method blank must contain the parameter of interest at levels of less than the reporting limit for that parameter. If the method blank contains levels over the reporting limits, the samples must be reanalyzed. The exception to this rule is when the samples to be reported contain greater than 10 times

the method blank level. In addition, if all the samples are less than a client required limit and the method blank is also less than that limit, then the results can be reported as less than that limit.

4.12 METHOD DETECTION LIMITS (MDLS). MDLs should be established for all appropriate methods, using a solution spiked at approximately 3 times the estimated detection limit. To determine the MDL values, take seven replicate aliquots of the spiked sample and process through the entire analytical method. The MDL is calculated by multiplying the standard deviation of three replicate analyses by 3.14, which is the student's t value for a 99% confidence level. If more than seven replicates are analyzed, the appropriate student's t value must be used (refer to 40 CFR pt 136, App. B). MDLs should be determined annually or after major maintenance to the instrument.

4.13 REAGENT GRADE: Analytical reagent (AR) grade, ACS reagent grade, and reagent grade are synonymous terms for reagents which conform to the current specifications of the Committee on Analytical Reagents of the American Chemical Society.

4.14 REAGENT WATER - water in which an interferant is not observed at or above the minimum quantitation limit of the parameters of interest. Accutest uses deionized water (municipal water which passes through Accutest's DI treatment system).

4.15 RELATIVE PERCENT DIFFERENCE (RPD) - As used in this SOP to compare two values, the relative percent difference is based on the mean of the two values, and is reported as an absolute value, i.e., always expressed as a positive number or zero. In contrast, see percent difference.

4.16 PERCENT DIFFERENCE (%D) - As used in this SOP and elsewhere to compare two values, the percent difference indicates both the direction and the magnitude of the comparison, i.e., the percent difference may be either negative, positive, or zero. (In contrast, see relative percent difference.)

4.17 REFERENCE MATERIAL: A material containing known quantities of target analytes in solution or in a homogeneous matrix. It is used to document the bias of the analytical process.

4.18 STANDARD CURVE: A plot of concentrations of known analyte standards versus the instrument response to the analyte. Calibration standards are prepared by successively diluting a standard solution to produce working standards, which cover the working range of the instrument. Standards should be prepared at the frequency specified in the appropriate section. The calibration standards should be prepared using the same type of acid or solvent and at the same concentration as will result in the samples following sample preparation. This is applicable to organic and inorganic chemical analyses.

5.0 HEALTH & SAFETY

5.1 The analyst should follow normal safety procedures as outlined in the Accutest Laboratory Employee Safety Plan and Chemical Hygiene Plan, which include the use of safety glasses and lab coats. In addition, all acids are corrosive and should be handled with care. Flush spills with plenty of water. If acids contact any part of the body, flush with water and contact the supervisor.

5.2 The furnace operates at high temperature and the furnace should be allowed to cool down before doing any system maintenance or troubleshooting. If there are any signs of a system blockage, open the sample introduction port and turn off the furnace to prevent build up of back pressure.

5.3 The toxicity or carcinogenicity of each reagent used in this method has not been precisely determined; however, each chemical should be treated as a potential health hazard. Exposure to these reagents should be reduced to the lowest possible level.

6.0 PRESERVATION & HOLDING TIME

6.1 Collection and Preservation

6.1.1 Containers: Samples should be collected in 2 oz glass jars with Teflon-lined lids.

6.1.2 Preservation: Samples should be stored at 4°C ± 2°C.

6.2 Holding time is 28 days from date of collection.

6.3 A 14 day holding time should be followed when analyzing TOC soils following the EPA Region II Lloyd Kahn method.

7.0 INTERFERENCES

7.1 High results may be obtained if the inorganic carbon is not completely removed from the sample before analysis. To ensure that all of the inorganic carbon is removed, heat an acidified sample at least 10 minutes at 75°C before starting the analysis. Some volatile organics may be lost in this heating step, resulting in a low bias in the TOC result.

8.0 APPARATUS

The following items are needed for the analysis of samples following the method outlined below:

8.1 Shimadzu TOC-L analyzer with soil analysis module or equivalent.

8.1.1 Each day of analysis, the humidifier/water trap should be checked to ensure that the water level is within 1 inch of the top of the humidifier.

8.1.2 Each day of analysis, the baseline should be checked to make sure that it is stable and near zero.

8.1.3 Whenever calibration check recoveries or blanks are out of compliance, the flow and the condition of the catalyst should be checked. If the catalyst appears bad (contains many small fines), it should be cleaned and replaced. Refer to the instrument manuals for additional information on system maintenance.

8.2 Syringes, 0.100, or 0.250 ml size.

8.3 Analytical balance, capable or weighing to 0.1 mg. The calibration of the analytical balance should be verified each day before use.

8.4 Volumetric glassware, class A. for standards preparation.

8.5 Ceramic boats. Ceramic Fiber

8.6 Drying oven, capable of being set to 75°C

8.7 Desiccator with metallic shelf

9.0 REAGENTS

All chemicals listed below are reagent grade unless otherwise specified. Deionized water taken from the DI taps in the organics lab should be used whenever water is required. Make sure to properly label all reagents and record the reagent preparation in the reagent logbook (including vendor, lot number, date of preparation, any calculations, and initials). Vendors typically used by Accutest include Fisher Scientific, VWR, Accustandard, Supelco, Absolute Standards, Ultra, and ERA. Additional vendors may be utilized as necessary.

9.1 Calibration Stock Solution, Already made KHP Calibration standards are purchased from Absolute Standards, Inc.

TOC 50000 mgC/L
TOC 25000 mgC/L
TOC 20000 mgC/L
TOC 10000 mgC/L
TOC 5000 mgC/L
TOC 2000 mgC/L
TOC 1000 mgC/L

9.2 Initial Calibration Verification (ICV). Already made KHP ICV standard is purchased from Absolute Standards, Inc. This second source standard is made at TOC concentration of 20000 mgC/L

9.3 Nitric Acid, reagent grade. Used for acidifying samples to remove inorganic carbon.

9.4 Oxygen Gas, high purity.

9.5 Phosphoric Acid, reagent grade. Used to acidify samples to remove inorganic carbon (For Lloyd Khan Method).

10.0 PROCEDURE

Below is the procedure to be followed for the analysis of soil samples for total organic carbon using the Shimadzu TOC soil analyzer.

10.1 Turn on the oxygen. The pressure in the soil module should be set at 200 kPa and the carrier gas should be set as marked on the dial (0.5 l/min). The oxygen pressure at the tank must be at least 80 psi to maintain sufficient pressure at the instrument. Check to make sure that the water trap contains sufficient water. It should be filled to within approximately 1 inch of the top of the humidifier/water trap. (The humidifier is located under the magnetic plate on the right side of the instrument)

10.2 Turn on the instrument using the main power at the right backside towards the top of the TOC instrument and then turn on the power at the side of the soil modules. After a few seconds the TOC instrument will shut down which means the instrument ready condition is not established. Turn on the power switch on the left of the instrument front door. The lit power switch will change from orange to green when ready.

10.3 Go into the TOC software by double clicking the TOC-Control L icon on windows desk top. Double click on sample table editor, enter "**Wetchem**" for name, and "**TOC2012**" for password. Note: software is case sensitive.

10.4 Go to **H/W Setting** on the sample table and double click on it. **TOC AQ** and **TOC SO** will appear below the **H/W Setting**. If Soil samples are to be analyzed choose **TOC SO**. Using a right click on the mouse, choose the **TOC SO** icon and a menu will appear that follows:

Line1: Connect
Line2: Background Monitor Setting
Line3: Shutdown
Line4: Maintenance
Line5: Instrument Setting

10.4.1 Click on **Connect**, a **Sequence** window will pop up. It indicates the sequence which is currently in progress. Also, the open port in this window should indicate 100% otherwise the instrument and the PC are not communicating.

NOTE: If the open port is not at 100%, shut down the software and the instrument and then reboot up the PC. Restart the start up process beginning with step 10.2 through 10.4.

10.4.2 Once the communication is established, click on the **Instrument Setting** in the 10.4 menu and the **Instrument Properties** window will appear. Go to the **SSM** tab in **Instrument Properties** and click on **SSM TC Furnace and IC Furnace**, then click

OK. The SSM module furnace will now begin to heat. The TC Furnace temperature must reach 900°C on the SSM module display before analysis may start.

10.4.3 While the furnace is heating up, go to the 10.4 menu and click on **Background Monitor Setting.** A window will come into view that is called the **peak view window.** Set the view setting at 50x and monitor the baseline. Let the instrument warm up for 30 to 45 minutes. Once the instrument is ready the baseline will be flat or in a slight seesaw motion.

10.5 Verify the carrier gas flow rate displayed on the **TOC** tab in the **Background Monitor** window reads 150 ml/min. Never allow the carrier gas supply pressure to exceed 250 KPA.

10.6 **Calibration**

If the instrument has not been calibrated within the last 3 month, then the instrument must be calibrated at this point (A new calibration is required at least once per quarter). Confirm the temperature of SSM has reached 900°C and stabilized. See section 9.1 for the calibration standards used for each curve. Calibration curve must be analyzed separately and without any attached samples.(this will enable the calibration curves to be printed out and included with future analysis run packages).

If a new calibration curve is to be run:

10.6.1 In the "**Toc-Control L Sample Table** "; click on **New** and the "**Select H/W Setting"** window will open. At **System** comb box select **TOC SO** and click **OK.** A blank template will open. Save the template by going to the toolbar and click on **file**. Next, click on **save as**, then name the file in the following format; TOC day month year S (for solid). Save the file. Example: **TOC101801S**. On the top toolbar, go to "**insert**", then click on "**calibration curve**". A window will open with several calibration curve templates. Choose **TOC Soil Cal Template** and click on it. The calibration curve file will install on the current template. On the toolbar, click on the **Start** button. The "**TOC Measurement**" window will now open. Click "**Connect**" and then click "**Start**" on this window. The "**Enter Sample Amount**" window will now open. Enter in the appropriate volume for Standards (STDs) and place the loaded ceramic boat with the standard into the SSM. Click on start in the "**Enter Sample Amount**" window.

10.6.2 A dialog box will appear to instruct the user to push the sample to the measurement position marked on SSM. Meanwhile, the measurement peaks may be viewed during the analysis with the sample window which can be accessed from the "**view**" on the toolbar or the second icon at the top right corner of the current templates. The software will indicate when to move the sample back to the cooling position. After staying in the cool position, the software will instruct the analyst to move the sample to the preparation position.

10.6.3 The "**Toc Measurement**" window now will be displayed and the previously analyzed sample results can be viewed. In this box you may also "**Repeat**", "**Excluded**", "**Stop**" or go to the "**Next**" sample.

10.6.4 Select the option to repeat the injection and repeat the steps outlined above (Minimum of two injections with CV less than 15% or RPD less than 20%). In "**TOC Measurement**" window you may also "**Exclude**" or "**Stop**".

10.6.5 When the last replicate of the last standard has been completed, click "**stop**" in the "**TOC measurement**" window.

10.6.6 If a correlation coefficient of greater than 0.995 is obtained, then save the curve using the file, save option. Check to make sure that the intercept, calculated using a weight of 1.0 g for Lloyd Kahn or using a weight of 0.1 g for other TOC analyses, is less than the reporting limit for each method (100 mg/kg for Lloyd Kahn or 1000 mg/kg for other TOC soils.) Note: If either the correlation coefficient or the intercept does not meet the above criteria, than recalibrate before proceeding with the samples.

10.7 **Sample Preparation**

10.7.1 Begin preparing the samples following the procedure outlined below.

Weigh out from 100 to 1000 mg of sample (wet weight) into a ceramic boat using a 4-place analytical balance. For samples that contain high levels of TOC smaller sample sizes may be needed. For unknown samples, start with a sample size of 100 mg. (All method blanks and spike blanks should be calculated assuming a 100 mg sample size.) Samples that contain non-homogeneous particulates should be homogenized with a mortar and pestle before weighing out the sample aliquot. All samples must be homogenized prior to preparation (refer to the Procedure for Representative Aliquots SOP MQA247). If a client is requiring a detection limit lower than 1000 mg/kg, then larger sample sizes are required. A detection limit of 100 mg/kg requires a weight of 1000 mg. A smaller sample size may be used only to bring the sample to within the range of the calibration curve.

10.7.2 Add nitric acid drop wise to the sample until no additional effervescence is observed and the surface of the sample is covered with the acid. Heat the acidified sample in an oven at 75°C for a minimum of 10 minutes.

10.7.3 With each batch of 20 samples or less, a MS and MSD should be analyzed. On each analysis day, a method blank and spike blank must be analyzed. All of these quality control points must be analyzed in duplicate. Prepare the method blank by treating a small amount (approximately 100 mg) of pre-baked Ottawa with nitric acid and heating at 75 º C for a minimum of 10 minutes.

10.7.4 Prepare the spike blank in the same manner as the method blank, but spike it with 100 ul of a 20000 mgC/l standard or external solution before adding the

acid.

10.7.5 Prepare the matrix spike duplicate in the same manner as a matrix spike. (10.7.6)

10.7.6 Prepare the matrix spike by adding 100 ul of a 20000 mgC/l standard or external solution to a sample aliquot before adding the acid and heating the sample.

10.8 **Sample Analysis**

10.8.1 Refer to section 10.1 through 10.5.

10.8.2 If a calibration curve is performed on the same day as the samples then, in the "**TOC-Control L Sample Table**" window, go to **TOC SO** folder and double click on **TOC SOLID TEMP** template. Select and highlight all samples in the template and go to "**edit**" and click on "**cut**". All the samples will be removed and the template is blank. Once a file is created it can no longer be overwritten. Go to "**insert**" on the toolbar and click on "**Multi Samples**".

10.8.3 A wizard will now open. On page one of the "**Sample Group Wizard (page 1) sample source**", click on calibration curve option button and then click on the browse button. A window will open. On "**File of Types**" comb box, select "**All Cal Curve Files (*.cal)**" to see all the past calibration curves. Select the curve that was analyzed the same day. The format of the curve is as follows:

Example: "TOC soil 2013_02_18_14_10_10.cal"

Notice that the original template name is followed by the year, month, day, hour, min, sec that the calibration curve was analyzed. Double click on the appropriate calibration curve to be used. Press "**Next**" to move to page two of the "**Sample Group Wizard (Page 2) Sample Parameter**.

10.8.4 On second page of the wizard, enter the number of samples including all QC, and CCV's. It is a good practice to choose more samples than needed as the run may be stopped at any time and more samples added to the analysis sequence for that day. Next remove any name that appears in the sample name field (for instance STD) and click "**finish**".

10.8.5 Now the template is ready to be used for the current day or any subsequent day that calibration isn't performed. Go to the toolbar and click on the save icon to save the file under TOC SOLID TEMP template. Do not use "save as". The save as file does not work here.

10.8.6 Next, save the file by clicking on "**file**", "**save as**", in the following format: Toc, month, day, year, S (for solids) and the number of the batch for that day. Example: Toc051313S1 or Toc051313S2 for the second run of the day.

10.8.7 Type in the sequence in "**sample field name**" as follows: CCV, ICV, QC samples (method blank, spike blank, matrix spike, and matrix spike duplicate) and then samples. After every 10 samples, analyze a CCV. After the sequence is entered, on the toolbar click "**Connect**" and then click "**start**". The "**Enter Sample Amount**" window will now open. Enter in the appropriate weight for sample and place the loaded ceramic boat with the sample into the SSM. Click on start in the "**Enter Sample Amount**" window.

10.8.8 Follow sections 10.6.2, 10.6.3, and 10.6.4.

10.9 If calibration curve is not performed the same day as the samples, the calibration must be verified with a low and a high standard and a blank (method blank can be used as blank) before proceeding on each analysis day. The low standard must be within 30 percent and the high within 10 percent of the true value. Make sure to use duplicate injections for all analyses.

10.9.1 After the calibration checks are completed, then analyze the continuing calibration verification standard (CCV). CCV should be a standard near the mid range of the curve. CCV results should agree within 10 percent of the true value. If the CCV is not within 10% of the true value, then no sample can be reported in the area bracketed by this CCV, unless the CCV is biased high (110 to 150%) and the sample results to be reported are less than the reporting limit. Analyze this standard after every 10 samples.

10.9.2 Analyze an external check standard (ICV). This standard must agree within 10 percent of the true value. If it is not within this range, determine the source of the problem, correct the problem, and recalibrate.

10.9.3 If the duplicate sample injections have a coefficient of variation (CV) of greater than 15 percent or 20% RPD, then repeat the analysis with 2 additional injections (unless the two injections are less than RL). If on the repeated analysis, a high CV or %RPD is still obtained, then report the average of 4 injections. The sample results should be reported with a flag due to possible sample non-homogeneity.

$$CV = (\text{Std Dev}_{n-1} / \text{mean}) \times 100$$

10.9.4 The final sample results are calculated using the equation shown below. The calculation is done automatically in the Shimadzu TOC software except for the percent solids correction. The percent solids correction is added when the data is transferred in the LIMS system. See area supervisor or manager for further details.

Organic Carbon, Total (mg/kg) =

$$\frac{\text{Conc. from curve}}{\%\text{sol}/100}$$

10.10 **GLASSWARE CLEANING**

10.10.1 Brush any adhering material from the boat and wash with soap and water and then Well rinsed with DI water. Soak in 2 M HCl solution for few minutes and rinse with DI water several times. Place the boats in muffle furnace at 900 Deg. C for 15 min.Cool, and store in desiccator.
Note: it is recommended to prepare the boats at the end of each analysis for next analysis date to conserve time.

11.0 QUALITY CONTROL

Below is a summary of the quality control requirements for this method. Make sure to check with the laboratory supervisor or manager for any additional client specific quality control requirements.

11.1 Calibration Curve. The instrument must be calibrated a minimum of once per quarter. It is recommended that the instrument be calibrated at least once per month. If the instrument is not calibrated on a given day, then the curve must be verified using a low and a high standard and a blank before proceeding on each analysis day. The low standard must be within 30 percent of the true value. All other check standards must be within 10 percent of the true value. The blank must contain less than the reporting limit for TOC.

11.2 Method Blank. The laboratory must prepare and analyze a method blank with each set of samples. A minimum of one method blank is required for every 20 samples. The method blank must contain the analyte at less that the reporting limit. If the method blank contains over that limit, the samples must reanalyzed. The exception to this rule is when the samples to be reported contain greater than 10 times the method blank level. In addition, if all the samples are less than a client required limit and the method blank is also less than that limit, then the results can be reported as less than that limit. (Note: For Florida samples, blanks should be prepared for 1 in 10 samples.)

11.3 Spike Blank. The laboratory must prepare and analyze a spike blank with each set of 20 or less samples. For a running batch, a new spike blank is required for each different analysis day. The laboratory should assess laboratory performance of the spike blank against recovery limits of 80 to 120 percent. (If the spike blank is used in place of the external, then it must be within recovery limits of 90 to 110 percent.) If the lab control recovery is high and the results of the samples to be reported are less than the reporting limit, then the sample results can be reported with no flag. In all other situations, all samples associated with a spike blank outside of recovery limits must be reanalyzed. (Note: For Florida samples, spikes should be prepared for 1 in 10 samples.)

11.4 Matrix Spike. The laboratory must add a known amount of each analyte to a minimum of 1 in 10 samples. (Note: For Florida samples, spikes should be prepared for 1 in 10 samples.)

11.4.1 The spike recovery should be assessed using in house limits. Until these limits can be generated, then default limits of 75 to 125 percent recovery should be applied. If a matrix spike is out of control, then the results should be flagged with the appropriate footnote. If the matrix spike amount is less than one fourth of the sample amount, then the sample cannot be assessed against the control limits and should be footnoted to that effect.

11.4.2 The matrix spike recovery should be calculated as shown below.

$$\frac{\text{(Spiked Sample Result - Sample Result)}}{\text{(Amount Spiked)}} \times 100 = \text{MS Recovery}$$

11.5 Matrix Spike Duplicate. The laboratory must add a known amount of each analyte to a minimum of 1 in 20 samples. The relative percent difference (RPD) between the matrix spike duplicate and the matrix spike should be assessed. (Note: For Florida samples, spikes should be prepared for 1 in 10 samples.)

11.5.1 The matrix spike duplicate RPD should be assessed using in house limits. Until these limits can be generated, then default limits of 20 percent RPD should be applied. If a duplicate is out of control, then the results should be flagged with the appropriate footnote.

11.5.2 The duplicate RPD should be calculated as shown below.

$$\frac{\text{(I MS Result - MSD Result I) x 100}}{\text{(MS Result + MSD Result) x 0.5}} = \text{\% RPD}$$

11.6 Quality Control Sample (also referred to as Initial Calibration Verification Standard, (ICV). A standard from a separate source than the calibration should be run at the beginning of each run to verify the initial calibration. This ICV should be within 10 percent of the true value. If it is not, the problem must be resolved before any samples can be analyzed. Note: The spike blank may be used in place of the ICV as long as a separate source standard is used and the 10 percent criterion is met.

11.7 Continuing Calibration Verification (CCV). Analyze the continuing calibration verification solution after every tenth sample and at the end of the sample run. If the CCV solution is not within 10 percent of the true value, then no samples can be reported in the area bracketed by that CCV. (Note: the exception is if the CCV is biased high (111 to 150%) and the samples are less than the detection limit. In that case, the samples can be reported with no flag.) The CCV concentration should be at or near the mid-range of the calibration curve.

11.8 Continuing Calibration Blank (CCB). For some clients, a continuing calibration blank (CCB) may be required. This is not required as part of the normal TOC protocol. If it is required, than it should be run after each CCV check. The results of the CCB must be less than the reporting limit for TOC. If the CCB is not less than the reporting limit, then no samples can be reported in the area bracketed by this CCB unless the sample results to be reported are less than the reporting limit.

11.9 A Precision and accuracy (P&A) study is performed as an initial determination of capability and on an annual basis (continuing demonstration of capability – a successful PT result may be used in place of a P&A for continuing DOC), and if any significant changes have been made to the instrument. In general, 4 replicates or blank spikes are analyzed using the same

procedures and conditions for sample analysis. The percent recovery is compared to the blank spike acceptance criteria. The standard deviation of the 4 replicate percent recoveries are compared to either ±20 or to in-house limits once established. If percent recovery or standard deviation criteria are not met, corrective action must be taken to bring the system back into control.

11.10 Quality Control data is generated (control charts) and reviewed on an annual basis by Quality Assurance (blank spike/ matrix spike recoveries and matrix spike duplicate RPDs).

12.0 DOCUMENTATION

12.1 All analytical data should be labeled with the sample ID and any dilutions made. The analyst should double check to make sure that all comments dealing with the run are recorded on the raw data.

12.2 The analytical logbook is a record of the analysis sequence; the logbook must be completed daily. Each instrument will have a separate logbook.

12.3 If samples require reanalysis, a brief explanation of the reason should be documented in this log.

12.4 The standard preparation logbook must be completed for all standard preparations. All information requested must be completed; the page must be signed and dated by the respective person.

12.5 The Accutest lot number must be cross-referenced on the standard vial/container.

12.6 The instrument Maintenance logbook must be completed when any type of maintenance is performed on the instrument. Each instrument will have a separate log.

12.7 All laboratory logbooks must be routinely reviewed and initialed or signed by the lab manager.

12.8 Any corrections to laboratory data must be done using a single line through the error. The initials of the person and date of correction must appear next to the correction.

13 DATA REVIEW

13.1 The analyst conducts the primary review of all data. This review begins with a check of all method quality control and processes through sample quality control concluding with a check to assure that the client's requirements have been executed. The analyst has the authority and responsibility to perform corrective action for any out-of-control parameter of non- conformance.

13.2 A secondary review is performed by department managers, and it includes review of the data produced by their department. All manual calculations, QC

criteria, and a comparison of the data package to client specified requirements are checked. The department manager may reject data, initiate reanalysis, take additional corrective action, or reprocess data.

13.3 The laboratory director performs a full tertiary review of the data package following its assembly. This review includes an evaluation of QC data against acceptance criteria and a check of the data package contents to assure that all analytical requirements and specifications were executed.

13.4 Spot check reviews are performed by the Quality Assurance Officer focusing on all elements of the deliverable including the client's specifications and requirements, analytical quality control, sample custody documentation and sample identification.

14 REPORTING

14.1 A results page including positive results and/or RLs, units, methodology, analysis dates, and data qualifiers are reported. Additional quality control data including calibration summaries, MS/MSD percent recoveries and RPDs, blank spike recoveries, and method blank results may be reported upon request of the client.

14.2 Data may be submitted to the client in a specified electronic format (EDD). Lab results are also available electronically via e-hardcopy upon request of the client.

14.3 Once the data is approved by the laboratory manager, it may be accessed by clients via LabLink™.

14.4 Procedures for handling non-conforming data.

14.4.1 If quality control data does not meet criteria the non-conformance must be discussed in a case narrative and footnoted on the applicable quality control report summary.

14.4.2 If preservation or holding time criteria is not met and the samples are analyzed the result page must be footnoted with this information, and the non-conformance must be discussed in a case narrative or other suitable communication (telephone conversation log or email). Client notification documentation should be included with the data (telephone conversation log, fax, or email).

15.0 POLLUTION PREVENTION & WASTE MANAGEMENT.

15.1 Pollution Prevention. Users of this method must perform all procedural steps that control the creation and/or escape of wastes of hazardous materials to the environment. The amounts of standards, reagents, and solvents must be limited to the amounts specified in this SOP. All safety practices designed to limit the escape of vapors, liquids, or solids to the environment must be followed. All method users must be familiar with the waste management practices described in section 15.2.

15.2 Waste Management. Individuals performing this method must follow established waste management procedures as described in the Sample and Waste Disposal SOP. This document describes the proper disposal of all waste materials generated during the testing of samples as follows:

15.2.1 Non-hazardous aqueous wastes
15.2.2 Hazardous aqueous wastes
15.2.3 Chlorinated organic solvents
15.2.4 Non-chlorinated organic solvents
15.2.5 Hazardous solid wastes
15.2.6 Non-hazardous solid wastes

16.0 METHOD PERFORMANCE

16.1 Method performance is evaluated by the annual QC limits (control charts) generated by QA, and the annual MDL study results. Refer to section 3.5 for MDLs, and section 11.10 for QC limits.

17.0ADDITIONAL REFERENCES

17.1There are no additional references for this SOP.

Lab Manager: Brad Madadian

QA Officer: Robert Treggiari

TITLE: METALS BY INDUCTIVELY COUPLED PLASMA ATOMIC EMISSION SPECTROMETRY SW846 6010C

TEST METHOD REFERENCE: SW846 6010C, Revision 3, February 2007

REVISED SECTIONS: 8.15, 11.10

1.0 SCOPE & APPLICATION

1.1 This method is applicable for the determination of Total metals in ground waters, domestic and industrial wastes, TCLP leachates, sludges, soils, sediments, and various other wastes.

NOTE: Dissolved elements are determined after filtration with 0.45 micron filter paper and preserved with Nitric acid for 24 hours prior to analysis.

1.2 Test Codes: A variety of metals can be analyzed by ICP. These include: Al, Sb, As, Au, B, Ba, Be, Cd, Ca, Cr, Cu, Co, Fe, Pb, Pd, Pt, Li, Mn, Mg, Mo, Ni, K, Se, Ag, Na, Si, Sn, Sr, Ti, Tl, V, W, Zn Zr.

2.0 SUMMARY

2.1 Prior to analysis, samples must be digested using the appropriate digestion method. When analyzing groundwater samples for dissolved constituents, acid digestion is not necessary if the samples are filtered and acid preserved prior to analysis.

2.2 This method describes multi-elemental determinations by ICP/AES using sequential or simultaneous optical systems and axial or radial viewing of the plasma. The instrument measures characteristic emission spectra by optical spectrometry. Samples are nebulized and the resulting aerosol is transported to the plasma torch. Element-specific emission spectra are produced by a radio-frequency ICP. Background correction is required for trace element determination.

3.0 METHOD DETECTION AND REPORTING LIMITS

3.1 Reporting limits are established at the lowest concentration standard. RL's may vary depending on matrix difficulties, sample volumes or weight, percent moisture. Detected concentrations below this concentration cannot be reported without qualification. See below table for analytes' RL:

Analyte	RL (ug/L)	Analyte	RL (ug/L)
Al	200	Mo	100
Sb	6	Ni	40
As	4	Pd	50
Ba	50	Pt	50
Be	4	K	5000
B	100	Se	10
Cd	4	Si	100
Ca	5000	Ag	5
Cr	10	Na	5000
Co	50	Sr	10
Cu	25	Tl	5
Au	50	Sn	100
Fe	100	Ti	50
Pb	5	W	100
Li	5000	V	10
Mg	5000	Zn	20
Mn	15	Zr	50

3.2 Method Detection Limits

3.2.1 Detection limits are experimentally determined using the procedures described in 40 CFR, Part 136, Appendix B. Actual reported MDLs incorporate the sample weight or volume analyzed and sample dilutions if needed, which may cause MDL variations from sample to sample.

3.2.2 In general, MDLs are determined through the analysis of at least 7 replicate blank spikes (using the same procedures for sample analysis). The MDL is calculated by multiplying the standard deviation of the replicate concentrations by the appropriate Student's t value (3.143 for 7 replicates). If more than 7 replicates are analyzed refer to 40 CFR, Part 136, Appendix B for the appropriate student's t value. MDL studies are performed on an annual basis or after any major changes to the instrumentation. For additional detail regarding MDL studies, refer to the MDL SOP MQA245.

3.2.3 The MDL represents the lowest reportable concentration of an individual analyte that meets the method qualitative identification criteria.

3.2.4 Current MDL studies are filed with Quality Assurance. Obsolete MDL studies are archived with the QA files. Electronic MDL data is found in the annual "MDL" folder on the QA server (LINUXMA1).

4.0 DEFINITIONS

4.1 ALIQUOT - a measured portion of a sample, or solution, taken for sample preparation and/or analysis.

4.2 BACKGROUND CORRECTION – a technique to compensate for variable background contribution to the instrument signal in the determination of trace elements.

4.3 BATCH – a group of samples which behave similarly with respect to the sampling or the testing procedures being employed and which are processed as a unit. For QC purposes, if the number of samples in a group is greater than 20 (or 10 for certain methods), then each group of 20 samples (or 10 samples for certain methods) or less will all be handled as a separate batch.

4.4 CALIBRATION – the establishment of an analytical curve based on the absorbance, emission intensity, or other measured characteristic of known standards. The calibration standards must be prepared using the same type of acid or concentration of acids as used in the sample preparation.

4.5 CALIBRATION BLANK - a volume of acidified deionized/distilled water.

4.6 CALIBRATION STANDARDS – a series of known solutions used by the analyst for calibration of the instrument (i.e., preparation of the analytical curve).

4.7 CONTINUING CALIBRATION – analytical standard run every 10 samples or 2 hours, whichever is more frequent, to verify the calibration of the analytical system.

4.8 DISSOLVED METALS – elements in an aqueous sample which will pass through a 0.45 um filter.

4.9 DRY WEIGHT – the weight of a sample based on percent solids. The weight after drying in an oven.

4.10 FIELD SAMPLE - a portion of material to be analyzed that is contained in single or multiple containers and identified by a unique sample number.

4.11 FIELD BLANK – this is any sample that is submitted from the field and is identified as a blank. This includes trip blanks, rinsates, equipment blanks, etc.

4.12 HOLDING TIME – the elapsed time expressed in days from the date of sampling until the date of its analysis.

4.13 INDUCTIVELY COUPLED PLASMA (ICP) – a technique for the simultaneous or sequential multi-element determination of elements in solution. The basis of the method is the measurement

of atomic emission by an optical spectroscopic technique. Characteristic atomic line emission spectra are produced by excitation of the sample in a radio frequency ICP.

4.14 INSTRUMENT CHECK SAMPLE – a solution containing both interfering and analyte elements of known concentration that can be used to verify background and interelement correction factors.

4.15 INSTRUMENT CHECK STANDARD – a multi-element standard of known concentrations prepared by the analyst to monitor and verify instrument performance on a daily basis.

4.16 INTERFERENTS – substances which affect the analysis for the element of interest.

4.17 MATRIX - the predominant material of which the sample to be analyzed is composed. For the purpose of this SOP, a sample matrix is either water or soil/sediment. Matrix is not synonymous with phase (liquid or solid).

4.18 MATRIX EFFECT - in general, the effect of a particular matrix (water or soil/sediment) on the constituents with which it contacts. This is particularly pronounced for clay particles which may adsorb chemicals and catalyze reactions. Matrix effects may prevent extraction of target analytes, and may affect surrogate recoveries. In addition, non-target analytes may be extracted from the matrix causing interferences.

4.19 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. The matrix spike recovery is calculated as shown below.

$$\frac{(\text{Spiked Sample Result} - \text{Sample Result})}{(\text{Amount Spiked})} \times 100 = \text{Matrix Spike Recovery}$$

4.20 MATRIX SPIKE DUPLICATE - a second aliquot of the original sample that is spiked in order to determine the precision of the method. The matrix spike duplicate RPD is calculated as shown below.

$$\frac{(|\text{MS Result} - \text{MSD Result}|) \times 100}{(\text{MS Result} + \text{MSD Result})/2} = \text{MSD RPD}$$

4.21 METHOD BLANK- an analytical control consisting of all reagents, that is carried throughout the entire analytical procedure. The method blank is used to define the level of laboratory, background, and reagent contamination.

4.22 PERCENT DIFFERENCE (%D) - as used in this SOP to compare two values, the percent difference indicates both the direction and the magnitude of the comparison, i.e., the percent difference may be either negative, positive, or zero.

4.23 REAGENT WATER- water that has been generated by any method which would achieve the performance specifications for ASTM Type II water. Water in which an interferant is not observed at or above the minimum quantitation limit of the parameters of interest. Accutest uses deionized water (municipal water which passes through Accutest's DI treatment system).

4.24 SERIAL DILUTION – the dilution of a sample by a factor of five. When corrected by the dilution factor, the diluted sample must agree with the original undiluted sample within specified limits. Serial dilution may reflect the influence of interferents.

4.25 SOIL - used herein synonymously with soil/sediment and sediment.

4.26 TOTAL METALS – analyte elements which have been digested prior to analysis.

4.27 Linear Dynamic Range (Linearity studies) -the concentration range over which the instrument response to an analyte is linear.

5.0 HEALTH & SAFETY

5.1 All safety practices must be followed as outlined in the Accutest Laboratories Employee Safety Handbook and Chemical Hygiene Plan. Safety glasses, gloves, and lab coats must be worn. All samples, solutions, and extracts must be treated as unknown and potentially hazardous. In addition, all acids are corrosive and should be handled with care. Flush spills with plenty of water. If acids contact any part of the body, flush with water and contact the supervisor.

5.2 The toxicity or carcinogenicity of each reagent used in this method has not been precisely determined; however, each chemical should be treated as a potential health hazard. Exposure to these reagents should be reduced to the lowest possible level.

6.0 COLLECTION, PRESERVATION, & HOLDING TIMES

6.1 Preservation. All aqueous samples should be preserved with nitric acid at the time of collection. Both soils and aqueous samples should be kept under refrigeration at 4° ± 2°C.

6.2 Holding Time. All samples should be analyzed within 6 months of the time of collection.

7.0 APPARATUS and MATERIALS

7.1 Currently there is one solid state ICP (Thermo 6500 ICP. Software iTEVA, Issue 8) available for use in the laboratory. The unit has been optimized to obtain low detection limits for a wide range of elements. Since it is solid state system, different lines may be included for elements to obtain the best analytical results. See below table for the lines in use.

Element	Wavelength	View	Internal	Wavelength
Ag	328	Axial	Yttrium	360.0
Al	396.1	Radial	Yttrium	371.0
As	189	Axial	Yttrium	224.3
Au	242.7	Axial	Yttrium	360.0
B	208.9	Axial	Yttrium	224.3
Ba	455.4	Radial	Yttrium	371.0
Be	313	Radial	Yttrium	371.0
Ca	317.9	Radial	Yttrium	371.0
Cd	228.8	Axial	Yttrium	224.3
Co	228.6	Axial	Yttrium	224.3
Cr	267.7	Axial	Yttrium	360.0
Cu	324.7	Axial	Yttrium	360.0
Fe	259.9	Radial	Yttrium	371.0
Li	610.3	Radial	Yttrium	371.0
K	766.4	Radial	Yttrium	371.0
Mg	279	Radial	Yttrium	371.0
Mn	257.6	Axial	Yttrium	360.0
Mo	202	Axial	Yttrium	224.3
Na	589.5	Radial	Yttrium	371.0
Ni	231.6	Axial	Yttrium	224.3
Pb	220.3	Axial	Yttrium	224.3
Pd	340.4	Axial	Yttrium	360.0
Pt	265.9	Axial	Yttrium	360.0
Sb	206.8	Axial	Yttrium	224.3
Se	196	Axial	Yttrium	224.3
Si	212.4	Axial	Yttrium	224.3

Sn	189.9	Axial	Yttrium	224.3
Sr	407.7	Radial	Yttrium	371.0
Ti	334.9	Axial	Yttrium	360.0
Tl	190.8	Axial	Yttrium	224.3
V	292.4	Axial	Yttrium	360.0
W	239.7	Axial	Yttrium	360.0
Zn	206.2	Axial	Yttrium	224.3
Zr	339.1	Axial	Yttrium	224.3

7.2 Peristaltic pump

7.3 Auto-sampler

7.4 Volumetric flasks of suitable precision and accuracy

7.5 Argon gas supply - Liquid, high purity grade (99.995%) supplied by Air Products, Inc.

7.6 Instrument maintenance

7.6.1 Recommended periodic maintenance includes the items outlined below. All maintenance should be recorded in the instrument maintenance log.

7.6.2 Change the pump tubing weekly or as needed.

7.6.3 Clean the filter on the re-circulating pump every one to two weeks and dust off the power supply vents every one to two weeks.

7.6.4 Clean the nebulizer, torch, and injector tube every week or more often as required.

7.6.5 Clean the pump once per month.

7.6.6 Change the sampler tip as needed.

7.6.7 Clean the recirculating pump lines as needed.

8.0 STANDARDS AND REAGENTS

8.1 All chemicals listed below are reagent or trace grade unless otherwise specified. Distilled, deionized water from Accutest's DI system should be used whenever water is required.

8.2 Concentrated hydrochloric acid, trace metal grade.

8.3 Concentrated nitric acid, trace metal grade.

8.4 Stock metals standard solution (generally 1000 Mg/L or 10000 Mg/L), ICAP grade.

NOTE: Combined stock standards can be ordered or made from ICAP purity standards.

8.5 Hydrochloric acid (1:1) - Add 500 ml conc. HCl to 400 ml of DI. Cool and dilute to 1 liter.

8.6 Nitric acid (1:1) - Add 500 ml of conc. HNO_3 to 400 ml of DI. Cool and dilute to 1 liter.

8.7 Rinse solution - To 800 ml DI water, add 50 ml of conc. HNO_3 and 50 ml of conc. HCl. Dilute to 1 liter.

8.8 Calibration Blank - To 800 ml DI, add 50 ml of conc. HNO_3 and 50 ml of conc. HCl. Dilute to 1 liter.

8.9 Calibration Standards

8.9.1 Premixed stocks purchased from Absolute standards and other vendors are used in this section along with individual standards and subsequent dilutions.

8.9.2 Calibration Std # 1 contains the following analytes and concentration:

Analyte	Conc. (Mg/L)
Sb, As, Ba, Be, B, Cd, Cr, Co, Cu, Pb, Mn, Mo, Ni, Se, Tl, Zn	**1000**
Ag	**12.5**

8.9.3 Calibration Std # 2 contains the following analytes and concentration:

Analyte	Conc. (Mg/L)
Al, Ca, Fe, Mg, K, Na	**1000**

8.10.4 Calibration Std # 3 contains the following analytes and concentration:

Analyte	Conc. (Mg/L)
Sr, Sn, Ti, V	**1000**

8.10.5 Individual 1000 ppm analytes.

8.10.6 In general there are 3 different standards are recommended to be used for calibration. Other combinations and concentrations may be used.

STANDARD#1:

Analyte	Conc. (Mg/L)
Sb, As. Ba, Be, B, Cd, Cr, Co, Cu, Pb, Mn, Mo, Ni, Se, Tl, Zn	4.0
Ag	0.50

STANDARD#2:

Analyte	Conc. (Mg/L)
Al, Ca, Fe, Mg, K, Na, Li	20

STANDARD #3:

Analyte	Conc. (Mg/L)
Sr, Sn, Ti, V, Au, Pd, Pt, Si, W, Zr	4.0

NOTE: 1) Since addition of silver may result in an initial precipitation, you may warm the flask until solution clears. Cool to room temperature and use.

8.11 Continuing Calibration Verification (CCV)

Prepare mixed CCV solution by combining appropriate volumes of the individual stock standards or by using the premixed stock purchased from Absolute Standard and subsequent dilutions. The CCV must be prepared in the same acid matrix using the same standards used for calibration at a concentration near the mid point of the calibration curve. Below are the recommended concentrations of CCV to use with the above calibration standards.

NOTE: Since addition of silver may result in an initial precipitation, you may warm the flask until the solution clears. Cool to room temperature and use.

The CCV will contain the following analytes and concentrations:

Analyte	Conc. (Mg/L)
Sb, As. Ba, Be, B, Cd, Cr, Co, Cu, Pb, Mn, Mo, Ni, Se, Tl, Zn, Sr, Sn, Sr, Ti, V, Si, W, Au, Pd, Pt, Zr	2.0
Al, Ca, Fe, Mg, K, Na, Li	10
Ag	0.25

8.12 Initial Calibration Verification/ Quality Control Standard (ICV/QCS)- This standard is prepared by combining compatible elements from a standard source different than the calibration standards and the concentrations should be at or near the mid-range of the calibration curve.

Below are the recommended concentrations of ICV. Dilute the individual stocks, and premixed standards such as ICQ500-19 or the equivalents. (ICQ500-19 is a multi element standard purchased and contains multiple analytes). The ICV will contain the following analytes and concentrations:

Analyte	Conc. (Mg/L)
Sb, As. Ba, Be, B, Cd, Cr, Co, Cu, Pb, Mn, Mo, Ni, Se, Tl, Zn, Sr, Sn, Ti, V, Si, W, Au, Pd, Pt, Zr	3.0
Al, K, Na, Li	15
Ca, Fe, Mg	18
Ag	0.50

8.13 Spectral Interference Check I (ICSA) - Dilute the following listed aliquot volumes of individual stock standards to 1 Liter with calibration blank (8.8).

Analyte	Stock Conc. (Mg/L)	Aliquot Vol. (Ml)	Final Conc. (Mg/L)
Al, Ca, Mg	10000	50	500

Fe	**10000**	**20**	**200**

8.14 Spectral Interference Check II (ICSAB) - Dilute the following listed aliquot volumes of individual stock standards and 10 ml of CLPILM 030 Analytes B standard to 1 Liter with calibration blank (8.8).

- CLPILM030 Analytes B standard contains the following analytes and concentrations:

Analyte	Conc. (Mg/L)
Ag, Cd, Ni, Pb, Zn	**100**
Ba, Be, Co, Cr, Cu, Mn, V	**50**

ICSAB solution contains the following analytes and concentrations:

Analyte	Stock Conc. (Mg/L)	Aliquot Vol. (Ml)	Final Conc. (Mg/L)
Al, Ca, Mg	**10000**	**50**	**500**
Fe	**10000**	**20**	**200**
As, Se, Tl, Sb	**1000**	**2.0**	**2.0**
Ag, Cd, Ni, Pb, Zn	**100**	**10**	**1.0**
Ba, Be, Co, Cr, Cu, Mn, v	**50**		**0.50**
Mo, Sr, Sn, B	**1000**	**1.0**	**1.0**
Ti, Au*, Pd*, Pt*	**1000**	**0.50**	**0.50**
Si*, W *	**1000**	**2.0**	**2.0**
Zr*	**1000**	**0.5**	**0.5**

*When the element is requested ICSAB will be made accordingly. Internal Standard/Ionization suppressant solution (5 ppm Yttrium, 1000 ppm Lithium) In a 1000 ml volumetric flask, dilute 5 ml of 1000 ppm Yttrium standard and 100 ml of 10000 ppm Lithium standard to the mark with calibration blank (8.8)

Note: If Na, Ca, Mg and K are not being analyzed Li can be excluded in the internal standard mix. If Li requested, use 5 ppm Yttrium and 50 ppm Indium solution mix as internal standard.

8.15 Low level Check (CRI). The CRI standard contains the elements of interest at levels near the low end of the curve. Typically the concentration of CRI is at the reporting limits. Check with the metals supervisor or the client tech specs, to see which CRI needs to be analyzed. The acceptance criteria of 70 to 130percent will be applied unless there are specific instructions set by client or program.

Program/Client	CRI Concentration Level	CRI Acceptance Criteria	Non Conformance
6010C	At the RL	+/- 30%	Re-calibrate & re-analyze
DoD	At the RL	+/- 20%	Re-calibrate & re-analyze
RCP	At the RL	+/- 30% *	Re-calibrate & re-analyze

* Except +/-50% for As, Sb, Tl, Co

. See below table for CRI solution preparation and the final concentration at the instrument.

Low Levels Check Solution	Element	Stock Conc. in mg/l	Amt of Stock used in ml	Final Vol. of CRI Stock Solution in ml	Conc. of CRI Stock Solution in ug/l	Amt of CRI Stock Solution used in ml	Final Vol. of CRI Solution in ml	Final Conc. at the instrument in ug/l
ICP CRI 6010C	Sb	6.00	0.50	500.00	N/A	N/A	N/A	6.00
	As	4.00			N/A			4.00
	Ba	50.00			N/A			50.00
	Be	4.00			N/A			4.00
	B	100.00			N/A			100.00
	Cd	4.00			N/A			4.00
	Cr	10.00			N/A			10.00
	Co	50.00			N/A			50.00
	Cu	25.00			N/A			25.00
	Pb	5.00			N/A			5.00
	Mn	15.00			N/A			15.00
	Mo	100.00			N/A			100.00
	Ni	40.00			N/A			40.00
	Se	10.00			N/A			10.00

	Ag	5.00			N/A		5.00
	Sr	10.00			N/A		10.00
	Tl	5.00			N/A		5.00
	Sn	100.00			N/A		100.00
	Ti	50.00			N/A		50.00
	V	10.00			N/A		10.00
	Zn	20.00			N/A		20.00
ICP CRI MINERAL	Al	20.00	5.00	500.00	N/A	N/A	200.00
	Fe	10.00					100.00
	Ca	500.00					5000.00
	Mg	500.00					5000.00
	K	500.00					5000.00
	Na	500.00					5000.00
ICP CRI OTHER	Au	1000.00	0.10	100.00	1000.00	5.00	50.00
	Pd	1000.00	0.10		1000.00		50.00
	Pt	1000.00	0.10		1000.00		50.00
	Sl	1000.00	0.20		2000.00		100.00
	W	1000.00	0.20		2000.00		100.00
	Zr	1000.00	0.10		1000.00		50.00
ICP CRI LI	Li	10000.00	0.05	100.00	N/A	N/A	5000.00

9.0 INTERFERENCES

9.1 Several types of interference effects may cause inaccuracies in the determination of an analyte in this method. These interferences can be summarized as follows:

9.1.1 Spectral Interferences: Spectral interferences are caused by overlap of a spectral line from another element, unresolved overlap of molecular band spectra, background contribution from stray light from the line emission of high concentration elements. Corrections for these interferences can be made by using interfering element corrections, by choosing an alternate analytical line, and/or by applying background correction points.

9.1.2 Physical Interferences: Physical interferences are generally considered to be effects associated with the sample introduction (nebulization and transport processes). Such properties as change in viscosity or surface tension can cause significant inaccuracies especially in samples which may contain high dissolved solids and/or high acid concentrations. The use of a peristaltic pump, sample dilution and/or utilization of standard addition techniques will reduce these interferences.

9.1.3 Chemical Interferences: Chemical interferences are not pronounced with the ICAP techniques due to the high temperature of plasma, however, if they are present, they can be reduced by optimizing the analytical condition (i.e. power level, torch height).

9.1.4 Memory Interferences: Memory interferences result when analytes in a previous sample contribute to the signal measured in the new sample. This result from sample builds up in the plasma torch spray chamber. This can be reduced by flushing the system between samples with rinse solution (8.7). A minimum of 60 seconds rinse time must be applied.

9.2 The occurrence of interferences described above are primarily attributed to the sample matrix. To ensure the absence of any type of interferences, the following precautions may be taken:

9.2.1 Serial Dilution: See section 11.9.

9.2.2 Analyte Addition (Post Digestion Spike – PDS): An analyte spike added to a portion of prepared sample, or its dilution, should be recovered to within 80% to 120% (Note, some cleints or programs (i.e., DoD), the recovery is 75% to 125%) of the known value. The spike addition should produce a minimum level of 10 times and a maximum of 100 times the IDL for that element.

9.2.3 Method of Standard Addition (MSA). If the PDS does not meet criteria, the MSA may be used. Standards are added at one or more levels to portions of a prepared sample. This technique compensates for enhancement or depression of an analyte signal by a matrix. It will not correct for additive interferences, such as contamination, interelement interferences, or baseline shifts. This technique is valid in the linear range when the interference effect is constant over the range, the added analyte responds the same as the endogenous analyte, and the signal is corrected for additive interferences. The simplest version of this technique is the single addition method. This procedure calls for two identical aliquots of the sample solution to be taken. To the first aliquot, a small volume of standard is added; while to the second aliquot, a volume of acid blank is added equal to the standard addition. The sample concentration is calculated by: multiplying the intensity value for the unfortified aliquot by the volume (Liters) and concentration (mg/l or mg/kg) of the standard addition to make the numerator; the difference in intensities for the fortified sample and unfortified sample is multiplied by the volume (Liters) of the sample aliquot for the denominator. The quotient is the sample concentration.

9.2.4 An alternative to using the method of standard addition is the internal standard technique. Add yttrium to the standards, samples, and blanks at a concentration to be sufficient for optimum precision, but not so high as to alter the salt concentration of the matrix. The element intensity is used by the instrument as an internal standard to ratio the analyte intensity signals for both calibration and quantitiation.

9.2.5 Wavelength Scanning: Wavelength scanning of the sample can be performed and compared to the scan of the analyte to detect potential spectral interferences.

10.0 PROCEDURE

General procedure on how to operate the SS6500 is described below. Refer to the Thermo 6500 operation manual for further details.

10.1 Before bringing up the instrument, make sure that the sample tubings, the nebulizer, and the spray chamber are clean and that there are no leaks in the torch area.

10.2 Turn on the recirculating cooler.

10.3 Engage the peristaltic pump.

10.4 Make sure reagent reservoirs (rinse solution and internal std solution) are filled with enough solution to last for a full days run. The rinse reservoir is filled with rinse solution (8.7) and internal standard solution (8.14).

NOTE: If the internal standard solution runs out during the run, instrument must be recalibrated with the new standard.

10.5 Ignite the plasma and let the instrument warm up for 30 minutes before starting analysis. New tubing may need an hour to stabilize.

10.6 Type up the auto sampler sequence, and set up the trays.

10.7 CALIBRATION

10.7.1 Prior to calibration, make sure a minimum of 60-second rinse time and 60 seconds up take time is set up in the method/auto-sampler.

10.7.2 Calibrate the instrument using calibration blank, standard #1, standard #2, and standard #3.

10.8 DAILY CALIBRATION

10.8.1 Calibrate the instrument using calibration blank (8.8) and calibration standards (8.10.5).

10.8.2 When calibration is complete, a printout of slopes will be printed for each line and calibration is automatically approved.

10.9 Dilutions.

10.9.1 The pH of all aqueous samples must be verified to be <2 prior to aliquot for processing. If samples have a pH of >2 add additional nitric acid and wait 24 hours before rechecking the pH. If a sample result exceeds the upper linear range, a dilution must be made using rinse solution (8.7). The diluted result should be within the upper linear dynamic range.

10.10 Filtration.

10.10.1 If particulate were observed in the samples, then they should be re-filtered along with the associated blanks.

11.0 QUALITY ASSURANCE

NOTE: 1) The system must be rinsed with the calibration blank solution between each sample analysis for a minimum of 60 seconds.

11.1 Set up the auto-sampler sequence as described below.

11.2 Analyze the mid-level initial calibration verification (ICV - 8.12). The analyzed value of each analyte for the mid-level ICV must be within 90%-110% of its expected value and the RPD between replicates should be less than 5%. If not, recalibrate.

11.3 Analyze the Initial Calibration Blank (ICB - 8.8). The ICB results should be less than the reporting limits for an element). If not, re-calibrate.

11.4 Analyze the Continuing Calibration Verification (CCV - 8.11) and Continuing Calibration Blank (CCB - 8.8). Also analyze the CCV and CCB after every 10 samples and at the end of the sample run. The analyzed value of each analyte in the CCV should be within 90% to 110% of its true value and the RPD between replicates should be less than 5%. If not, rerun one more time. If an analyte value is still outside the range, the instrument should be recalibrated and all samples following the last acceptable CCV should be reanalyzed. All CCBs should be less than the reporting limits for each element. If not, it can be reanalyzed one more time, if still out, no samples can be reported in the area bracketed by the failing CCB for the failing elements.

11.5 Analyze the CRI check standards at the beginning and the end of the run with each new calibration.

11.5.1 The low-level initial calibration verification (LLICV) is used as CRI check standard at the beginning of the run. The analyzed value of each analyte for the LLICV must be within 70%-130% of its expected value. Note, for some clients or programs (i.e., DoD), the recovery is 80%-120%, check with Dept. manager for specific project requirement. It acceptance criteria not met, reanalyze once. If acceptance criteria not met after second analysis perform corrective action and recalibrate. The LLICV can be prepared by using the same source as the calibration standards, but must at a concentration expected to be at the RL.

11.5.2 The low-level continuing calibration verification (LLCCV) is used as CRI check standard at the end of the run. It is recommended that a LLCCV be analyzed after every 10 samples and at the end of each analysis batch. The acceptance criteria for LLCCV should be within 70%-130% of its true value. Note, for some clients or programs (i.e., DoD), the

recovery is 80%-120%, check with Dept. manager for specific project requirement. It acceptance criteria not met, reanalyze once. If acceptance criteria not met after second analysis, no samples for failed analyte in LLCCV can be reported. The LLCCV should be prepared from the same source as the initial calibration standards at a concentration of the RL.

11.6 Analyze the Spectral Interference Check I (ICSA) and Spectral Interference Check II (ICSAB)

11.6.1 The analyzed value of Al, Ca, Mg, and Fe must be between 80-120%. The analyzed value of each remaining analyte in ICSA should be less than twice the reporting limit, unless specified by client or program. If not, rerun one more time, if still not, either recalibrate or no sample for failed analyte in ICSA can be reported. You may have to perform inter-element correction of interfering elements.

11.6.2 The analyzed value of each analyte in ICSAB should be within ± 20% of its expected value. If not, repour and reanalyze. If still outside the limit, recalibrate. If after recalibration ICSAB is still outside the limit, you may have to perform inter-element correction of interfering elements and recalibrate.

11.6.3 The ICSA and ICSAB should be run prior to sample analysis and at the end of the run.

11.7 If all initial quality control steps mentioned above were satisfied, you may start the analysis of the samples.

11.8 For each analysis run a laboratory reagent blank (method blank), laboratory fortified blank (spike blank), laboratory control sample (LCS – for soil samples), laboratory fortified sample (matrix spike), a matrix spike duplicate must be analyzed. If samples that were prepared on several different days are analyzed, make sure that the method blanks and spike blanks from all of the preparation dates are also analyzed. (A matrix spike, a matrix spike duplicate, a spike blank, and a method blank are prepared with each set of 20 samples).

Analyte recovery: Until sufficient data becomes available the following limits should be exercised:

Spike Blank Recovery	± 20%
Spike Blank Duplicate RPD	≤ 20% (AQ) ≤ 30% (SO)
Matrix Spike Recovery	± 25% *
RPD	± 20%

Note: The Spike Blank Duplicate is an MCP/RCP requirement. A project-specific matrix duplicate or matrix spike duplicate may be used in lieu of the spike blank duplicate for MCP.

* If the % Recovery of an analyte falls outside the ± 25% limit (Note, for some clients or programs (i.e., DoD), the Matrix Spike recovery is +/- 20%, check with Dept. manager for project specific requirement) but spike blank recovery falls within the accepted range, then the recovery problem is judged to be matrix related, not system related. Sample results

need to be footnoted and a post spike must be analyzed. (Analyze the post digestion spike according to section 9.2.2) Note, for some clients , the unspiked aliquot of the sample should be spiked at two –times the indigenous level or two times their specific required detection limit, which ever is greater. The results of post spike must be reported in their summary report (check with Dept. manager for client specific lists) If post spike recoveries are not within the 80%-120% criteria, a matrix effect should be suspected. Follow step 11.9.

For the soil LCS – the manufacturer QC limits should be used for evaluation.

The method blank results must be less than the reporting limit for an element. Note, for some clients the method blank results must be less then ½ of the RL. If not, any sample results in the associated batch has a positive result for that element, the batch must be re-digested and reanalyzed.

11.9 Serial Dilution- The analysis of a (1:5) dilution should agree within 10% of the original sample result. If the analyte concentration is not high enough and the serial dilution is not within 10 % of the original sample but less than 50 times the IDL for that analyte, serial dilution results are acceptable. In addition, serial dilution should be done on every sample that is significantly different matrix or if a matrix interference is suspected. If analysis of the dilution does not meet the 10% criteria, a matrix interference may be suspected. For some clients or programs (i.e., DoD), if 1:5 dilution does not agree within +/- 10% of the original measurement for samples with concentrations greater than 50 times of LOD, perform post digestion spike (check with Dept. manager for project specific requirement for post spike criteria).

11.10 Internal standard-Internal standard is added to all standards and samples. Acceptance criteria for the internal standard are ±30% as compared to the Calibration Blank.

11.11 A Precision and accuracy (P&A) study is performed as an initial determination of capability, on an annual basis, and if any major changes have been made to the instrument. Four replicates or blank spikes are analyzed using the same conditions for sample analysis. The percent recoveries are compared to limits as described in section 11.8 (spike blank). The standard deviation results of the 4 blank spike recoveries should be ≤ 20%. If percent recovery or standard deviation criteria are not met, corrective action must be taken to bring the system back into control.

11.12 Linearity studies should be determined quarterly and whenever there is significant change in instrument response. The study must be performed using minimum 5 different concentration standards across the range, and 1 standard must be near the upper limit. The acceptance criteria for linearity studies should be within ±10% of its true value.

11.13 MDLs are determined annually, or if significant maintenance has been performed on the instrument. Refer to the MDL SOP (MQA245) for details.

11.14 IDLs are performed quarterly. The IDLs can be estimated by calculating the average of the standard deviations of three runs on three non-consecutive days from the analysis of a reagent

blank solution with seven consecutive measurements per day. The IDL concentration must be ≤ the MDL concentration. The study must be repeated for any analytes that do not meet this criteria.

11.15 The lower limit of quantitation sample (LLQC) should be analyzed after establising the lower laboratory reporting limits and on an as needed basis to demonstrate the desired detection capability. The LLQC must be prepared at the same concentrations as the RL, and the LLQC must be carried through the entire preparation and analytical procedure. The lower quantitation limits are verfied when all analytes in the LLQC are detected within ±30% of their true value. This check should be used to both establish and confirm the lowest quantitation limit.

11.16 Interelement spectral interference determination routine must be verified every 6 months.

11.17 Quality control data are generated at least on an annual basis by QA using an in-house program. Blank spike and MS/Dup data are pooled for the previous year (or other specified time frame) and the data is processed and evaluated by QA. The annual QC data is filed with QA.

11.18 All NELAC-accredited target compounds must be spiked in the blank spike and matrix spike within a two-year period. All target compounds reported for a project are spiked and evaluated in the blank spike and MS/MSD

12 DOCUMENTATION

12.1 Make sure that all sample ID's and standard lot# are recorded in analysis log book. All comments and edits MUST BE clearly documented and initialed. Generate run logs from the LIMS system along with all quality control data.

12.2 Each analyst should review all data and assemble a data package consisting of :

- Print out of automated digestion prep sheet
- Print out of automated analysis runlog
- LIMS generated run log
- Raw data
- LIMS generated batch list

12.3 If samples require reanalysis, a brief explanation of the reason should be documented in this log.

12.4 The Accutest lot number must be cross-referenced on the standard bottle. Expiration date must be noted on standard bottle.

12.5 The instrument Maintenance logbook must be completed daily. Each instrument will have a separate log.

12.6 All laboratory logbooks must be reviewed and initialed or signed by the lab manager. A signed or initialed copy of the logbook page filed with the daily batch is sufficient.

12.7 Any corrections to laboratory data must be done using a single line through the error. The initials of the person and date of correction must appear next to the correction.

12.8 The inter-element spectral interference determination routine must be kept on file.

12.9 Linearity studies must be kept on file.

13 DATA REPORTING

13.1 A results page including positive results and/or RLs for all target elements, units, methodology, dates of digestion and analysis, data qualifiers, are reported. Additional quality control data including calibration summaries, MS/MSD percent recoveries and RPDs, blank spike recoveries, method and calibration blank results, and any associated raw support data may be reported upon request of the client.

13.2 Data may be submitted to the client in a specified electronic format (EDD).

13.3 Data may be submitted to the client in PDF via e-hardcopy.

13.4 Once the data is approved by the laboratory manager, it may be accessed by clients via LabLink™.

13.5 Procedures for handling non-conforming data.

13.5.1 If quality control data does not meet criteria the non-conformance must be discussed in a case narrative and footnoted on the applicable quality control report summary.

13.5.2 If preservation or holding time criteria is not met and the samples are analyzed the result page must be footnotes with this information, and the non-conformance must be discussed in a case narrative or other suitable communication (telephone conversation log or email). Client notification documentation should be included with the data (telephone conversation log, fax, or email).

14.0 DATA REVIEW

14.1 The analyst conducts the primary review of all data. This review begins with a check of all Instrument and method quality control and progresses through sample quality control concluding with a check to assure that the client's requirements have been executed. The analyst has the authority and responsibility to perform corrective action for any out-of-control parameter of non-conformance.

14.2 A secondary review is performed by department managers, and it includes review of the data produced by their department. All manual calculations, QC criteria, and a comparison of the data package to client specified requirements are checked. The department manager may reject data, initiate reanalysis, take additional corrective action, or reprocess data.

14.3 The laboratory director performs a full tertiary review of the data package following its assembly. This review includes an evaluation of QC data against acceptance criteria and a check of the data package contents to assure that all analytical requirements and specifications were executed.

14.4 Spot-check reviews are performed by the Quality Assurance Officer focusing on all elements of the deliverable including the client's specifications and requirements, analytical quality control, sample custody documentation and sample identification

15.0 POLLUTION PREVENTION & WASTE MANAGEMENT

15.1 Pollution Prevention. Users of this method must perform all procedural steps that control the creation and/or escape of wastes of hazardous materials to the environment. The amounts of standards, reagents, and solvents must be limited to the amounts specified in this SOP. All safety practices designed to limit the escape of vapors, liquids, or solids to the environment must be followed. All method users must be familiar with the waste management practices described in section 15.2.

15.2 Waste Management. Individuals performing this method must follow established waste management procedures as described in the Sample and Waste Disposal SOP. This document describes the proper disposal of all waste materials generated during the testing of samples as follows:

15.2.1 Non-hazardous aqueous wastes
15.2.2 Hazardous aqueous wastes
15.2.3 Chlorinated organic solvents
15.2.4 Non-chlorinated organic solvents
15.2.5 Hazardous solid wastes
15.2.6 Non-hazardous solid wastes

16.0 METHOD PERFORMANCE

16.1 Method performance is evaluated by the annual quality control data generated by QA, and the annual MDL study results. Refer to section 3.0 for MDLs, and section 11.9.10 for QC data.

17.0 ADDITIONAL REFERENCES

17.1 None.

Lab Manager: Brad Madadian

QA Manager: Robert Treggiari

TITLE: **AUTOMATED COLD VAPOR ANALYSIS OF MERCURY FOR WASTE WATER SAMPLES**

TEST METHOD: SW846 7470A, Rev. 1, Sept. 1994

REVISED SECTION: 8.2, 8.3, 8.5, 10.1.5

1.0 SCOPE & APPLICATION

1.1 The method can be applied for the analysis of mercury in water, wastewater, and TCLP& SPLP leachates.

1.2 Test Code: HG

1.3 Reporting limits (RLs) are established at the lowest concentration standard. The RL for mercury is 0.2 ug/l.

2.0 SUMMARY

2.1 Prior to analysis, all water samples must be prepared according to the procedure discussed in this SOP.

2.2 A cold-vapor atomic absorption technique, is based on the absorption of radiation at 253.7-nm by mercury vapor. The mercury is reduced to the elemental state 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. Results are quantified by comparison to a daily calibration curve.

3.0 METHOD REPORTING AND DETECTION LIMIT

3.1 Reporting limits (RLs) are established at the lowest concentration standard. The RL for mercury is 0.2 ug/L.

3.2 The Method Detection Limits (MDL) represents the lowest reportable concentration of an individual analyte that meets the method qualitative identification criteria.

3.3 Method Detection limits are experimentally determined using the procedures described in 40 CFR, Part 136, Appendix B. Actual reported MDLs incorporate the sample volume analyzed and sample dilutions if needed, which may cause MDL variations from sample to sample.

3.4 In general, MDLs are determined through the analysis of at least 7 replicate blank spikes (using the same procedures for sample analysis). The MDL is calculated by multiplying the standard deviation of the replicate concentrations by the appropriate Student's t value (3.143 for 7 replicates).

If more than 7 replicates are analyzed refer to 40 CFR, Part 136, Appendix B for the appropriate student's t value. MDLs are determined initially (prior to analysis), on an annual basis, and after major maintenance to equipment. MDL data is archived with Quality Assurance. Refer to the most recent study for current MDLs.

4.0 DEFINITIONS

4.1 ALIQUOT - a measured portion of a sample, or solution, taken for sample preparation and/or analysis.

4.2 BATCH - A group of samples which behave similarly with respect to the sampling or the testing procedures being employed and which are processed as a unit. For QC purposes, if the number of samples in a group is greater than 20 (or 10 for certain methods), then each group of 20 samples (or 10 samples for certain methods) or less will all be handled as a separate batch.

4.3 CALIBRATION – the establishment of an analytical curve based on the absorbance, emission intensity, or other measured characteristic of known standards. The calibration standards must be prepared using the same type of acid or concentration of acids as used in the sample preparation.

4.4 CALIBRATION BLANK - a volume of acidified deionized/distilled water.

4.5 CALIBRATION STANDARDS – a series of known solutions used by the analyst for calibration of the instrument (i.e., preparation of the analytical curve).

4.6 CONTINUING CALIBRATION VERIFICATION – analytical standard run every 10 samples or 2 hours, whichever is more frequent, to verify the calibration of the analytical system.

4.7 CONTAMINATION - a component of a sample or an extract that is not representative of the environmental source of the sample. Contamination may stem from other samples, sampling equipment, while in transit, from laboratory reagents, laboratory environment, or analytical instruments.

4.8 CORRELATION COEFFICIENT – A NUMBER (r) which indicates the degree of dependence between two variables (concentration – absorbance). The more dependent they are the closer the value to one.

4.9 CRI (LOW CALIBRATION CHECK) – a check standard, which is prepared at the low point of the calibration, and is analyzed to check accuracy at the low end of the calibration curve. Similar to the CRDL (CLP - Contract Required Detection Limit standard).

4.10 FIELD SAMPLE - a portion of material to be analyzed that is contained in single or multiple containers and identified by a unique sample number.

4.11 FIELD BLANK – this is any sample that is submitted from the field and is identified as a blank. This includes rinsates and equipment blanks, etc.

4.12 HOLDING TIME – the elapsed time expressed in days from the date of sampling until the date of its analysis.

4.13 INTERFERENTS – substances which affect the analysis for the element of interest.

4.14 INITIAL CALIBRATION VERIFICATION - analytical standard run after every initial calibration to verify the calibration of the analytical system. Must be obtained from a different vendor or lot number from the initial calibration standards.

4.15 INSUFFICIENT QUANTITY - when there is not enough volume (water sample) to perform any of the required operations: sample analysis or extraction, MS/MSD, etc.

4.16 MATRIX - the predominant material of which the sample to be analyzed is composed. For the purpose of this SOP, a sample matrix is water. Matrix is not synonymous with phase (liquid).

4.17 MATRIX EFFECT - in general, the effect of a particular matrix (water or soil/sediment) on the constituents with which it contacts. This is particularly pronounced for clay particles which may adsorb chemicals and catalyze reactions. Matrix effects may prevent extraction of target analytes, and may affect surrogate recoveries. In addition, non-target analytes may be extracted from the matrix causing interferences.

4.18 MATRIX SPIKE - aliquot of a matrix (water) 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. The matrix spike recovery is calculated as shown below.

$$\frac{(\text{Spiked Sample Result} - \text{Sample Result})}{(\text{Amount Spiked})} \times 100 = \text{Matrix Spike Recovery}$$

4.19 MATRIX SPIKE DUPLICATE - a second aliquot of the original sample that is spiked in order to determine the precision of the method. The matrix spike duplicate RPD is calculated as shown below.

$$\frac{(|\text{MS Result} - \text{MSD Result}|) \times 100}{(\text{MS Result} + \text{MSD Result})/2} = \text{MSD RPD}$$

4.20 METHOD BLANK - an analytical control consisting of all reagents that is carried throughout the entire analytical procedure. The method blank is used to define the level of laboratory, background, and reagent contamination.

4.21 REAGENT WATER - water in which an interferant is not observed at or above the minimum quantitation limit of the parameters of interest. Accutest uses deionized water (municipal water which passes through Accutest's DI treatment system).

4.22 RELATIVE PERCENT DIFFERENCE (RPD) - As used in this SOP to compare two values, the relative percent difference is based on the mean of the two values, and is reported as an absolute value, i.e., always expressed as a positive number or zero.

5.0 HEALTH & SAFETY

5.1 All safety practices must be followed as outlined in the Accutest Laboratories Chemical Hygiene Plan. Safety glasses, gloves, and lab coats must be worn. All samples, solutions, and extracts must be treated as unknown and potentially hazardous. Particular care must be taken when handling strong acids and oxidizing agents (5.2 and 5.3)

5.2 Potassium persulfate is a strong oxidizing agent - handle with caution.

5.3 Magnesium perchlorate is an oxidizer and an irritant. All handling of this chemical should be done in a hood.

6.0 COLLECTION, PRESERVATION, AND HOLDING TIMES

6.1 Preservation

6.1.1 Collect aqueous samples in a 500ml polyethylene bottle. All waste water samples should be preserved by acidification with nitric acid to a pH of 2 or lower.

6.2 Holding Times

6.2.1 All samples should be analyzed within 28 days of the date of collection.

7.0 APPARATUS AND MATERIALS

7.1 Currently, there are two Leeman HYDRA AA automated analyzer are available for analysis. Refer to the instrument manual for further details on this instrumentation, including proper venting and safety requirements.

7.2 Digestion tubes with caps - 60 ml capacity.

7.3 Volumetric pipettes

7.4 Volumetric flasks

7.5 Graduated cylinders

7.6 Digestion Block capable of maintaining 90-95 Deg. C. (Environmental Express)

8.0 STANDARDS AND REAGENTS

8.1 All chemicals listed below are reagent grade unless otherwise specified. Distilled, deionized water should be used whenever water is required. All applicable standard/reagent preparation information, including vendor, lot number, date of preparation, calculations, and initials must be entered in the appropriate standard/reagent preparation logbook. Vendors typically used by Accutest include

Fisher Scientific, VWR, Accustandard, Supelco, Chemservices, Ultra, and ERA. Additional vendors may be utilized as necessary.

8.2 Hydrochloric acid, concentrated. Trace metal grade

8.3 Hydrochloric acid, 0.5 N. Add 14.0 ml of concentrated Hydrochloric acid to 0.5 liters of water. Dilute to 1 liter with water and mix well.

CAUTION!! ADD ACID TO WATER.

8.4 Nitric acid, concentrated – trace metal grade.

8.5 Stannous chloride. Add 50 g of stannous chloride to 500 ml of 0.5 N Hydrochloric acid and dissolve. This compound does not dissolve well and should be stirred continuously when in use. Stannous sulfate may be used in place of stannous chloride.

8.6 Sodium chloride-Hydroxylamine sulfate. Add 120 g of sodium chloride and 120 g of hydroxylamine sulfate to 750 ml of water. Dilute to 1000 ml and mix well. Hydroxylamine hydrochloride may be used in place of hydroxylamine sulfate. .

8.7 Potassium Permanganate. Add 50 g of potassium permanganate to 1000 ml of water and mix well.

CAUTION!! POTASSIUM PERMANGANATE IS A STRONG OXIDIZING AGENT. HANDLE WITH CARE.

8.8 Potassium persulfate. Add 50 g of potassium persulfate to 1000 ml of water and mix well.

CAUTION!! POTASSIUM PERSULFATE IS A STRONG OXIDIZING AGENT. HANDLE WITH CARE.

8.9 Mercury standard solutions - to be made fresh daily.

8.9.1 10 ppm Hg calibration solution. Using a 1 ml volumetric pipette, add 1 ml of 1000 ppm stock (to be purchased from a vendor) to a 100 ml volumetric flask containing approximately 75 mL of water and 2 ml of concentrated nitric acid. Dilute to volume with water and mix well. This standard maybe held up to 3 month

8.9.2 100 ppb Hg calibration solution. Using 1 ml volumetric pipette, add 1 ml of 10 ppm Hg solution (8.9.1) to a 100 ml volumetric flask containing approximately 75 mL of DI water and 2 ml of concentrated nitric acid. Dilute to volume with Deionized water and mix well, (to be made fresh daily).

8.9.3 10 ppb Hg calibration solution. Using 10 ml volumetric pipette, add 10 ml of 100 ppb Hg solution (8.9.2) to a 100 ml volumetric flask containing approximately 75 ml of DI water and 2

ml of concentrated nitric acid. Dilute to volume with DI water and mix well,(to be made fresh daily).

8.9.4 1.0 ppb Hg calibration solution. Using 10 ml volumetric pipette, add 10 ml of 10 ppb Hg solution (8.9.3) to a 100 ml volumetric flask containing approximately 75 ml of DI water and 2 ml of concentrated nitric acid. Dilute to volume with DI water and mix well,(to be made fresh daily).

8.9.5 10 ppm ICV Hg solution. Using a 1 mL volumetric pipette, add 1 mL of 1000 ppm stock (to be purchased from a vendor) to a 100 ml volumetric flask containing approximately 75 mL of water and 2 mL of concentrated nitric acid. Dilute to volume with water and mix well.

Note: Make sure that the ICV stock is from an alternate source than calibration stock.

8.9.6 100 ppb ICV Hg solution. Using a 1 ml volumetric pipette, add 1 ml of 10 ppm ICV Hg solution (8.9.5) to a 100 ml volumetric flask containing approximately 75 ml of water and 2 ml of concentrated nitric acid. Dilute to volume with deionized water and mix well, (to be made fresh daily).

8.9.7 10 ppb ICV Hg solution. Using a 10 ml volumetric pipette, add 10 ml of 100 ppb ICV Hg solution (8.9.6) to a 100 ml volumetric flask containing approximately 75 ml of water and 2 ml of concentrated nitric acid. Dilute to volume with deionized water and mix well, (to be made fresh daily).

8.9.8 Rinse solution / Dilution acid solution. In a 1000 ml volumetric flask, add 20 ml of concentrated hydrochloric acid to 700 ml of DI water. Dilute to mark with DI water. Mix well..

9.0 INTERFERENCES

9.1 Possible interference from sulfide is eliminated by the addition of potassium permanganate. Concentrations of sulfide as sodium sulfide as high as 20 mg/l do not interfere with mercury recoveries when following this method. High copper concentrations (> 10 mg/l) may also interfere with mercury recoveries. Samples that are high in chloride such as seawater, brine, and industrial effluent may require as much as 5 ml of additional permanganate.

NOTE: **When chloride concentrations are high, hydroxylamine sulfate and stannous sulfate should be used in place of the corresponding chlorides.**

9.2 Certain volatile organic materials will also absorb at this wavelength and can interfere. It can be determined if this type of interference is present by doing a preliminary run without reagents.

10.0 PROCEDURE

10.1 Digestion. Below is a step-by-step procedure for the digestion and analysis for mercury.

10.1.1 All standards and QC must be digested along with the samples.

10.1.2 Make up the standard curve as shown below. Make sure to clearly label each bottle with a bottle number. The actual standard ID's should be recorded on the mercury log book.

ML of 10 ppb Hg (8.9.3)	ML of 1.0 ppb Hg (8.9.4)	Final Volume of DI water (ML)	ppb of Hg
0.0	-	20.0	(Cal Blk)
-	4.0	16.0	0.20
-	10	10.0	0.50
2.0	-	18.0	1.00
4.0	-	16.0	2.00
6.0	-	14.0	3.00
8.0	-	12.0	4.00
10	-	10.0	5.00
12	-	8.0	6.00

10.1.3 Make up the quality control samples as shown below. Make sure to clearly label each bottle.

Sample ID	ML of 100 ppb Hg (8.9.2)	ML of 10 ppb Hg (8.9.3)	Final Volume of DI water (ML)	ppb of Hg
Spike Blank	0.60	-	19.4	3.0
CCV	-	6	14.0	3.0
CCB	0.0	-	20.0	0.0
Method Blank	0.0	-	20.0	0.0
Matrix Spike	0.60	-	0.0	3.0+ sample
Matrix Spike DUP	0.60	-	0.0	3.0+ sample
Original	-	-	0.0	-

10.1.4. In addition, an external QC sample (from an alternate source than the above curve) should be set up so that the final level is close to the mid-range of the curve.

	ML of 10 ppb Hg (8.9.7)	Final Volume of DI water (ML)	ppb of Hg
ICV	8	12.0	4.0

10.1.5 Samples. The pH of all aqueous samples must be verified to be <2 prior to aliquot for processing. If samples have a pH of >2 add additional nitric acid and wait 24 hours before rechecking the pH. If no information is available about the level of mercury in the samples to be analyzed, set up a 20.0 ml sample size. If information is available, select a sample size that will result in an analysis value near the mid-range of the curve. Record the volume used on the sample analysis data sheet. Shake the sample to homogenize.

10.1.6 To all samples, QC and standards add the following reagents **(perform under the hood):**

10.1.6.1 1.0 ml of conc. sulfuric acid.
10.1.6.2 0.50 ml of conc. nitric acid.
10.1.6.3 3.0 ml of permanganate solution.

10.1.7 Swirl the samples well after each addition of reagent. More potassium permanganate solution may be required for some samples. Enough should be added so that the purple color persists for at least 15 minutes (Up to 5 ml additional permanganate). Adjust/add extra permanganate to method blank, and spike blank.

10.1.8 Add 1.6 ml of persulfate solution and mix.

10.1.9 Cover the samples and place them in digestion block. Set the temperature to between 90 to 95°C. After the block has reached this temperature range, heat the samples for 2 hours and record the temperature on the temperature log. Then remove and cool. Add 1.2 ml of hydroxylamine sulfate solution to all and mix. Then dilute all to 30 ml with DI water.

10.1.10 While the samples are digesting, begin setting up the mercury analyzer following the steps outlined below. Further details are available in the instrument manual.

10.1.10.1 Turn the nitrogen gas on, if it is not already on. Place the vent line under the hood.

10.1.10.2 Inspect all pump tubing and change if needed. Put the tubing on the cassettes on the Pump head. Check the drying line and make sure it is clean. Place fresh stannous chloride solution in the bottle. Fill the rinse bottle with 2% hydrochloride acid.

10.1.10.3 Double click on the WINHG icon on desktop. This will open the WinHg runner. Click on the

Control tab and click on " turn on gas" option and also "turn on pump" option as well.

10.1.10.4 Click on the **DB** ↓ button. This will open the WinHg Database window. Go to file and choose NEW PROTOCOL. Type the date (ie. 040502) in the box. This will be the protocol for that day. In WinHgDatabase window, click on "LINE INFO" tab and enter the calibration standard concentrations and click on "Apply Button". Click on **RN** ↑ button and this will upload all informations into the WinHgRunner window.

10.1.10.5 Click on the "STANDARD" tab in WinHgRunner window. And click on **S1** through **S7** (no. of standards). Click on "REP1" button. Click on "STAND AUTO" button and calibration will start.

10.1.10.6 While calibration is being performed, type the analysis sequence by clicking on the Rack Button. Enter the samples and appropriate QC. ICV, CCV, CRI and CCB. Save the sequence as Hg –day-month-matrix-run no.(ie, Hg0405w1). After calibration is performed and accepted, click on the Sample tab in WinHg Runner and click on "START NEW BATCH " button. Type the auto-sampler sequence file name as above (ie,Hg0405w1).

10.1.10.7 In auto-Sampler Run rack name drop down window, choose the file name you are Analyzing. Assign the location of the start cup and the End cup, click the RUN AUTO button.

10.2 **Calibration**

10.2.1 Verify that the curve is within acceptable QC limits (0.995 corr.). If the curve is not within acceptable limits, consult with your laboratory supervisor.

10.2.2 Immediately after the system has been calibrated, an ICV and ICB should be analyzed. The ICV results must be within 90 to 110 percent of the true value and ICB must be less than the reporting limit. If not, re-analyze one more time. If still outside the range, analysis must be terminated, and the problem corrected and recalibrate.

10.2.3 Continuing Calibration Verification. Analyze the continuing calibration verification solution and the continuing calibration blank after ICV/ICB, after every tenth sample and at the end of the sample run. CCV checks should be within 20% for SW846 7470A. If the CCV is not within these ranges, reanalyze one more time. If still outside the range, no samples can be reported in the area bracketed by the failing CCV. Resolve the problem, recalibrate. If multiple CCV's are prepared, combine all into one source.

10.2.4 Continuing Calibration Blank. Analyze the continuing calibration BLANK (CCB) solution after every CCV. If results for the CCB are not less than the reporting limit, then no samples can be reported in the area bracketed by the failing CCB. If multiple CCB's are prepared, combine all into one source.

10.2.5 CRI (low calibration check). Analyze the CRI after the initial CCV and at the end of the run. If the results are not within ±20%, reanalyze one more time. If still outside the range then no samples can be reported. Note: If a CRDL standard is required (prepared at 2 times the level of the low standard) see the supervisor.

10.3 Sample Dilutions

10.3.1 Establish dilution of sample in order to fall within the mid to upper level of the calibration range. If available, use historical data to obtain proper dilution.

10.3.2 If undiluted sample result is above the upper range of the calibration curve, perform a dilution on the digestate.

11.0 QUALITY ASSURANCE

11.1 Quality Control Sample (QCS/ICV). It is recommended that this sample be analyzed at the start of each run. For the QCS, the instrument result must be within 10% of the true value. The quality control sample must be from a different source than the calibration standards.

11.2 Method Blank. The laboratory must digest and analyze a method blank with each set of samples. A minimum of one method blank is required for every 20 samples. The method blank must not contain mercury at greater than the reporting limit. If the method blank contains levels over the reporting limits, the samples must be redigested or reanalyzed (Specific clients may have different criteria for method blanks – such as ½ the RL for acceptable contaminant levels). The exception is if the samples are non-detected for mercury.

11.3 Lab Control Sample (blank spike). The laboratory must digest and analyze a laboratory control sample with each set of samples. A minimum of one lab control sample is required for every 20 samples. Until sufficient lab control data becomes available the laboratory should assess laboratory performance against recovery limits of 80-120%. When sufficient data is available, develop control limits from the percent mean recovery and the standard deviation of the mean recovery. The upper control limit should be equal to the mean plus 3 times the standard deviation. The lower control limit should be equal to the mean minus 3 times the standard deviation. The generated limits must meet or exceed 80-120%. If the blank spike is outside of the control limits, all samples must be redigested and reanalyzed . The exception is if the lab control recovery is high and the results of the samples to be reported are less than the reporting limit. In that case, the sample results can be reported with no flag.

11.4 Matrix Spike. The laboratory must add a known amount of each analyte to a minimum of 1 per 20 samples. The matrix spike recovery is calculated as shown below. The laboratory should apply control limits of 75-125% to assess whether a spike is in control. If a matrix spike is out of control, then the results should be flagged with the appropriate footnote (if the matrix spike recovery is out of control but the blank spike recovery is within acceptance criteria – matrix interference is indicated).

$$\frac{Spiked\ Sample\ Result - Sample\ Result}{Amount\ Spiked} \times 100 = Matrix\ Spike\ Recovery$$

11.5 Matrix Spike Duplicate. The laboratory must digest a Matrix Spike Duplicate for a minimum of 1 in 10 samples. This second aliquot of the original sample that is spiked in order to determine the precision of the method. The relative percent difference (RPD) between the Matrix Spike duplicate and the Matrix Spike should be assessed using in house limits. Until these limits can be generated, then default limits of ±20% RPD should be applied.

$$\frac{(\,|MS\ Result - MSD\ Result|\,) \times 100}{(\,MS\ Result + MSD\ Result\,)/2} = MSD\ RPD$$

11.6 A Precision and accuracy (P&A) study is performed as an initial determination of capability, on an annual basis, and if any significant changes have been made to the instrument. In general, 4 replicates or blank spikes are analyzed using the same procedures and conditions for sample analysis. The percent recovery is compared to 80-120% until in-house control limits are established. If percent recovery criteria are not met, corrective action must be taken to bring the system back into control.

12.0 DOCUMENTATION

12.1 The standard preparation logbook must be completed for all standard preparations. All information requested must be completed; the page must be signed and dated by the respective person.

12.2 The Accutest lot number must be cross-referenced on the standard vial/container.

12.3 The instrument Maintenance logbook must be completed when any type of maintenance is performed on the instrument. Each instrument will have a separate log.

12.4 Document all information in the mercury analysis log book. This information must include start time, end time, temperature, reagent lot# for all digestions solutions, as well as sample ID, volume, and any dilution factors during analysis. Note any unusual sample characteristics in the comment section. Make sure that all sample ID's and standard lot# are recorded in analysis log book. All comments and edits MUST BE clearly documented and initialed. Generate run logs from the LIMS system along with all quality control data.

12.5 Each analyst should review all data and assemble a data package consisting of :

- Copy of the prep log book
- Copy of the analysis log book
- LIMS generated run logs and QC
- Raw data
- LIMS generated work group list

12.6 Any corrections to laboratory data must be done using a single line through the error. The initials of the person and date of correction must appear next to the correction.

12.7 All laboratory logbooks must be routinely reviewed and initialed or signed by the lab manager.

13.0 DATA REVIEW

13.1 The analyst conducts the primary review of all data. This review begins with a check of all Instrument and method quality control and progresses through sample quality control concluding with a check to assure that the client's requirements have been executed. The analyst has the authority and responsibility to perform corrective action for any out-of-control parameter of non-conformance.

13.2 A secondary review is performed by department managers, and it includes review of the data produced by their department. Manual calculations, QC criteria, and a comparison of the data package to client specified requirements are checked. The department manager may reject data, initiate reanalysis, take additional corrective action, or reprocess data.

13.3 The laboratory director performs a full tertiary review of the data package following its assembly. This review includes an evaluation of QC data against acceptance criteria and a check of the data package contents to assure that all analytical requirements and specifications were executed

13.4 Spot-check reviews are performed by the Quality Assurance Officer focusing on all elements of the deliverable including the client's specifications and requirements, analytical quality control, sample custody documentation and sample identification

14.0 DATA REPORTING

14.1 A results page including positive result and/or RL for mercury, units, methodology, date of digestion and analysis, and data qualifiers are reported. Additional quality control data including calibration summaries, MS/MSD percent recoveries and RPDs, blank spike recoveries, and method blank results may be reported upon request of the client.

14.2 Data may be submitted to the client in a specified electronic format (EDD).

14.3 Data may be submitted to the client as a PDF (e-hardcopy).

14.4 Once the data is approved by the laboratory manager, it may be accessed by clients via LabLink™.

15.0 POLLUTION PREVENTION & WASTE MANAGEMENT

15.1 Pollution Prevention. Users of this method must perform all procedural steps that control the creation and/or escape of wastes of hazardous materials to the environment. The amounts of standards, reagents, and solvents must be limited to the amounts specified in this SOP. All

safety practices designed to limit the escape of vapors, liquids, or solids to the environment must be followed. All method users must be familiar with the waste management practices described in section 15.2

15.2 Waste Management. Individuals performing this method must follow established waste management procedures as described in the Sample and Waste Disposal SOP. This document describes the proper disposal of all waste materials generated during the testing of samples as follows:

15.2.1 Non-hazardous aqueous wastes
15.2.2 Hazardous aqueous wastes
15.2.3 Chlorinated organic solvents
15.2.4 Non-chlorinated organic solvents
15.2.5 Hazardous solid wastes
15.2.6 Non-hazardous solid wastes

Lab Manager: Brad Madadian
QA Manager: Robert Treggiari

TEST NAME: METALS BY INDUCTIVELY COUPLED PLASMA – MASS SPECTROMETRY (ICP-MS)

METHOD REFERENCE: SW846 6020A, Revision 1, February 2007.

Revised Sections: 9.8.1

1.0 SCOPE AND APPLICATION

1.1 This method is applicable for the determination of total and dissolved metals in water samples and in waste extracts or in solid or aqueous digests.

2.0 SUMMARY

2.1 Samples are prepared for analysis by digestion. The prepared samples are introduced into radio frequency plasma by pneumatic nebulization. There the energy transfer processes cause desolvation, atomization, and ionization. The ions are extracted from the plasma through a differentially pumped vacuum interface and separated on the basis of their mass to charge ratio by a quadrupole mass spectrometer. The ions transmitted through the quadrupole are detected by an electron multiplier and the ion information is processed by a data handling system.

3.0 REPORTING LIMIT AND METHOD DETECTION LIMIT

3.1 Reporting Limit. Current reporting limits for this method have been established at the levels listed in Table 1. The reporting limits are dependent upon the metal being analyzed and are in all cases greater than the IDL and the MDL for each element. Note: Many clients require special reporting limits. Refer to the scheduling sheets and check with the metals supervisor for additional information.

3.2 Method Detection Limit. Experimentally determine MDLs using the procedure specified in 40 CFR, Part 136, Appendix B. This value represents the lowest reportable concentration of an individual compound that meets the method qualitative identification criteria.

3.3 Experimental MDLs are determined initially (prior to analysis), on an annual basis, and after major maintenance to equipment. MDL data is archived with Quality Assurance. Refer to the most recent study for current MDLs. Refer to the SOP for MDLs (MQA245) for additional detail regarding MDL study procedures. Electronic MDL data is found in the annual “MDL” folder on the QA server (LINUXMA1).

4.0 DEFINITIONS

BATCH. A group of 20 samples or less that behave similarly with respect to the sampling or the testing procedures being employed and which are processed as a unit within a 24 hour period. For QC purposes, if the number of samples in a group is greater than 20, then each group of 20 samples or less will all be handled as a separate batch.

CALIBRATION CHECK STANDARD. The calibration check standard is a mid-range calibration standard. It is recommended that the calibration check standard be run at a frequency of 10 percent or every 2 hours during an analysis run, whichever is more frequent, and at the end of the analysis sequence. For this method, the mid-level calibration check standard criteria is ± 10 percent of the true value and the relative standard deviation for the replicates that are greater than 5 times the reporting limit is less than 5 percent. The exception to this rule is if the recovery on the calibration check standard is high and the samples to be reported are less than the reporting limit.

EXTERNAL CHECK STANDARD. The external check standard is a standard from a separate source than the calibration curve that is used to verify the accuracy of the calibration standards. It must be run after each calibration. The external check standard criteria is ± 10% of the true value and the replicates that are greater than 5 times the reporting limit should have a relative standard deviation of less than 5 percent. If the external check is outside of the control limits for a given parameter, all samples must be reanalyzed for that parameter after the problem has been resolved.

SPIKE BLANK OR LAB CONTROL SAMPLE. Digest and analyze a laboratory control sample or spike blank with each set of samples. A minimum of one lab control sample or spike blank is required for every 20 sample batch. A sample batch is defined as a maximum of 20 field samples in a preparation batch over a time period of 24 hours. Assess laboratory performance against the control limits of 80 to 120 percent. In house limits should also be generated once sufficient data is available to support the default limits. If the lab control or spike blank is outside of the control limits for a parameter, all samples must be redigested and reanalyzed for that parameter. The exception is if the lab control or spike blank recovery is high and the results of the samples to be reported are less than the reporting limit. In that case, the sample results can be reported with a sample case narrative.

MATRIX: The component or substrate (e.g., water, soil) which contains the analyte of interest.

MATRIX SPIKE: The laboratory must add a known amount of each analyte to a minimum of 1 in 20 samples. The matrix spike recovery is calculated as shown below. Assess laboratory performance against default limits of 75 to 125 % recovery. In house limits should be generated once sufficient data is available. If a matrix spike is out of control, then the results should be flagged with the appropriate footnote. If the matrix spike amount is less than one fourth of the sample amount, then the sample cannot be assessed against the control limits and should be footnoted to that effect.

$$\frac{(\text{Spiked Sample Result} - \text{Sample Result}) \times 100}{(\text{Amount Spiked})} = \text{Matrix Spike Recovery}$$

MATRIX SPIKE DUPLICATES: Intralaboratory split samples spiked with identical concentrations of target analyte(s). The spiking occurs prior to sample preparation and analysis. They are used to document the precision and bias of a method in a given sample matrix. A matrix spike duplicate is digested at a minimum of 1

in 10 samples. The relative percent difference (RPD) between the matrix spike duplicate and the matrix spike should be assessed. The matrix spike duplicate RPD is calculated as shown below.

$$\frac{(|\text{Spiked Sample Result} - \text{Spiked Duplicate Result}|) \times 100}{(\text{Spiked Sample Result} + \text{Spiked Duplicate Result})/2} = \text{Spike Duplicate RPD}$$

METHOD BLANK. The laboratory must digest and analyze a method blank with each set of samples. A minimum of one method blank is required for every 20 sample batch. If no digestion step is required, then the method blank is equivalent to the reagent blank. The method blank must contain the parameter of interest at levels of less that the reporting limit for that parameter. If the method blank contains levels over the reporting limits, the samples must be redigested and reanalyzed. The exception to this rule is when the samples to be reported contain greater than 10 times the method blank level. In addition, if all the samples are less than a client required limit and the method blank is also less than that limit, then the results can be reported as less than that limit.

METHOD DETECTION LIMITS (MDLS). The minimum concentration of a substance that can be measured and reported with 99% confidence that the analyte concentration is greater than zero and is determined from analysis of a sample in a given matrix containing the analyte. MDLs should be determined approximately once per year for frequently analyzed parameters.

REAGENT BLANK. The reagent blank is a blank that has the same matrix as the samples, i.e., all added reagents, but did not go through sample preparation procedures. The reagent blank is an indicator for contamination introduced during the analytical procedure. (Note: for methods requiring no preparation step, the reagent blank is equivalent to the method blank.) Either a reagent blank or a method blank must be analyzed with each batch of 20 samples or less. The concentration of the analyte of interest in the reagent blank must be less than the reporting limit for that analyte. If the reagent blank contains levels over the reporting limits, the samples must be reanalyzed. The exception to this rule is when the samples to be reported contain greater than 10 times the reagent blank level. In addition, if all the samples are less than a client required limit and the reagent blank is also less than that limit, then the results can be reported as less than that limit.

REAGENT GRADE. Analytical reagent (AR) grade, ACS reagent grade, and reagent grade are synonymous terms for reagents which conform to the current specifications of the Committee on Analytical Reagents of the American Chemical Society.

REAGENT WATER. Water that has been generated by any method which would achieve the performance specifications for ASTM Type II water.

STANDARD ADDITION. The practice of adding a known amount of an analyte to a sample immediately prior to analysis. It is typically used to evaluate interferences.

STANDARD CURVE: A plot of concentrations of known analyte standards versus the instrument response to the analyte. Calibration standards are prepared by successively diluting a standard solution to produce working standards which cover the working range of the instrument. Standards should be prepared at the frequency specified in the appropriate section. The calibration standards should be prepared using the same type of acid or solvent and at the same concentration as will

result in the samples following sample preparation. This is applicable to organic and inorganic chemical analyses.

5.0 HEALTH & SAFETY

5.1 The analyst must follow normal safety procedures as outlined in the Accutest Health and Safety Plan and Personal Protection Policy, which include the use of safety glasses and lab coats. In addition, all acids are corrosive and must be handled with care. Flush spills with plenty of water. If acids contact any part of the body, flush with water and contact the supervisor.

5.2 The toxicity or carcinogenicity of each reagent used in this method has not been precisely determined; however, each chemical must be treated as a potential health hazard. Exposure to these reagents must be reduced to the lowest possible level. The laboratory is responsible for maintaining a current awareness file of OSHA regulations regarding the safe handling of the chemicals specified in this method. A reference file of data handling sheets must be made available to all personnel involved in these analyses.

6.0 PRESERVATION & HOLDING TIME

6.1 All water samples should be preserved with nitric acid to a pH of 2 or less. All solid samples should be stored in a refrigerator at 4 degrees C until digestion.

6.2 All samples should be analyzed within 6 months of the date of collection.

7.0 INTERFERENCES

7.1 Several types of interferences can cause inaccuracies in trace metals determinations by ICP-MS. These interferences are discussed below.

7.2 Isobaric elemental interferences are caused by isotopes of different elements which form singly or doubly charged ions of the same nominal mass-to-charge ratio and which cannot be resolved by the mass spectrometer in use. If isobaric interferences are present in the ion being analyzed, then the data must be corrected by measuring the signal from another isotope of the interfering element and subtracting the appropriate signal ratio from the element of interest.

7.3 Abundance sensitivity is a property that defines the degree to which the wings of a mass peak contribute to adjacent masses and is affected by ion energy and quadrupole operating pressure. Wing overlap interferences may result when a small ion peak is being measured next to a large one. Spectrometer resolution should be adjusted to minimize these interferences.

7.4 Isobaric polyatomic ion interferences are caused by ions consisting of more than one atom which have the same nominal mass-to-charge ratio as the isotope of interest, and which cannot be resolved by the mass spectrometer in use. Refer to method 200.8 and 6020 for lists of common interferences and correction equations to be applied. If these interferences

cannot be avoided by the use of different isotopes, then correction equations should be applied to the data. Alternatively, collision/reaction cell technology can be applied to physically and chemically remove interferences.

7.5 Physical interferences can occur during the transfer of the solution to the nebulizer (viscosity effects).

7.6 Memory interferences can be caused by build up on the sampler and skimmer cones, and from buildup of sample material in the torch and spray chamber. Some elements, such as mercury, can suffer from severe memory effects. In that case, gold is added to the rise solution to decrease the Hg rinse out time.

8.0 APPARATUS

8.1 Currently there is one ICP-MS instrument available for use in the lab. The Agilent 7500CX ICP-MS with collision/reaction cell capacity and the associated autosampler.

8.2 Class A volumetric glassware as needed and instrument autosampler tubes.

8.2.1 All glassware must be washed with soap and tap water and then soaked in a 10% nitric acid bath for several hours. It must then be rinsed at least 3 times with distilled, deionized water.

8.3 Polypropylene bottles for standard storage. These bottles must also be cleaned as outlined above.

9.0 REAGENTS

9.1 All chemicals listed below are reagent grade unless otherwise specified. Deionized water must be used whenever water is required. Note: All reagents can be scaled up or down proportionately if different final volumes are required.

9.2 Hydrochloric acid, trace metals grade.

9.3 Nitric acid, trace metals grade. Note – ultra trace grade may be required if lower detection limits than normal are needed.

9.4 Standard stock solutions available from Inorganic Ventures, Ultra Scientific, Agilent or equivalent. Note: All standards must be ICP-MS quality standards or must be demonstrated to be free of interferences at the levels of use. Standards should come labeled with an expiration date and certificate of concentrations from the manufacturer. If both of these items are not received, then the manufacturer should be contacted before use of the standard.

9.5 Calibration Standards: These can be made up by diluting the stock solutions to the appropriate concentrations. Fresh calibration standards should be prepared a minimum of every two weeks.

9.5.1 Standards should be made in a low acid matrix. Concentrations of 1 to 2 percent nitric acid and 0 to 0.6 percent hydrochloric acid are suggested, although any acid concentration that provides good analytical results may be used. High chloride concentrations may cause interferences so chloride concentrations should be limited. HCl may be omitted if silver and antimony are not elements of interest.

9.5.2 Refer to the Reagent Application for the make-up and concentrations of standards and stock solutions being used to calibrate the ICP-MS. Suggested standard levels are shown in Table 2. Calibrations must consist of a minimum of a blank and a high standard. The calibration must be verified with a low check at the reporting limit at the time of analysis

9.5.3 All standards should be stored in acid washed FEP fluorocarbon bottles.

9.6 Pulse/Analog (P/A) Factor and Tuning/Performance Check Solution. Mix 1.0 ml of PA Tuning 1 solution and 1.0 ml of PA Tuning 2 solution (available from Agilent, part number 5188-6524) and bring to 100 ml final volume with a solution of 1% nitric acid and 0.6% HCl. This final solution contains 200 ppb of As, Be, Cd, Zn; 100 ppb of Mg, Ni, and Pb; 50 ppb of Al, Ba, Bi, Co, Cr, Cu, In, Li^6, Lu, Mn, Na, Sc, Sr, Th, Tl, U, and V; and 25 ppb of Y and Yb; 100 ppb of Ge, Mo, Pd, Ru, Sb, Sn ; and 50 ppb of Ir and Ti.

9.7 Tuning Standard. This solution is used to verify mass calibration and thermal stability and must contain a mix of elements representing all of the mass regions of interest. Elements include 1 ppb Ce, Co, Li, Mg, Tl, and Y.

9.8 Internal Standards. Internal standards are added to all calibration standards, quality control, and samples during analysis, normally using a second channel of the peristalic pump and a mixing manifold. For full mass range scans, a minimum of three internal standards must be used. It is recommended that all elements have an internal within a mass range of 50.

9.8.1 A stock solution containing 100 ppm of Li, Sc, Ge, Y,Rh, In, Tb, and Bi. 1 ml stock solution to 100 ml final volume with a solution of 1% HNO3 and 0.6% HCl. The concentration of this final solution is 1 ppm, with a 0.25 mm IS pump tubing equates to approximately 50 ppb in the plasma. Refer to Table 3 for internal standard masses and associated Tune.

9.9 Calibration /Rinse Blank. The calibration and rinse blanks are prepared by diluting acids to the same concentrations found in the standards. The calibration blank is used to establish the analytical calibration curve and the rinse blank is used to flush the instrument between samples in order to reduce memory interferences.

9.10 Continuing Calibration Verification Check (CCV). This solution is prepared by adding either mixed or single element metals solutions to a solution containing the same acid matrix as the calibration standards. The metals should be at concentrations near the middle of the calibration curve. (Note: This check is run after the calibration, after every 10 samples or

every 2 hours during an analysis run, whichever is more frequent, and at the end of the sample run.) Refer to Table 2 for suggested concentrations for the CCV.

9.10.1 Method 6020 does not specify the source of the CCV check. However, it is recommended that these be prepared from the same source as the calibration as it required in method 200.8

9.11 Matrix Spike and Spike Blank Solution. Suggested levels for the final concentrations of the spike are shown in Table 4. This solution is prepared by adding either mixed or single element metals solutions to a solution containing 1 % HNO3 and 0 to 0.6 % HCl and diluting to a fixed final volume with this acid mixture. 0.5 ml of this stock solution should be added to spike blank and the matrix spike before they are digested and brought to a final volume of 50 ml. For this particular method, a lab control (Section 9.12) is used more frequently than a spike blank. In situations where any odd elements, such as B, Sr, and Sn, is of interest for a specific project, besides a lab control, a spike blank spiked with these elements is also digested.

9.12 Lab Control Solution. This solution is prepared by adding either mixed or single element metal solutions to a solution containing 1 % HNO3 and 0 to 0.6 % HCl and diluting to a fixed final volume with this acid mixture. 50 ml of this solution is digested and brought to a final volume of 50 ml.

9.13 Interference Element Check Solutions. The purpose of the ICSA and ICSAB solutions is to demonstrate the magnitude of interferences and provide an adequate test of any corrections. It is recommended that the following solutions be purchased commercially.

9.13.1 ICSA Solution. The ICSA solution contains only the interfering elements. The recommended concentrations are shown below. The ICSA solution must be made fresh weekly.

Al	100 mg/L
Ca	100 mg/L
Fe	100 mg/L
Mg	100 mg/L
Na	100 mg/L
P	100 mg/L
K	100 mg/L
S	100 mg/L
C	200 mg/L
Cl	1000 mg/L
Mo	2.00 mg/L
Ti	2.00mg/L

9.13.2 ICSAB Solution. The ICSAB solution contains both the interferents and the analytes of interest. The recommended concentrations are shown below. The ICSAB solution must be made fresh weekly.

Al	100 mg/L
Ca	100 mg/L
Fe	100 mg/L
Mg	100 mg/L
Na	100 mg/L
P	100 mg/L
K	100 mg/L
S	100 mg/L
C	200 mg/L
Cl	1000 mg/L
Mo	2.00 mg/L
Ti	2.00mg/L
As	0.020 mg/l
Cd	0.020 mg/l
Cr	0.020 mg/l
Co	0.020 mg/l
Cu	0.020 mg/l
Mn	0.020 mg/l
Ni	0.020 mg/l
Ag	0.020 mg/l
Zn	0.020 mg/l

9.14 Initial Calibration Verification (ICV) or Quality Control Sample (QCS). The metals in this solution should be at final concentrations that are at the mid-point of the calibration curve. This solution is prepared by adding either mixed or single element metals solutions to a solution containing 1 % nitric acid and 0 to 0.6 % hydrochloric acid and diluting to a fixed final volume with this acid mixture. The ICV sample must be from a separate source from the calibration standards. This solution should be stored in a FEP fluorocarbon or previously unused polyethylene bottle. Refer to Table 2 for suggested concentrations for the ICV.

9.15 CRI Standards (also referred to as LLCCV). The CRI standard must contain the elements of interest at (or below) the reporting limit for each element. The CRI level is at the reporting limit as shown in Table 1. This should be prepared by diluting calibration standard(s) to the reporting limit level for each element. They should be made in the same matrix as the calibration standards. Note: The CRI must be verified at the RL before any dilutions are applied. For example, Be is verified at 0.5 ug/l and the water reporting limit is 1.0 ug/l with a 1:2 dilution.

9.16 Liquid Argon or Argon Gas. Argon, high purity grade (99.995%), is supplied by Air Products, Inc in the large outdoor tank. No lab monitoring of the tank is normally necessary.

9.17 Helium Gas. Helium, high purity grade (99.995%), is supplied by Air Products, in a small cylinder tank. The lab monitoring of the tank is necessary. This is required for running the reaction cell.

10.0 INITIAL INSTRUMENT SET-UP PROCEDURE FOR THE AGILENT 7500CX ICP-MS

10.1 A general procedure on how to operate the Agilent 7500CX ICP-MS is given below. Refer to the operation manual for further details.

10.2 Before bringing up the instrument, make sure that the lines, the torch, the nebulizer, and the spray chamber are clean, and that there are no leaks in the torch area.

10.3 Turn the vacuum pump and the heat exchanger on and verify that the liquid argon is turned on and the helium gas is turned on.

10.4 Connect the pump tubing and engage the peristaltic pump.

10.5 Put a new solution of acid rinse into the rinse reservoir. (Note: the composition of the rinse solution may be periodically changed to minimize sample introduction problems and sample carryover.) Make sure that sufficient internal standard solution is present.

10.6 Open the ICP-MS Chem Station Top software. Click on the instrument and open the instrument control panel. Click the plasma on. The instrument will automatically go through the start-up cycle. Then let the instrument warm up for at least 30 minutes.

10.7 Every one to two days or as needed, tune the instrument. Tuning must always be done after moving the position of the torch or the cones. Tuning can be done either manually or by following autotune procedures. It is recommended that autotune procedures be followed initially and then manual tuning be done as a second step. The purpose of tuning is to optimize the instrument for the highest sensitivity while obtaining low levels of oxides and doubly charged species. After the tune is complete, make sure to save the optimized parameters.

10.7.1 Open the ICP-MS top software, click on the instrument, and open the ICP-MS tuning page

10.7.2 Click file and open the NOGAS.U file. Keep the internal standard line in a solution of 1% nitric acid and 0.6% hydrochloric acid. Using the ALS (autosampler) send the probe to the 1 ppb Agilent tuning solution (see 9.7). On the tuning page, click start under the RTD window to see the counts and RSD values. Do not start the tune process until the count and mean have similar readings and the RSD is < 5%. Click stop under RTD window.

10.7.3 On the tuning page, go to file, select Generate Multi_Mode Report, type the date on the pop-up window, and click OK. This will perform the tuning of the instrument using both the NOGAS and the Helium mode. Print the tune and save it as MAXXXXX_Tune.pdf.

10.7.4 Also tune the instrument for NOGAS and the Helium mode separately if necessary, then generate Multi_Mode report.

10.7.4.1 On the tuning page, open the NOGAS.U file, go to file, select Generate Report, type the date on the pop-up window, and click OK. This will perform the tuning of the instrument using NOGAS mode.

10.7.4.2 On the tuning page, open the He.U file, go to file, select Generate Report, type the date on the pop-up window, and click OK. This will perform the tuning of the instrument using Helium mode.

10.8 On a daily basis, perform a cross calibration to align the pulse and analog signals.

10.8.1 Go to the ICP-MS Top portion of the software and be sure that the NOGAS.U file is open. Using ALS, send the probe to the P/A factor solution. This solution is diluted from a concentrated mixture of PA Tuning 1 and PA Tuning 2 solution which can be purchased from Agilent Technologies.

10.8.2 In the ICP-MS Tuning page, under the Tune, click P/A factor. Click run in the popup window. Some elements may have too low or too high sensitivity. In this case, rerun the P/A factor process one or more times.

10.8.3 Print and save the P/A factor report as MAXXXX_PA.pdf.

10.9 Before calibrating, run and print out a performance test. This must include the following items.

10.9.1 Demonstrate instrument stability by running the tuning solution a minimum of five times. Relative standard deviations of the absolute signals must be less than 5 percent for all elements in the tuning solution. If this criteria is not met, correct the problem and then repeat the stability test. Print the results of this test and store with the raw data for the run.

10.9.2 Verify acceptable mass calibration by running the tuning solution and monitoring the peak width measured at 5% of peak maximum for 7Li, 59Co, 115In and 205Tl. If the peak widths are outside of the range of 0.65 to 0.85 and the masses are off more than 0.1 amu, then redo the mass calibration as outlined in 10.8 before proceeding.

10.9.3 To run this performance test, be sure that the NOGAS.U file is open. Set up the daily run sequence. Select TN_6020.M method and run. This method is set up to run 5 replicates. After the performance test is finished, click print (in case of TN200_8.M is also ran as part of performance test, combine the portions of the performance test together in a PDF converter window) and then save this as MAXXXX_perf.pdf.

10.10 Before starting sample analysis, set up the internal standards. Internal standards are added to all calibration standards, quality control, and samples during analysis, normally using a second channel of the peristaltic pump and a mixing manifold. Refer to Table 3 and Section 9.8 for additional information.

10.11 To start running samples, open the ICP-MS Top window, and then click method followed by load. Selection the method from the list. The normal method used for 6020 analyses is EPA6020V.M

10.11.1 Click sequence and then click load. Select the latest sequence or template from the list.

10.11.2 Click Edit the sample log table and type in the standards and samples. Save the sequence as MMDDYY.s (i.e: 063012.s). Be sure to load the saved sequence again. Click Position and Run to start the run.

10.11.2.1 At the pop-up window, the data Bach directory line will show file name as C:\ICPMH\1\DATA\12F30XXX.B\. (i.e: 12F30XXX.B, 12 is the year; F is the month, in this case is 6; 30 is the date) Click on run sequence. This will open the data analysis page.

10.12 Calibrate the instrument using a minimum of a calibration blank and three non-zero standards that bracket the desired sample concentration range. (Note: The calibration standards may be included in the autosampler program or they may be run separately.) A correlation coefficient of 0.998 or better must be obtained using a first order (linear) curve fit. A minimum of three replicate integrations are required for all data acquisitions.

10.12.1 In between each analysis of a separate standard or sample, a rinse blank must be run through the lines of the sample introduction system. Each sample or standard should be aspirated for a minimum of 30 seconds prior to the acquisition of data to allow equilibrium to be established.

10.12.2 Alternatively, a calibration may be done with a blank and a high standard. This calibration must then be confirmed with low level and mid-level calibration standards that are run immediately after the calibration is complete. The low level check must have recoveries within 70 to 130 % to be acceptable and the mid-level check must have recoveries within 90 to 110 %to be acceptable.

10.13 After the instrument is properly calibrated, begin by analyzing the ICV solution. The ICV must be run after each calibration. For the ICV, all elements to be reported must be within 10 % of the true value and the replicates that are greater than 5 times the reporting limit should have a relative standard deviation of less than 5 %. If the ICV is outside of criteria, then the problem must be identified and corrected before samples can be run and reported for the element(s) that are outside of criteria. Correction of the problem can be verified by rerunning the check standard(s) and showing that they meet QC criteria.

10.13.1 An ICB may be run after the ICV, but is not required for this method. If it is run, then all elements must be less than reporting limit (lower limit of quantitation) for each element.

10.13.2 Run the CRI (LLCCV) solution right after the ICV and ICB, (or any other place at the beginning of the run after the ICV, ICB and before any real samples are analyzed). For the CRI, all elements of interest must be within 30% of the true value or within client specified limits.

10.14 Then analyze the CCV and CCB check standards. For the CCV, all elements to be reported must be within 10 % of the true value and the replicates that are greater than 5 times the reporting limit should have a relative standard deviation of less than 5 %. For the CCB, all elements to be reported must normally be less than the reporting limit (lower limit of quantitation). If either the CCV or CCB do not meet criteria, then elements failing this criteria must not reported in the area bracketed by this QC.

10.14.1 The internal standard levels in the CCV and CCB must also be within 30% of the internal standard level for the initial calibration. If they are outside of these levels, then no samples can be reported in the area bracketed by this QC.

10.15 After the initial QC is completed and before any samples are analyzed, the ICSA and ICSAB solutions must be analyzed. The method does not list specific criteria for the ICSA and ICSAB, but in house criteria will be applied. For all the spiked elements, the analyzed results must be within 20 % of the true value. For unspiked elements, the interfering element solution should contain less than the absolute value of 3 times the reporting limit for each element. If these criteria are not met, then samples with significant interferences can not be reported until the instrument is optimized and the ICSA and ICSAB are within specifications.

10.15.1 If the run is longer than 12 hours, a second ICSA, ICSAB pair must be analyzed before the next 12 hours is started.

10.15.2 If mass changes are made for the analysis of an element, all QC criteria must be met for the new mass and it must be verified that appropriate correction factors are in place.

10.15.3 The Agilent 7500CX includes collision/reaction cell technology. The instrument is tuned both in regular (non-cell) mode and in helium (collision/reaction) cell mode. This technology is used to minimize interferences during analysis. If this technology is not applied, then correction factors for interferences must be added into the method. Table 1 includes which elements are run using collision/reaction cell technology.

10.16 After the initial analytical quality control has been analyzed, the samples and the preparation batch quality control should be analyzed. Depending on the type of digestion and the sample matrix, samples and the associated QC should normally be diluted by a factor of from 2 to 5 before analysis. This dilution factor should be indicated in the sample ID file on the instrument.

10.16.1 Each sample analysis must be a minimum of 3 integrations. For samples containing levels of elements greater than approximately 5 times the reporting limits, the relative standard deviations for the replicates should be less than 10%. If not, reanalyze the sample. If, upon reanalysis, the RSDs are acceptable, then report the data from the reanalysis. If RSD's are not acceptable on reanalysis, then the results for that element may, on the reviewer's discretion, be footnoted that there are possible analytical problems indicated by a high RSD between replicates. In some cases, an additional dilution analysis may be needed. Check with the area supervisor or manager for additional information.

10.16.2 The internal standard levels must be monitored for all samples and quality control. If the internal standard is not within 30% of the internal standard level for the initial calibration blank, then the sample must be diluted by a factor of 5 to bring the internal standard to within the correct range. If the internal standard is still outside of the range after the initial 1:5 dilution, then additional dilutions must be done until the internal standard is within the appropriate range.

10.16.2.1 If an internal standard is present in a sample, then do not use that internal standard. For example, Y is sometimes seen in real samples. If the Y recoveries are high relative to the other internal standards, then do not use the Y internal standard.

10.16.3 For any readings that exceed the linear range for a given element, a dilution is required. After a high reading, the sample following the high one must be examined for possible carryover. A verification may be necessary by rinsing the lines with an acid solution and then rereading the sample.

10.16.4 Indicate dilution factors for samples using df followed by the dilution factor after the sample ID. There should be a space between the sample number and the df.

10.17 Between each sample, flush the nebulizer and solution uptake system with a blank rinse solution for a minimum of 30 seconds or for the required period of time to ensure that analyte memory effects are not occurring. (60 seconds is recommended for normal methods excluding Hg and B. Longer times may be needed when Hg and B are being analyzed.)

10.18 Analyze the continuing calibration verification solution and the continuing calibration blank after every ten samples and at the end of the sample run.

10.18.1 For the CCV, all elements to be reported must be within 10 % of the true value and the replicates that are greater than 5 times the reporting limit should have a relative standard deviation of less than 5 %. If the CCV solution is not within 10 % of the true value, no samples can be reported in the area bracketed by the failing CCV for the failing element.

10.18.2 For the CCB, all elements to be reported must be less than the reporting limit (lower limit of quantitation).

10.18.3 The internal standard levels in the CCV and CCB must also be within 30% of the internal standard level for the initial calibration. If they are outside of these levels, then no samples can be reported in the area bracketed by this QC.

10.19 The CRI (LLCV) must be analyzed at the end of each calibration (analysis) batch. The acceptance criterion for the CRI check is 70 to 130% recovery. If an element does not meet this criterion, then all samples for that element in the concentration range between the CRI and the CCV must be reanalyzed. Samples containing concentrations higher than the CCV may be reported as long as CCV criteria are met.

10.19.1 More frequent CRI (LCCV) checks may be analyzed during the course of the run if system stability at the low end of the calibration is questionable or if the lab wants to ensure that fewer samples will have to be submitted for reanalysis if there is a failed CRI at the end of a run.

10.19.2 It is recommended that the CRI check be run bracketing every 4 to 8 hour period of analysis. It may be run as frequently as every 10 samples if the supervisor or manager deems that this is necessary.

10.20 After the run is completed, convert the data file to a CSV format using the option on the results screen. First save the file on the local drive using the file naming system described below. Update the run in the LIMS and enter the run name into the workgroup using lower case characters. Then copy the data from the local drive to the LIMS drive.

10.20.1 The file should be named as followed- initial instrument indicator (XA), date (MMDD), year, run type (soil, water, or mixed), and sequential run number for that day. For example, the first water run from 06/30/12 would be designated xa063012w1.csv.

10.21 Calculations are done in the LIMS using the calculations shown below.

10.21.1 Calculation for aqueous samples.

$$\text{Original sample concentration of metal } (\mu g/l) = \frac{(\text{conc. in the digestate } (\mu g/l)) \times (\text{final digestate volume (ml)})}{(\text{Initial sample volume (ml)})}$$

10.21.2 Calculation for solid samples.

$$\text{Original sample concentration of metal (mg/kg)} = \frac{(\text{conc. in the digestate } (\mu g/l)) \times (\text{final digestate volume (ml)})}{(\text{Initial sample weight (g)}) \times (\%\text{sol}/100)}$$

10.22 At the end of the analysis day the ICP-MS must be brought down using the following sequence.

- Rinse the tip in a solution of 1 % nitric acid and 0.6 % hydrochloric acid for 10 minutes and in DI water for 20 minutes. (Note: a stronger acid solution may be needed depending on the matrix of the samples that were analyzed.)
- Turn off the plasma using off button.
- Release the tension on the pump tubing.
- Turn off the heat exchanger.
-

11.0 QC REQUIREMENTS

11.1 This section outlines the QA/QC requirements necessary to meet the method 6020.

11.2 Instrument Detection Limits (IDLs). IDLs must be established for all analytes a minimum of once per quarter. They are calculated by taking the average of the standard deviations of three runs on three non-consecutive days from the analysis of a reagent blank solution with seven consecutive measurements per day.

11.3 LLQC (Lower Limit of Quantitation Check Sample) or LOQ Verification sample. A sample must be digested and analyzed initially and on an as needed basis to verify the quantitation limits for the method. Recoveries of this check must be within 70 to 130% of the true value. If recoveries are outside of this level, then the reporting limit must be increased to a level that can be verified.

11.3.1 For DOD work, the LOQ verification must be analyzed quarterly.

11.4 Method Detection Limits (MDLs). MDLs should be established for all analytes, using a solution spiked at approximately 2 to 5 times the estimated detection limit. To determine the MDL values, take seven replicate aliquots of the spiked sample and process through the entire analytical method. The MDL is calculated by multiplying the standard deviation of the replicate analyses by 3.14, which is the student's t value for a 99% confidence level. MDLs should be determined approximately once per year or whenever there is a significant change in the background or instrument response. In general, if the amount spiked for the MDL is greater than 10 times the actual MDL, then the MDL should be redone with a lower spike level.

11.5 Linear Calibration ranges. The upper limit of the linear dynamic range needs to be established for each wavelength used by determining the signal responses from a minimum of three, preferably five, different concentration standards across the linear range. The linear calibration range which may be used for the analysis of samples should be judged by the analyst from the resulting data. The data, calculations and rationale for the choice of range made must be documented and kept on file. A standard at the upper limit must be prepared, analyzed and quantitated against the normal calibration curve. The calculated value should be within 10% (±10%) of the true value. Linear calibration ranges should be determined whenever there is a significant change in instrument response. They must be done at least every six months. For any readings that exceed the linear range for a

given element, a dilution is required. In addition, if there significant interferences generated from elements above the linear range, than these elements must also be diluted so that accurate interfering element corrections can be applied. Normal linear range values by element are shown in Table 2.

11.6 Initial Calibration Verification (ICV) or Quality Control Sample (QCS) and Initial Calibration Blank (ICB). After every new calibration, an ICV must be analyzed. The analysis of the ICV may be followed by the analysis of the ICB, although this is not required by the method.

11.6.1 For the ICV, all elements to be reported must be within 10 % of the true value and the replicates that exceed 5 times the reporting limit should have a relative standard deviation of less than 5 %. The ICV must be from a different source than the calibration standards and must be near the mid-point of the calibration curve. If the ICV does not meet criteria, then the problem must be identified and corrected before samples can be run and reported for the element(s) that are outside of criteria. Correction of the problem can be verified by rerunning the check standard and showing that it meets QC criteria.

11.6.2 If an ICB is analyzed, than all elements to be reported must be less than the RL (LLOQ). If the ICB is outside of criteria, then the problem must be identified and corrected before samples can be run and reported for the element(s) that are outside of criteria. Correction of the problem can be verified by rerunning the check standard and showing that it meets QC criteria. Analysis of a CCB before running any reportable samples can be used to verify that the system meets calibration blank requirements.

11.7 Continuing Calibration Verification (CCV) and Continuing Calibration Blank (CCB). Analyze the continuing calibration verification solution and the continuing calibration blank after every 10 sample and at the end of the sample run.

11.7.1 For the CCV, all elements to be reported must be within 10 % of the true value and the replicates that are greater than 5 times the reporting limit should have a relative standard deviation of less than 5 %. The CCV should be made from the same source as the calibration standards at a concentration near the mid-level of the calibration curve. If an element does not meet the recovery criteria of the CCV, than no samples can be reported for that element in the area bracketed by the CCV.

11.7.2 For the CCB, all elements to be reported must be less than the reporting limit (LLOQ). If an element does not meet this criterion, then no samples can be reported for that element in the area bracketed by the CCB.

11.7.3 The internal standard levels in the CCV and CCB must also be within 30% of the internal standard level for the initial calibration. If they are outside of these levels, then no samples can be reported in the area bracketed by this QC.

11.8 Interference Check Standards. After the initial QC is completed and before any samples are analyzed, the ICSA and ICSAB solutions must be analyzed. The method does not give

specific criteria for the ICSA and ICSAB, but in house criteria should be applied. For all the spiked elements, the analyzed results must be within 20 % of the true results. For unspiked elements, the interfering element solution should contain less than the absolute value of 3 times the reporting limit for each element. If these criteria are not met, then samples with significant interferences can not be reported until the correction factors are optimized and the ICSA and ICSAB are within specifications.

11.8.1 If the run is longer than 12 hours, a second ICSA, ICSAB pair must be analyzed before the next 12 hours is started.

11.8.2 If mass changes are made for the analysis of an element, all QC criteria must be met for the new mass and it must be verified that appropriate correction factors are in place.

11.9 Low Level Calibration Verification (CRI or LLCCV). The CRI standard containing the elements of interest at (or below) the reporting level for each element. The CRI (LLCV) must be analyzed at the beginning and end of each calibration (analysis) batch. The acceptance criterion for the CRI check is 70 to 130% recovery. If an element does not meet this criterion, then all bracketed samples for that element in the concentration range between the CRI and the CCV must be reanalyzed. Samples containing concentrations higher than the CCV may be reported as long as CCV criteria are met.

11.9.1 More frequent CRI (LCCV) checks may be analyzed during the course of the run if system stability at the low end of the calibration is questionable or if the lab wants to ensure that fewer samples will have to be submitted for reanalysis if there is a failed CRI at the end of a run.

11.9.2 It is recommended that the CRI check be run bracketing every 4 to 8 hour period of analysis. It may be run as frequently as every 10 samples if the supervisory staff deems that this is necessary.

11.10 Method Blank. The laboratory must digest and analyze a method blank with each set of samples. A minimum of one method blank is required for every 20 sample batch. If the method blank does not contain target analytes at a level that interferes with the project-specific DQOs, then the method blank is considered acceptable.

11.10.1 The default SOP limit for the method blank is that is must be less than one half of the reporting limit.

11.10.2 In addition, the blank is considered acceptable if it is less than 10% of the regulatory limit, or less than 10% of the lowest sample concentration for each analyte in a given preparation batch, whichever is greater.

11.10.3 If the method blank does not meet criteria, then it can be reanalyzed along with any associated samples. If it is still unacceptable, then all associated samples must be redigested and reanalyzed along with the other appropriate batch QC samples.

11.11 Lab Control Sample or Spike Blank. The laboratory must digest and analyze a laboratory control sample or spike blank with each set of samples. A minimum of one lab control sample or spike blank is required for every 20 sample batch. The laboratory should assess laboratory performance of the lab control and spike blank against recovery limits of 80 to 120 %. In house lab control and spike blank limits may also be generated to support these default limits. If the lab control or spike blank is outside of the control limits for a given element, all samples must be redigested and reanalyzed for that element.

11.11.1 If solid lab controls are used, then the manufacturer's limits should be applied.

11.12 Matrix Spike. The laboratory must add a known amount of each analyte to a minimum of 1 in 20 samples. The matrix spike recovery is calculated as shown below. Recoveries should be assessed against default limits of 75 to 125 %. In house limits may be generated for this method for informational purposes only. If a matrix spike is out of control, then the results should be flagged with the appropriate footnote and it is recommended that a post-digest spike be analyzed for the out of control element(s). If the matrix spike amount is less than one fourth of the sample amount, then the sample cannot be assessed against the control limits and should be footnoted to that effect. Note: Both the matrix spike amount and the sample amount are calculated to the IDL for any given element. Any value less than the IDL is treated as zero.

$$\frac{\text{(Spiked Sample Result - Sample Result)}}{\text{Amount Spiked}} \times 100 = \text{matrix spike recovery}$$

11.12.1 If a post-digest spike is required, the sample should be spiked with approximately 2 times the sample level or two times the reporting limits, whichever is greater. Limits of 80 to 120 % are normally applied. The serial dilution is used to confirm any matrix effects. The post-digest spike recovery must be footnoted on the matrix spike recovery or otherwise noted in the quality control summary report.

11.13 Matrix Spike Duplicate (MSD) or Matrix duplicate (DUP). The laboratory must digest a matrix spike duplicate or matrix duplicate sample for a minimum of 1 in 20 samples. The relative percent difference (rpd) between the MSD and the MS or between the DUP and the sample should be assessed. The rpd is calculated as shown below. The control limit for the duplicate rpd is method defined as 20%. If the sample and the duplicate are less than 5 times the reporting limits and are within a range of $\pm$ the reporting limit, then the duplicate is considered to be in control. Note: Both the duplicate amount and the sample amount are calculated to the IDL for any given element. Any value less than the IDL is treated as zero.

11.13.1 If a MSD or duplicate is out of control, then the data should be checked carefully to confirm that the high rpd for a given element is not a result of an analytical problem. If an analytical problem is suspected, the MSD or duplicate must be reanalyzed for confirmation. If the initial and reanalysis are in agreement (within 20%), then the high rpd is a result of preparation or sample issues and further analysis of the initial preparation is not required. If the initial and reanalysis are not in agreement due to an analytical problem, then any affected samples in the associated batch should also be reanalyzed for that element.

11.13.2 If more than 50% of the elements in a sample (that have levels of at least 5 times the reporting limit) have a high RPD, then the MSD or duplicate should be redigested for confirmation, unless the sample matrix is such that the non-homogeneity of the sample is visually apparent. If the results confirm, the results from the original MSD or duplicate should be flagged as indicative of possible sample non-homogeneity. If the results do not confirm, then the whole batch should be digested and reanalyzed.

11.13.3 If 50% or less of the elements in a sample (that have levels of at least 5 times the reporting limit) have a high rpd, then the high rpd(s) should be footnoted as indicating possible sample non-homogeneity unless other problems are suspected. If problems are suspected, the reviewer will initiate redigestion and reanalysis of the batch.

11.13.4 The calculations used to calculate RPD are shown below.

$$\frac{(|\text{MS Result} - \text{MSD Result}|) \times 100}{(\text{MS Result} + \text{MSD Result})/2} = \text{MSD RPD}$$

$$\frac{(|\text{Sample Result} - \text{Duplicate Result}|) \times 100}{(\text{Sample Result} + \text{Duplicate Result})/2} = \text{Duplicate RPD}$$

11.14 Serial Dilution. A serial dilution is required on a frequency of one in 20 samples. It is normally done on the same sample as is used for the matrix spike. If the analyte concentration is within the linear dynamic range of the instrument and sufficiently high (minimally a factor of at least 100 times greater than the concentration in the reagent blank), then an analysis of a fivefold (1+4) dilution must agree to within ±10% of the original determination. If not, an interference effect must be suspected and the serial dilution result for the element with the suspected interference must be footnoted. The serial dilution is calculated as shown below.

$$\frac{(\text{Sample result} - \text{Serial dilution result}) \times 100}{\text{Sample result}} = \text{Serial dilution percent difference}$$

11.14.1 Results of less than the IDL are treated as 0. The concentration in the reagent blank is normally < 3 times the IDL, so the factor of 100 times the concentration in the reagent blank (listed above) so the limits should be applied to sample concentrations of greater than 300 times the IDL.

11.15 Lower Limit of Quantitation check sample (LLQC). The LLQC is a sample at the reporting limit that is taken through the entire preparation and analytical process. This standard must be analyzed when reporting limits are initial established and on an as needed basis after that. The LLQC is equivalent to the LOQ (Limit of quantitation) standard which must be analyzed quarterly for the DOD QSM program. The limits of quantitation are verified when all analytes in the LLQC sample are detected within 30% of their true value. If the limits cannot

be verified at the spiked level, then the quantitation limit must be adjusted to a level where verification is successful.

13.0 DOCUMENTATION REQUIREMENTS

13.1 If samples or QC checks require reanalysis, a brief explanation of the reason must be documented on the run log. All instrument data should be exported to the LIMS system.

13.2 All standard preparations must be entered and completed in the Reagent Application. All information requested must be completed. All standards must have a lot number that is generated by Reagent Application on the bottle before being used.

13.3 The Instrument Maintenance Logbook must be completed when any type of maintenance is performed on the instrument daily.

13.4 Any corrections to laboratory data must be done using a single line through the error. The initials of the person and date of correction must appear next to the correction.

13.5 Supervisory (or peer) personnel must routinely review (approximately once per month) all laboratory logbooks to ensure that information is being recorded properly. Additionally, the maintenance of the logbooks and the accuracy of the recorded information should also be verified during this review.

14.0 INSTRUMENT MAINTENANCE

14.1 Recommended periodic maintenance includes the items outlined below.

14.1.1 Change the pump tubing weekly or as needed.

14.1.2 Clean the nebulizer, torch, and injector tube every two to four weeks or more often as needed.

14.1.3 Change the sampler tip as needed (every one to two months).

14.1.4 Clean the recirculating pump lines as needed.

15.0 POLLUTION PREVENTION & WASTE MANAGEMENT

15.1 Users of this method must perform all procedural steps in a manner that controls the creation and/or escape of wastes or hazardous materials to the environment. The amounts of standards, reagents, and solvents must be limited to the amounts specified in this SOP. All safety practices designed to limit the escape of vapors, liquids or solids to the environment must be followed. All method users must be familiar with the waste management practices described in section 15.2

15.2 Waste Management. Individuals performing this method must follow established waste management procedures as described in the waste management SOP. This document describes the proper disposal of all waste materials generated during the testing of samples as follows:

15.2.1 Non hazardous aqueous wastes.

15.2.2 Hazardous aqueous wastes

15.2.3 Chlorinated organic solvents

15.2.4 Non-chlorinated organic solvents

15.2.5 Hazardous solid wastes

15.2.6 Non-hazardous solid wastes

16.0 ADDITIONAL REFERENCES

Refer to other SOP's for ICP-MS analysis (EPA 200.8).

TABLE 1: ELEMENTS, MASSES, AND REPORTING LIMIT

Mass and Element	Associated Tune (1 = no gas, 2= helium)	CRI (LOQLL) CRI Check	Normal Digested Aqueous Sample Reporting Limit (Dilution Factor of 2) in ug/l.	Normal Digested Solid Sample Reporting Limit (Dilution Factor of 5) in mg/kg.	Comments
9Be	1	0.5	1	0.25	
11B	1	5	10	2.5	
23Na	1	250	500	125	
24Mg	1	250	500	125	
27Al	1	25	50	12.5	
39K	1	250	500	125	
44Ca	1	250	500	125	
47Ti	1	1	2	0.5	
51V	2	1	2	0.5	
52Cr	2	1	2	0.5	
55Mn	2	0.5	1	0.25	
56Fe	2	25	50	12.5	
59Co	2	0.5	1	0.25	
60Ni	2	1	2	0.5	
63Cu	2	1	2	0.5	
66Zn	2	2	4	1	

75As	2	0.5	1	0.25	
78Se	2	0.5	1	0.25	
88Sr	1	5	10	2.5	
98Mo	1	1	2	0.5	.
107Ag	1	0.5	1	0.25	
111Cd	2	0.5	1	0.25	
120Sn	1	5	10	2.5	
121Sb	1	0.5	1	0.25	123Sb is the method recommended line, but 121Sb is used instead. Xe is a possible interference for 123Sb and is sometimes found as a contaminant in argon.
137Ba	1	1	2	0.5	
182W	1				
205Tl	1				
208Pb	1	0.5	1	0.25	206Pb,207Pb, and 208Pb summed and reported under 208Pb

TABLE 2: RECOMMENDED STANDARDS AND ICV AND CCV LEVELS AND NORMAL LINEAR RANGES

Mass and Element	STDA	STDB	STDC	STDD	STDE	STDF	STDG	STDH	LINEAR RANGE	ICV	CCV
9Be	0	0.5	5	25	50	100	0	0	1000	60	50
11B	0	0.5	5	25	50	100	0	0	1000	60	50
23Na	0	0.5	5	25	50	100	1000	10000	100000	5500	5000
24Mg	0	0.5	5	25	50	100	1000	10000	100000	5500	5000
27Al	0	0.5	5	25	50	100	1000	10000	100000	5500	5000
39K	0	0.5	5	25	50	100	1000	10000	100000	5500	5000
44Ca	0	0.5	5	25	50	100	1000	10000	100000	5500	5000
47Ti	0	0.5	5	25	50	100	0	0	1000	60	50
51V	0	0.5	5	25	50	100	0	0	1000	60	50
52Cr	0	0.5	5	25	50	100	0	0	1000	60	50
55Mn	0	0.5	5	25	50	100	0	0	1000	60	50
56Fe	0	0.5	5	25	50	100	1000	10000	100000	5500	5000
59Co	0	0.5	5	25	50	100	0	0	1000	60	50
60Ni	0	0.5	5	25	50	100	0	0	1000	60	50
63Cu	0	0.5	5	25	50	100	0	0	1000	60	50
66Zn	0	0.5	5	25	50	100	0	0	1000	60	50
75As	0	0.5	5	25	50	100	0	0	1000	60	50
78Se	0	0.5	5	25	50	100	0	0	1000	60	50
88Sr	0	0.5	5	25	50	100	0	0	1000	60	50
95Mo	0	0.5	5	25	50	100	0	0	1000	60	50
107Ag	0	0.5	5	25	50	100	0	0	200	60	50
111Cd	0	0.5	5	25	50	100	0	0	1000	60	50
120Sn	0	0.5	5	25	50	100	0	0	1000	60	50
121Sb	0	0.5	5	25	50	100	0	0	1000	60	50
137Ba	0	0.5	5	25	50	100	0	0	1000	60	50
182W	0	0.5	5	25	50	100	0	0	1000	60	50
205Tl	0	0.5	5	25	50	100	0	0	1000	60	50
208Pb	0	0.5	5	25	50	100	0	0	1000	60	50

TABLE 3: INTERNAL STANDARD MASSES AND ELEMENTS

Mass and Element	Associated Tune for ISTD (1 = no gas, 2= helium)	Comments
6Li	1	
45Sc	1, 2	
72Ge	1,2	
89Y	1, 2	Sometimes found in soil matrices. Monitor recoveries with other internal standards. Optional
103Rh	1	
115In	1, 2	
159Tb	1	
175Lu	1	Optional
209Bi	1	

TABLE 4: MS AND BLANK SPIKE CONCENTRATIONS

Element	Soils Final Concentration in mg/kg	Aqueous Final Concentration in µg/l
Ag	20	200
Al	200	2000
As	50	500
B	100	1000
Ba	200	2000
Be	50	500
Ca	2500	25000
Cd	50	500
Co	50	500
Cr	50	500
Cu	50	500
Fe	200	2000
K	2500	25000
Mg	2500	25000
Mn	50	500
Mo	100	1000
Na	2500	25000
Ni	100	500
Pb	50	1000
Sb	50	500
Se	50	500
Tl	50	500
V	50	500
Zn	50	500
Sn	100	1000
Sr	50	500
Ti	50	500

Lab Manager: Brad Madadian

QA Officer: Robert Treggiari

TITLE: DIGESTION OF NON-POTABLE WATERS FOR FLAME AND ICP ANALYSIS INCLUDING ANTIMONY (Sb).

TEST METHODS REFERENCE: SW846 3010A Rev. 1 July 1992

REVISED SECTIONS: 11.3

1.0 SCOPE & APPLICATION

1.1 This method is applicable for the digestion of aqueous, TCLP extracts, and wastes that contain small amount of suspended solids. After digestion, the samples can be analyzed by ICP. This digestion method is based upon SW846 method.

Note: this method must not be used for drinking water samples. Please refer to the drinking water SOP

1.2 Test Code: METDIG

2.0 SUMMARY

2.1 A representative aliquot of sample (50 ml) is digested with nitric acid and dilute hydrochloric acid until the digestate is light in color or until its color has stabilized. After the digestate has been brought to low volume, it is cooled and diluted to final volume of 50 ml with DI water. Sample is then mixed and filtered if it contains suspended solids.

3.0 METHOD REPORTING AND DETECTION LIMITS

3.1 See the determinative method for detection limits.

4.0 DEFINITIONS

4.1 ALIQUOT - a measured portion of a sample, or solution, taken for sample preparation and/or analysis.

4.2 BATCH – A group of samples which behave similarly with respect to the sampling or the testing procedures being employed and which are processed as a unit. For QC purposes, if the number of samples in a group is greater than 20 then each group of 20 samples or less will all be handled as a separate batch.

4.3 CONTAMINATION - a component of a sample or an extract that is not representative of the environmental source of the sample. Contamination may stem from other samples, sampling

equipment, while in transit, from laboratory reagents, laboratory environment, or analytical instruments.

4.4 DEIONIZED WATER (DI water) - water that has passed through Accutest's deionization system. Used as reagent water (water that an interferant is not observed at or above the minimum quantitation limit of the parameters of interest). Also called reagent water.

4.5 FIELD SAMPLE - a portion of material to be analyzed that is contained in single or multiple containers and identified by a unique sample number.

4.6 INSUFFICIENT QUANTITY - when there is not enough volume to perform any of the required operations: sample digestion or analysis, MS/MSD, etc.

4.7 MATRIX - the predominant material of which the sample to be analyzed is composed. For the purpose of this SOP, a sample matrix is water. Matrix is not synonymous with phase (liquid or solid).

4.8 MATRIX EFFECT - in general, the effect of a particular matrix (water or soil/sediment) on the constituents with which it contacts. This is particularly pronounced for clay particles which may adsorb chemicals and catalyze reactions. Matrix effects may prevent extraction of target analytes. In addition, non-target analytes may be extracted from the matrix causing interferences.

4.9 MATRIX SPIKE - aliquot of a matrix (water or soil) fortified (spiked) with known quantities of specific analytes and subjected to the entire analytical procedure in order to indicate the appropriateness of the method for the matrix by measuring recovery. The matrix spike recovery is calculated as shown below.

$$\frac{(\text{Spiked Sample Result} - \text{Sample Result})}{(\text{Amount Spiked})} \times 100 = \text{Matrix Spike Recovery}$$

4.10 MATRIX SPIKE DUPLICATE - a second aliquot of the original sample that is spiked in order to determine the precision of the method. The matrix spike duplicate RPD is calculated as shown below.

$$\frac{(|\text{MS Result} - \text{MSD Result}|) \times 100}{(\text{MS Result} + \text{MSD Result})/2} = \text{MSD RPD}$$

4.11 METHOD BLANK - an analytical control consisting of all reagents that is carried throughout the entire digestion and analytical procedure. The method blank is used to define the level of laboratory, background, and reagent contamination.

4.12 RELATIVE PERCENT DIFFERENCE (RPD) - as used in this SOP to compare two values, the relative percent difference is based on the mean of the two values, and is reported as an absolute value, i.e., always expressed as a positive number or zero.

4.13 SPIKE BLANK – DI water fortified (spiked) with known quantities of specific analytes and subjected to the entire analytical procedure in order to indicate the accuracy of the analysis.

5.0 HEALTH & SAFETY

5.1 All safety practices must be followed as outlined in the Accutest Laboratories Employee Safety Handbook and Chemical Hygiene Plan. Safety glasses, gloves, and lab coats must be worn. All samples, solutions, and extracts must be treated as unknown and potentially hazardous.

5.2 The toxicity or carcinogenicity of each reagent used in this method has not been precisely determined; however, each chemical should be treated as a potential health hazard. Exposure to these reagents should be reduced to the lowest possible level.

5.3 All acid digestion procedures will take place under a working hood. Verify the hood is working before use. Tape a strip of kim-wipe or plastic to the hood sash for visual verification of hood function.

6.0 PRESERVATION & HOLDING TIME

6.1 Preservation: Samples must be preserved to < 2.0 pH with nitric acid at the time of collection or upon receipt by the laboratory. Sample must be cooled to 4°C ±2°C upon collection.

6.2 Holding Time: Samples must be digested and analyzed within 6 months of the time of collection.

6.3 Samples should be collected in 250-ml plastic bottles.

7.0 APPARATUS & MATERIALS

The apparatus needed for this digestion procedure are listed below. It should be noted that hot plates and beakers with watch glasses may be used in place of the digestion block and digestion tubes.

7.1 Equipment

7.1.1 Digestion block. Designed to hold sample digestion tubes and capable of temperature control. Environmental Express HOT BLOCK or equivalent.

7.2 Materials

7.2.1 Sample digestion tubes. 60 ml polypropylene tubes.
7.2.2 Ribbed watch glass. Polypropylene.
7.2.3 Automatic pipetter bottles.
7.2.4 Polypropylene filter funnels.
7.2.5 Whatman #41 filter paper or equivalent
7.2.6 Filtermate 2u Teflon

8.0 STANDARDS & REAGENTS

All chemicals listed below are trace metal grade unless otherwise specified. Distilled, deionized water should be used whenever water is required.

8.1 Nitric Acid. Baker instra-analyzed or equivalent.

8.2 Hydrochloric Acid. Baker instra-analyzed or equivalent.

8.3 Hydrochloric acid (1 +1). Add 500 ml of concentrated HCl to 400 ml of DI water. Cool and dilute to 1 liter.

8.4 Metals Spiking Solutions. All metals spiking solutions should be made up in a solution of 2 % nitric acid following the procedures outlined in the table 1.

Spiking Solution	Element	Stock Conc. in mg/l	Vol. of Stock in ml	Final Vol. of Spiking Solution in ml	Spiking Solution Conc. in mg/l
Ag Spike Solution	Ag	1000.00	2.00	100.00	20.00
ICP Spike Solution 1	As	1000.00	5.00	100.00	50.00
	Be	1000.00	5.00		50.00
	Cd	1000.00	5.00		50.00
	Co	1000.00	5.00		50.00
	Cr	1000.00	5.00		50.00
	Cu	1000.00	5.00		50.00
	Mn	1000.00	5.00		50.00
	Ni	1000.00	5.00		50.00
	Sb	1000.00	5.00		50.00
	Se	1000.00	5.00		50.00
	Sr	1000.00	5.00		50.00
	Ti	1000.00	5.00		50.00
	Tl	1000.00	5.00		50.00
	V	1000.00	5.00		50.00
	Zn	1000.00	5.00		50.00
ICP Spike Solution 2	Al	10000.00	2.00	100.00	200.00
	Ba	1000.00	20.00		200.00
	Fe	10000.00	2.00		200.00
	Pb	1000.00	10.00		100.00
ICP Mineral Spike	Ca	10000.00	25.00	100.00	2500.00
	Mg	10000.00	25.00		2500.00
	K	10000.00	25.00		2500.00
	Na	10000.00	25.00		2500.00
ICP B Spike	Au	1000.00	10.00	100.00	100.00
ICP Au Spike	B	1000.00	10.00	100.00	100.00
ICP Mo Spike	Mo	1000.00	5.00	100.00	50.00
ICP Pd Spike	Pd	1000.00	10.00	100.00	100.00
ICP Pt Spike	Pt	1000.00	10.00	100.00	100.00
ICP Si Spike	Si	1000.00	10.00	100.00	100.00

ICP Sn Spike	Sn	1000.00	10.00	100.00	100.00
ICP W Spike	W	1000.00	10.00	100.00	100.00
ICP Zr Spike	Zr	1000.00	5.00	100.00	50.00
ICP Li Spike	Li	10000.00	10000.00	100.00	2500.00

Table 1

9.0 INTERFERENCES

9.1 Aqueous samples can contain diverse matrix types which may contain a variety of interferences. Spiked samples can be used to determine if these interferences are adequately treated in the digestion process. For a discussion of other interferences, refer to specific analytical methods.

10.0 PROCEDURE

10.1 Measure out 50 ml of each sample into a labeled digestion tube. Make sure that the sample has been thoroughly mixed. Make sure that the sample identification is accurately recorded with the digestion tube numbers on the sample digestion log. In addition to the samples, a Matrix Spike, a Matrix Spike Duplicate, a Spike Blank and a Method Blank should be set up with each batch of 20 samples. Add 0.50 ml of the spiking solutions to the Matrix Spike, Matrix Spike Duplicate and Spike Blank. Check with the metals supervisor for the spiking solutions (refer to Table 1) to use for each batch.

10.2 Add 1.5 ml of concentrated nitric acid to all quality control and samples.

10.3 Place the numbered tubes into a digestion block. Heat the block until the samples are at a gentle reflux (90-95°C). Record the temperature.

10.4 Reduce the volume of each sample to approximately 5 to 10 mls.

10.5 Add an additional 1.5 ml of concentrated nitric acid to all quality control and samples. Cover with watch glasses. Heat the samples at a gentle reflux until the volume is again at approximately 5 to 10 mls.

Note: This step may be omitted if the sample appears completely digested at the end of step 10.4. Signs of a complete digestion are if the digestate is light in color and/or the appearance does not change with continued refluxing.

10.6 Add 5 ml of 1+1 HCl to each sample and reflux for additional 15 minutes. Cool.

10.7 Filter the samples through Whatman #41 filter paper or by using Filtermate 2u Teflon (if needed), dilute to final volume of 50 ml with distilled, deionized water and mix. The sample is now ready for analysis by ICP or FLAME AAS.

10.8 Glassware cleaning

10.8.1 All glassware should be washed with soap and tap water and then soaked in 5 % nitric acid. It should then be rinsed at least 3 times with distilled, deionized water. Store upside down or in sealed bins to prevent accumulation of dust.

11.0 QUALITY ASSURANCE

11.1 Below is a summary of the quality control requirements, performance criteria and general corrective action guidelines for this method .Make sure to check with the laboratory supervisor for any additional client specific quality control requirements.

11.2 **Method Blank.** The laboratory must digest and analyze a method blank with each set of samples. A minimum of one method blank is required for every 20 samples. For a running batch, a new method blank is required for each different day. The method blank must contain the analyte at a level less than the reporting limit(less than ½ the reporting limit for certain clients). If the method blank contains over that limit, the samples must be reanalyzed.

11.3 **Spike Blank.** The laboratory must analyze a spike blank with each set of samples. A minimum of one spike blank is required for every 20 samples. For a running batch, a new spike blank is required for each different analysis day. Until sufficient lab control data becomes available (usually a minimum of 20-30 analyses) the laboratory should assess the laboratory performance of the spike blank against recovery limits of 80-120 %. If the lab control recovery is high and the results of the samples to be reported are less than the reporting limit, then the sample results can be reported with a sample case narrative. If the samples are above the reporting limit or if the lab control recovery is low, report to the laboratory supervisor. In most cases the lab control and the samples must be re-prepped and reanalyzed.

11.4 **Matrix Spike.** The laboratory must add a known amount of each analyte to a minimum of 1 in 20 samples. The spike recovery should be assessed using in house limits. Until these limits can be generated, default limits of 75-125 % recovery should be applied. If a matrix spike is out of control, then the results should be flagged with the appropriate footnote. If the matrix spike amount is less than one fourth of the sample amount, then the sample can not be assessed against the control limits and should be footnoted to that effect.

11.5 **Matrix Spike Duplicate.** The laboratory must analyze a Matrix Spike Duplicate for a minimum of 1 in 20 samples. This second aliquot of the original sample that is spiked in order to determine the precision of the method. The relative percent difference (RPD) between the Matrix Spike duplicate and the Matrix Spike should be assessed using in house limits. Until these limits can be generated, then default limits of ±20% RPD should be applied.

12.0 DOCUMENTATION

12.1 Record all digestion information in Metal Digestion automated logbook. The information required includes the digestion tube number, the sample identification, the initial sample volume, the final sample volume, the acids used, the spikes used, and the temperature. The analyst should write additional information such as unusual sample characteristics in the comments section. All spiking solution information should be entered in the metals reagents and standards logbook.

12.2 The standard preparation logbook must be completed for all standard preparations. All information requested must be completed; the page must be signed and dated by the respective person.

12.3 The Accutest lot number must be cross-referenced on the standard vial/container. The expiration date must be noted on the standard vial/container

12.4 All laboratory logbooks must be routinely reviewed and initialed or signed by the lab manager.

12.5 Any corrections to laboratory data must be done using a single line through the error. The initials of the person and date of correction must appear next to the correction.

13.0 DATA REVIEW & REPORTING

13.1 See the determinative method SOP for data review and reporting. The Laboratory Manager and Quality Assurance Officer should review the digestion logbook and reagents and standards logbook on a periodic basis.

14.0 POLLUTION PREVENTION & WASTE MANAGEMENT

14.1 Pollution Prevention. Users of this method must perform all procedural steps that control the creation and/or escape of wastes of hazardous materials to the environment. The amounts of standards, reagents, and solvents must be limited to the amounts specified in this SOP. All safety practices designed to limit the escape of vapors, liquids, or solids to the environment must be followed. All method users must be familiar with the waste management practices described in section 14.2

14.2 Waste Management. Individuals performing this method must follow established waste management procedures as described in the Sample and Waste Disposal SOP. This document describes the proper disposal of all waste materials generated during the testing of samples as follows:

14.2.1 Non-hazardous aqueous wastes
14.2.2 Hazardous aqueous wastes
14.2.3 Chlorinated organic solvents
14.2.4 Non-chlorinated organic solvents
14.2.5 Hazardous solid wastes
14.2.6 Non-hazardous solid wastes

15.0 ADDITIONAL REFERENCES

15.1 No additional references are required for this method.

Lab Manager: Brad Madadian

QA Officer: Robert Treggiari

TITLE: DIGESTION OF SOILS FOR FLAME AND ICP ANALYSIS

TEST METHOD: SW846 3050B Rev. 2 December, 1996

REVISED SECTIONS: 10.4; 11.4

1.0 SCOPE & APPLICATION

1.1 This method is applicable for the digestion of sediments, soils, sludge, and solids wastes. After digestion, the samples can be analyzed by ICP or by flame AAS. This digestion method is based upon SW846 method 3050B.

1.2 Test Code: METDIG

1.3 See the determinative method for reporting limits.

2.0 SUMMARY

2.1 A representative sample (1 –2g) is digested in nitric acid and hydrogen peroxide. The digestate is then refluxed with diluted hydrochloric acid. All samples are diluted to 100 ml with DI water. Samples are mixed and filtered for analysis.

3.0 METHOD DETECTION LIMIT

3.1 The reporting limit (RL) is based on the lowest calibration standard. RL's may vary depending on matrix difficulties, sample volumes or weights, and percent moisture. Detected concentrations below this concentration cannot be reported without qualification.

3.2 The Method Detection Limit (MDL) represents the lowest reportable concentration of an individual analyte that meets the method qualitative identification criteria.

3.3 Method Detection limits (MDLs) are experimentally determined using the procedures described in 40 CFR, Part 136, Appendix B. Actual reported MDLs incorporate the sample volume analyzed and sample dilutions if needed, which may cause MDL variations from sample to sample.

3.4 In general, MDLs are determined through the analysis of at least 7 replicate blank spikes (using the same procedures for sample analysis). The MDL is calculated by multiplying the standard deviation of the replicate concentrations by the appropriate Student's t value (3.143 for 7 replicates).

If more than 7 replicates are analyzed refer to 40 CFR, Part 136, Appendix B for the appropriate student's t value. MDLs are determined initially (prior to analysis), on an annual basis, and after major maintenance to equipment. MDL data is archived with Quality Assurance. Refer to the most recent study for current MDLs.

4.0 DEFINITIONS

4.1 ALIQUOT - a measured portion of a sample, or solution, taken for sample preparation and/or analysis.

4.2 BATCH – A group of samples which behave similarly with respect to the sampling or the testing procedures being employed and which are processed as a unit. For QC purposes, if the number of samples in a group is greater than 20 (or 10 for certain methods), then each group of 20 samples (or 10 samples for certain methods) or less will all be handled as a separate batch.

4.3 CONTAMINATION - a component of a sample or an extract that is not representative of the environmental source of the sample. Contamination may stem from other samples, sampling equipment, while in transit, from laboratory reagents, laboratory environment, or analytical instruments.

4.4 FIELD SAMPLE - a portion of material to be analyzed that is contained in single or multiple containers and identified by a unique sample number.

4.5 INSUFFICIENT QUANTITY - when there is not enough volume to perform any of the required operations: sample digestion or analysis, MS/MSD, etc.

4.6 MATRIX - the predominant material of which the sample to be analyzed is composed. For the purpose of this SOP, a sample matrix is soil or sediment. Matrix is <u>not</u> synonymous with phase (liquid or solid).

4.7 MATRIX EFFECT - in general, the effect of a particular matrix (water or soil/sediment) on the constituents with which it contacts. This is particularly pronounced for clay particles which may adsorb chemicals and catalyze reactions. Matrix effects may prevent extraction of target analytes. In addition, non-target analytes may be extracted from the matrix causing interferences.

4.8 MATRIX SPIKE - aliquot of a matrix (water or soil) fortified (spiked) with known quantities of specific analytes and subjected to the entire analytical procedure in order to indicate the appropriateness of the method for the matrix by measuring recovery. The matrix spike recovery is calculated as shown below.

$$\frac{(\text{Spiked Sample Result} - \text{Sample Result})}{(\text{Amount Spiked})} \times 100 = \text{Matrix Spike Recovery}$$

4.9 MATRIX SPIKE DUPLICATE - a second aliquot of the original sample that is spiked in order to determine the precision of the method. The matrix spike duplicate RPD is calculated as shown

below.

$$\frac{(\,|\text{MS Result} - \text{MSD Result}|\,) \times 100}{(\,\text{MS Result} + \text{MSD Result}\,)/2} = \text{MSD RPD}$$

4.10 METHOD BLANK - an analytical control consisting of all reagents that is carried throughout the entire digestion and analytical procedure. The method blank is used to define the level of laboratory, background, and reagent contamination.

4.11 PERCENT MOISTURE - an approximation of the amount of water in a soil/sediment sample made by drying an aliquot of the sample at 105 □C. The percent moisture determined in this manner also includes contributions from all compounds that may volatilize at or below 105 □C, including water. Percent moisture may be determined from decanted samples and from samples that are not decanted.

4.12 RELATIVE PERCENT DIFFERENCE (RPD) - As used in this SOP to compare two values, the relative percent difference is based on the mean of the two values, and is reported as an absolute value, i.e., always expressed as a positive number or zero.

4.13 SPIKE BLANK – DI water fortified (spiked) with known quantities of specific analytes and subjected to the entire analytical procedure in order to indicate the accuracy of the analysis.

4.14 LABORATORY CONTROL SAMPLE (LCS)- A solid laboratory control sample obtained from ERA,or Absolute, or any other vendor, must be digested and analyzed. The results must be within the limits provided by the vendor.

5.0 HEALTH & SAFETY

5.1 The analyst should follow normal safety procedures as outlined in the Accutest Laboratory Chemical Hygiene Plan which includes the use of safety glasses, gloves and lab coats. In addition, all acids are corrosive and should be handled with care. Flush spills with plenty of water. If acids contact any part of the body, flush with water and contact the supervisor.

5.2 All acid digestion procedures will take place under a working hood. Verify the hood is working before use. Tape a strip of kim-wipe to the hood sash for visual verification of hood function.

6.0 PRESERVATION & HOLDING TIME

6.1 Preservation: Collect sample in a 4 or 8 oz. glass sample jar. Samples must be cooled to 4°C ±2°C upon collection.

6.2 Holding Time: Samples must be digested and analyzed within 6 months of the time of collection.

7.0 APPARATUS & MATERIALS

The apparatus needed for this digestion procedure are listed below. It should be noted that hot plates and beakers with watch glasses may be used in place of the digestion block and digestion tubes.

7.1 Equipment

7.1.1 Digestion block. Designed to hold sample digestion tubes and capable of temperature control. Environmental Express HOT BLOCK or equivalent.

7.1.2 Top loader balance. Capable of accurate weighing to 0.01g.

7.2 Materials.

7.2.1 Sample digestion tubes. 120 ml polypropylene tubes.
7.2.2 Ribbed watch glass. Polypropylene.
7.2.3 Automatic piptter bottles.
7.2.4 100 ml volumetric flasks
7.2.5 Polypropylene filter funnels.
7.2.6 Whatman #41 filter paper or equivalent
7.2.7 FilterMate 2u Teflon
7.2.8 Glass Beads or Teflon Chips

8.0 STANDARDS & REAGENTS

All applicable standard/reagent preparation information, including vendor, lot number, date of preparation, calculations, and initials must be entered in the appropriate standard/reagent preparation logbook. Vendors typically used by Accutest include Fisher Scientific, VWR, Accustandard, Absolute Standards, Supelco, Chemservices, Ultra, and ERA. Additional vendors may be utilized as necessary. All chemicals listed below are trace metal grade unless otherwise specified. Distilled, deionized water should be used whenever water is required.

8.1 Nitric Acid. (1+1). Add 500 ml of concentrated HNO3 (Baker Instra-analyzed trace metals or equivalent to 500 ml of DI water. Mix. Cool.

8.2 Hydrochloric acid (1+1). Add 500 ml of concentrated HCl (Baker Instra-analyzed trace metals or equivalent) to 500 ml of DI water. Mix. Cool.

8.3 Hydrogen peroxide (30%).

8.4 Metals Spiking Solutions. All metals spiking solutions should be made up in a solution of 2 % nitric acid following the procedures outlined in the table 1.

Table 1

Spiking Solution	Element	Stock Conc. in mg/l	Vol. of Stock in ml	Final Vol. of Spiking Solution in ml	Spiking Solution Conc. in mg/l
Ag Spike Solution	Ag	1000.00	2.00	100.00	20.00
ICP Spike Solution 1	As	1000.00	5.00	100.00	50.00
	Be	1000.00	5.00		50.00
	Cd	1000.00	5.00		50.00
	Co	1000.00	5.00		50.00
	Cr	1000.00	5.00		50.00
	Cu	1000.00	5.00		50.00
	Mn	1000.00	5.00		50.00
	Ni	1000.00	5.00		50.00
	Sb	1000.00	5.00		50.00
	Se	1000.00	5.00		50.00
	Sr	1000.00	5.00		50.00
	Ti	1000.00	5.00		50.00
	Tl	1000.00	5.00		50.00
	V	1000.00	5.00		50.00
	Zn	1000.00	5.00		50.00
ICP Spike Solution 2	Al	10000.00	2.00	100.00	200.00
	Ba	1000.00	20.00		200.00
	Fe	10000.00	2.00		200.00
	Pb	1000.00	10.00		100.00
ICP Mineral Spike	Ca	10000.00	25.00	100.00	2500.00
	Mg	10000.00	25.00		2500.00
	K	10000.00	25.00		2500.00
	Na	10000.00	25.00		2500.00
ICP B Spike	Au	1000.00	10.00	100.00	100.00
ICP Au Spike	B	1000.00	10.00	100.00	100.00
ICP Mo Spike	Mo	1000.00	5.00	100.00	50.00
ICP Pd Spike	Pd	1000.00	10.00	100.00	100.00
ICP Pt Spike	Pt	1000.00	10.00	100.00	100.00
ICP Si Spike	Si	1000.00	10.00	100.00	100.00
ICP Sn Spike	Sn	1000.00	10.00	100.00	100.00
ICP W Spike	W	1000.00	10.00	100.00	100.00
ICP Zr Spike	Zr	1000.00	5.00	100.00	50.00
ICP Li Spike	Li	10000.00	10000.00	100.00	2500.00

9.0 INTERFERENCES

9.1 Soil samples can contain diverse matrix types which may contain a variety of interferences. Spiked samples can be used to determine if these interferences are adequately treated in the digestion process. For a discussion of other interferences, refer to specific analytical methods.

10.0 PROCEDURE

10.1 Weigh out a 1.00 g portion of sample to the nearest 0.01 g in a labeled digestion tube. The sample should be weighed out using a top loader balance. For samples with low percent solids, a larger sample size may be weighed. Make sure that the sample has been thoroughly stirred before weighing out the representative sample (refer to the Representative Aliquot SOP – MQA247-03). Discard rocks, sticks, etc. from the sample. Make sure that the sample identification is accurately recorded with the digestion tube numbers on the sample digestion log. In addition to the samples, a Matrix Spike, a Matrix Spike Duplicate, Spike Blank, LCS and a Method Blank should be set up with each batch of 20 samples. For LCS weigh about 1.0 gram of solid standard, and for Method Blank, and Spike Blank weigh about 1.0 gram of Teflon chips or glass beads. Add 1.0 ml of the spiking solutions to the Matrix Spike, Matrix Spike Duplicate and Spike Blank. Check with the metals supervisor for the spiking solutions (refer to Table 1) to use for each batch.

10.2 Add 10 ml of 1:1 nitric acid to all quality control and samples.

10.3 Place the numbered tubes into a digestion block. Heat the block until the samples are at a gentle reflux (90-95°C). Record the temperature in the digestion logbook. Heat the samples for 10 to 15 minutes. Allow the samples to cool.

10.4 Add an additional 5 ml of concentrated nitric acid to all quality control and samples. Heat the samples at a gentle reflux for an additional 2 hours. **Cool.** If brown fumes are generated, indicating oxidation of the sample by HNO_3 repeat step 10.4 until sample no longer fumes.

Note #2: make sure samples are cooled

10.5 Add 2 ml of water and 3 ml of 30 % hydrogen peroxide to each sample and reflux until effervescence subsides. **Cool**. Continue to add 30 % hydrogen peroxide in 1 ml aliquots with warming until the effervescence is minimal or until the general sample appearance is unchanged.

Note #3: Do not add more than a total of 10 ml of 30 % hydrogen peroxide.

Note #4: make sure samples are cooled

10.6 Add 10 ml of (1+1) HCl and reflux at 90 - 95°C for an additional 15 minutes.

10.7 Filter the sample through Whatman #41 filter paper into 100 ml volumetric flasks. Make sure to rinse the digestion tubes and the filter paper well with distilled, deionized water. Dilute to volume with distilled, deionized water and mix. The sample is now ready for analysis by ICP or flame AAS.

11.0 QUALITY ASSURANCE

11.1 Below is a summary of the quality control requirements, performance criteria and general corrective action guidelines for this method. Make sure to check with the laboratory supervisor for any additional client specific quality control requirements.

11.2 **Laboratory Control Sample (LCS)**. Upon request or as necessary, a solid standard from a different source must be analyzed. The true values and acceptance limits for LCS is provided by the vendor. The laboratory control sample could be substituted for the spike blank. See spike blank (11.4) for corrective actions associated with LCS.

11.3 **Method Blank.** The laboratory must digest and analyze a method blank with each set of samples. A minimum of one method blank is required for every 20 samples. The method blank must contain the analyte at a level less than the reporting limit. If the method blank contains over that limit, the samples must be reanalyzed.

11.4 **Spike Blank.** The laboratory must analyze a spike blank with each set of samples. A minimum of one spike blank is required for every 20 samples. For a running batch, a new spike blank is required for each different analysis day. Until sufficient lab control data becomes available (usually a minimum of 20-30 analyses) the laboratory should assess the laboratory performance of the spike blank against recovery limits of 80-120 %. If the lab control recovery is high and the results of the samples to be reported are less than the reporting limit, then the sample results can be reported with a sample case narrative. If the samples are above the reporting limit or if the lab control recovery is low, report to the laboratory supervisor. In most cases the lab control and the samples must be re-prepped and reanalyzed.

11.5 **Matrix Spike.** The laboratory must add a known amount of each analyte to a minimum of 1 in 20 samples. The spike recovery should be assessed using in house limits. Until these limits can be generated, default limits of 75-125 % recovery should be applied. If a matrix spike is out of control, then the results should be flagged with the appropriate footnote. If the matrix spike amount is less than one fourth of the sample amount, then the sample can not be assessed against the control limits and should be footnoted to that effect.

11.6 **Matrix Spike Duplicate.** The laboratory must analyze a Matrix Spike Duplicate for a minimum of 1 in 20 samples. This second aliquot of the original sample that is spiked in order to determine the precision of the method. The relative percent difference (RPD) between the Matrix Spike duplicate and the Matrix Spike should be assessed using in house limits. Until these limits can be generated, then default limits of $\pm$20% RPD should be applied.

12.0 DOCUMENTATION

12.1 Record all digestion information in Metal Digestion automated logbook. The information required includes the digestion tube number, the sample identification, the initial sample weight, the dry sample weight, the final sample volume, the acids used, the spikes used, and the temperature. The analyst

should write additional information such as unusual sample characteristics in the comments section. All spiking solution information should be entered in the metals reagents and standards logbook.

12.2 All laboratory logbooks must be routinely reviewed and initialed or signed by the lab manager.

12.3 Any corrections to laboratory data must be done using a single line through the error. The initials of the person and date of correction must appear next to the correction.

13.0 DATA REVIEW & REPORTING

13.1 See the determinative method SOP for data review and reporting. The Laboratory Manager and Quality Assurance Officer should review the digestion logbook and reagents and standards logbook on a periodic basis.

14.0 POLLUTION PREVENTION & WASTE MANAGEMENT

14.1 Pollution Prevention. Users of this method must perform all procedural steps that control the creation and/or escape of wastes of hazardous materials to the environment. The amounts of standards, reagents, and solvents must be limited to the amounts specified in this SOP. All safety practices designed to limit the escape of vapors, liquids, or solids to the environment must be followed. All method users must be familiar with the waste management practices described in section 14.2

14.2 Waste Management. Individuals performing this method must follow established waste management procedures as described in the Sample and Waste Disposal SOP. This document describes the proper disposal of all waste materials generated during the testing of samples as follows:

14.2.1 Non-hazardous aqueous wastes
14.2.2 Hazardous aqueous wastes
14.2.3 Chlorinated organic solvents
14.2.4 Non-chlorinated organic solvents
14.2.5 Hazardous solid wastes
14.2.6 Non-hazardous solid wastes
14.2.7 Microbiological wastes

15.0 METHOD PERFORMANCE

15.1 Refer to the determinative method.

16.0 ADDITIONAL REFERENCES

16.1 None

Lab Manager: Doug Yargeau

QA Officer: Robert Treggiari

TITLE: DETERMINATION OF VOLATILE ORGANICS USING GC/MS SYSTEM

TEST METHOD REFERENCE: SW846 8260C, Rev. 3, August 2006

REVISED SECTIONS: 10.2.3

1.0 SCOPE & APPLICATION

1.1 The following method describes the analytical procedures which are utilized by Accutest to aquire samples for the analysis of volatile organic compounds. Refer to Tables 8, 9 or use the Compound List function in LIMS to view the compounds analyzed by this method. Additional compounds may be appended to the SOP.

1.2 This analytical method is designed for nearly all types of samples, regardless of water content, including groundwater, aqueous sludges, oily waste, sediments, and soils.

1.3 The purgeable organics can be quantitated by Gas Chromatograph/Mass Spectrometer (GC/MS) following purge and trap utilizing the internal standard technique.

1.4 The Reporting (RL) is based on the lowest calibration standard. RL'S may vary depending on matrix difficulties and sample volumes or weight and percent moisture. Additionally, RL's will vary between some compounds.

2.0 SUMMARY

2.1 This method is performed in accordance with EPA methodologies 8260C and 5030B (purge and trap), from SW-846, 3rd edition.

2.2 An inert gas is bubbled through a 5 ml sample contained in a specifically designed purging chamber at ambient temperature. The purgeables are efficiently transferred from the aqueous phase to the vapor phase. The vapor is swept through a sorbent column where the purgeables are trapped. After purging is completed, the sorbent column is heated and back flushed with the inert gas to desorb the purgeables onto a gas chromatographic (GC) column.

2.3 The volatile compounds are separated by the temperature programmed GC column and detected using a mass spectrometer, which is used to provide both qualitative and quantitative information.

2.4 The peaks detected are qualitated by comparison to characteristic ions and retention times specific to the known target list of compounds.

2.5 Once identified the compound is quantitated by internal standard technique with an average response factor generated from a calibration curve containing a minimum of five points. . Additional points may be added to meet client requirements.

2.6 Additional unknown peaks with a response > 10 % of the closest internal standard may be processed through a library search with comparison to a data base of approximately 70,000 spectra. An estimated concentration is quantitated by assuming a response factor of 1.

3.0 METHOD DETECTION LIMIT

3.1 The reporting limit (RL) is based on the lowest calibration standard. RL's may vary depending on matrix difficulties, sample volumes or weights, and percent moisture. Detected concentrations below this concentration cannot be reported without qualification.

3.2 The Method Detection Limit (MDL) represents the lowest reportable concentration of an individual analyte that meets the method qualitative identification criteria.

3.3 Method Detection limits (MDLs) are experimentally determined using the procedures described in 40 CFR, Part 136, Appendix B. Actual reported MDLs incorporate the sample volume analyzed and sample dilutions if needed, which may cause MDL variations from sample to sample.

3.4 In general, MDLs are determined through the analysis of at least 7 replicate blank spikes (using the same procedures for sample analysis). The MDL is calculated by multiplying the standard deviation of the replicate concentrations by the appropriate Student's t value (3.143 for 7 replicates). If more than 7 replicates are analyzed refer to 40 CFR, Part 136, Appendix B for the appropriate student's t value. MDLs are determined initially (prior to analysis), on an annual basis, and after major maintenance to equipment. MDL data is archived with Quality Assurance. Refer to the most recent study for current MDLs. Refer to the MDL SOP (MQA245) for additional detail of procedures.

3.5 Current MDLs are entered into the LIMS, and can be viewed by printing out the compound list from the LIMS. Additionally, MDLs are reported on the result page upon client request. Current MDL studies are filed with Quality Assurance. Obsolete MDL studies are archived with the QA files. Electronic MDL data is found in the annual "MDL" folder on the QA server (LINUXMA1).

4.0 DEFINITIONS

4.1 ALIQUOT - a measured portion of a sample, or solution, taken for sample preparation and/or analysis.

4.2 BATCH – A group of samples which behave similarly with respect to the sampling or the testing procedures being employed and which are processed as a unit. For QC purposes, if the number of samples in a group is greater than 20 (or 10 for certain methods), then each group of 20 samples (or 10 samples for certain methods) or less will all be handled as a separate batch.

4.3 CALIBRATION – the establishment of an analytical curve based on the absorbance, emission intensity, or other measured characteristic of known standards. The calibration standards must be prepared using the same type of acid or concentration of acids as used in the sample preparation.

4.4 CALIBRATION STANDARDS – a series of known solutions used by the analyst for calibration of the instrument (i.e., preparation of the analytical curve).

4.5 CONTINUING CALIBRATION – analytical standard run every 12 hours to verify the initial calibration of the analytical system.

4.6 DRY WEIGHT – the weight of a sample based on percent solids. The weight after drying in an oven.

4.7 CONTAMINATION - a component of a sample or an extract that is not representative of the environmental source of the sample. Contamination may stem from other samples, sampling equipment, while in transit, from laboratory reagents, laboratory environment, or analytical instruments.

4.8 DIFFICULT COMPOUNDS – Historically poor-performing compounds (and defined by the MA DEP MCP CAM): Acetone, MEK, MIBK, 2-Hexanone, Dichlorodifluoromethane, Bromomethane, Chloromethane, and 1,4-Dioxane.

4.9 FIELD SAMPLE - a portion of material to be analyzed that is contained in single or multiple containers and identified by a unique sample number.

4.10 FIELD BLANK – this is any sample that is submitted from the field and is identified as a blank. This includes trip blanks, rinsates, equipment blanks, etc.

4.11 HOLDING TIME – the elapsed time expressed in days from the date of sampling until the date of its analysis.

4.12 INTERFERENTS – substances which affect the analysis for the analyte of interest.

4.13 GAS CHROMATOGRAPH (GC) - the instrument used to separate analytes on a stationary phase within a chromatographic column. The analytes are volatized directly from the sample (VOA water and low-soil) volatized from the sample extract (VOA medium soil), or injected as extracts (SVOA and PEST). In VOA and SVOA analysis, the compounds are detected by a Mass Spectrometer (MS). In PEST analysis, the compounds are detected by an Electron Capture (EC) detector. In the screening procedure (all fractions), the Flame Ionization Detector (FID) is used as the detector.

4.14 INITIAL CALIBRATION - analysis of analytical standards for a series of different specified concentrations; used to define the linearity and dynamic range of the response of the mass spectrometer or electron capture detector to the target compounds.

4.15 INITIAL CALIBRATION VERIFICATION – analysis of a check standard from a second source (either vendor or lot) from the initial calibration standards to verify the initial calibration.

4.16 INTEGRATION TIME RANGE - the retention time at the beginning of the area of integration to the retention time at the end of the area of integration.

4.17 INSUFFICIENT QUANTITY - when there is not enough volume (water sample) or weight (soil/sediment) to perform any of the required operations: sample analysis or extraction, percent moisture, MS/MSD, etc.

4.18 MATRIX - the predominant material of which the sample to be analyzed is composed. For the purpose of this SOP, a sample matrix is either water or soil/sediment. Matrix is not synonymous with phase (liquid or solid).

4.19 MATRIX EFFECT - in general, the effect of a particular matrix (water or soil/sediment) on the constituents with which it contacts. This is particularly pronounced for clay particles which may adsorb chemicals and catalyze reactions. Matrix effects may prevent extraction of target analytes, and may affect surrogate recoveries. In addition, non-target analytes may be extracted from the matrix causing interferences.

4.20 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.

4.21 MATRIX SPIKE DUPLICATE - a second aliquot of the same matrix as the matrix spike (above) that is spiked in order to determine the precision of the method.

4.22 MASSACHUSETTS CONTINGENCY PLAN (MCP) – A Massachusetts Department of Environmental Protection (MA DEP) program which deals with environmental cleanups within the state. For the purposes of this SOP MCP specifically refers to data quality and reporting guidelines established under the MCP Data Enhancement Program.

4.23 METHOD BLANK - an analytical control consisting of all reagents, internal standards, and surrogate standards (or SMCs for VOA), that is carried throughout the entire analytical procedure. The method blank is used to define the level of laboratory, background, and reagent contamination.

4.24 METHANOL PRESERVATION DILUTION EFFECT – target analyte concentrations in solid samples preserved with methanol are subject to a systematic negative bias if the potential increase of the total solvent volume during the methanol extraction process is not considered. This increase in extraction solvent volume is a direct result of the solubility of the entrained sample moisture (water) in the methanol. The total solvent volume is the additive sum of the volume of methanol and the entrained sample moisture that partitions into the methanol during extraction. The volume of water partitioned is estimated from the percent moisture determination (and the assumption that 1 g of water occupies a volume of 1 ml). This is a conservative correction regarding calculated VOC concentrations because some fraction of the samples percent moisture may not partition into the methanol, due to various physiochemical binding forces. The total solvent/water volume (V_m) is calculation using the equation described in section 10.4.7. The potential for under reporting VOC concentrations is more pronounced the greater the percent moisture content of the sample.

4.25 PERCENT DIFFERENCE (%D) - As used in this SOW and elsewhere to compare two values, the percent difference indicates both the direction and the magnitude of the comparison, i.e., the percent difference may be either negative, positive, or zero. (In contrast, see relative percent difference.)

4.26 PERCENT MOISTURE - an approximation of the amount of water in a soil/sediment sample made by drying an aliquot of the sample at 105 °C. The percent moisture determined in this manner also includes contributions from all compounds that may volatilize at or below 105 °C, including water.

Percent moisture may be determined from decanted samples and from samples that are not decanted.

4.27 PURGE AND TRAP (DEVICE) - analytical technique (device) used to isolate volatile (purgeable) organics by stripping the compounds from water or soil by a stream of inert gas, trapping the compounds on an adsorbent such as a porous polymer trap, and thermally desorbing the trapped compounds onto the gas chromatographic column.

4.28 PURGEABLES - volatile compounds.

4.29 REAGENT WATER - water in which an interferant is not observed at or above the minimum quantitation limit of the parameters of interest. Accutest uses deionized water (municipal water which passes through Accutest's DI treatment system).

4.30 RELATIVE PERCENT DIFFERENCE (RPD) - As used in this SOP to compare two values, the relative percent difference is based on the mean of the two values, and is reported as an absolute value, i.e., always expressed as a positive number or zero. In contrast, see percent difference.

4.31 RELATIVE RESPONSE FACTOR (RRF) - a measure of the relative mass spectral response of an analyte compared to its internal standard. Relative Response Factors are determined by analysis of standards and are used in the calculation of concentrations of analytes in samples. RRF is determined by the following equation:

$$RRF = \frac{A_x}{A_{is}} x \frac{C_{is}}{C_x}$$

Where,

A = area of the characteristic ion measured
C = concentration, or amount (mass)
is = internal standard
x = analyte of interest

4.32 RELATIVE RETENTION TIME (RRT) - the ratio of the retention time of a compound to that of a standard (such as an internal standard).

$$RRT = \frac{RT_c}{RT_{is}}$$

Where,

RT_c = Retention time for the volatile target or surrogate compound in continuing calibration.
RT_{is} = Retention time for the internal standard in calibration standard or in a sample.

4.33 RESPONSE - or Instrumental Response: a measurement of the output of the GC detector (MS, EC, or FID) in which the intensity of the signal is proportionate to the amount (or concentration) detected. Measured by peak area or peak height.

4.34 SOIL - used herein synonymously with soil/sediment and sediment.

4.35 SURROGATES (Surrogate Standard) – for volatiles, semivolatiles and pesticides/Aroclors, compounds added to every blank, sample, matrix spike, matrix spike duplicate, and standard; used to evaluate analytical efficiency by measuring recovery. Surrogates are brominated, fluorinated, or isotopically labeled compounds not expected to be detected in environmental media.

4.36 TWELVE-HOUR TIME PERIOD - The twelve (12) hour time period for GC/MS system instrument performance check, standards calibration (initial or continuing calibration), and method blank analysis begins at the moment of injection of the DFTPP or BFB analysis that the laboratory submits as documentation of instrument performance. The time period ends after 12 hours have elapsed according to the system clock. For pesticide/Aroclor analyses performed by GC/EC, the twelve hour time period in the analytical sequence begins at the moment of injection of the instrument blank that precedes sample analyses, and ends after twelve hours have elapsed according to the system clock.

4.37 VOLATILE COMPOUNDS - compounds amenable to analysis by the purge and trap technique. Used synonymously with purgeable compounds.

4.38 RETENTION TIME (RT) - the time a target analyte is retained on a GC column before elution. The identification of a target analyte is dependent on a target compound's retention time falling within the specified retention time window established for that compound. Retention time is dependent on the nature of the column's stationary phase, column diameter, temperature, flow rate, and other parameters.

4.39 DEIONIZED WATER (DI water) - water that has passed through Accutest's deionization system. Used as reagent water (water that an interferant is not observed at or above the minimum quantitation limit of the parameters of interest).

4.40 SPIKE BLANK OR LABORATORY CONTROL SAMPLE (LCS) – A blank spiked with a known concentration of analyte (from a second source from the calibration standard) or an external quality control standard with a known concentration of analyte used to determine accuracy of the method.

5.0 HEALTH & SAFETY

5.1 All safety practices must be followed as outlined in the Accutest Laboratories Employee Safety Handbook and Chemical Hygiene Plan. Safety glasses, gloves, and lab coats must be worn. All samples, solutions, and extracts must be treated as unknown and potentially hazardous.

5.1 The toxicity or carcinogenicity of each reagent used in this method has not been precisely determined; however, each chemical should be treated as a potential health hazard. Exposure to these reagents should be reduced to the lowest possible level.

6.0 COLLECTION, PRESERVATION, AND HOLDING TIMES

6.1 Collection and Preservation

6.1.1 Soil/Sediment: Refer to SOP MSM207 (Collection and Preservation of Solid Samples for Volatile Organics Analysis by SW846 5035 Methodology).

6.1.2 Aqueous: Samples are collected in certified pre-cleaned 40 ml VOA vials equipped with a teflon-lined silicone septum cap. Samples must be preserved with 1: 1 HCL to a pH of < 2. The pH of the samples are checked using wide-range pH paper after the sample is analyzed. A clean Pasteur pipette is used to obtain a small aliquot of the sample, and it is applied to the wide-range pH paper. The pH is noted in the analysis log. Water samples must be checked for headspace. The client must be notified of any samples exhibiting headspace greater than "pea size" (>6mm). The client must provide permission to analyze any samples with headspace greater than "pea-size", and this permission must be documented with the project.

Note: The pH of aqueous volatile samples must be checked AFTER analysis. If sample is not properly preserved, this information must be communicated to the client.

6.1.3 Volatile samples must be protected from light and stored segregated from samples for other analyses at a temperature of 4°C ± 2°C from the time of receipt to analysis.

6.2 Hold Time: Samples must be analyzed within 14 days of sampling.

7.0 APPARATUS & MATERIALS

7.1 SYRINGE

7.1.1 10, 25, 50, 100, 500 and 5000 ul graduated syringes, manually held (Hamilton or equiv.).

7.1.2 5 ml glass gas tight syringes with Luerlok end, if applicable to the purging device.

7.2 BALANCE

7.2.1 Analytical balance capable of weighing 0.0001 gram.

7.2.2 Top-loading balance capable of weighing 0.1 g.

7.3 PURGE AND TRAP DEVICES

7.3.1 Tekmar LSC2000, Solatek 72, Velocity XPT, Aquatek 70 and O.I. Analytical 4552, 4560, 4660 (Eclipse), and 4551-a are used for purging, trapping and desorbing the sample into GC column.

7.3.2 The sample purge vessel must be designed to accept 5 ml samples with a water column at least 3 cm deep.

7.3.3 The trap must be at least 25 cm long and have an inside diameter of at least 0.105 inch. The trap must be packed to contain the following absorbents (3-ring):

7.3.3.1 2,6-Diphenylene oxide polymer.
7.3.3.2 Silica gel.
7.3.3.3 Charcoal packing.
7.3.3.4 Or equivalent

7.3.4 The trap should be conditioned according to manufacturer specifications by back flushing with a Helium gas flow of at least 20 ml/min prior to use.

7.3.5 The desorber should be capable of rapidly heating the trap to 180°C for desorption.

7.3.6 O.I. 4552 and Solatek 72 is equipped with sample heater for analyzing low-level soils.

7.4 GAS CHROMATOGRAPH/MASS SPECTROMETER SYSTEM

7.4.1 Gas Chromatograph.

7.4.1.1 An alytical system complete with a temperature programmable gas chromatograph and all required accessories including syringes, analytical columns, and gases.

7.4.2 Column.

7.4.2.1 For 5890: 75 m x 0.53 mm I.D., 3 um film DB-624; J&W Scientific. Or equivalent. For 6890: 60 m x 0.25 mm I.D., 1.4 um film DB-VRX; J&W Scientific. Or equivalent.

7.4.3 Mass Spectrometer.

7.4.3.1 Capable of scanning from 35-260 amu every second or less utilizing a 70 volt (nominal) electron energy in the electron impact ionization mode.

7.4.3.2 Capable of producing a mass spectrum which meets all the criteria in Table 2 when injecting 50 ng of Bromofluorobenzene(BFB).

7.5 DATA SYSTEM

7.5.1 A computer system is interfaced to the mass spectrometer which allows the continuous acquisition and storage on machine readable media (disc) of all mass spectra obtained throughout the duration of the chromatographic program.

7.5.2 The computer utilizes software which allows searching any GC/MS data file for target analytes which display specific fragmentation patterns.

7.5.3 The Enviroquant data system is capable of quantitation using multipoint calibration and multipoint internal standards.

7.5.4 The recent version of the EPA/NIH mass spectral library (70,000 compounds) is being used for non target peak tentative identification.

7.5.5 Data can be archived to magnetic tape for long term storage.

8.0 STANDARDS & REAGENTS

Note: All applicable standard/reagent preparation information, including vendor, lot number, date of preparation, date of expiration, calculations, and initials must be entered in the appropriate standard/reagent preparation logbook. Vendors typically used by Accutest include Fisher Scientific, VWR, Accustandard, Supelco, Chemservices, Ultra, and ERA. Additional vendors may be utilized as necessary.

8.1 Solvent

8.1.1 Methanol: Omni Solvent or equivalent. Store apart from other solvents.

8.2 Reagent water

8.2.1 Reagent water is defined as water in which an interferant is not observed at the method detection limit of the parameters of interest.

8.2.1.1 Reagent water is generated by either passing tap water through a bed of approximately one pound of activated carbon or by using the water purification system at Accutest which is a series of deionizers and carbon cartridges.

8.3 Stock standard solutions

8.3.1 Commercially prepared standards used:

Calibration Standards:

- Ethanol standard M-502A-R-10X @ 100,000 ug/ml
- M-502A-R-10X 2.0 mg/ml
- M-502B-10X @ 2.0 mg/ml
- Cus-6580 Ultra
- Cus-6581 Ultra
- O-Cymene @ 2470 ug/ml

Spike standards (second source)

- Ethanol standard @ 100,000 ug/ml
- Cus-6580-ICV @ various conc.
- Cus 6581-ICV @ various conc.
- M-502-10X
- O-cymene @ 2150 ug/ml

Or equivalent standards.

8.3.2 Stock standard solutions (except gases) must be replaced after 6 months or according to manufacturers expiration date if comparison with quality control check samples indicates a problem.

8.3.2.1 The purgeable gases standard may need to be replaced weekly or sooner if comparison to quality control samples indicates a problem. Purgeable gas standards may be kept longer than a week if acceptability can be documented by the calibration check standard.

8.3.2.2 Stock standard solutions should be stored according to manufacturers specifications. Opened ampules should be stored at -10°C to -20°C except for acrolein/acrylonitrile (solution in water) – should be refrigerated at 4°C ± 2°C (or stored according to the manufacturer).

8.4 Internal Standard and Surrogate Solution.

8.4.1 Four internal standards (see Table 3) are used that exhibit similar analytical behavior to the compounds of interest:

Internal Std: Ultra, Internal Standard Mixture # STM-341N ; 2.0 mg/ml in methanol or equivalent; TBA-d9 ISTD Restek, 50,000 ug/ml or equivalent.

Surrogate: Ultra, Surrogate Standard Mixture # STM-330N; 2.0 mg/ml in methanol or equivalent. Refer to section 8.3.2.2 for storage conditions.

8.5 Working standards

8.5.1 A 200 ug/ml working standard is used for the O.I. 4552 and 4551a autosamplers. A 1:10 dilution of these standards may also be prepared to use in the 3 lowest calibration levels.

8.5.1.1 The blank spike and matrix spike solutions are prepared independently from the calibration standards using a different vendor or lot number. Note: It is recommended to use a different vendor – if unavailable verify that the source materials are from separate lots.

8.5.2 See Table 10 for preparation of working standard.

8.5.4 Working standard solutions should be stored at -10°C to -20°C.

9.0 INTERFERENCES

9.1 The data from all blanks, samples, and spikes must be evaluated for interferences.

9.2 Impurities in the purge gas, organic compounds out-gassing from the plumbing ahead of the trap, and solvent vapors in the laboratory 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 tubing, non-TFE thread sealants, or flow controllers with rubber components in the purging device should be avoided.

9.3 Samples can be contaminated by diffusion of volatile organics (particularly methylene chloride and fluorocarbons) through the septum seal into the sample 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.

9.4 Contamination by carry-over can occur whenever high level and low level samples are sequentially analyzed. Whenever an unusually concentrated sample is encountered, it should be followed by an analysis of reagent water to check for cross contamination.

10.0 PROCEDURE

10.1 CALIBRATION (See Section 10.1.6.4 before proceeding)

10.1.1 The calibration range covered by the standards is 0.5, 1, 2, 5, 25, 50, 100, 200, 400 ug/l. The ICAL analyzed for Army Corps project samples must have a low standard no lower than 5 ppb. Refer to the specific Army Corps project QAPP for additional guidance for the ICAL analysis. The Analyte Reporting Limit (RL) can not be lower than the lowest calibration level utilized.

10.1.2 The linear range covered by this calibration is 100 % of highest concentration standard (up to 400 ug/L).

10.1.3 For the O.I. autosamplers internal standards are added from a reservoir at a concentration of 250 ug/ml. For the Tekmar autosamplers internal and surrogate standards are added from a reservoir at a concentration of 125 ug/ml. 1 ul is added to the standard, sample or blank prior to analyses (O.I), and 2 ul is added to the standard, sample, or blank prior to analyses (Tekmar). Surrogate standards are added to each calibration standard at the same level as corresponding target analytes.

10.1.4 Each analyte is quantitatively determined by internal standard technique using the closest eluting internal standard and the corresponding area of the major ion. See Table 7.

10.1.5 The Response Factor (RF) is defined in section 10.4.1.

10.1.6 Initial calibration

The following criteria must be met for the initial calibration to be valid.

10.1.6.1 The percent relative standard deviation (% RSD) (see section 10.4.2) of must be ≤ 20 % for all target compounds for quantitation versus an average response factor to take place. For compounds with % RSD > 20, linear regression or quadratic curve may be used provided the linear correlation coefficient is greater than or equal to 0.99. A minimum of five calibration levels must be used for a Linear regression and a minimum of six levels must be used for a Quadratic curve. Forcing the curve through the origin (zero) is acceptable in many instances (If used – verify acceptability with the department supervisor).

10.1.6.2 If linear regression is used, the low point (reporting limit) must be verified by re-calculating the concentration of the low point against the curve. Acceptance criteria is 70-130% recovery. If this criteria is not met a higher RL which meets the acceptance criteria should be used – or otherwise data reported to the RL should be reported as estimated.

10.1.6.3 The minimum average response factors (RFs) listed in Table 8 of SW846 8260C (see Table 9 of SOP) must be met for the lowest calibration concentration and for the average RF. RF acceptance criteria for compounds not listed in these tables should be developed with a minimum RF of 0.05. Target compounds with an RF of <0.05 may result in rejection of non-detected results.

10.1.6.4 Evaluation of retention times. The relative retention time of each target analyte in each calibration standard should agree within 0.06 relative retention time units.

10.1.6.5 Internal standard should not drift by more than ±10 seconds as compared to the mid-level standard. All standards run during the initial calibration must pass this criteria.

10.1.6.6 The analyst is allowed to remove a low point or a high point on a curve to meet criteria. Middle points may not be removed unless there is clear evidence of some obvious error (such as a poor purge or an incorrect spike concentration) – and this must be confirmed by a supervisor and documented in the instrument run logbook. Removal of a low point to meet criteria will raise the reporting limit, while removal of a high point will lower the calibration range. The minimum of 5 (or 6 points for quadratic) points must be maintained.

10.1.6.7 The concentration of the calibration standard at the upper limit of the curve will be reduced if the instrument response indicates signs of detector saturation.

10.1.6.8 An initial calibration is a single event. Reanalysis of a calibration standard must occur within the same tuning period as the initial calibration – and before sample analysis occurs.

10.1.6.9 For Selective Ion Monitoring (SIM) analysis the initial calibration curve uses lower concentration calibration standards than normal analyses: 0.1, 0.5, 1.0, 5.0, 20, 50, and

100 ppb (may vary). The SIM ICAL is evaluated using the same criteria as normal scan.

10.1.7 Initial calibration verification (ICV)

10.1.7.1 The initial calibration is verified for accuracy immediately following the last standard with a verification standard from a source independent of the calibration standards (either a lot or vendor different from the calibration standards – preferably a different vendor). ICV acceptance criteria is 70-130% recovery except for compounds determined to be "difficult" (refer to Definitions section) – which ICV acceptance criteria is 40-160%.

10.1.7.2 The SIM ICV is analyzed at a concentration of 5.0 ppb (may vary). Acceptance criteria is the same as for normal scan.

10.1.8 Calibration Check (CBCHK)

10.1.8.1 A continuing calibration check standard at mid-level concentration (100 ug/ml) must be acquired every 12 hrs. All continuing and initial calibration standards are prepared in volumetric flasks and transferred to 40 ml vials.

10.1.8.2 The RF's generated for each parameter must be compared to the average RF in the Initial calibration for each analyte to determine the percent difference (% D) (see section 10.4.3).

10.1.8.3 The minimum RFs listed for each target compound in Table 8 of SW846 8260C (see Table 9 of SOP) must be met. RF acceptance criteria for compounds not listed in these tables should be developed with a minimum RF of 0.05. Target compounds with an RF of <0.05 may result in rejection of non-detected results.

10.1.8.4 The % D for all target compounds must be less than or equal to 20%. Up to 20% of compounds may exceed the 20% D criteria as long as the %D is <40%.

10.1.8.5 If the above specified criteria are met, the continuing calibration is considered valid.

10.1.8.6 If either of the criteria fail, corrective action must be performed. Standard data is evaluated to determine if an analytical system problem exists. If there is problem which does not require making major changes to the system, then those changes are made and the continuing calibration is re-analyzed. If a major problem exists or major changes need to be performed, then the Supervisor is notified for further instruction.

10.1.8.7 If any of the internal standard areas change by a factor of two (- 50% to + 100%) from the last mid-point initial calibration standard, the analytical system must be inspected for malfunctions and corrections will be made, as appropriate.

10.1.8.8 If the retention time for the Internal Standards change by more than 10 seconds from the most recent mid-point initial calibration standard, the system must be inspected for malfunctions. When corrections are made the sample must be reanalyzed.

10.1.8.9 A calibration check may only be repeated once. If the second analysis fails, a new initial calibration must be performed. In situations where the first check fails to meet criteria, the instrument logbook should have clear documented notations as to what the problem was and what the corrective actions was performed to enable the second analysis to meet criteria.

10.1.8.10 If the calibration verification is being performed using an autosampler for night batch analysis, two vials of standard solution may be set up for analysis. The second standard must meet calibration check criteria. The second check may be discarded due to poor purge or incorrect spike concentration – however, the first check must meet criteria. This scenario must be approved by the supervisor, and documented in the instrument run logbook.

10.1.8.11 The SIM calibration check is analyzed at a concentration of 5.0 ppb (may vary). Acceptance criteria is the same as for normal scan.

10.2 ANALYSIS

10.2.1 Instrument conditions.

10.2.1.1 Recommended instrument conditions are listed in Table 1 Modifications are allowed as long as criteria of calibration are met.

10.2.1.2 For SIM analysis, the Scan Parameters are changed. Scanning windows are established in the instrument run method which use a minimum of one target ion and one secondary ion for each target analyte. Appropriate time is allowed for the elution of each peak of interest.

10.2.2 Purge and Trap Conditions.

10.2.2.1 Recommended instrument conditions are listed in Table 1.

10.2.3 Procedure for the evaluation of the BFB tune.

NOTE: It may be necessary at this step of the procedure to analyze a conditioning blank (section 4.5) to condition the instrumentation prior to any analysis. This step is usually necessary when instrumentation has been idle for a period of time.

10.2.3.1 Every 24 hours, inject 2 µl (50 ng) of BFB solution directly on column.

Note: The BFB Tune may be performed either as a separate analysis or evaluated from the daily calibration or blank check.

10.2.3.2 The GC/MS system must be checked to verify acceptable performance criteria are achieved (see Table 2) for BFB.

10.2.3.3 This performance test must be passed before any samples, blanks or standards are analyzed.

10.2.3.4 If all the criteria are not achieved, the analyst must retune the mass spectrometer and repeat the test until all criteria are met.

10.2.3.5 The injection time of the acceptable tune analysis, is considered the start of the 24 hour clock.

10.2.4.6 Three scans (the peak apex scan and the scans immediately preceding and following the apex) are acquired and averaged.

10.2.4.7 Background subtraction is required, and must be accomplished using a single scan acquired within 20 scans of the elution of BFB. The background subtraction should be designed only to eliminate column bleed or instrument background ions. Do not subtract part of the BFB peak.

10.2.4.8 As a secondary procedure, an individual scan (the apex or the scans preceding or following the apex) may be used. Background subtraction must be used in the same manner as the first approach.

10.2.4 Daily calibration check

10.2.4.1 See section 10.1.8

10.2.5 Method blank (reagent water)

10.2.5.1 An acceptable method blank must be analyzed for every 12 hour time period.

10.2.5.2 For O.I. and Tekmar autosamplers, fill a clean 40 ml voa vial with deionized water. Replace teflon lined cap being sure not to leave any air bubbles in vial. Analyze as per 10.2.

10.2.5.3 Surrogates must meet in-house acceptance limits (or other regulatory program/client QAPP limits as applicable).

10.2.5.4 Evaluate the method blank for target compound contamination to the MDL. If target compounds are detected in the method blank above ½ the RL, the source of contamination must be identified and corrective action must be taken (and documented in the run log) before continuing with sample analysis. The method blank must be re-analyzed and evaluated before sample analysis can begin (must meet surrogate and contamination criteria). Common laboratory contaminants such as acetone, methylene chloride, 2-butanone, and chloroform may not be present in the method blank greater than 5 times the RL.

10.2.6 Sample analysis

10.2.6.1 Rinse 5 ml syringes at least three times with organic-free water (reagent water).

10.2.6.2 Establish dilution of sample in order to fall within the upper portion of the calibration range.

10.2.6.2.1 from acquired sample data.
10.2.6.2.2 from history program.
10.2.6.2.3 sample characteristics (appearance, sheen, etc.)

10.2.6.3 Water sample

10.2.6.3.1 Place 40 ml vial into appropriate position on autosampler.
10.2.6.3.2 Record pH using 0-14 pH paper and record in logbook.
10.2.6.3.3 The pH is checked after analysis is completed (refer to section 6.1.2).

10.2.6.4 Sediment/ soil sample

Low-level soil method (Requires separate initial and continuing calibrations)

- Low level initial calibrations and calibration checks are prepared in sodium bisulfate solution.

10.2.6.4.1 Low level soils are collected in the field preserved in sodium bisulfate. If sample is not preserved – the analyst adds 5 mls of DI water to 5 g of sample (and a stir bar) in a 40 ml voa vial. Soil samples are generally purged on O.I. 4552 instrumentation. Surrogates and IS are loaded onto the instrument (in vials) and automatically injected into the sample. The samples are heated, stirred, and purged onto the trap.
10.2.6.4.2 Alternately, accurately weigh approximately 5 g (or less) sample into clean 40 ml voa vial.
10.2.6.4.3 Add 5 ml reagent water and 1 stir bar to voa vial.
10.2.6.4.4 Place vial into autosampler.

Medium-level soil method

- Medium level initial calibrations and calibration checks are prepared in water containing methanol equivalent to a 1:50 dilution.

The sample should be extracted in methanol if sample contains analytes above working calibration range or exhibits severe matrix interference.

10.2.6.4.5 weigh 10 g sample into VOA vial containing 10 ml methanol and seal with Teflon lined septum.
10.2.6.4.6 mix by hand shaking vigorously for 1 minute.
10.2.6.4.7 let settle.
10.2.6.4.8 aliquot proper amount of extract by using gas tight microsyringe.
10.2.6.4.9 add aliquoted sample (extract) to a 50 ml volumetric flask (filled to mark with DI water). Cap and swirl gently (or invert gently 3 times) to mix.

10.2.6.5 For medium-level soil analyses, add 5 ul of 50 ug/ml internal standard (I.S.) to syringe containing sample. The concentration of each I.S. should be 50 ug/l without any dilution factors.

10.2.6.6 Fill a 40 ml voa vial with the diluted sample extract (no headspace) and place in autosampler.

10.2.6.7 For analysis of low-level soils soils, heat the sample vial to 40°C while purging and stirring the sample for 11 minutes with Helium. Water samples are not heated. Sample dry purge time is 2 minutes for both matrices.

10.2.6.8 Desorb the sample for 1-4 minutes by rapidly heating the trap to 180°C while backflushing with Helium. The desorb time should be set based on the trap manufacturer's specification.

10.2.6.9 Bake the trap for 12 minutes at 225°C or at the manufacturers specifications to remove any residual purgeable compounds.

10.2.6.10 If the quantitation value for any analyte exceeds the working range of the GC/MS system, dilute the sample and re-analyze.

Note: Internal standard and surrogate is added by the autosampler

10.3 DATA INTERPRETATION

10.3.1 Qualitative identification.

10.3.1.1 The targeted compounds shall be identified by analyst with competent knowledge in the interpretation of mass spectra by comparison of the sample mass spectrum to the mass spectrum of a standard of the suspected compound. The analyst or supervisor may use professional judgement to supercede identification criteria in certain cases (especially when evaluating low-level results). The criteria required for a positive identification are:

10.3.1.2 The sample component must elute at the same relative retention time (RRT) as the daily standard. Criteria is the RRT of sample component must be within ± 0.06 RRT units of the standard.

10.3.1.3 All ions present in the standard mass spectra at a relative intensity greater than 10% (major abundant ion in the spectrum equals 100%) must be present in the sample spectrum.

10.3.1.4 The relative intensities of these ion must agree within ± 30% between the daily 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%).

10.3.1.5 Structural isomers that produce very similar mass spectra should be identified as individual isomers if they have sufficiently different GC retention times. Sufficient GC resolution is achieved if the height of the valley between the two isomer peaks is less than 50% of the average of the two peak heights.

10.3.2 Quantitative analysis.

10.3.2.1 When a target compound has been identified, concentration (see section 10.4) will be based on the integrated area of the quantitation ion, normally the base peak (see Table 7).

10.3.2.2 If the sample produces an interference for the primary ion, use a secondary ion to quantitate (see Table 7). This is characterized by an excessive background signal of the same ion which distorts the peak shape beyond a definitive integration. Also an interference could severely inhibit the response of the internal standard ion.This secondary ion must also be used to generate new calibration response factors.

10.3.3 Library search for tentatively identified compounds.

10.3.3.1 If a library search is requested, the analyst should perform a forward library search of NBS mass spectral library to tentatively identify 15 non-reported compounds.

10.3.3.2 Guidelines for making tentative identification are:

10.3.3.2.1 These compounds should have a response greater than 10% of the nearest internal standard. The response is obtained from the integration for peak area of the Total Ion Chromatogram (TIC).

10.3.3.2.2 The search is to include a spectral printout of the 3 best library matches for a particular substance. The results are to be interpreted by analyst.

10.3.3.2.3 Molecular ions present in the reference spectrum should be present in the sample spectrum.

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

10.3.3.2.5 The relative intensities the major ions should agree within ± 20%.

10.3.3.2.6 Ions present in the sample spectrum but not in the reference spectrum should be reviewed for possible background contamination or presence of coeluting compounds.

10.3.3.2.7 Ions present in the reference spectrum but not in the sample spectrum should be reviewed for possible background subtraction from the sample spectrum because of background contamination or coeluting peaks.

10.3.3.2.8 Quantitation of the tentatively identified compounds is obtained from the total ion chromatogram based on a response factor of 1 and is to be tabulated on the library search summary data sheet.

10.3.3.2.9 Quantitation will be performed by using the nearest internal standard.

10.3.3.2.10 Report result as estimated.

10.4 CALCULATION

10.4.1 Response Factor (RF)

$$RF = \frac{As \times Cis}{Ais \times Cs}$$

where: As = Area of the characteristic ion for the compound being measured.
Ais = Area of the characteristic ion for the specific internal standard.
Cs = Concentration of the compound being measured (ug/l).
Cis = Concentration of the specific internal standard (ug/l).

10.4.2 Percent Relative Standard Deviation (% RSD)

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

where: SD = Standard Deviation
RFav = Average response factor from initial calibration.

10.4.3 Percent Difference (% D)

$$\%D = \frac{|RFav - RFc|}{RFav} \times 100$$

where: RFc = RF from continuing calibration (CBCHK)

10.4.4 Concentration (Conc.)

For water:

$$Conc.\ (ug/l) = \frac{Ac \times Cis \times Vp}{Ais \times RFav \times Vi}$$

For soil/sediment (on a dry weight basis):

$$Conc.\ (ug/kg) = \frac{Ac \times Cis \times Vp}{Ais \times RFav \times Ws \times M}$$

Where: Ac = Area of characteristic ion for compound being measured.
Vp = 5 ml (Total Purge Volume)
Vi = Initial volume of water purged (ml).
Ws = Weight of sample extracted (g).
M = (100 - % moisture in sample) / 100 or % solids / 100

10.4.5 Percent Recovery (% R)

$$\%R = \frac{Concentration\ Found}{Concentration\ Spiked} \times 100$$

10.4.6 Relative Percent Difference (RPD)

$$RPD = \frac{|MSC - MSDC|}{(\frac{1}{2})(MSC + MSDC)} \times 100$$

Where: MSC = Matrix Spike Concentration
MSDC = Matrix Spike Duplicate Concentration

10.4.7Data Correction for Target Analyte Calculations for Methanol Preservation Dilution Effect.

10.4.7.1 Results for soil/sediment samples must be corrected for the Methanol Preservation Dilution Effect. The potential for under reporting results is more pronounced the greater the percent moisture content of the sample if this procedure is not used.

10.4.7.2 ml solvent/water (Vm) = ml methanol + ((%moisture/100) X g of sample) – this calculation is automatically performed by the LIMS.

11.0 QUALITY ASSURANCE

11.1 QC Requirements Summary

BFB	Every 12 hrs.

ICV	Every ICAL
Calibration Check std.	Every 12 hrs.
Method blank	Every 12 hrs. and/or every batch*
Blank Spike*	Every 12 hrs. and/or every batch*
Blank Spike Duplicate**	Every 12 hrs. and/or every batch*
Matrix Spike	one per 20 samples.
Matrix Spike Duplicate	one per 20 samples.
Blank Spike	one per batch.*
Surrogate	every sample and standard.
Internal Standard	every sample and standard.

*The maximum number of samples per analytical batch is twenty.
**MCP Requirement.

11.2 Daily GC/MS performance check - refer to section 10.2.3

11.3 ICV – refer to section 10.1.7

11.4 Daily calibration check - refer to section 10.1.8

11.5 Method blank (reagent water) - refer to section 10.2.5

11.6 Matrix Spike(MS)/Matrix Spike Duplicate(MSD).

11.6.1 One sample is selected at random from each analytical batch of similar matrix types and spiked in duplicate with select compounds to check precision and reproducibility.

11.6.2 Matrix spikes are prepared by spiking an actual sample at a concentration of 50 ug/l or 50 ug/kg based on 5 g dry weight. This is analyzed as outlined in 10.2.

11.6.3 Percent recoveries (% R) (see section 10.4.5) are compared to 70-130% or to in-house control limits.

11.6.4 A relative percent deviation (RPD) (see section 10.4.6) is calculated and compared to acceptance criteria of 30 or to in-house control limits.

11.6.5 If matrix spikes do not meet criteria and the QC check sample (blank spike) passed acceptance criteria, a matrix interference is to be assumed and the data is reportable and must be footnoted.

11.6.6 A relative percent deviation (RPD) (see section 10.4.6) is calculated and must $\leq$ 20.

11.6.7 If matrix spikes do not meet criteria and the QC check sample (blank spike) passed acceptance criteria (in-house limits), a matrix interference is to be assumed and the data is reportable and must be footnoted.

11.7 Blank Spike/Blank Spike Duplicate.

NOTE: **The BSPD is an MCP requirement.**

11.7.1 Reagent water is used for the Blank Spike.

11.7.2 Blank Spikes are prepared by adding 5uls of blank Spike solution (prepared independently and from a second source as the calibration standards) to 5mls reagent water. For O.I. 4552 and 4551a , 10 uls are added to 40 mls. reagent water. See Table 10 for standard preparation. For the MCP, the blank spike is prepared and analyzed in duplicate.

11.7.3 Percent recoveries are compared to 70-130% or in-house acceptance limits. The RPD acceptance criteria is less than or equal to 25. If RPD criteria is not met, the source of the non-conformance must be determined and resolved before sample analysis can proceed. If samples have been analyzed, the non-conformance must be discussed in the case narrative.

11.7.4 Blank spike values are used to verify results when Matrix spike/matrix spike duplicate results indicate a potential problem due to sample matrix.

11.7.5 If blank spike recoveries are biased high, and sample results are non-detected, these results may be reported without qualification. If blank spike recoveries are biased low, the samples (and blank spike) must be re-analyzed.

11.8 Surrogate

11.8.1 All blanks, samples, and standards contain surrogate compounds which are used to monitor method performance.

11.8.2 If the recovery of any surrogate compound does not meet the control limits specified by in-house acceptance limits the calculation must be checked.

11.8.3 The sample must be reanalyzed if the recovery of any one surrogate is out of control limits of 70-130% or in-house control limits.

11.8.4 Above conditions (section 11.8.3) are not required for samples having severe matrix interference problems.

11.8.5 If surrogate recoveries are acceptable upon reanalysis, the data from the reanalysis is reported. If the reanalysis date did not meet the hold time, then both sets of data have to submitted with the reanalysis reported.

11.8.6 If surrogates are still outside control limits upon reanalysis, then both sets of data should be submitted with the first analysis reported.

11.9 Internal Standard.

11.9.1 Retention time for all internal standard must be within ± 30 seconds of the corresponding internal standard in the latest continuing calibration or 100 ug/l standard of initial calibration if samples are analyzed directly following an initial calibration.

11.9.2 The area (Extracted Ion Current Profile) of the internal standard in all analyses must be within 50 to 200% of the corresponding area in the latest calibration standard (12 hr. time period).

11.9.3 If area of internal standard does not meet control limits, the calculations must be checked. If a problem is not discovered, the sample must be reanalyzed.

11.9.4 If areas are acceptable upon reanalysis, the reanalysis data is reported.

11.9.5 If areas are unacceptable upon reanalysis, then both set of data are submitted with the original analysis reported.

11.10 A Precision and accuracy (P&A) study is performed as an initial determination of capability, on an annual basis (continued demonstration of capability – a successful PT result may be used in place of a P&A for continued DOC), and if any significant changes have been made to the instrument. In general, 4 replicates or blank spikes are analyzed using the same procedures and conditions for sample analysis. The percent recoveries are compared to 70-130% or in-house control limits. The standard deviation of the 4 replicate percent recoveries are compared to 30 or in-house limits. If percent recovery or standard deviation criteria are not met, corrective action must be taken to bring the system back into control.

11.11 Quality control limits are generated at least on an annual basis by QA using an in-house program. Blank spike, MS/MSD, and surrogate QC data are pooled for the previous year (or other specified time frame) and the data is processed and evaluated by QA. The new limits are maintained on the QA server. The annual QC limit data is filed with QA.

11.12 All NELAC-accredited target compounds must be spiked in the blank spike and matrix spike within a two-year period. All target compounds reported for a project are spiked and evaluated in the blank spike and MS/MSD.

11.13 Cleanup blanks.

11.13.1 Blanks may be analyzed in certain situations in order to clean up the analytical system or prevent carryover to client samples. However, the analysis of these cleanup blanks must be performed in a manner which does not bias analytical results or create unfair treatment of quality assurance samples.

11.13.2 A clean up blank may be analyzed after the calibration check and blank spike or MS/MSD in order to prevent low-level (< the RL) carry-over of heavier compounds such as naphthalene and trimethylbenzenes. It may not be necessary to analyze a clean up blank after the MS/MSD if the client does not require estimated (“J” flag) results or if the heavier compounds are not to be reported.

11.13.3 A clean up blank should be analyzed after a suspected highly contaminated sample in order to prevent carryover. If samples analyzed after the clean up blank exhibit concentrations consistent with carryover (and the clean up blank also exhibits these low concentrations) then either the affected samples should be re-analyzed or results narrated.

12.0 DOCUMENTATION

12.1 The analytical logbook is a record of the analysis sequence; the logbook must be completed daily. Each instrument will have a separate logbook.

12.2 If samples require reanalysis, a brief explanation of the reason should be documented in this log.

12.3 The standard preparation logbook must be completed for all standard preparations. All information requested must be completed; the page must be signed and dated by the respective person.

12.4 The Accutest lot number must be cross-referenced on the standard vial.

12.5 The instrument Maintenance logbook must be completed when any type of maintenance is performed on the instrument. Each instrument will have a separate log.

12.6 All laboratory logbooks must be reviewed and initialed or signed by the lab manager.

12.7 Any corrections to laboratory data must be done using a single line through the error. The initials of the person and date of correction must appear next to the correction.

13.0 DATA REVIEW

13.1 The analyst conducts the primary review of all data. This review begins with a check of all Instrument and method quality control and progresses through sample quality control concluding with a check to assure that the client's requirements have been executed. The analyst has the authority and responsibility to perform corrective action for any out-of-control parameter of non-conformance.

13.2 A secondary review is performed by department managers, and it includes review of the data produced by their department. All manual calculations, QC criteria, and a comparison of the data package to client specified requirements are checked. The department manager may reject data, initiate reanalysis, take additional corrective action, or reprocess data.

13.3 The Client Services Staff performs a tertiary review on all data prior to release to the client that includes a check of the data package contents to assure that all analytical requirements and specifications were executed.

13.4 Spot-check reviews are performed by the Quality Assurance Officer focusing on all elements of the deliverable including the client's specifications and requirements, analytical quality control, sample custody documentation and sample identification.

13.5 Follow the client or regulatory program – specified technical specifications for quality control evaluation as applicable. These technical specifications are found in the "Client Tech Specs" folder on the QA server (LINUXMA1). Any special requirements – including client or regulatory program technical specifications – will be described in the project comments field in the daily work list.

14.0 DATA REPORTING

14.1 A results page including positive results and/or RLs, units, methodology, surrogate recoveries, analysis dates, and data qualifiers are reported. Additional quality control data including calibration summaries, MS/MSD (or duplicate) percent recoveries and RPDs, blank spike recoveries, and method blank results may be reported upon request of the client. Raw data may be reported to the client on request.

14.2 Data may be submitted to the client in a specified electronic format (EDD).

14.3 Once the data is approved by the laboratory manager, it may be accessed by clients via LabLink™.

14.4 Data assessment and acceptance criteria for quality control. For samples collected under the MCP: If QC do not meet acceptance criteria (method blank, blank spike, surrogate recoveries, and internal standard area counts) and are reanalyzed outside of the recommended holding time – the lab must report results of both the initial and reanalysis. For surrogate recoveries and internal standard area counts– if the sample is not reanalyzed due to obvious interference the lab must provide a copy of the chromatogram.

14.5 Procedures for handling non-conforming data.

14.5.1 If quality control data does not meet criteria the non-conformance must be discussed in a case narrative and footnoted on the applicable quality control report summary.

14.5.2 If preservation or holding time criteria is not met and the samples are analyzed the result page must be footnoted with this information, and the non-conformance must be discussed in a case narrative or other suitable communication (telephone conversation log or email). Client notification documentation should be included with the data (telephone conversation log, fax, or email).

15.0 POLLUTION PREVENTION & WASTE MANAGEMENT

15.1 Pollution Prevention. Users of this method must perform all procedural steps that controls the creation and/or escape of wastes of hazardous materials to the environment. The amounts of standards, reagents, and solvents must be limited to the amounts specified in this SOP. All safety practices designed to limit the escape of vapors, liquids, or solids to the environment must be followed. All method users must be familiar with the waste management practices described in section 15.2

15.2 Waste Management. Individuals performing this method must follow established waste management procedures as described in the Sample and Waste Disposal SOP. This document describes the proper disposal of all waste materials generated during the testing of samples as follows:

15.2.1 Non-hazardous aqueous wastes
15.2.2 Hazardous aqueous wastes

15.2.3 Chlorinated organic solvents
15.2.4 Non-chlorinated organic solvents
15.2.5 Hazardous solid wastes
15.2.6 Non-hazardous solid wastes

16.0 METHOD PERFORMANCE

16.1 Method performance is evaluated by the annual quality control limits generated by QA, and the annual MDL study results. Refer to section 3.5 for MDLs, and section 11.11 for QC limits.

17.0 ADDITIONAL REFERENCES

17.1 SW846 5030B, Rev. 2, December, 1996, SW846 5035 Rev. 1, December, 1996, SW846 5035A, Draft Rev. 1, December 1996, SW846 8000C, Rev. 3, March, 2003, MA DEP WSC-CAM-IIA, Rev. 4, 5/28/04

Table 1
RECOMMENDED OPERATING CONDITION

Gas Chromatograph/ Mass Spectrometer	5890/5970MSD	6890/5975/ 6890/5973
Carrier Gas(linear velocity)	Helium at 30 cm/sec	1.2 ml/min
Mass range	35 - 260 amu	"
Electron Energy	70 volts (nominal)	"
Scan time	not to exceed 7 sec. per scan	"
Injection port temperature	250degC	"
Source temperature	200 – 250degC	"
Transfer line temperature	250 – 300degC	"
Analyzer temperature	220 – 260degC	"
Initial temperature	36degC	42
Time 1	3 minutes	2 minutes
Column temperature rate	8deg/min.	10deg/min to 80degC 14deg/min to 210degC 16deg/min to 240degC
Final temperature	200degC. 3 min. hold	80degC 2.9min hold
Total run time	30 minutes	20 minutes

Purge and Trap Unit

Water Samples

Purge flow	40 mls/ min.
Purge time	11 min.
Dry Purge	2 min
Desorb preheat	175degC
Desorb	1-4 min. at 180 degC
Bake	8-12 min. at 225degC
Transfer line	100 – 110degC
Valve temperature	approx. transfer line temp.

Purge and Trap Unit

Soil Samples

Purge flow	40 mls/ min.
Purge time	11 min.
Dry Purge	2 min
Desorb preheat	175degC
Desorb	4 min. at 180degC
Bake	8-12 min. at 200 - 225degC
Transfer line	100 – 110degC
Valve temperature	approx. transfer line temp.
Preheat	40degC
Preheat time	3 min.

These conditions are recommendations and may be optimized.

Table 2

BFB KEY IONS AND ION ABUNDANCE CRITERIA

Mass	Ion Abundance Criteria
50	15 – 40% of mass 95
75	30 – 60% of mass 95
95	Base peak, 100% relative abundance
96	5 - 9% of mass 95
173	<2% of mass 174
174	>50% of mass 95
175	5 - 9% of mass 174
176	>95% and <101% of mass 174
177	5 - 9% of mass 176

Table 3

INTERNAL STANDARD

Internal Standard	Prim./Sec. Ions
Pentafluorobenzene	168
Chlorobenzene-d5	117 / 82, 119
TBA-d9	65/46
1,4-Difluorobenzene	114 / 63, 88
1,4-Dichlorobenzene-d4	152, 115, 150

Table 4

SURROGATES

Compound	(Prim./Sec. ions)	Control Limit (%)	
		Water	Soil
Dibromofluoromethane	(113)	70-130%	70-130%
Toluene-d8	(92 / 91,65)	70-130%	70-130%
4-Bromofluorobenzene	(95 / 174,176)	70-130%	70-130%

Table 5
Volatile Internal Standards with Corresponding Analytes Assigned for Quantitation

Pentafluorobenzene	ions
Acetone	(58/43)
Acrylonitrile	(53/52,51)
Acrolein	(56/55,58)
Bromochloromethane	(128,49,130)
Bromomethane	(94/96,79)
Carbon disulfide	(76/78)
Chloroethane	(64/66,49)
Chloroform	(83/85,47)
Chloromethane	(50/52,49)
Dichlorodifluoromethane	(85/87,50)
1,1-Dichloroethane	(63/65,83)
2,2-Dichloropropane	(77,97)
1,2-Dichloroethane	(surr.)
1,1-Dichloroethene	(96/61,98)
Iodomethane	(142/127,141)
Methylene chloride	(84/49,51)
Vinyl chloride	(62/64,61)
trans-1,2-Dichloroethene	(96/61,98)
Trichlorofluoromethane	(101/103,66)
Tertiary butyl alcohol	(59/41)
Vinyl Acetate	(43,86)

Chlorobenzene-d5	ions
1,1,2-Trichloroethane	(97,99,61)
Chlorobenzene	(112/114,77)
Ethylbenzene	(106/91)
1,3-Dichloropropane	(76,78)
*Ethyl methacrylate	(69/41,99)
2-Hexanone	(43/58,57)
Styrene	(104/78,103)
Tetrachloroethene	(164/129,131)
Toluene-d8	(surr.)
Xylenes	(106/91)
Chlorodibromomethane	(129/208,206)
1,1,1,2-Tetrachloroethane	(131,133,206)
Bromoform	(173/171,175,252)
Trans-1,4-dichloro-2-butene	(53)

cis-1,2-Dichloroethene	(96,61,98)
Tetrahydrofuran	(42/71/72)
Acetonitrile	(41)
Di-isopropyl ether	(45)
Isobutyl alcohol	(43)
Ethyl-tert butyl ether	(59)
Cyclohexane	(56)
Chloroprene	(53)
Ethanol	(45)
Ethyl acetate	(43)
Freon 113	(101)
Ethyl ether	(59)
Methacrylonitrile	(41)

1,4-Difluorobenzene	ions	1,4-Dichlorobenzene-D4	ions
Benzene	(78/52,77)	Isopropylbenzene	(105,120)
Bromodichloromethane	(83/85,129)	Bromobenzene	(156,77,158)
2-Butanone	(72/57,43)	1,2,3-Trichloropropane	(75/110,77,61)
Carbon tetrachloride	(117/119,121)	n-Propylbenzene	(91,120)
2-Chloroethyl vinyl ether	(63/65,106)	1,1,2,2 Tetrachloroethane	(83/85,131,133)
Dibromomethane	(93/174,95)	2-chlorotoluene	(91,126)
1,4-Dichloro-2-butene	(75/53,89)	4-chlorotoluene	(91,126)
1,2-Dichloropropane	(63/62,41)	1,3,5-Trimethylbenzene	(105,120)
cis-1,3-Dichloropropene	(75/77,390	t-Butlybenzene	(119,91,134)
trans-1,3-Dichloropropene	(75/77,39)	sec-Butlybenzene	(105,134)
1,1,1-Trichloroethane	(97/99,117)	1,3-Dichlorobenzene	(146/148,111)
Trichloroethene	(130/95,97,132)	1,3-Dichlorobenzene	(146/148,111)
Vinyl acetate	(43/86)	1,2-Dichlorobenzene	(146/148,111)
Methyl tert butyl ether	(73/57)	p-Isopropyltoluene	(119,134,91)
1,4-Dioxane	(88/58)	n-Butylbenzene	(91,92,134)
Ethyl acetate	(43/45,61)	1,2-Dibromo-3-chloropropane	(75,155,157)
Bromofluorobenzene	(surr.)	1,2,4-Trichlorobenzene	(180,182,145)
1,1-Dichloropropane	(75,110,77)	Naphthalene	(128)
1,2-Dichloroethane	(62,64,98)	Hexachlorobutadiene	(225,223,227)
Toluene	(91/92,65)	1,2,3-Trichlorobenzene	(180,182,145)
4-methyl-2-pentanone	(43/58,100)		
1,2-Dibromoethane	(107, 109)		
Tert-amyl ethyl ether	(73)		
Ethyl methacrylate	(69)		
Methyl methacrylate	(69)		
Methylcyclohexane	(83)		

TBA-d9	
Tert butyl alcohol (TBA)	(59)

Table 6

Compounds reported by 8260B

COMPOUND	CAS #
ACETONE	67-64-1
ACETONITRILE	75-05-8
ACROLEIN	107-02-08
ACRYLONITRILE	107-13-1
ALLYL CHLORIDE	107-05-1
BENZENE	71-43-2
BROMOBENZENE	108-86-1
BROMOCHLOROMETHANE	74-97-5
BROMODICHLOROMETHANE	75-27-4
BROMOFORM	75-25-2
BROMOMETHANE	74-83-9
2-BUTANONE	78-93-3
n-BUTLYBENZENE	104-51-8
sec-BUTYLBENZENE	135-98-8
tert-BUTYLBENZENE	98-06-6
CARBON DISULFIDE	75-15-0
CARBON TETRACHLORIDE	56-23-5
CHLOROBENZENE	108-90-7
CHLOROETHANE	75-00-3
CHLOROFORM	67-66-3
CHLOROMETHANE	74-87-3
CHLOROPRENE	126-99-8
CYCLOHEXANE	110-82-7
2-CHLOROETHYL VINYL ETHER	110-75-8
2-CHLOROTOLUENE	95-49-8
4-CHLOROTOLUENE	106-43-4
DIBROMOCHLOROMETHANE	124-48-1
1,2-DIBROMO-3-CHLOROPROPANE	96-12-8
1,2-DIBROMOETHANE	106-93-4
DIBROMOMETHANE	74-95-3
1,2-DICHLOROBENZENE	95-50-1
1,3-DICHLOROBENZENE	540-73-1
1,4-DICHLOROBENZENE	106-46-7

DICHLORODIFLUOROMETHANE	75-71-8
1,1-DICHLOROETHANE	75-34-3
DICHLOROFLUOROMETHANE	75-71-8
1,2-DICHLOROETHANE	107-06-02
1,1-DICHLOROETHYLENE	75-35-4
1,4-DIOXANE	123-91-1
DI-ISOPROPYL ETHER	108-20-3
cis-1,2-DICHLOROETHYLENE	156-59-2
trans-1,2-DICHLOROETHYLENE	156-60-5

Table 6 (cont.)

Compounds reported by 8260B

COMPOUND	CAS #
1,3-DICHLOROPROPANE	142-28-9
1,2-DICHLOROPROPANE	78-87-5
2,2-DICHLOROPROPANE	594-20-7
1,1-DICHLOROPROPENE	563-58-6
cis-1,3-DICHLOROPROPENE	10061-01-5
trans-1,3-DICHLOROPROPENE	10061-02-6
ETHYLBENZENE	100-41-4
ETHYL METHACRYLATE	97-63-2
ETHYL ACETATE	141-78-6
FREON 113	76-13-1
FREON 114	76-14-2
HEXACHLOROBUTADIENE	87-68-3
ETHANOL	64-17-5
ETHYL ETHER	60-29-7
2-HEXANONE	591-78-6
IODOMETHANE	74-88-4
ISOPROPYLBENZENE	98-82-8
ISOBUTYL ALCOHOL	78-83-1
p-ISOPROPYLTOLUENE	99-87-6
METHYLENE CHLORIDE	75-09-2
4-METHYL-2-PENTANONE	108-10-1
METHACRYLONITRILE	126-98-7
METHYL METHACRYLATE	80-62-6
METHYLCYCLOHEXANE	108-87-2
METHYL ACETATE	79-20-9
NAPHTHALENE	99-20-3
METHYL TERT BUTYL ETHER	1634-04-4
n-PROPYLBENZENE	103-65-1
PROPIONITRILE	107-12-0
PENTACHLOROETHANE	76-01-7
STYRENE	100-42-5

1,1,1,2-TETRACHLOROETHANE	630-20-6
1,1,2,2-TETRACHLOROETHANE	79-34-5
TETRACHLOROETHYLENE	127-18-4
TETRAHYDROFURAN	109-99-9
TOLUENE	108-88-3
TRANS-1,4-DICHLORO-2-BUTENE	110-57-6
CIS-1,4-DICHLORO-2-BUTENE	1476115
TERT-AMYL METHYL ETHER	994-05-8
TERT-BUTYL ETHYL ETHER	637-92-3
1,2,3-TRICHLOROBENZENE	87-61-6
1,2,4-TRICHLOROBENZENE	120-82-1

Table 6 (cont.)

Compounds reported by 8260B

1,1,1-TRICHLOROETHANE	71-55-6
1,1,2-TRICHLOROETHANE	79-00-5
TRICHLOROETHYLENE	79-01-6
TRICHLOROFLUOROMETHANE	75-69-4
1,2,3-TRICHLOROPROPANE	96-18-4
1,2,4-TRIMETHYLBENZENE	95-63-6
1,3,5-TRIMETHYLBENZENE	108-67-8
TERT BUTYL ALCOHOL	637-92-3
VINYL ACETATE	108-05-4
VINYL CHLORIDE	75-01-4
m-XYLENE	108-38-3
p-XYLENE	106-42-3
o-XYLENE	95-47-6
TOTAL XYLENES	1330-20-7

Note: Analysis of additional compounds may be appended to this SOP.

Table 7
SINGLE LABORATORY ACCURACY AND PRECISION DATA FOR VOLATILE ORGANIC COMPOUNDS IN WATER DETERMINED WITH A WIDE-BORE CAPILLARY COLUMN *

COMPOUND	CONC. RANGE (UG/L)	RECOVERY (%)	STANDARD DEVIATION OF REC.	PERCENT RSD
ACETONE	ND **	ND	ND	ND
ACROLEIN	ND	ND	ND	ND
ACRYLONITRILE	ND	ND	ND	ND
BENZENE	0.1-10	97	6.5	5.7
BROMOBENZENE	0.1-10	100	5.5	5.5
BROMOCHLOROMETHANE	0.5-10	90	5.7	6.4
BROMODICHLOROMETHANE	0.1-10	95	5.7	6.1
BROMOFORM	0.5-10	101	6.4	6.3
BROMOMETHANE	0.5-10	95	7.8	8.2
2-BUTANONE	ND	ND	ND	ND
n-BUTLYBENZENE	0.5-10	100	7.6	7.6
sec-BUTYLBENZENE	0.5-10	100	7.6	7.6
tert-BUTYLBENZENE	0.5-10	102	7.4	7.3
CARBON DISULFIDE	ND	ND	ND	ND
CARBON TETRACHLORIDE	0.5-10	84	7.4	8.8
CHLOROBENZENE	0.1-10	98	5.8	5.9
CHLOROETHANE	0.5-10	89	8.0	9.0
CHLOROFORM	0.5-10	90	5.5	6.1
CHLOROMETHANE	0.5-10	93	8.3	8.9
2-CHLOROETHYL VINYL ETHER	ND	ND	ND	ND
2-CHLOROTOLUENE	0.1-10	90	5.6	6.2
4-CHLOROTOLUENE	0.1-10	99	8.2	8.3
DIBROMOCHLOROMETHANE	0.1-10	92	6.5	7.0
1,2-DIBROMO-3-CHLOROPROPANE	0.5-10	83	16.6	19.9
1,2-DIBROMOETHANE	0.5-10	102	4.0	3.9
DIBROMOMETHANE	0.5-10	100	5.6	5.6
1,2-DICHLOROBENZENE	0.1-10	93	5.8	6.2
1,3-DICHLOROBENZENE	0.5-10	99	6.8	6.9
1,4-DICHLOROBENZENE	0.2-20	103	6.6	6.4
DICHLORODIFLUOROMETHANE	0.5-10	90	6.9	7.7
1,1-DICHLOROETHANE	ND	ND	ND	ND
1,2-DICHLOROETHANE	ND	ND	ND	ND
1,1-DICHLOROETHYLENE	ND	ND	ND	ND
cis-1,2-DICHLOROETHYLENE	ND	ND	ND	ND
trans-1,2-DICHLOROETHYLENE	ND	ND	ND	ND
1,2-DICHLOROPROPANE	0.1-10	97	5.9	6.1
1,3-DICHLOROPROPANE	0.1-10	96	5.7	6.0
2,2-DICHLOROPROPANE	0.5-10	86	14.6	16.9

1,1-DICHLOROPROPENE	0.5-10	98	8.7	8.9
cis-1,3-DICHLOROPROPENE	ND	ND	ND	ND

Table 7 (cont.)

COMPOUND	CONC. RANGE (UG/L)	RECOVERY (%)	STANDARD DEVIATION OF REC.	PERCENT RSD
trans-1,3-DICHLOROPROPENE	ND	ND	ND	ND
ETHYLBENZENE	0.1-10	99	8.4	8.6
HEXACHLOROBUTADIENE	0.5-10	100	6.8	6.8
2-HEXANONE	ND	ND	ND	ND
IODOMETHANE	ND	ND	ND	ND
ISOPROPYLBENZENE	0.5-10	101	7.7	7.6
p-ISOPROPYLBENZENE	0.1-10	99	6.7	6.7
METHYLENE CHLORIDE	0.1-10	95	5.0	5.3
4-METHYL-2-PENTANONE	ND	ND	ND	ND
NAPHTHALENE	0.1-100	104	8.6	8.2
n-PROPYLBENZENE	0.1-10	100	5.8	5.8
STYRENE	0.1-100	102	7.3	7.2
1,1,1,2-TETRACHLOROETHENE	ND	ND	ND	ND
1,1,2,2-TETRACHLOROETHANE	0.1-10	91	5.7	6.3
TETRACHLOROTOLUENE	ND	ND	ND	ND
TOLUENE	0.5-10	102	8.1	8.0
1,2,3-TRICHLOROBENZENE	0.5-10	109	9.4	8.6
1,2,4-TRICHLOROBENZENE	0.5-10	108	9.0	8.3
1,1,1-TRICHLOROETHANE	0.5-10	98	7.9	8.1
1,1,2-TRICHLOROETHANE	0.5-10	104	7.6	7.3
TRICHLOROETHYLENE	0.5-10	90	6.5	7.3
TRICHLOROFLUOROMETHANE	0.5-10	89	7.2	8.1
1,2,3-TRICHLOROPROPANE	0.5-10	108	15.6	14.4
1,2,4-TRIMETHYLBENZENE	0.5-10	99	8.0	8.1
1,3,5-TRIMETHYLBENZENE	0.5-10	92	6.8	7.4
VINYL ACETATE	ND	ND	ND	ND
VINLY CHLORIDE	0.5-10	98	6.5	6.7
m-XYLENE	0.1-10	97	6.3	6.5
p-XYLENE	0.5-10	104	8.0	7.7
o-XYLENE	0.1-31	103	7.4	7.2

*** Criteria from SW846 method 8260B**
**** ND; not determined**

Table 8
WORKING STANDARD PREPARATION

Internal standard and Surrogate mixture O.I. (250 ug/ml)		**Tekmar (125 ug/ml)**
Internal Standard (2000 ug/ml)	1250 ul	1250 ul
Surrogates (2000 ug/ml)	1250 ul	1250 ul
TBA (50,000 ug/ml)	500 ul	500 ul
Methanol	7.0 mls	17.0 mls
Final volume	10ml	20 mls

Calibration mixture

2 ml Ethanol Standard @ 100,000 ug/ml

1 ml M-502A-R-10X @ 2.0 mg/ml

1 ml M-502-B-10X @ 2.0 mg/ml

1 ml Cus-6580 Ultra

1 ml Cus-6581 Ultra

0.8 ml 0-Cymene @ 2470 ug/ml

3200 ul Methanol

FV = 10 ml

Blank Spike/ Matrix Spike (2nd source)

2 mls Ethanol Standard @ 100,000 ug/ml

1 ml Cus-5680-ICV @ various conc.

1ml Cus-6581 @ various conc.

1ml M-502-10X

0.93 ml O-Cymene @ 2150 ug/ml

4070 ul Methanol

FV = 10 ml

Table 9
Minimum RFs

Compound	Minimum RF
Dichlorodifluoromethane	0.100
Chloromethane	0.100
Vinyl Chloride	0.100
Bromomethane	0.100
Chloroethane	0.100
Trichlorofluoromethane	0.100
1,1-Dichloroethene	0.100
1,1,2-Trichloro-1,2,2-trifluoroethane	0.100
Acetone	0.100
Carbon Disulfide	0.100
Methyl Acetate	0.100
Methylene Chloride	0.100
Trans-1,2-Dichloroethene	0.100
cis-1,2-Dichloroethene	0.100
Methyl tert Butyl Ether	0.100
1,1-Dichloroethane	0.200
2-Butanone	0.100
Chloroform	0.200
1,1,1-Trichloroethane	0.100
Cyclohexane	0.100
Carbon Tetrachloride	0.100
Benzene	0.500
1,2-Dichloroethane	0.100
Trichloroethene	0.200
Methylcyclohexane	0.100
1,2-Dichloropropane	0.100
Bromodichloromethane	0.200
cis-1,3-Dichloropropene	0.200
trans-1,3-Dichloropropene	0.100
4-Methyl-2-pentanone	0.100
Toluene	0.400
1,1,2-Trichloroethane	0.100
Tetrachloroethene	0.200
2-Hexanone	0.100
Dibromochloromethane	0.100
1,2-Dibromoethane	0.100
Chlorobenzene	0.500

Ethylbenzene	0.100
meta-/para-Xylene	0.100
ortho-Xylene	0.300
Styrene	0.300
Bromoform	0.100
Isopropylbenzene	0.100
1,1,2,2-Tetrachloroethane	0.300
1,3-Dichlorobenzene	0.600
1,4-Dichlorobenzene	0.500
1,2-Dichlorobenzene	0.400
1,2-Dibromo-3-chloropropane	0.050
1,2,4-Trichlorobenzene	0.200

Lab Manager: Douglas Yargeau

QA Officer: Robert Treggiari

TITLE: DETERMINATION OF SEMIVOLATILE ORGANIC COMPOUNDS USING GC/MS SYSTEM

TEST METHOD REFERENCE: SW846 8270D Rev. 4, February 2007

REVISED SECTIONS: defined linear and quadratic regression; conditioning blank; updated section 8.0 reagent notation; updated Table 1; revised 10.2.3

1.0 SCOPE & APPLICATION

1.1 The following method describes the analytical procedure which is utilized by Accutest to analyze semivolatile organic compounds using a gas chromatograph/mass spectrometer (GC/MS). This SOP includes full scan and single ion monitoring (SIM) procedures. Applicable matrices include soil, water, and solid waste. Refer to Tables 7, 10, 11, or 12 or use the Compound List function in LIMS to view the compounds analyzed by this method.

2.0 SUMMARY

2.1 This method is performed in accordance with extraction methodologies: SW-846 3rd Edition – 3510C, 3545A, 3520C, 3540C and 3550B.

2.2 The resultant methylene chloride extract is injected into a tuned and calibrated GC/MS system equipped with a fused silica capillary column.

2.3 The peaks detected are qualitated by comparison to characteristic ions and retention times specific to the known target list of compounds.

2.4 Once identified the compound is quantitated by internal standard techniques with an average response factor generated from a 5 point curve.

2.5 Additional unknown peaks with a response > 10 % of the closest internal standard may be processed through a library search with comparison to a data base of approximately 40,000 spectra. An estimated concentration is quantitated by assuming a response factor of 1.

3.0 REPORTING LIMIT AND METHOD DETECTION LIMIT

3.1 The Reporting (RL) is based on Accutest laboratory extraction procedure and lowest calibration standard. RL'S may vary depending on matrix difficulties and sample volumes or weight and percent moisture.

3.2 The Method Detection Limit (MDL) represents the lowest reportable concentration of an individual analyte that meets the method qualitative identification criteria.

3.3 Method Detection limits (MDLs) are experimentally determined using the procedures described in 40 CFR, Part 136, Appendix B. Actual reported MDLs incorporate the sample volume analyzed and sample dilutions if needed, which may cause MDL variations from sample to sample.

3.4 In general, MDLs are determined through the analysis of at least 7 replicate blank spikes (using the same procedures for sample analysis). The MDL is calculated by multiplying the standard deviation of the replicate concentrations by the appropriate Student's t value (3.143 for 7 replicates). MDLs are determined initially (prior to analysis) and on an annual basis. Refer to the MDL SOP (MQA245) for further MDL study details.

3.5 Current MDLs are entered into the LIMS, and can be viewed by printing out the compound list from LIMS. Additionally, MDLs are reported on the result page upon client request. Current MDL studies are filed with Quality Assurance. Obsolete MDL studies are archived with the QA files. Electronic MDL data is found in the annual "MDL" folder on the QA server (LINUXMA1).

4.0 DEFINITIONS

4.1 ALIQUOT - a measured portion of a sample, or solution, taken for sample preparation and/or analysis.

4.2 BATCH – A group of samples which behave similarly with respect to the sampling or the testing procedures being employed and which are processed as a unit. For QC purposes, if the number of samples in a group is greater than 20 (or 10 for certain methods), then each group of 20 samples (or 10 samples for certain methods) or less will all be handled as a separate batch.

4.3 CALIBRATION – the establishment of an analytical curve based on the absorbance, emission intensity, or other measured characteristic of known standards. The calibration standards must be prepared using the same type of acid or concentration of acids as used in the sample preparation.

4.4 CALIBRATION STANDARDS – a series of known solutions used by the analyst for calibration of the instrument (i.e., preparation of the analytical curve).

4.5 CONDITIONING BLANK – defined as any matrix blank analyzed on instrumentation prior to or following any analytical quality control or samples. This blank is used to condition the instrumentation, normally as the first analysis of any day, or following an analytical sequence, such that all instrumentation or column conditions are stabilized prior to analysis.

4.6 CONTINUING CALIBRATION – analytical standard run every 12 hours to verify the initial calibration of the analytical system.

4.7 DRY WEIGHT – the weight of a sample based on percent solids. The weight after drying in an oven.

4.8 DIFFICULT COMPOUNDS – Compounds that historically perform poorly (and as defined by the MA DEP MCP CAM): 4-Chloroaniline, 4-Nitrophenol, Phenol, and 2,4-Dinitrophenol.

4.9 CONTAMINATION - a component of a sample or an extract that is not representative of the environmental source of the sample. Contamination may stem from other samples, sampling

equipment, while in transit, from laboratory reagents, laboratory environment, or analytical instruments.

4.10 EXTRACTABLE - a compound that can be partitioned into an organic solvent from the sample matrix and is amenable to gas chromatography. Extractables include semivolatile (BNA) and pesticide/Aroclor compounds.

4.11 FIELD SAMPLE - a portion of material to be analyzed that is contained in single or multiple containers and identified by a unique sample number.

4.12 FIELD BLANK – this is any sample that is submitted from the field and is identified as a blank. This includes trip blanks, rinsates, equipment blanks, etc.

4.13 HOLDING TIME – the elapsed time expressed in days from the date of sampling until the date of its analysis.

4.14 INTERFERENTS – substances which affect the analysis for the analyte of interest.

4.15 GAS CHROMATOGRAPH (GC) - the instrument used to separate analytes on a stationary phase within a chromatographic column. The analytes are volatized directly from the sample (VOA water and low-soil) volatized from the sample extract (VOA medium soil), or injected as extracts (SVOA and PEST). In VOA and SVOA analysis, the compounds are detected by a Mass Spectrometer (MS). In PEST analysis, the compounds are detected by an Electron Capture (EC) detector. In the screening procedure (all fractions), the Flame Ionization Detector (FID) is used as the detector.

4.16 INITIAL CALIBRATION - analysis of analytical standards for a series of different specified concentrations; used to define the linearity and dynamic range of the response of the mass spectrometer or electron capture detector to the target compounds.

4.17 INITIAL CALIBRATION VERIFICATION – analysis of a check standard from a second source (either vendor or lot) from the calibration standards (4.14) to verify the initial calibration.

4.18 INTEGRATION TIME RANGE - the retention time at the beginning of the area of integration to the retention time at the end of the area of integration.

4.19 INSUFFICIENT QUANTITY - when there is not enough volume (water sample) or weight (soil/sediment) to perform any of the required operations: sample analysis or extraction, percent moisture, MS/MSD, etc

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4.20 LINEAR REGRESSION – an analytical calibration regression where the concentrations of the analytes and the instrument response for each standard can be fit to a straight line. This yields a model described by the equation:

$$Y = mx + b$$

Where Y is the instrument response, m represents instrument sensitivity, and b is a constant that describes the instrument background. The analyte concentration (x) of unknown samples can then be calculated.

4.21 MASSACHUSETTS CONTINGENCY PLAN (MCP) – A Massachusetts Department of Environmental Protection (MA DEP) program which deals with environmental cleanups within the state. For the purposes of this SOP MCP specifically refers to data quality and reporting guidelines established under the MCP Data Enhancement Program.

4.22 MATRIX - the predominant material of which the sample to be analyzed is composed. For the purpose of this SOP, a sample matrix is either water or soil/sediment. Matrix is not synonymous with phase (liquid or solid).

4.23 MATRIX EFFECT - in general, the effect of a particular matrix (water or soil/sediment) on the constituents with which it contacts. This is particularly pronounced for clay particles which may adsorb chemicals and catalyze reactions. Matrix effects may prevent extraction of target analytes, and may affect surrogate recoveries. In addition, non-target analytes may be extracted from the matrix causing interferences.

4.24 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.

4.25 MATRIX SPIKE DUPLICATE - a second aliquot of the same matrix as the matrix spike (above) that is spiked in order to determine the precision of the method.

4.26 METHOD BLANK - an analytical control consisting of all reagents, internal standards, and surrogate standards that is carried throughout the entire analytical procedure. The method blank is used to define the level of laboratory, background, and reagent contamination.

4.27 PERCENT DIFFERENCE (%D) - As used in this SOP and elsewhere to compare two values, the percent difference indicates both the direction and the magnitude of the comparison, i.e., the percent difference may be either negative, positive, or zero. (In contrast, see relative percent difference.)

4.28 PERCENT MOISTURE - an approximation of the amount of water in a soil/sediment sample made by drying an aliquot of the sample at 105 °C. The percent moisture determined in this manner also includes contributions from all compounds that may volatilize at or below 105 °C, including water. Percent moisture may be determined from decanted samples and from samples that are not decanted.

4.29 PURGE AND TRAP (DEVICE) - analytical technique (device) used to isolate volatile (purgeable) organics by stripping the compounds from water or soil by a stream of inert gas, trapping the compounds on an adsorbent such as a porous polymer trap, and thermally desorbing the trapped compounds onto the gas chromatographic column.

4.30 QUADRATIC REGRESSION – a nonlinear model where in the basic form the functional part of the model in not linear with respect to the unknown parameters, and the method of least squares is used to estimate the values of the unknown parameters.

$$Y = ax^2 + bx + c$$

Where x represents a variable or an unknown, and a, b, and c are constants with, a not equal to zero.

4.31 REAGENT WATER - water in which an interferant is not observed at or above the minimum quantitation limit of the parameters of interest. Accutest uses deionized water (municipal water which passes through Accutest's DI treatment system).

4.32 RELATIVE PERCENT DIFFERENCE (RPD) - As used in this SOP to compare two values, the relative percent difference is based on the mean of the two values, and is reported as an absolute value, i.e., always expressed as a positive number or zero. In contrast, see percent difference.

4.33 RELATIVE RESPONSE FACTOR (RRF) - a measure of the relative mass spectral response of an analyte compared to its internal standard. Relative Response Factors are determined by analysis of standards and are used in the calculation of concentrations of analytes in samples. RRF is determined by the following equation:

$$RRF = \frac{A_x}{A_{is}} x \frac{C_{is}}{C_x}$$

Where,

A = area of the characteristic ion measured
C = concentration, or amount (mass)
is = internal standard
x = analyte of interest

4.34 RELATIVE RETENTION TIME (RRT) - the ratio of the retention time of a compound to that of a standard (such as an internal standard).

$$RRT = \frac{RT_c}{RT_{is}}$$

Where,

RT_c = Retention time for the semivolatile target or surrogate compound in continuing calibration.
RT_{is} = Retention time for the internal standard in calibration standard or in a sample.

4.35 RESPONSE - or Instrumental Response: a measurement of the output of the GC detector (MS, EC, or FID) in which the intensity of the signal is proportionate to the amount (or concentration) detected. Measured by peak area or peak height.

4.36 SOIL - used herein synonymously with soil/sediment and sediment.

4.37 SURROGATES (Surrogate Standard) - for semivolatiles and pesticides/Aroclors, compounds added to every blank, sample, matrix spike, matrix spike duplicate, and standard; used to evaluate analytical efficiency by measuring recovery. Surrogates are brominated, fluorinated, or isotopically labeled compounds not expected to be detected in environmental media.

4.38 TWELVE-HOUR TIME PERIOD - The twelve (12) hour time period for GC/MS system instrument performance check, standards calibration (initial or continuing calibration), and method blank analysis begins at the moment of injection of the DFTPP or BFB analysis that the laboratory submits as documentation of instrument performance. The time period ends after 12 hours have elapsed according to the system clock. For pesticide/Aroclor analyses performed by GC/EC, the twelve hour time period in the analytical sequence begins at the moment of injection of the instrument blank that precedes sample analyses, and ends after twelve hours have elapsed according to the system clock.

4.39 RETENTION TIME (RT) - the time a target analyte is retained on a GC column before elution. The identification of a target analyte is dependent on a target compound's retention time falling within the specified retention time window established for that compound. Retention time is dependent on the nature of the column's stationary phase, column diameter, temperature, flow rate, and other parameters.

4.40 DEIONIZED WATER (DI water) - water that has passed through Accutest's deionization system. Used as reagent water (water that an interferant is not observed at or above the minimum quantitation limit of the parameters of interest).

4.41 SPIKE BLANK OR LABORATORY CONTROL SAMPLE (LCS) – A blank spiked with a known concentration of analyte (from a second source from the calibration standard) or an external quality control standard with a known concentration of analyte used to determine accuracy of the method.

5.0 HEALTH & SAFETY

5.1 All safety practices must be followed as outlined in the Accutest Laboratories Employee Safety Handbook and Chemical Hygiene Plan. Safety glasses, gloves, and lab coats must be worn. All samples, solutions, and extracts must be treated as unknown and potentially hazardous.

5.1 The toxicity or carcinogenicity of each reagent used in this method has not been precisely determined; however, each chemical should be treated as a potential health hazard. Exposure to these reagents should be reduced to the lowest possible level.

6.0 COLLECTION, PRESERVATION, AND HOLDING TIMES

6.1 PRESERVATION

6.1.1 Aqueous

6.1.1.1 Container - 2 (two) 1 liter glass bottles with teflon insert in cap. Client must provide additional bottles if sample is to be used for QC.

6.1.2 Soil

6.1.2.1 Container – 1 (one) 300-500 ml amber glass bottle

6.1.3 Sample should be taken with care so as to prevent any portion of the collected sample coming in contact with the sampler's gloves, thus avoiding possible phthalate contamination.

6.1.4 The samples must be protected from light and refrigerated at 4° C (± 2° C) from the time of receipt until extraction and analysis.

6.1.5 Store extracts at -10°C to -20°C protected from light in sealed vials equipped with unpierced PTFE lined septa.

6.2 HOLDING TIME

6.2.1 Aqueous samples must be extracted within 7 days of sampling.

6.2.2 Soil samples must be extracted within 14 days of sampling.

6.2.3 Extracts must be analyzed within 40 days following extraction.

7.0 APPARATUS & MATERIALS

7.1 GAS CHROMATOGRAPH/MASS SPECTROMETER SYSTEM

7.1.1 Gas Chromatograph.

7.1.1.1 The analytical system which is complete with a temperature programmable gas chromatograph and all required accessories including syringes, analytical columns, and gases. The injection port is designed for splitless injection with capillary columns. The capillary column is directly coupled to the source.

7.1.2 Column.

7.1.2.1 30 m x 0.25 mm fused silica (0.25 um film thickness) DB-5MS capillary column. Condition the column as per manufacturers directions.

7.1.3 Mass Spectrometer.

7.1.3.1 Capable of scanning from 35-500 amu every second or less utilizing a 70 volt (nominal) electron energy in the electron impact ionization mode. Capable of producing a mass spectrum which meets all the criteria in Table 2 when injecting 50 ng of Decafluorotriphenyl phosphine (DFTPP).

7.2 DATA SYSTEM

7.2.1 A computer system is interfaced to the mass spectrometer which allows the continuous acquisition and storage on machine readable media (disc) of all mass spectra obtained throughout the duration of the chromatographic program.

7.2.2 The computer utilizes software which allows searching any GC/MS data file for analytes which display specific fragmentation patterns.

7.2.3 The ENVIROQUANT data system is capable of quantitation using multipoint calibration and multipoint internal standards.

7.2.4 The recent version (rev E) of the EPA/NIH mass spectral library (75,000 compounds) is being used.

7.2.5 Data can be archived to disc for long term storage.

7.3 SYRINGE

7.3.1 10 ul graduated, manually held (Hamilton or equiv.).

7.3.2 10 ul graduated, auto sampler (Hamilton or equiv.).

8.0 STANDARDS & REAGENTS

Note: All chemicals listed are reagent grade unless otherwise specified. Distilled, deionized water should be used whenever water is required. All applicable standard/reagent preparation information, including vendor, lot number, date of preparation, calculations, and initials must be entered in the appropriate standard/reagent preparation logbook. Vendors typically used by Accutest include Fisher Scientific, VWR, Accustandard, Absolute Standards, Supelco, Chemservices, Ultra, and ERA. Additional vendors may be utilized as necessary.

8.1 Solvents - Ultra pure, chromatography grade.

8.1.1 Methylene chloride, Methanol, and Acetone.

8.2 Stock standard solutions.

8.2.1 Commercially prepared standards used.

8.2.1.1 Accustandard ,Chemserve and Ultra Scientific: (or equivalent)

8.2.1.1.1 Base Neutrals.

8.2.1.1.1.1 Accustandard CLP-HC-BN-R 2.0mg/ml in MeCl2
8.2.1.1.1.2 Accustandard Z-014e-R3 2.0mg/ml in MeCle2
8.2.1.1.1.3 Accustandard Z-014F 2.0mg/ml in MeOH

8.2.1.1.2 Acids.

8.2.1.1.2.1 Accustandard CLP-HC-A-R 2.0mg/ml in MeCl2
8.2.1.1.2.2 Accustandard APP-9-050-10x 1.0mg/ml in MeCl2

8.2.1.2 DFTPP tune stock – Accustandard M-625-TS, 50ug/ml in ch2cl2 or equivalent.

8.2.1.3 Stock standard solutions must be replaced after 1 year or sooner if comparison with quality control check samples indicates a problem. All standards should be stored at -10°C to -20°C or according to manufacturer's specifications.

8.3 Internal Standard Solution.

8.3.1 Internal standard solution – Accustandard: Z-014J 4000ug/ml in methylene chloride or equivalent. Store at -10°C to -20° C or according to the manufacturer recommended storate temperature and holding time.

8.4 Surrogate standards.

8.4.1 Acid/Base/neutral surrogate solution, Accustandard, M8270-SS Surrogate Standard. 4.0 mg/ml in methylene chloride or equivalent. Surrogate standards should be stored according to the manufacturer's recommended storage temperature and holding time.

8.5 Calibration standards.

8.5.1 Calibration standards are prepared at the following concentration levels, including surrogates, from the stock standards. Additional levels may be added if necessary. Calibration standards should be stored at -10°C to -20°C and replaced after one year or sooner if check standards indicate a problem.

8.5.1.1 Seven levels : 1, 5, 10, 50, 80, 120, 160 ug/ml

8.5.2 The concentration of surrogate shall be same as each calibration level.

8.5.3 See Table 9 for preparation of working standard.

8.6 Decafluorotriphenylphosphine (DFTPP).

8.6.1 The DFTPP solution can be used directly from the 50 ug/ml stock solution. Store at -10°C to -20°C or according to the manufacturer's recommended storage temperature and holding time.

8.7 Matrix Spike Solution.

8.7.1 The base/neutral matrix spike solution is prepared at a concentration of 50 ug/ml. The Acid matrix spike solution is prepared at 100 ug/ml. Refer to 8270C Prep Method, SOP MOP013.1

8.8 Blank Spike Solution.

8.8.1 The base/neutral Blank spiking solution is prepared at a concentration of 50 ug/ml. The acid Blank spiking solution is at 100 ug/ml. Refer to 8270C Prep Method, SOP MOP013.1

8.9 Store spiking solutions according to the manufacturer's specifications.

9.0 INTERFERENCES

9.1 The data from all blanks, samples, and spikes must be evaluated for interferences.

9.2 Method interferences may be caused by contaminants in solvents, reagents, glassware, and other stages of sample processing. Refer to "The Preparation of Glassware for Extraction of organic contaminants" SOP for practices utilized in the extraction department.

10.0 PROCEDURE

10.1 CALIBRATION

10.1.1 The calibration range covered by the standards is 1,5,10, 20, 50, 80, 120, 160, 200 ug/ml (refer to Table 10).

10.1.2 The linear range is covered by this calibration is the highest concentration standard (up to 160 ug/ml).

10.1.3 Aliquot the proper amount of each calibration standard into a 2 ml crimp top vial.

10.1.4 The concentration of each internal standard should be 40 ug/ml (refer to Table 10).

10.1.5 Each analyte is quantitatively determined using the closest eluting internal standard. (See Table 7.).

10.1.6 The Response Factor (RF) is defined in section 10.4.8.1.

10.2 Initial calibration.

10.2.1 The following criteria must be met for the initial calibration to be valid.

10.2.2 The percent relative standard deviation (% RSD) (see section 10.4.8.2) of each compound must be $\leq$ 20%. Up to 10% of the compounds may exceed the 20% RSD criteria (or "r" criteria – see below) but no greater than 40% RSD or >0.98 r.

10.2.3 Alternatively, a linear or quadratic curve may be generated (refer to definitions section). A correlation coefficient of 0.99 or higher must be achieved for the curve to be valid. A minimum of five calibration points must be used for a linear curve and six calibration points for a quadratic curve. For linear regression, the curve may be forced through the origin in many circumstances. Forcing the curve through the origin may be acceptable or prohibited

depending on the client or regulatory program. See the manager to verify acceptability prior to performing this procedure.

10.2.4 If linear regression is used the low calibration point must be verified by re-calculating this point against the curve. Acceptance criteria is 70-130% R. If the acceptance criteria is not met the RL must be raised to the next calibration point that exhibits acceptable recoveries (70-130% R) when recalculated against the curve.

10.2.5 The minimum average RF of each compound and the RF of the lowest calibration standard is evaluated against Table 4 of 8270D. If Table 4 of 8270D does not list a compound then acceptance criteria for the RF should be established with the minimum being 0.05 (non-detected results for a compound with an RF of <0.05 may be rejected).

10.2.6 The concentration of the calibration standard at the upper limit of the curve will be reduced if the instrument response indicates signs of detector saturation.

10.2.7 An initial calibration is a single event. Reanalysis of a calibration standard must occur within the same tuning period as the initial calibration – and before sample analysis occurs.

10.2.8 Calibration points within the calibration curve (above the low point and below the high point) may not be removed for the sole purpose of meeting RSD or linear regression acceptance criteria. A point within the curve may be removed only if it can be determined to be an obvious problem with the analysis (eg. poor injection, etc.). The reasoning for removing a middle point must be clearly documented in the analysis logbook. The low or high point of the curve may be removed to meet RSD or linear regression criteria. However, by removing the low point the RL is subsequently raised; by removing the high point the quantitation range is lowered.

10.2.9 For Selective Ion Monitoring (SIM) analysis the initial calibration curve uses lower concentration calibration standards than normal analyses. Typical ICAL levels are 0.05, 0.1, 0.5, 1.0, 5.0, 20, 40, 80, and 120 ug/L (reported concentration). For SIM compounds, the % RSD should be less than or equal to 20% or the correlation coefficient should be greater than or equal to 0.990.

10.3 Initial calibration verification (ICV).

10.3.1 An initial calibration verification standard at mid-level concentration (50 ug/L for Acids and 20 ug/L for Base/Nuetrals) obtained from a source independent from the calibration standards must be acquired every initial calibration.

10.3.2 The percent recoveries for the ICV must be evaluated.

10.3.3 The % recovery for all analytes must be 70-130% (or 40-160% for anlalytes determined to be difficult (see definitions section) .

10.3.4 If the above criteria specified are met, the initial calibration is considered valid.

10.3.5 If the above criteria fails, corrective action must be performed and another ICV must be run. Typical maintenance might include injection port cleaning, seal and glass sleeve cleaning or replacement and column clipping.

10.3.6 If any of the internal standard area change by a factor two (-50% to +100 %) from the last mid-level initial calibration standard, the mass spectrometer must be inspected for malfunctions and corrections will be made, as appropriate.

10.3.7 The retention times of the internal standards in the ICV cannot change by more than 30 seconds from the mid-level initial calibration standard.

10.3.8 The SIM ICV is analyzed at a lower concentration, and may vary. Typical acid ICV concentration is 40 ug/L and base/neutral concentration is 20 ug/L (reported concentration). Acceptance criteria for the SIM ICV is the same as for normal scan.

10.4 Calibration Check (CBCHK).

10.4.1 A calibration check standard at mid-level concentration (50 ug/L Acids and 20 ug/L Base Nuetrals) must be acquired every 12 hrs. For SIM, the calibration check concentration is either 20 ug/L or 5.0 ug/L. Acceptance criteria for the SIM calibration check is the same as for normal scan.

10.4.2 The RF's generated for each parameter must be compared to the average RF for the initial calibration to determine percent difference (% D) (see section 10.4.8.3).

10.4.3 The minimum RF of each analyte is compared to Table 4 of the 8270D.

10.4.4 The % D or % drift for each compound must be $\leq$ 20%. Up to 20% of the compounds (or 15% compounds from the acid or base fractions) may exceed the 20% criteria but must be <40%.

10.4.5 The internal standard area counts n the calibration check must be between 50%-200% of the area counts of the mid-level initial calibration standard.

10.4.6 If the above criteria specified are met, the continuing calibration is considered valid.

10.4.7 If any of the criteria fail, corrective action must be performed and another continuing calibration check run. Typical maintenance might include injection port cleaning, seal and glass sleeve cleaning or replacement and column clipping.

10.4.8 If any of the internal standard area change by a factor two (-50% to +100 %) from the last mid-level initial calibration standard, the mass spectrometer must be inspected for malfunctions and corrections will be made, as appropriate.

10.4.9 The retention times of the internal standards in the continuing calibration cannot change by more than 30 seconds from the last mid-level initial calibration standard.

10.4.9.1 RF acceptance criteria for compounds not listed in these tables should be developed with a minimum RF of 0.05. Target compounds with an RF of <0.05 may result in rejection of non-detected results.

10.5 ANALYSIS

10.5.1 Instrument conditions.

10.5.1.1 Recommended instrument conditions are listed in Table 1 Modifications are allowed as long as criteria of calibration are met.

10.5.1.2 For SIM analysis, the Scan Parameters are changed. Scanning windows are established in the instrument run method which use a minimum of one target ion and one secondary ion for each target analyte. Appropriate time is allowed for the elution of each peak of interest.

10.5.2 Sample preparation.

10.5.2.1 Refer to the appropriate extraction SOP (MOP013, MOP209, MOP214).

10.5.3 Daily GC/MS performance check

10.5.3.1 Every 12 hours, inject 1 ul (50 ng) of DFTPP solution directly on to the column.

10.5.3.2 The GC/MS system must be checked to verify that acceptable performance criteria are achieved (see Table 2).

10.5.3.3 Pentachlorophenol and Benzidene are should be at their normal responses and mininimal peak tailing should be present. The peak tailing factor for pentachlorophenol and benzidine must be <2.0. DDT breakdown must be ≤20%. If excessive tailing or poor peak response is present, maintenance may be required to correct the problem.

10.5.3.4 This performance test must be passed before any samples, blanks or standards are analyzed.

10.5.3.5 If all the criteria are not achieved, the analyst must retune the mass spectrometer and repeat the test until all criteria are met.

10.5.3.6 The injection time of the acceptable tune analysis, is considered the start of the 12 hour clock.

10.5.4 Daily calibration check.

10.5.4.1 See section 10.4.

10.5.5 Sample analysis.

10.5.5.1 The internal standard (I.S.) must be added to sample extract and mixed thoroughly, immediately before injection into the instrument. This minimizes losses due to adsorption, chemical reaction or evaporation.

10.5.5.2 The concentration of each I.S. should be 40 ug/ml. Add 10 ul Internal Standard Solution (4000 ug/ml) into 1 ml extract.

10.5.5.3 One microliter is injected into the GC system and data collection is started.

10.5.5.4 If the response for any ion exceeds the working range of the GC/MS system, dilute the extract and re-analyze (refer to section 10.5.9).

10.5.5.5 When the extracts are not being used for the analyses, store them at -10°C to -20° C protected from light in crimp-cap vials equipped with unpierced Teflon-lined septa.

10.5.6 Data interpretation.

10.5.6.1 Qualitative identification.

10.5.6.1.1 the targeted compounds shall be identified by analyst with competent knowledge in the interpretation of mass spectra by comparison of the sample mass spectrum to the mass spectrum of a standard of the suspected compound. The Criteria required for a positive identification are:

10.5.6.1.2 The sample component must elute at the same relative retention time (RRT) as the daily standard. Criteria is the RRT of sample component must be within ± 0.06 RRT units of the standard.

10.5.6.1.3 All ions present in the standard mass spectra at a relative intensity greater than 10 % (major abundant ion in the spectrum equals 100 %) must be present in the sample spectrum.

10.5.6.1.4 The relative intensities of these ion must agree within ± 30 % between the initial calibration mid-point 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 %.

10.5.7 Quantitative analysis.

10.5.7.1 When a target compound has been identified, concentration (see section 10.4) will be based on the integrated area of the quantitation ion, which is normally the base peak.

10.5.7.2 The sample may produce an interference for the primary ion. This may be characterized by an excessive background signal of the same ion which distorts the peak shape beyond a definitive integration. The interference could also, severely inhibit the response of the internal standard ion. If an interference is is apparent the secondary ion must be used to generate a new calibration factor.

10.5.7.3 Library search for tentatively identified compounds. If a library search is requested, the analyst should perform a forward library search of the NBS mass spectral library to tentatively identify 10 to 15 non-reported compounds (15 for base, 10 for acid, 25 for base/acid fraction).

10.5.7.4 Guidelines for making tentative identification are:

10.5.7.5 These compounds should have a response greater than 10 % of the nearest internal standard. The response is obtained from the Total Ion Chromatogram (TIC).

10.5.7.6 The search is to include a spectral printout of the 3 best library matches for a particular substance. The results are to be interpreted by analyst.

10.5.7.7 Molecular ions present in the reference spectrum should be present in the sample spectrum.

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

10.5.7.9 The relative intensities the major ions should agree within ± 20 %.

10.5.7.10 Ions present in the sample spectrum but not in the reference spectrum should be reviewed for possible background contamination or presence of coeluting compounds.

10.5.7.11 Ions present in the reference spectrum but not in the sample spectrum should be verified by performing further manual background subtraction to eliminate the interference created by coeluting peaks and/or matrix interference.

10.5.7.12 Quantitation of the tentatively identified compounds is obtained from the total ion chromatogram based on a response factor of 1 and is to be tabulated on the library search summary data sheet.

10.5.7.13 Quantitation will be performed on the nearest internal standard.

10.5.8 CALCULATION

10.5.8.1 Response Factor (RF).

$$RF = \frac{As \times Cis}{Ais \times Cs}$$

where: As = Area of the characteristic ion for the compound being measured.

Ais = Area of the characteristic ion for the specific internal standard.

Cs = Concentration of the compound being measured (ug/ml).

Cis = Concentration of the specific internal standard (ug/ml).

10.5.8.2 Percent Relative Standard Deviation (%RSD).

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

where: SD = Standard Deviation.
RFav = Average response factor from initial calibration.

10.5.8.3 Percent Difference (%D).

$$\%D = \frac{(RFav - RFc)}{RFav} \times 100$$

where: RFc = RF from continuing calibration (CBCHK).

10.5.8.4 Concentration (Conc.).

for water:

$$\text{Conc. (ug/l)} = \frac{Ac \times Cis \times Vf \times D \times 1000}{Ais \times RFav \times Vi}$$

for soil/sediment (on a dry weight basis):

$$\text{Conc. (ug/Kg)} = \frac{Ac \times Cis \times Vf \times D \times 1000}{Ais \times RFav \times Ws \times M}$$

where: Ac = Area of characteristic ion for compound being measured.

Vf = Final Volume of total extract (ml).

D = Secondary dilution factor.

Vi = Initial volume of water extracted (ml).

Ws = Weight of sample extracted (g).

M = (100 - % moisture in sample) / 100.

10.5.8.5 Percent Recovery (%R).

$$\%R = \frac{\text{Concentration found}}{\text{Concentration spiked}} \times 100$$

10.5.8.6 Relative Percent Difference (RPD).

$$RPD = \frac{(MSC - MSDC)}{(1/2)(MSC+MSDC)} \times 100$$

where: MSC = Matrix Spike Concentration.

MSDC = Matrix Spike Duplicate Concentration.

10.5.9 Sample Dilution

10.5.9.1 Sample results greater than the upper limit of the initial calibration (160 ug/L) must be re-analyzed at a dilution.

10.5.9.2 Dilution is accomplished by use of an appropriate-sized micro syringe. An aliquot of the sample extract is added to an aliquot of the proper solvent (methylene chloride) in a I ml crimp-cap (autosampler) vial. The dilution is recorded in the analysis logbook.

10.5.9.3 The sample dilution results should be within the mid to upper range of the initial calibration.

11.0 QUALITY ASSURANCE

11.1 QC Requirements Summary.

DFTPP	Every 12 hrs.
ICV	Every ICAL
Calibration Check std.	Every 12 hrs.
Method blank	one per batch*
Matrix Spike	one per batch*.
Matrix Duplicate	one per batch*.
Blank Spike	one per batch*.
Blank Spike Duplicate	one per batch*
Surrogate	every sample and standard.
Internal Standard	every sample and standard.

* The maximum number of samples per extraction batch is twenty. One per batch or every twenty samples. ***Note – the blank spike duplicate is an MCP requirement.***

11.2 DFTPP Evaluation Procedure

11.2.1 Three scans (the peak apex scan and the scans immediately preceding and following the apex) are acquired and averaged.

11.2.2 Background subtraction is required, and must be accomplished using a single scan acquired no more than 20 scans prior to the elution of DFTPP. The background subtraction should be designed only to eliminate column bleed or instrument background ions. Do not subtract part of the DFTPP peak.

11.2.3 As a secondary procedure, an individual scan (the apex or the scans preceding or following the apex) may be used. Background subtraction may be used in the same manner as the first approach.

11.3 Daily GC/MS performance check - refer to section 10.5.3.

11.4 ICV – refer to section 10.3

11.5 Daily calibration check - refer to section 10.4.

11.6 Method blank

11.6.1 The method blank should be carried through all stages of the sample preparation and measurement.

11.6.2 An acceptable method blank must be analyzed once for each extraction batch.

11.6.3 Evaluate the method blank for target compounds to the MDL. If target compounds are detected in the method blank above ½ the RL, the samples must be re-extracted and reanalyzed unless the samples are non-detected for the contaminant. Common laboratory contaminants (such as phthalates) may not be detected in the method blank above 5 times the RL.

11.6.4 Surrogates must meet acceptance criteria (11.7).

11.6.5 If the method blank does not meet surrogate or contamination criteria, the entire batch must be reextracted. However, if contamination criteria is not met, and the samples are non-detected for the contaminant – the sample results may be reported.

11.6.6 Clean up blanks may be analyzed in certain situations in order to clean up the analytical system or prevent carryover to client samples. However, the analysis of these cleanup blanks must be performed in a manner which does not bias analytical results or create unfair treatment of quality assurance samples.

11.6.7 A clean up blank should be analyzed after a suspected highly contaminated sample in order to prevent carryover. If samples analyzed after the clean up blank exhibit concentrations consistent with carryover (and the clean up blank also exhibits these low concentrations) then either the affected samples should be re-analyzed or results narrated.

11.7 Matrix Spike(MS)/Matrix Spike Duplicate(MSD)/Blank spike/Blank Spike Duplicate

11.7.1 One sample from each analytical batch is selected and spiked in duplicate with select compounds to check precision and reproducibility.

11.7.2 Matrix spikes are prepared by spiking an actual sample at a concentration of 50 ug/l for base/neutral and 100 ug/l for acids.

11.7.3 Percent recoveries (%R) (see section 10.5.8.5) are compared to in-house generated limits. If samples are collected under the MCP or RCP, percent recovery default limits of 40-140% recovery for base-neutrals and 30-130% for acid compounds are used for comparison (in-house generated limits are used if they are within the MCP/RCP limits).

11.7.4 A relative percent deviation (RPD) (see section 10.5.8.6) is calculated and compared to RPD levels of in-house generated limits. If samples are collected under the MCP or RCP, default RPD limits of 20 for waters and 30 for soils are used for comparison (in-house generated limits are used if they are within the MCP limits).

11.7.5 Blank spike. An aliquot of clean matrix (sand or water) is spiked with matrix spike and prepared with each batch of samples (spiking solution must be prepared from a source independent from the calibration standards – a second source). For the MCP, the blank spike is prepared in duplicate. Percent recoveries are compared to in-house generated limits. For samples collected under the MCP or RCP, percent recovery default limits of 40-140% for base-neutrals and 30-130 for acid compounds are used (in-house generated limits may be used for comparison if they are within the MCP/RCP limits. Additionally, the RPD should be evaluated for the BSP/BSPD. Acceptance criteria is less than or equal to 20% RPD for waters and less than or equal to 30% RPD for soils. Corrective action should be taken to resolve RPD exceedences. Any exceedence should be discussed in the case narrative.

11.7.6 If matrix spike recoveries are not met, and the QC check sample (blank spike) passed acceptance criteria, a matrix interference is to be assumed and the data is reportable. If the blank spike recoveries do not meet acceptance criteria, the samples and QC must be re-extracted. The exception to this is if the blank spike recoveries are above acceptance criteria, and the samples are non-detected for those compounds.

Note for samples collected under the MCP or RCP: The lab may use in-house generated QC limits – even if they exceed MCP default limits – as long as the compound is designated "difficult" or "problem" (based on the in-house generated data) by the lab. The lab must maintain the data to support this designation, and have available for audit review.

11.8 Surrogate

11.8.1 All blanks, samples, and matrix spikes contain surrogate compounds, which are used to monitor method performance.

11.8.2 If the recovery of any surrogate compounds does not meet acceptance limits, the calculation must be checked. If needed the sample will be reanalyzed and possibly reextraced. For samples collected under the MCP or RCP, percent recovery default limits of 30-130 (for soils), 30-130 (base-neutral - waters), and 15-110 (acid - waters) are used for comparison (in-house generated limits may be used if within the MCP/RCP limits).

11.8.3 The sample must be reanalyzed or re-extracted if the following conditions exists:

11.8.3.1 Recovery of any one surrogate compound in either base/neutral or acid fraction is below 10 %.

11.8.3.2 The above conditions (section 11.7.2 and 11.7.3.1) are not required for samples having matrix interference problems. The client may request chromatograms in the event of matrix-affected surrogate recoveries.

11.8.3.3 If surrogate recoveries are acceptable upon reanalysis, the data from the reanalysis is reported. If the sample is re-extracted and the second extraction date did not meet the hold time, then both sets of data have to submitted.

11.8.3.4 If the surrogates are still outside control limits upon reextraction and reanalysis, then both sets of data should be submitted with the first analysis reported.

11.9 Internal Standard.

11.9.1 Retention time of the internal standard in the calibration check standard must be within ± 30 seconds of the most recent mid-level initial calibration standard.

11.9.2 If this is exceeded, instrument maintenance must occur and the calibration check standard must be re-analyzed.

11.9.3 The area (Extracted Ion Current Profile) of the internal standard in the CCV's must be within 50 to 200 % of the corresponding area of the latest mid-level initial calibration standard.

11.9.4 Internal standard areas of samples, blanks and QC samples must be within 50 to 100 % of the daily calibration check standard.

11.9.5 If the area of internal standard does not meet control limits, the calculations must be checked. If a problem is not discovered, the sample must be reanalyzed.

11.9.6 If the areas are acceptable upon reanalysis, the reanalysis data is reported.

11.9.7 If the areas are unacceptable upon reanalysis, then both sets of data are submitted with the original analysis reported.

11.10 A Precision and accuracy (P&A) study is performed as an initial determination of capability, on an annual basis (continued demonstration of capability – a successful PT result may be used in place of a P&A for continued DOC), and if any significant changes have been made to the instrument. In general, 4 replicates or blank spikes are analyzed using the same procedures and conditions for sample analysis. The percent recovery and standard deviation (of the 4 replicate percent recoveries) are compared to in-house control limits. If percent recovery or standard deviation criteria are not met, corrective action must be taken to bring the system back into control.

11.11 Data assessment and acceptance criteria for quality control. Quality control limits are generated and updated on at least on an annual basis by QA using an in-house program. Blank spike, MS/MSD, and surrogate QC data are pooled for the previous year (or other specified time frame) and the data is processed and evaluated by QA. The new limits are entered into the LIMS where they can be viewed directly or by printing out a compound list (QC limits must be requested). The annual QC limit data is filed with QA.

12.0 DOCUMENTATION

12.1 The analytical logbook is a record of the analysis sequence; the logbook must be completed daily. Each instrument will have a separate logbook.

12.2 If samples require reanalysis, a brief explanation of the reason should be documented in this log. Dilutions must be recorded in the analysis logbook.

12.3 The standard preparation logbook must be completed for all standard preparations. All information requested must be completed; the page must be signed and dated by the respective person.

12.4 The Accutest lot number must be cross-referenced on the standard vial.

12.5 The instrument Maintenance logbook must be completed when any type of maintenance is performed on the instrument. Each instrument will have a separate log.

12.6 All laboratory logbooks must be reviewed and initialed or signed by the lab manager.

12.7 Any corrections to laboratory data must be done using a single line through the error. The initials of the person and date of correction must appear next to the correction.

13.0 DATA REVIEW

13.1 The analyst conducts the primary review of all data. This review begins with a check of all Instrument and method quality control and progresses through sample quality control concluding with a check to assure that the client's requirements have been executed. The analyst has the authority and responsibility to perform corrective action for any out-of-control parameter of non-conformance.

13.2 A secondary review is performed by department managers, and it includes review of the data produced by their department. All manual calculations, QC criteria, and a comparison of the data package to client specified requirements are checked. The department manager may reject data, initiate reanalysis, take additional corrective action, or reprocess data.

13.3 The laboratory director performs a full tertiary review of the data package following its assembly. This review includes an evaluation of QC data against acceptance criteria and a check of the data package contents to assure that all analytical requirements and specifications were executed.

13.4 Spot-check reviews are performed by the Quality Assurance Officer focusing on all elements of the deliverable including the client's specifications and requirements, analytical quality control, sample custody documentation and sample identification.

14.0 DATA REPORTING

14.1 A results page including positive results and/or RLs, units, methodology, surrogate recoveries, analysis dates, and data qualifiers are reported. Additional quality control data including calibration summaries, MS/MSD (or duplicate) percent recoveries and RPDs, blank spike recoveries, and method blank results may be reported upon request

of the client or program (MCP/RCP). Raw data may be reported to the client on request.

14.2 Data may be submitted to the client in a specified electronic format (EDD).

14.3 Once the data is approved by the laboratory manager, it may be accessed by clients via LabLink™.

14.4 Procedures for handling non-conforming data.

14.4.1 If quality control data does not meet criteria the non-conformance must be discussed in a case narrative and footnoted on the applicable quality control report summary.

14.4.2 If preservation or holding time criteria is not met and the samples are analyzed the result page must be footnoted with this information, and the non-conformance must be discussed in a case narrative or other suitable communication (telephone conversation log or email). Client notification documentation should be included with the data (telephone conversation log, fax, or email).

15.0 POLLUTION PREVENTION & WASTE MANAGEMENT

15.1 Pollution Prevention. Users of this method must perform all procedural steps that controls the creation and/or escape of wastes of hazardous materials to the environment. The amounts of standards, reagents, and solvents must be limited to the amounts specified in this SOP. All safety practices designed to limit the escape of vapors, liquids, or solids to the environment must be followed. All method users must be familiar with the waste management practices described in section 15.2

15.2 Waste Management. Individuals performing this method must follow established waste management procedures as described in the Sample and Waste Disposal SOP. This document describes the proper disposal of all waste materials generated during the testing of samples as follows:

15.2.1 Non-hazardous aqueous wastes
15.2.2 Hazardous aqueous wastes
15.2.3 Chlorinated organic solvents
15.2.4 Non-chlorinated organic solvents
15.2.5 Hazardous solid wastes
15.2.6 Non-hazardous solid wastes

16.0 METHOD PERFORMANCE

16.1 Method performance is evaluated by the annual quality control limits generated by QA, and the annual MDL study results. Refer to section 3.5 for MDLs, and section 11.10 for QC limits.

17.0 ADDITIONAL REFERENCES

17.1 SW846 Method 8000C, Revision 3, March, 2003; MA DEP WSC-CAM-IIB, Revision No. 4, 8/13/04; CT RCP SW846 8270C Version 2.0 , July, 2006

Table 1

RECOMMENDED OPERATING CONDITION (FULLL SCAN AND SIM)

Injection type	Split/Splitless
Split Injection Ratio	4:1 or 5:1
Injection Pulse Pressure	20 psi until 0.4 min.
Carrier Gas(linear velocity)	Helium at 1.5 mL/min
Septum purge flow	3 ml/min.
Mass range	35 - 550 amu
Electron Energy	70 volts (nominal)
Scan time	2 – 3 scans/sec

Injection port temperature	250 - 300° C
Source temperature	220 - 270° C
Transfer line temperature	300° C
Quadropole temperature	200° C
Initial temperature	45° C
Time 1	1 minutes
Column temperature rate	30 degrees C/min; 20 degrees C/min
Final temperature	according to column type; 325 C
Total run time	15 mins

Table 2

DFTPP KEY IONS AND ION ABUNDANCE CRITERIA

Mass	Ion Abundance Criteria
51	10-80% of Base Peak
68	<2 % of mass 69
70	<2 % of mass 69
127	10-80% of Base Peak
197	<2 % of mass 198
198	Base peak, or >50% of Mass 442
199	5-9 % of mass 198
275	10-60 % of Base Peak
365	>1 % of mass 198
441	Present but <24% of mass 442
442	Base Peak, or >50% of mass 198
443	15-24% of mass 442

Table 3

INTERNAL STANDARD

Internal standard	Prim/Sec. Ions
1,4-Dichlorobenzene-d4	152 / 150, 115
Naphthalene-d8	136 / 68
Acenaphthene-d10	164 / 162, 160
Phenanthrene-d10	188 / 94, 80
Chrysene-d12	240 / 120, 236
Perylene-d12	264 / 260, 265

Table 4

SURROGATES

BASE/NEUTRAL SURROGATES :

Compound	(Prim/Sec. ions)	Control limit(%) Water	Soil
Nitrobenzene-D5	(82/128,65)	Refer to current in-house limits	Refer to current in-house limits
2-Fluorobiphenyl	(172 / 171)		
Terphenyl-d14	(244/122,212)		

ACID SURROGATES :

Compound	(Prim/Sec. ions)	Control limit(%) Water	Soil
2-Fluorophenol	(112 / 64)	Refer to current in-house limits	Refer to current in-house limits
Phenol-d6	(99/ 42,71)		
2,4,6-Tribromophenol	(330/332,141)		

Table 5

Criteria for CCC and SPCC

Initial Calibration: Maximum % RSD for CCC is 30 %

Continuing Calibration: Maximum % D for CCC is 20 %

Minimum acceptable average relative response factor (RRF) is 0.050 for SPCC.

Calibration check compounds (CCC)

Base Neutral	Acid
1,4-Dichlorobenzene	Phenol
Hexachlorobutadiene	2,4-Dichlorophenol
Acenaphthene	2-Nitrophenol
Fluoranthene	p-Chloro-m-cresol
Di-n-octyl phthalate	Pentachlorophenol
Benzo(a)pyrene	2,4,6-Trichlorophenol
Diphenylamine	

System Performance Check Compounds (SPCC)

Base Neutral	Acid
N-Nitroso-di-n-propylamine	2,4-Dinitrophenol
Hexachlorocyclopentadiene	4-Nitrophenol

Table 6

MATRIX SPIKE RECOVERY and RSD LIMITS

Base Neutral fraction

Matrix Spike Compound	RPD * Water	RPD * Soil	% Recovery limit* Water	% Recovery limit* Soil
1,4-Dichlorobenzene	Refer to current in-house limits	----------	----------	----------
Acenaphthene				
2,4-Dinitrotoluene				
Pyrene				
N-Nitroso-di-N-propylamine				
1,2,4-Trichlorobenzene				

Acid fraction

Matrix Spike Compound	RPD * Water	RPD * Soil	% Recovery limit* Water	% Recovery limit* Soil
Phenol	Refer to current in-house limits	----------	----------	----------
2-Chlorophenol				
4-Chloro-3-methylphenol				
4-Nitrophenol				
Pentachlorophenol				

Table 7

Semivolatile Internal Standards with Corresponding Analytes Assigned for Quantitation

1,4-Dichlorobenzene-d4	ions	Naphthalene-d8	ions
Aniline	(93/66,65)	*Acetophenone	(105/77,51)
Benzyl alcohol	(108/79,77)	Benzoic acid	(184/92,185)
Bis(2-chloroethyl)ether	(93/63,95)	Bis(2-chloroethoxy)methane	(93/95,123)
Bis(2-chloroisopropyl)ether	(77/121)	4-chloroaniline	(129,65,92)
2-Chlorophenol	(128/64,130)	4-Chloro-3methylphenol	(107/144)
1,3-Dichlorobenzene	(146/148,111)	2,4-Dichlorophenol	(162/164,98)
1,4-Dichlorobenzene	(146/148,111)	*2,6-Dichlorophenol	(162/164,98)
1,2-Dichlorobenzene	(146/148,111)	2,4-Dimethylphenol	(122/107)
*Ethyl methanesulfonate	(79/109,97)	*a,a-Dimethyl-phenethylamine	(58/91,42)
2-Fluorophenol	(SURR.)	Hexachlorobutadiene	(225/223,227)
Hexachloroethane	(117/201,199)	Isophorone	(82/95,138)
*Methyl methanesulfonate	(80/79,64)	2-Methylnaphthalene	(142/141)
2-Methylphenol	(108/107,79)	Naphthalene	(128/129,127)
4-Methylphenol	(108/107,79)	Nitrobenzene	(77/123,65)
N-Nitrosodimethylamine	(74/42)	Nitrobenzene-d8	(SURR.)
N-Nitroso-di-n-propylamine	(70/101,130)	N-Nitroso-di-n-butylamine	(84/57/41)
Phenol-d6	(SURR.)	2-Nitrophenol	(139/109,65)
*2-Picoline	(93/66,92)	*N-Nitrosopiperidine	(42/114,55)
Phenol		1,2,4-Trichlorobenzene	(180/182,145)
pyridine			

* non-routine target compound

Table 7 (Continued)

Semivolatile Internal Standards with Corresponding Analytes Assigned for Quantitation

Acenaphthene-d10	ions
Acenaphthene	(154/153,152)
Acenaphthylene	(152/151,153)
*1-Chloronaphthalene	(162/127,164)
2-Chloronaphthalene	(162/127,164)
4-Chlorophenylphenyl ether	(204/206,141)
Dibenzofuran	(168/139)
Diethyl phthalate	(149/177,150)
Dimethyl phthalate	(163/149,164)
2,4-Dinitrophenol	(184/63,154)
2,4-Dinitrotoluene	(165/63,89)
2,6-Dinitrotoluene	(165/63,89)
Fluorene	(166/165,167)
Hexachlorocyclopentadiene	(237/235,272)
2-Fluorobiphenyl	(SURR.)
*1-Naphthylamine	(143/115,116)
*2-Naphthylamine	(143/115,116)
2-Nitroaniline	(65/92,138)
3-Nitroaniline	(138/108,92)
4-Nitroaniline	(138/108,92)
4-Nitrophenol	(139/109,65)
Pentachlorobenzene	(250/252,248)
*1,2,4,5-Tetrachlorobenzene	(216/214,218)
*2,3,4,6-Tetrachlorphenol	(232/230,131)
2,4,6-Tribromophenol	(SURR.)
2,4,6-Trichlorophenol	(196/198,200)
2,4,5-Trichlorophenol	(196/198,200)

Phenanthrene-d10	ions
*4-Aminobiphenyl	(169/168,170)
Anthracene	(178/176,179)
4-Bromophenyl phenyl ether	(248/250,141)
Di-n-Butyl phthalate	(149/150)
4,6-Dinitro-2-methylphenol	(198/51,105)
*Diphenylamine	(169/168,167)
1,2-Diphenylhydrazine	(77/105)
Fluoranthene	(202/101,203)
Hexachlorobenzene	(284/142,249)
N-Nitrosodiphenylamine	(169/168,167)
Pentachlorophenol	(266/264,268)
*Pentachloronitrobenzene	(295/237,142)
*Phenacetin	(108/109,179)
Phenanthrene	(178/179,176)
*Pronamide	(173/175,145)

Chrysene-d12	ions
Benzidine	(184/92,185)
Benzo(a)anthracene	(228/229/226)
Bis(2-ethylhexyl)phthalate	(149/167,279)
Butylbenzyl phthalate	(149/91)
Chrysene	(228/226,229)
3,3'-Dichlorobenzidine	

Perylene-d12	ions
Benzo(b)fluoranthene	(252/125)
Benzo(k)fluoranthene	(252/125)
Benzo(g.h.i)perylene	(276/138,277)
Benzo(a)pyrene	(252/253,125)
*Dibenz(a,j)acridine	(279/280)
Dibenz(a,h)anthracene	

	(252/254,126)		(278/139,279)
*p-Dimethylaminoazobenzene		*7,12-Dimethylbenz(a)anthracene	
	(120/225,77)		(256/241,257)
Pyrene	(202/200,203)	Indeno(1,2,3-d)pyrene	(276)
Terphenyl-d14	(SURR.)	*3-Methylchloanthrene	(268/253)
Di-n-octyl-phthalate	(167/43)		

* non-routine target compound

Table 8

REPORTING LIMITS (RL)

Compound	Cas Number	RL Water ug/l	RL Soil Ug/kg
Phenol	108-95-2	10	660
bis(2-Chloroethyl)ether	111-44-4	10	660
2-Chlorophenol	95-57-8	10	660
1,3-Dichlorobenzene	541-73-1	10	660
1,4-Dichlorobenzene	106-46-7	10	660
Benzyl Alcohol	100-51-6	20	1300
1,2-Dichlorobenzene	95-50-1	10	660
2-Methylphenol(o-Cresol)	95-48-7	10	660
bis(2-Chloroisopropyl)ether	39638-32-9	10	660
4-Methylphenol (p-Cresol)	106-44-5	10	660
N-Nitroso-Di-N-Propylamine	621-64-7	10	660
Hexachloroethane	67-72-1	10	660
Nitrobenzene	98-95-3	10	660
Isophorone	78-59-1	10	660
2-Nitrophenol	88-75-5	10	660
2,4-Dimethylphenol	105-67-9	10	660
Benzoic Acid	65-85-0	50	3300
bis(2-Chloroethoxy)methane	111-91-1	10	660
2,4-Dichlorophenol	120-83-2	10	660
1,2,4-Trichlorobenzene	120-82-1	10	660
Naphthalene	91-20-3	10	660
4-Chloroaniline	106-47-8	20	1300
Hexachlorobutadiene	87-68-3	10	660
4-Chloro-3-methylphenol	59-50-7	20	1300
2-Methylnaphthalene	91-57-6	10	660
Hexachlorocyclopentadiene	77-47-4	10	660
2,4,6-Trichlorophenol	88-06-2	10	660
2,4,5-Trichlorophenol	95-95-4	10	660
2-Chloronaphthalene	91-58-7	10	660
2-Nitroaniline	88-74-4	50	3300
Dimethyl phthalate	131-11-3	10	660
Acenaphthylene	208-96-8	10	660
3-Nitroaniline	99-09-2	50	3300
Acenaphthene	83-32-9	10	660
2,4-Dinitrophenol	51-28-5	50	3300
4-Nitrophenol	100-02-7	50	3300
Dibenzofuran	132-64-9	100	660
2,4-Dinitrotoluene	121-14-2	10	660
2,6-Dinitrotoluene	606-20-2	10	660

Table 8 (Continued)

REPORTING LIMITS (RL)

Compound	Cas Number	RL Water ug/l	RL Soil Ug/kg
Diethylphthalate	84-66-2	10	660
4-Chlorophenyl phenyl ether	7005-72-3	10	660
Fluorene	86-73-7	10	660
4-Nitroaniline	100-01-6	50	3300
4,6-Dinitro-2-methylphenol	534-52-1	50	3300
Nitrosodiphenylamine	86-30-6	10	660
4-Bromophenyl phenyl ether	101-55-3	10	660
Hexachlorobenzene	118-74-1	10	660
Pentachlorophenol	87-86-5	50	3300
Phenanthrene	85-01-8	10	660
Anthracene	120-12-7	10	660
Di-n-butylphthalate	84-74-2	10	660
Fluoranthene	206-44-0	10	660
Pyrene	129-00-0	10	660
Butyl benzyl phthalate	85-68-7	10	660
3,3'-Dichlorobenzidine	91-94-1	20	1300
Benzo(a)anthracene	56-55-3	10	660
bis(2-ethylhexyl)phthalate	117-81-7	10	660
Chrysene	218-01-9	10	660
Di-n-octyl phthalate	117-84-0	10	660
Benzo(b)fluoranthene	205-99-2	10	660
Benzo(k)fluoranthene	207-08-9	10	660
Benzo(a)pyrene	50-32-8	10	660
Indeno(1,2,3-cd)pyrene	193-39-5	10	660
Dibenz(a,h)anthracene	53-70-3	10	660
Benzo(g,h,i)perylene	191-24-2	10	660

Table 9

CALIBRATION/SPIKE STANDARD PREPARATION

	Calibration Level(ug/ml)						
A/BN calibration standard	1	5	10	50	80	120	160
CLP-HC-BN-R (2000 ug/ml)				25	40	60	80
Z-014F (2000 ug/ml)	6.25*			25	40	60	80
Z-014E-R3 (2000 ug/ml)		31.25*		25	40	60	80
CLP-HC-AR (2000 ug/ml)			62.5*	25	40	60	80
APP-9-050-10X (1000ug/ml)				50	80	120	160
M8270SS (4000 ug/ml)				12.5	20	30	40
Methylene Chloride	993.75	968.75	937.5	837.5	740	610	480
total vol.	1000	1000	1000	1000	1000	1000	900

All spike amounts in microliters
*** add 10 ul I.S. (4000 ug/ml) into 1000 ul total mixture ***
* amount of 160 ug/ml spiked prior to adding Internal Standard

Table 10

Compound List Report
Matrix: AQ Aqueous

Method List: AB8270 AQ

Report List: ABAIX ALL
RL/MDL Factor: 1

Method Ref: SW846 8270C
ABN Appendix 9
List

Compound	CAS No.
2-Chlorophenol	95-57-8
4-Chloro-3-methyl phenol	59-50-7
2,4-Dichlorophenol	120-83-2
2,4-Dimethylphenol	105-67-9
2,4-Dinitrophenol	51-28-5
2,6-Dichlorophenol	87-65-0
4,6-Dinitro-o-cresol	534-52-1
Dinoseb	88-85-7
2-Methylphenol	95-48-7
3&4-Methylphenol	
2-Nitrophenol	88-75-5
4-Nitrophenol	100-02-7
Pentachlorophenol	87-86-5
Phenol	108-95-2
2,3,4,6-Tetrachlorophenol	58-90-2
2,4,5-Trichlorophenol	95-95-4
2,4,6-Trichlorophenol	88-06-2
2-Acetylaminofluorene	53-96-3
4-Aminobiphenyl	92-67-1
Acenaphthene	83-32-9
Acenaphthylene	208-96-8
Acetophenone	98-86-2
Aniline	62-53-3
Anthracene	120-12-7
Aramite	140-57-8
A,A-Dimethylphenethylamine	122-09-8
Benzo(a)anthracene	56-55-3

Benzo(a)pyrene	50-32-8
Benzo(b)fluoranthene	205-99-2
Benzo(g,h,i)perylene	191-24-2
Benzo(k)fluoranthene	207-08-9
4-Bromophenyl phenyl ether	101-55-3
Butyl benzyl phthalate	85-68-7
Benzyl Alcohol	100-51-6
2-Chloronaphthalene	91-58-7
4-Chloroaniline	106-47-8
Chlorobenzilate	510-15-6
Chrysene	218-01-9
bis(2-Chloroethoxy)methane	111-91-1
bis(2-Chloroethyl)ether	111-44-4
bis(2-Chloroisopropyl)ether	108-60-1
4-Chlorophenyl phenyl ether	7005-72-3
1,2-Dichlorobenzene	95-50-1
1,3-Dichlorobenzene	541-73-1
1,4-Dichlorobenzene	106-46-7
2,4-Dinitrotoluene	121-14-2
2,6-Dinitrotoluene	606-20-2
3,3'-Dichlorobenzidine	91-94-1
3,3'-Dimethylbenzidine	119-93-7
7,12-Dimethylbenz(a)anthracene	57-97-6
Diallate	2303-16-4
Dibenzo(a,h)anthracene	53-70-3
Dibenzofuran	132-64-9
Dimethoate	60-51-5
Diphenylamine	122-39-4
Disulfoton	298-04-4
m-Dinitrobenzene	99-65-0
p-(Dimethylamine)azobenzene	60-11-7
Di-n-butyl phthalate	84-74-2
Di-n-octyl phthalate	117-84-0
Diethyl phthalate	84-66-2
Dimethyl phthalate	131-11-3
bis(2-Ethylhexyl)phthalate	117-81-7
Ethyl methanesulfonate	62-50-0
Famphur	52-85-7
Fluoranthene	206-44-0
Fluorene	86-73-7

Hexachlorobenzene	118-74-1
Hexachlorobutadiene	87-68-3
Hexachlorocyclopentadiene	77-47-4
Hexachloroethane	67-72-1
Hexachlorophene	70-30-4
Hexachloropropene	1888-71-7
Indeno(1,2,3-cd)pyrene	193-39-5
Isodrin	465-73-6
Isophorone	78-59-1
Isosafrole	120-58-1
Kepone	143-50-0
2-Methylnaphthalene	91-57-6
3-Methylcholanthrene	56-49-5
Methapyrilene	91-80-5
Methyl methanesulfonate	66-27-3
Methyl parathion	298-00-0
1,4-Naphthoquinone	130-15-4
1-Naphthylamine	134-32-7
2-Naphthylamine	91-59-8
2-Nitroaniline	88-74-4
3-Nitroaniline	99-09-2
4-Nitroaniline	100-01-6
5-Nitro-o-toluidine	99-55-8
Naphthalene	91-20-3
Nitrobenzene	98-95-3
n-Nitrosodimethylamine	62-75-9
4-Nitroquinoline 1-Oxide	56-57-5
N-Nitroso-di-n-propylamine	621-64-7
N-Nitrosodi-n-butylamine	924-16-3
N-Nitrosodiethylamine	55-18-5
N-Nitrosodiphenylamine	86-30-6
N-Nitrosomethylethylamine	10595-95-6
N-Nitrosomorpholine	59-89-2
N-Nitrosopiperidine	100-75-4
N-Nitrosopyrrolidine	930-55-2
O,O,O-Triethyl phosphorothioat	126-68-1
2-Picoline	109-06-8
Parathion	56-38-2
Pentachlorobenzene	608-93-5
Pentachloroethane	76-01-7

Pentachloronitrobenzene	82-68-8
Phenacetin	62-44-2
Phenanthrene	85-01-8
Phorate	298-02-2
Pronamide	23950-58-5
Pyrene	129-00-0
Pyridine	110-86-1
p-Phenylenediamine	106-50-3
Safrole	94-59-7
1,2,4,5-Tetrachlorobenzene	95-94-3
1,2,4-Trichlorobenzene	120-82-1
Thionazin	297-97-2
o-Toluidine	95-53-4
sym-Trinitrobenzene	99-35-4
Tetraethyl dithiopyrophosphate	3689-24-5
122 compounds reported in list ABAIX	

Table 11

Compound List Report
Product: AB8270PPL Semivolatiles, PPL
Matrix: AQ Aqueous

Method List: AB8270 AQ
Report List: ABPPL ALL
RL/MDL Factor: 1

Method Ref: SW846 8270C
ABN PPL List

Compound	CAS No.
2-Chlorophenol	95-57-8
4-Chloro-3-methyl phenol	59-50-7
2,4-Dichlorophenol	120-83-2
2,4-Dimethylphenol	105-67-9
2,4-Dinitrophenol	51-28-5
4,6-Dinitro-o-cresol	534-52-1
2-Nitrophenol	88-75-5
4-Nitrophenol	100-02-7
Pentachlorophenol	87-86-5
Phenol	108-95-2
2,4,6-Trichlorophenol	88-06-2
Acenaphthene	83-32-9
Acenaphthylene	208-96-8
Anthracene	120-12-7
Benzidine	92-87-5
Benzo(a)anthracene	56-55-3
Benzo(a)pyrene	50-32-8
Benzo(b)fluoranthene	205-99-2
Benzo(g,h,i)perylene	191-24-2
Benzo(k)fluoranthene	207-08-9
4-Bromophenyl phenyl ether	101-55-3
Butyl benzyl phthalate	85-68-7
2-Chloronaphthalene	91-58-7
4-Chloroaniline	106-47-8
Chrysene	218-01-9
bis(2-Chloroethoxy)methane	111-91-1
bis(2-Chloroethyl)ether	111-44-4
bis(2-Chloroisopropyl)ether	108-60-1
4-Chlorophenyl phenyl ether	7005-72-

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1,2-Dichlorobenzene	95-50-1
1,2-Diphenylhydrazine	122-66-7
1,3-Dichlorobenzene	541-73-1
1,4-Dichlorobenzene	106-46-7
2,4-Dinitrotoluene	121-14-2
2,6-Dinitrotoluene	606-20-2
3,3'-Dichlorobenzidine	91-94-1
Dibenzo(a,h)anthracene	53-70-3
Di-n-butyl phthalate	84-74-2
Di-n-octyl phthalate	117-84-0
Diethyl phthalate	84-66-2
Dimethyl phthalate	131-11-3
bis(2-Ethylhexyl)phthalate	117-81-7
Fluoranthene	206-44-0
Fluorene	86-73-7
Hexachlorobenzene	118-74-1
Hexachlorobutadiene	87-68-3
Hexachlorocyclopentadiene	77-47-4
Hexachloroethane	67-72-1
Indeno(1,2,3-cd)pyrene	193-39-5
Isophorone	78-59-1
Naphthalene	91-20-3
Nitrobenzene	98-95-3
n-Nitrosodimethylamine	62-75-9
N-Nitroso-di-n-propylamine	621-64-7
N-Nitrosodiphenylamine	86-30-6
Phenanthrene	85-01-8
Pyrene	129-00-0
1,2,4-Trichlorobenzene	120-82-1
58 compounds reported in list ABPPL	

Table 12

Compound List Report
Product: AB8270TCL Semivolatiles, TCL
Matrix: AQ Aqueous

Method List: AB8270 AQ
Report List: ABTCL ALL
RL/MDL Factor: 1

Method Ref: SW846 8270C
ABN TCL List

Compound	CAS No.
Benzoic Acid	65-85-0
2-Chlorophenol	95-57-8
4-Chloro-3-methyl phenol	59-50-7
2,4-Dichlorophenol	120-83-2
2,4-Dimethylphenol	105-67-9
2,4-Dinitrophenol	51-28-5
4,6-Dinitro-o-cresol	534-52-1
2-Methylphenol	95-48-7
3&4-Methylphenol	
2-Nitrophenol	88-75-5
4-Nitrophenol	100-02-7
Pentachlorophenol	87-86-5
Phenol	108-95-2
2,4,5-Trichlorophenol	95-95-4
2,4,6-Trichlorophenol	88-06-2
Acenaphthene	83-32-9
Acenaphthylene	208-96-8
Anthracene	120-12-7
Benzo(a)anthracene	56-55-3

Benzo(a)pyrene	50-32-8
Benzo(b)fluoranthene	205-99-2
Benzo(g,h,i)perylene	191-24-2
Benzo(k)fluoranthene	207-08-9
4-Bromophenyl phenyl ether	101-55-3
Butyl benzyl phthalate	85-68-7
Benzyl Alcohol	100-51-6
2-Chloronaphthalene	91-58-7
4-Chloroaniline	106-47-8
Carbazole	86-74-8
Chrysene	218-01-9
bis(2-Chloroethoxy)methane	111-91-1
bis(2-Chloroethyl)ether	111-44-4
bis(2-Chloroisopropyl)ether	108-60-1
4-Chlorophenyl phenyl ether	7005-72-3
1,2-Dichlorobenzene	95-50-1
1,3-Dichlorobenzene	541-73-1
1,4-Dichlorobenzene	106-46-7
2,4-Dinitrotoluene	121-14-2
2,6-Dinitrotoluene	606-20-2
3,3'-Dichlorobenzidine	91-94-1
Dibenzo(a,h)anthracene	53-70-3
Dibenzofuran	132-64-9
Di-n-butyl phthalate	84-74-2
Di-n-octyl phthalate	117-84-0
Diethyl phthalate	84-66-2
Dimethyl phthalate	131-11-3
bis(2-Ethylhexyl)phthalate	117-81-

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Fluoranthene	206-44-0
Fluorene	86-73-7
Hexachlorobenzene	118-74-1
Hexachlorobutadiene	87-68-3
Hexachlorocyclopentadiene	77-47-4
Hexachloroethane	67-72-1
Indeno(1,2,3-cd)pyrene	193-39-5
Isophorone	78-59-1
2-Methylnaphthalene	91-57-6
2-Nitroaniline	88-74-4
3-Nitroaniline	99-09-2
4-Nitroaniline	100-01-6
Naphthalene	91-20-3
Nitrobenzene	98-95-3
N-Nitroso-di-n-propylamine	621-64-7
N-Nitrosodiphenylamine	86-30-6
Phenanthrene	85-01-8
Pyrene	129-00-0
1,2,4-Trichlorobenzene	120-82-1
66 compounds reported in list ABTCL	

Note: 1,4-Dioxane is also reported by this SOP (SIM).

Table 9
Minimum RFs

Compound	Minimum RF
Benzadehyde	0.010
Phenol	0.800
Bis(2-chloroethyl)ether	0.700
2-Chlorophenol	0.800
2-Methylphenol	0.700
2,2-Oxybi-(1-chloropropane)	0.010
Acetophone	0.010
4-Methylphenol	0.600
N-Nitroso-di-n-propylamine	0.500
Hexachloroethane	0.300
Nitrobenzene	0.200
Isophorone	0.400
2-Nitrophenol	0.100
2,4-Dimethylphenol	0.200
Bis(2-chloroethoxy)methane	0.300
2,4-Dichlorophenol	0.200
Naphthalene	0.700
4-Chloroaniline	0.010
Hexachlorobutadiene	0.010
Caprolactam	0.010
4-Chloro-3-methylphenol	0.200
2-Methylnaphthalene	0.400
Hexachlorocyclopentadiene	0.050
2,4,6-Trichlorophenol	0.200
2,4,5-Trichlorophenol	0.200
1,1'-Biphenyl	0.010
2-Chloronaphthalene	0.800
2-Nitroaniline	0.010
Dimethyl phthalate	0.010
2,6-Dinitrotoluene	0.200
Acenaphthylene	0.900
3-Nitroaniline	0.010
Acenaphthene	0.900
2,4-Dinitrophenol	0.010
4-Nitrophenol	0.010

Dibenzofuran	0.800
2,4-Dinitrotoluene	0.200
Diethyl phthalate	0.010
1,2,4,5-Tetrachlorobenzene	0.010
4-Chlorophenyl-phenyl ether	0.400
Fluorene	0.900
4-Nitroaniline	0.010
4,6-Dinitro-2-methylphenol	0.010
4-Bromophenyl-phenyl ether	0.100
N-Nitrosodiphenylamine	0.010
Hexachlorobenzene	0.100
Atrazine	0.010
Pentachlorophenol	0.050
Phenanthrene	0.700
Anthracene	0.700
Carbazole	0.010
Din-n-butyl phthalate	0.010
Fluoranthene	0.600
Pyrene	0.600
Butylbenzylphthalate	0.010
3,3'-Dichlorobenzidine	0.010
Benzo(a)anthracene	0.800
Chrysene	0.700
Bis-(2-ethylhexyl)phthalate	0.010
Di-n-octyl phthalate	0.010
Benzo(b)fluoranthene	0.700
Benzo(k)fluoranthene	0.700
Benzo(a)pyrene	0.700
Indeno(1,2,3-cd)pyrene	0.500
Dibenz(a,h)anthracene	0.400
Benzo(g,h,i)perylene	0.500
2,3,4,6-Tetrachlorophenol	0.010

Laboratory Manager: Doug Yargeau

QA Officer: Robert Treggiari

TITLE: PREPARATION OF SOLIDS MATRICES BY ULTRASONIC EXTRACTION

TEST METHOD SW846 Method 3550B (Rev. 2 December 1996)

REVISED SECTIONS: removed section 14.0, revised 7.3.8, 9.6, 9.12, 9.13

1.0 SCOPE AND APPLICATION

1.1 This method is a procedure for extracting non-volatile and semivolatile organic compounds from solids such as soils, sludges, and wastes. The ultrasonic process ensures intimate contact of the sample matrix with the extraction solvent.

1.2 This method is divided into two sections based on the expected concentration of organics in the sample. The low concentration method (individual organic components of less than or equal to 20 mg/kg) uses a larger sample size and a more rigorous extraction procedure (lower concentrations are more difficult to extract). The medium/high concentration method (individual organic components of greater than 20 mg/kg) is much simpler and therefore faster.

1.3 Ultrasonic extraction is not as rigorous as other extraction methods for soils/solids. Therefore, it is critical that the method (including the manufacturers' instructions) be followed explicitly in order to achieve the maximum extraction efficiency.

1.4 Test Code: See the specific determinative method.

2.0 SUMMARY

2.1 Low concentration method

2.1.1 A 30g sample is mixed with anhydrous sodium sulfate to form a free-flowing powder. This is solvent extracted three times using ultrasonic extraction. The extract is separated from the sample by vacuum filtration or centrifugation. The extract is ready for cleanup and/or analysis following concentration.

2.2 Medium/high concentration method

2.2.1 A 2-g sample is mixed with anhydrous sodium sulfate to form a free-flowing powder. This is solvent extracted once using ultrasonic extraction. A portion of the extract is removed for cleanup and/or analysis.

3.0 METHOD DETECTION LIMIT

3.1 The reporting limit (RL) is based on the lowest calibration standard. RL's may vary depending on matrix difficulties, sample volumes or weights, and percent moisture. Detected concentrations below this concentration cannot be reported without qualification.

3.2 The Method Detection Limit (MDL) represents the lowest reportable concentration of an individual analyte that meets the method qualitative identification criteria.

3.3 Method Detection limits (MDLs) are experimentally determined using the procedures described in 40 CFR, Part 136, Appendix B. Actual reported MDLs incorporate the sample volume analyzed and sample dilutions if needed, which may cause MDL variations from sample to sample.

3.4 In general, MDLs are determined through the analysis of at least 7 replicate blank spikes (using the same procedures for sample analysis). The MDL is calculated by multiplying the standard deviation of the replicate concentrations by the appropriate Student's t value (3.143 for 7 replicates). If more than 7 replicates are analyzed refer to 40 CFR, Part 136, Appendix B for the appropriate student's t value. MDLs are determined initially (prior to analysis), on an annual basis, and after major maintenance to equipment. MDL data is archived with Quality Assurance. Refer to the most recent study for current MDLs.

3.5 See the determinative method for detection limits.

4.0 DEFINITIONS

4.1 ALIQUOT - a measured portion of a sample, or solution, taken for sample preparation and/or analysis.

4.2 BATCH – A group of samples which behave similarly with respect to the sampling or the testing procedures being employed and which are processed as a unit. For QC purposes, if the number of samples in a group is greater than 20 (or 10 for certain methods), then each group of 20 samples (or 10 samples for certain methods) or less will all be handled as a separate batch.

4.3 DRY WEIGHT – the weight of a sample based on percent solids. The weight after drying in an oven.

4.4 PERCENT MOISTURE - an approximation of the amount of water in a soil/sediment sample made by drying an aliquot of the sample at 105 °C. The percent moisture determined in this manner also includes contributions from all compounds that may volatilize at or below 105 °C, including water. Percent moisture may be determined from decanted samples and from samples that are not decanted.

4.5 EXTRACTABLE - a compound that can be partitioned into an organic solvent from the sample matrix and is amenable to gas chromatography. Extractables include semivolatile (BNA) and pesticide/Aroclor compounds.

4.6 FIELD SAMPLE - a portion of material to be analyzed that is contained in single or multiple containers and identified by a unique sample number.

4.7 FIELD BLANK – this is any sample that is submitted from the field and is identified as a blank. This includes rinsates and equipment blanks, etc.

4.8 HOLDING TIME – the elapsed time expressed in days from the date of sampling until the date of its analysis.

4.9 INSUFFICIENT QUANTITY - when there is not enough weight (soil/sediment) to perform any of the required operations: sample analysis or extraction, percent moisture, MS/MSD, etc.

4.10 MATRIX - the predominant material of which the sample to be analyzed is composed. For the purpose of this SOP, a sample matrix is either water or soil/sediment. Matrix is not synonymous with phase (or solid).

4.11 MATRIX EFFECT - in general, the effect of a particular matrix (soil/sediment) on the constituents with which it contacts. This is particularly pronounced for clay particles which may adsorb chemicals and catalyze reactions. Matrix effects may prevent extraction of target analytes, and may affect surrogate recoveries. In addition, non-target analytes may be extracted from the matrix causing interferences.

4.12 MATRIX SPIKE - aliquot of a matrix (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.

4.13 MATRIX SPIKE DUPLICATE - a second aliquot of the same matrix as the matrix spike (above) that is spiked in order to determine the precision of the method.

4.14 MATRIX DUPLICATE – a second aliquot of the original sample that is spiked in order to determine the precision of the method.

4.15 METHOD BLANK - an analytical control consisting of all reagents, internal standards, and surrogate standards, which are carried throughout the entire analytical procedure. The method blank is used to define the level of laboratory, background, and reagent contamination.

4.16 REAGENT WATER - water in which an interferant is not observed at or above the minimum quantitation limit of the parameters of interest.

4.17 SURROGATES (Surrogate Standard) - for semivolatiles and pesticides/Aroclors, compounds added to every blank, sample, matrix spike, matrix spike duplicate, and standard; used to evaluate analytical efficiency by measuring recovery. Surrogates are brominated, fluorinated, or isotopically labeled compounds not expected to be detected in environmental media.

5.0 HEALTH AND SAFETY

5.1 All safety practices must be followed as outlined in the Accutest Laboratories Employee Safety Handbook and Chemical Hygiene Plan. Safety glasses, gloves, and lab coats must be worn. All samples, solutions, and extracts must be treated as unknown and potentially hazardous.

5.2 The toxicity or carcinogenicity of each reagent used in this method has not been precisely determined; however, each chemical should be treated as a potential health hazard. Exposure to these reagents should be reduced to the lowest possible level.

5.3 All extractions and the use of organic solvent must be performed under a ventilation hood.

5.4 Use caution when operating the muffle furnace – extremely hot.

6.0 COLLECTION, PRESERVATION, AND HOLDING TIMES

6.1 See the specific determinative method for preservation and holding time requirements. Generally, these requirements are as follows:

6.1.1 Preservation. Samples are collected in a 300-ml amber glass sample bottle. Samples should be stored a 4°C until extraction.

6.1.2 Samples must be extracted within 14 days of sampling and must be analyzed within 40 days of extraction.

7.0 APPARATUS AND MATERIALS

7.1 Apparatus

7.1.1 Sonicator/sonic disrupter, 300 watt output minimum, pulsing capability, and with soundproof casing. Appropriate tips (micro tip and standard)

7.1.1.1 The sonicator must be tuned to manufacturer's specifications prior to each use.

7.1.2 Evaporating steam baths.

7.1.3 Nitrogen blow down apparatus.

7.1.4 Zymark Turbovap® concentrator

7.2 Glassware

7.2.1 Beakers - 150 ml.

7.2.2 Drying column – glass funnel.

7.2.3 Kuderna-Danish evaporator with 10 ml graduated concentrator tube.

7.2.4 Three-ball Snyder column.

7.2.5 2-ml crimp vials (with crimper).

7.2.6 Glass funnels.

7.2.7 Whatman 41 filter papers (or equivalent).

7.2.8 Zymark concentrator tubes.

7.2.9 Pasteur pipettes.

7.3 Standards and Reagents

7.3.1 Methylene chloride - reagent grade for trace organic analysis.

7.3.2 Sodium sulfate, granular anhydrous – Baked (muffled) at 400°C for at least 4 hours.

7.3.3 Nitrogen gas - purified, supplied by Air Products with valves attached to each ventilation hood.

7.3.4 Hexane – reagent grade for pesticide residue analysis.

7.3.5 Acetone – reagent grade for trace organic analysis.

7.3.6 1:1 Acetone/Methylene Chloride – mix equal parts of each solvent.

7.3.7 Spiking solutions appropriate for the specific extraction method.

7.3.8 All chemicals listed are reagent grade unless otherwise specified. Distilled, deionized water should be used whenever water is required. All applicable standard/reagent preparation information, including vendor, lot number, date of preparation, expiration date, calculations, and initials must be entered in the appropriate standard/reagent preparation logbook. Vendors typically used by Accutest include Fisher Scientific, VWR, Absolute Standards, Accustandard, Supelco, Chemservices, CPI, Ultra, and ERA. Additional vendors may be utilized as necessary.

8.0 INTERFERENCES

8.1 Method interferences may be caused by contaminants in the solvent reagents and glassware. Glassware must be scrupulously cleaned as outlined in “The Procedure for Washing Glassware” SOP. Solvents and reagents must be of the purest grade and monitored by the lot numbers. Solvents may be checked by concentrating them and analyzing them by GC to check for trace solvent contaminants.

8.2 Matrix interferences may be caused by contaminants that are coextracted from the sample. The extent of matrix interferences will vary from source to source. Interferences such as sulfur and phthalate may be treated with copper and alumina by organics preparation respectively.

8.3 Avoiding interferences with any plastic materials and checking all solvents and reagents for phthalate contamination can best minimize interference from phthalate esters.

9.0 PROCEDURE

9.1 Locate the soil samples for analysis. For every 20 samples, a matrix spike sample, matrix spike duplicate sample, spike blank and method blank. The method blank and spike blank are empty beakers, which should be processed as samples beginning with 9.5. See specific extraction method for required quality control samples.

Note: For PCB's in Oils the spiking matrix should be PCB free oil, such as transformer oil.

9.2 Each day, prior to use, the sonicator must be tuned per manufacturer's instructions. Daily tunes must be recorded in the logbook.

9.3 Decant and discard any water layer and mix sediment sample thoroughly. Discard any foreign objects such as sticks, leaves and rocks. Homogenize the sample in its container by mixing with a spatula. Refer to the Representative Aliquot SOP (MQA247) for details.

9.4 Weigh out the 2g (medium high concentration method) - and use micro tip - or 30g (low concentration method) – and use standard tip - weight of wet weight soil (per specific method) into a tared 150-ml beaker and record the weight to the nearest 0.01 g. Samples that do not have a free flowing texture (sandy) are mixed with approximately 60-g anhydrous sodium sulfate to form a free flowing mixture. A sample should be weighed in triplicate for the batch matrix spike and matrix spike duplicate.

Note: For PCB Oils, samples must be weighed to the nearest 0.0001g

Note: the micro tip must be used for medium to high level extraction and the standard tip must be used for low level extraction – they are <u>not</u> interchangeable.

9.5 A beaker with an aliquot of Ottawa sand (similar to the sample size being extracted) approximately 2-5 g (medium to high level method) or 20-30 g (low level method) of anhydrous sodium sulfate is used as a method blank and spike blank and should be treated as a sample.

9.6 Three extractions are performed with the appropriate solvents and the required horn tip for the testing desired. For the first extraction; add 100 ml of 1:1methylene chloride/acetone to each beaker under the hood. Proceed through the following steps and perform extractions 2 and 3 in step 9.10

9.7 Spike all QC and field samples with the appropriate surrogate (specific to the determinative method) each beaker. Also, add the appropriate matrix spike solution to the replicate aliquots of a sample you have designated for matrix spike, matrix spike duplicate and the blank spike.

NOTE: Spike Witnesses are required for all spiking procedures. The spike witness is required to verify that the addition of the correct spiking solution, volume, and concentration has occurred. The initials of the spike witness must be documented in the logbook within the spike witness field. For occasions where a spike witness is not present (weekends and/or evening shifts) the spike witness field must be given an "NA" (Not Applicable) designation and verified by a supervisor or manager when available.

9.8 Place the tip of the horn ½ inch below the solvent surface (but above the sample). Sonicate each sample for 3 minutes at 50% duty cycle and an output of 10 – pulse energy. Check with your supervisor on how to make adjustments. Very active mixing of the sample and the solvent must occur when the ultrasonic pulse is activated. The analyst must observe such mixing at some point during the extraction process.

9.9 Place a prepared Kuderna Danish apparatus under the drying funnel to collect the sample extract. Take the sample extract contained in the 150-ml beaker and pour it into the top of the drying funnel through a funnel containing filter paper and sodium sulfate. Make sure all apparatus is labeled accordingly.

9.10 Add a second 100-ml volume of 1:1 acetone/methylene chloride to beakers and sonicate a second time. Decant the extract into the same drying column and combine the extracts in the K-D flask. Perform a third extraction in the same manner.

9.11 After the final extract has passed through the sodium sulfate column, rinse down the funnel and the inside of the column with an excess of 30-ml of methylene chloride. Add a glass-boiling bead to the K-D flask and attach a three-ball Snyder column, prime the Snyder column by adding 1 ml of methylene chloride through the top.

9.12 Position the K-D concentrator setup on the hot water bath with only the 10-ml concentrator tube submersed in the water. Concentration process should be completed in 10 – 15 minutes. Adjust the column height and/or water bath temperature (80 - 90°C) accordingly. Proper distillation rate should result in an active chartering but the column chambers should not flood with solvent. Evaporate the extract to 1 – 2ml not allowing it to go to dryness. Remove the K-D apparatus from the bath and allow the solvents condensed in the Snyder column to drip back down into the concentrator tube. Allow the solvent to cool for approximately 10 minutes.

9.13 If solvent exchange is necessary, add the appropriate amount of solvent specific to the extraction method to the cooled methylene chloride and reconcenetrate the extract to less than 1 - 2ml. Aluminum foil may be placed around the lower rounded surface of the K-D flask to aid evaporation. Allow the solvent to cool and remove the Snyder column and K-D flask, while rinsing the connector joints with approximately 1 ml of hexane into the receiver. Bring the extract to the volume appropriate to the specific extraction method and mix well.

9.14 Bring the extract to volume appropriate to the specific extraction method and mix well. Proceed to sample cleanup (see specific extraction method) or transfer to 2 GC auto sampler vials with PTFE-lined crimp caps and label with sample ID and batch number. Store at 4°C ±2°C until analysis.

9.15 If further concentration is necessary, the nitrogen blow down technique may be used.

9.15.1 Place the concentrator tube in a warm bath (35°C) and evaporate the solvent to the final volume as specified by the specific extraction method using a gentle stream of clean, dry nitrogen (filtered through a column of activated carbon).

9.15.2 The internal wall of the tube must be rinsed several times with methylene chloride or appropriate solvent during the operation. During evaporation, the tube must be positioned to avoid water condensation (i.e., the solvent level should be below the level of the water bath). The extract must not be allowed to become dry.

Caution: When the volume of solvent is reduced below 1ml, semivolatile analytes may be lost.

9.16 Concentration using Zymark. Sample extracts may be concentrated by using the Zymark Turbovap® concentrator.

9.16.1 Prepare the Zymark by:

9.16.1.1 Check incoming gas supply (sufficient reserve and turned on).
9.16.1.2 Check water bath level (> sample level).
9.16.1.3 With cover down, turn on unit and set BATH TEMP.
9.16.1.4 Select the TEMPERATURE display (35°C - 40°C).
9.16.1.5 Allow bath to come to temperature (display stops blinking)
9.16.1.6 Fill concentrator tubes with samples.
9.16.1.7 Open cover and place tubes in water bath.
9.16.1.8 Close cover

9.16.2 Start Concentration

9.16.2.1 Press ENDPOINT SELECT until desired mode is selected (Sensor).
9.16.2.2 Set ENDPOINT TIME (NA).
9.16.2.3 Press SELECT DISPLAYED CONDITION until pressure is selected. Pull out regulator knob, adjust gas pressure, and push in to lock.
9.16.2.4 Press the START/STOP button for each cell position used.

9.16.3 Stop Concentration

9.16.3.1 To pause concentration for all tubes, raise the cover. To continue operation, lower the cover.
9.16.3.2 To stop concentration process for an individual cell (running manually or need to stop and choose one of the other operation modes), press the START/STOP key for the corresponding cell. CAUTION: This clears out the TIME ELAPSED.
9.16.3.3 Otherwise, when a cell reaches its selected endpoint, its corresponding light blinks and the beeper sounds briefly every 30 seconds.
9.16.3.4 Raise the cover to silence the beeper.

Rapid beeping during concentration indicates low gas pressure – check your gas supply.

9.16.3.5 For extracts that require a solvent exchange – add the appropriate volume of method-specific solvent.
9.16.3.6 Concentrate to <10 mls > 1.0 ml.
9.16.3.7 Remove the sample

9.16.4 Remove the sample

9.16.4.1 Prompt removal of the completed tube is important since highly volatile compounds may be lost of allowed to sit for an extended period of time, and steam from a water bath of over 50°C can condense on the cover and possibly drip into sample tubes if they are left in place.

9.16.5 Add the appropriate solvent to volume. Mix thoroughly.

9.16.6 Shut down

9.16.6.1 Replace the plastic closures in each cell position.
9.16.6.2 Leave the cover open to keep moisture from accumulating on the inside cover.
9.16.6.3 Clean the concentrator tubes according to procedures described in "The Procedures for Washing Glassware" SOP.
9.16.6.4 Turn the power and gas supply off (optional).

9.16.7 Proceed to cleanup procedures, if necessary (see specific extraction method), or transfer the extract to 2 GC auto sampler vials with PTFE-lined crimp caps. Store extracts according the specific method until analysis.

10.0 QUALITY ASSURANCE

10.1 Refer to the specific determinative method for details (including corrective actions).

10.2 The following QC must be extracted with each batch of samples (20 or less):

10.2.1 Method blank
10.2.2 Blank spike
10.2.3 Matrix spike/matrix spike duplicate

Note: For PCB Oils a sample batch is 10 samples with the same QC requirements described above.

10.3 Each sample and QC sample must be spiked with surrogate solution.

10.4 Prior to running samples, the laboratory must demonstrate initial proficiency by generating data of acceptable accuracy and precision (P&A study) for target analytes in a clean matrix (Ottawa sand). This procedure must be repeated on an annual basis, whenever new staff are trained, or when significant changes in instrumentation are made. Four blank spikes are prepared and analyzed (see determinative method) using the same procedures and conditions as samples. Calculate the average recovery and standard deviation of the recoveries of the analytes in each of the four blank spikes (see determinative method). Use the recoveries described in the specific determinative method as guidance for evaluating the results.

11.0 DOCUMENTATION

11.1 The preparation logbook must be completed in its entirety. Unused space must be "Zed" out. Any problems or anomalies must be clearly noted in the logbook.

11.2 The standard preparation logbook must be completed for all standard preparations. All information requested must be completed; the page must be signed and dated by the respective person.

11.3 The Accutest lot number must be cross-referenced on the standard vial/container. The expiration date must be noted on the standard vial/container.

11.4 The instrument Maintenance logbook must be completed when any type of maintenance is performed on the instrument. Each instrument will have a separate log.

11.5 All laboratory logbooks must be routinely reviewed and initialed or signed by the lab manager.

11.6 Any corrections to laboratory data must be done using a single line through the error. The initials of the person and date of correction must appear next to the correction.

11.7 A record of the daily sonicator tune is recorded in the sonicator tuning log.

12.0 DATA REVIEW AND REPORTING

12.1 See the specific determinative method SOP for data review and reporting. The department manager and Quality Assurance Officer should review and initial the extraction logbooks and reagents and standards logbooks on a periodic basis (note – a copy of the extraction logbook is initialed by the department manager and filed with the raw data – the original extraction logbook pages are not initialed). Copies of logbook pages are included with full deliverable data packages.

13.0 POLLUTION PREVENTION & WASTE MANAGEMENT

13.1 Pollution Prevention. Users of this method must perform all procedural steps that control the creation and/or escape of wastes of hazardous materials to the environment. The amounts of standards, reagents, and solvents must be limited to the amounts specified in this SOP. All safety practices designed to limit the escape of vapors, liquids, or solids to the environment must be followed. All method users must be familiar with the waste management practices described in section 13.2

13.2 Waste Management. Individuals performing this method must follow established waste management procedures as described in the Sample and Waste Disposal SOP. This document describes the proper disposal of all waste materials generated during the testing of samples as follows:

13.2.1 Non-hazardous aqueous wastes
13.2.2 Hazardous aqueous wastes
13.2.3 Chlorinated organic solvents
13.2.4 Non-chlorinated organic solvents
13.2.5 Hazardous solid wastes
13.2.6 Non-hazardous solid wastes
13.2.7 Microbiological wastes

Lab Manager: Douglas Yargeau

QA Officer: Robert Treggiari

TITLE: **SEPERATORY FUNNEL LIQUID-LIQUID EXTRACTION**

TEST METHOD: SW836 3510C, Rev. 3 December, 1996

REVISED SECTIONS: 8.9; 10.4

1.0 SCOPE & APPLICATION

1.1 This method describes the general procedures for isolating organic compounds from aqueous samples. The method also describes concentration techniques suitable for preparing the extract for the appropriate determinative method.

1.2 Test Codes: See the specific determinative method.

2.0 SUMMARY

2.1 A measured volume of sample, usually 1 liter, at a specified pH (refer to the specific method), is serially extracted with methylene chloride using a separatory funnel.

2.2 The extract is dried, concentrated (if necessary), and, as necessary, exchanged into a solvent compatible with the cleanup or determinative method to be used.

3.0 METHOD DETECTION LIMIT AND REPORTING LIMIT

3.1 The reporting limit (RL) is based on the lowest calibration standard. RL's may vary depending on matrix difficulties, sample volumes or weights, and percent moisture. Detected concentrations below this concentration cannot be reported without qualification.

3.2 The Method Detection Limit (MDL) represents the lowest reportable concentration of an individual analyte that meets the method qualitative identification criteria.

3.3 Method Detection limits (MDLs) are experimentally determined using the procedures described in 40 CFR, Part 136, Appendix B. Actual reported MDLs incorporate the sample volume prepared and analyzed and sample dilutions if needed, which may cause MDL variations from sample to sample.

3.4 In general, MDLs are determined through the analysis of at least 7 replicate blank spikes (using the same procedures for sample analysis). The MDL is calculated by multiplying the standard deviation of the replicate concentrations by the appropriate Student's t value (3.143 for 7 replicates). If more than 7 replicates are analyzed refer to 40 CFR, Part 136, Appendix B for the appropriate student's t value. MDLs are determined initially (prior to analysis), on an annual basis, and after major maintenance to equipment. MDL data is archived with Quality Assurance. Refer to the most recent study for current MDLs.

4.0 DEFINITIONS

4.1 ALIQUOT - a measured portion of a sample, or solution, taken for sample preparation and/or analysis.

4.2 BATCH – A group of samples which behave similarly with respect to the sampling or the testing procedures being employed and which are processed as a unit. For QC purposes, if the number of samples in a group is greater than 20 (or 10 for certain methods), then each group of 20 samples (or 10 samples for certain methods) or less will all be handled as a separate batch. A group of samples prepared at the same time in the same location using the same method.

4.3 EXTRACTABLE - a compound that can be partitioned into an organic solvent from the sample matrix and is amenable to gas chromatography. Extractables include semivolatile (BNA) and pesticide/Aroclor compounds.

4.4 FIELD SAMPLE - a portion of material to be analyzed that is contained in single or multiple containers and identified by a unique sample number.

4.5 FIELD BLANK – this is any sample that is submitted from the field and is identified as a blank. This includes rinsates and equipment blanks, etc.

4.6 HOLDING TIME – the elapsed time expressed in days from the date of sampling until the date of its extraction and analysis.

4.7 INSUFFICIENT QUANTITY - when there is not enough volume (water sample) to perform any of the required operations: sample analysis or extraction, percent moisture, MS/MSD, etc.

4.8 MATRIX - the predominant material of which the sample to be analyzed is composed. For the purpose of this SOP, a sample matrix is water. Matrix is <u>not</u> synonymous with phase (solid).

4.9 MATRIX EFFECT - In general, the effect of a particular matrix (water or soil) on the constituents with which it contacts. This is particularly pronounced for clay particles which may adsorb chemicals and catalyze reactions. Matrix effects may prevent extraction of target analytes, and may affect surrogate recoveries. In addition, non-target analytes may be extracted from the matrix causing interferences.

4.10 MATRIX SPIKE - aliquot of a matrix (water) 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.

4.11 MATRIX SPIKE DUPLICATE - a second aliquot of the same matrix as the matrix spike (above) that is spiked in order to determine the precision of the method.

4.12 MATRIX DUPLICATE – a second aliquot of the original sample in order to determine the precision of the method.

4.13 METHOD BLANK - an analytical control consisting of all reagents, internal standards, and surrogate standards that is carried throughout the entire extraction and analytical procedure. The method blank is used to define the level of laboratory, background, and reagent contamination.

4.14 REAGENT WATER - water in which an interferant is not observed at or above the minimum quantitation limit of the parameters of interest.

4.15 SURROGATES (Surrogate Standard) - for semivolatiles and pesticides/Aroclors, compounds added to every blank, sample, matrix spike, matrix spike duplicate, and standard; used to evaluate analytical efficiency by measuring recovery. Surrogates are brominated, fluorinated, or isotopically labeled compounds not expected to be detected in environmental media.

5.0 HEALTH & SAFETY

5.1 All safety practices must be followed as outlined in the Accutest Laboratories Employee Safety Handbook and Chemical Hygiene Plan. Safety glasses, gloves, and lab coats must be worn. All samples, solutions, and extracts must be treated as unknown and potentially hazardous.

5.2 The toxicity or carcinogenicity of each reagent used in this method has not been precisely determined; however, each chemical should be treated as a potential health hazard. Exposure to these reagents should be reduced to the lowest possible level.

5.3 **All extractions and the use of organic solvents must be performed under a properly functioning ventilation hood.**

6.0 COLLECTION, PRESERVATION, & HOLDING TIMES

6.1 See the specific determinative method for collection, preservation, and holding time requirements. General holding time requirements are as follows:

6.1.1 Generally, samples must be extracted within 14 days of sampling and must be analyzed within 40 days of extraction.

7.0 APPARATUS AND MATERIALS

7.1 Equipment

7.1.1 Evaporating steam baths.

7.1.2 Nitrogen blowdown apparatus (N-Evap®).

7.1.3 Zymark Turbovap® concentrator

7.2 Materials

7.2.1 Separatory funnels - 2 liter with Teflon stopcocks.

7.2.2 Drying column - chromatographic column with glass fritted disc, or glass funnel fitted with Whatman P5 filter paper.

7.2.3 Kuderna-Danish evaporator with 10 ml graduated concentrator tube.

7.2.4 Three-Ball Snyder column.

7.2.5 150 ml beakers.

7.2.6 2 ml crimp vials (with crimper).

7.2.7 5 ml volumetric flasks.

7.2.8 1000 ml graduated cylinder.

7.2.9 Glass stirring rods (or Teflon).

7.2.10 Pasteur pipettes.

7.2.11 Zymark concentrator tubes

8.0 STANDARDS AND REAGENTS

8.1 Reagent water - deionized and carbon filtered water.

8.2 Sodium hydroxide solution (ion)-dissolve 40 g of NaOH in reagent water and dilute to 100 ml.

8.3 Sulfuric acid (conc.).

8.4 Hexane - reagent grade for pesticide residue analysis.

8.5 Methylene chloride - reagent grade for trace organic analysis.

8.6 Sodium sulfate (granular anhydrous) - or muffled at 400°C for 4 hours.

8.7 Nitrogen gas (purified) - supplied by Air Products with valves attached to each ventilation hood.

8.8 Spiking solutions for the specific extraction method.

8.9 All chemicals listed are reagent grade unless otherwise specified. Distilled, deionized water should be used whenever water is required. All applicable standard/reagent preparation information, including vendor, lot number, date of preparation, expiration date, calculations, and initials must be entered in the appropriate standard/reagent preparation logbook. Vendors typically used by Accutest include

Fisher Scientific, VWR, Absolute Standards, Accustandard, Supelco, Chemservices, Ultra, and ERA. Additional vendors may be utilized as necessary.

9.0 INTERFERENCES

9.1 Method interferences may be caused by contaminants in the solvent reagents and glassware. Glassware must be scrupulously cleaned as outlined in the glassware prep SOP. Solvents and reagents must be of the purest grade and monitored by the lot numbers. Method Blanks are used to check for trace solvent contaminants.

9.2 Interferences by phthalate esters can be a problem with semivolatile analysis. All plastic should be eliminated from the extraction lab, and handling of glassware surfaces that come in contact with extraction solvents should be avoided.

10.0 PROCEDURE

10.1 Mark the sample meniscus on the sample bottle and pour the sample into a 2 liter separatory funnel.

10.2 Check the pH by using a glass or Teflon stirring rod to place a drop of the sample on broad range pH paper. Note: This procedure is normally performed by the Sample Management department, and is only necessary when pH adjustment is made by the organic prep lab – 10.3).

10.3 Adjust the pH as necessary using sulfuric acid or sodium hydroxide (according to the specific determinative method).

10.4 Add 60 ml of methylene chloride to the sample bottle. Cap, invert and gently swirl the methylene chloride within the sample bottle to thoroughly rinse the contents. Add this rinse to the separatory funnel.

10.5 Prepare a blank and blank spike by adding 1 liter of deionized water to two separatory funnels, to be used as the batch blank and spike (see specific determinative or extraction method for additional QC samples).

10.6 Select a sample for matrix spike/matrix spike duplicate (or matrix duplicate) and prepare each aliquot as described in 10.1 – 10.4.

10.7 Spike all QC and field samples with the appropriate surrogate (see the specific determinative or extraction method).

NOTE: Spike Witnesses are required for all spiking procedures. The spike witness is required to verify that the addition of the correct spiking solution, volume, and concentration has occurred. The initials of the spike witness must be documented in the logbook within the spike witness field. For occasions where a spike witness is not present (weekends and/or evening shifts) the spike witness field must be given an "NA" (Not Applicable) designation and verified by a supervisor or manager when available.

10.8 Spike all QC samples with the appropriate spiking solutions (see specific determinative or extraction method).

10.9 Cap the separatory funnel, invert it, and open the stopcock under the hood to vent it. Gently swirl the contents to mix while leaving the stopcock open.

10.10 Close the stopcock and shake the contents 2 or 3 times. Open the stopcock to vent the funnel. Repeat this process until there is no pressure buildup.

10.11 Extract by shaking the funnel vigorously for approximately 2 minutes. Release excess pressure by occasionally venting the funnel under the hood by opening and closing the Teflon stopcock. Allow the aqueous solvent layers to separate for about 5-10 minutes while preparing the sodium sulfate drying columns (10.13). Once the solvent layer has sufficiently separated, drain the (lower) solvent layer into a labeled 150-ml flask.

10.12 Add a second 60 ml volume of methylene chloride to the separatory funnel, and repeat the extraction procedure described in Sections 10.9 – 10.11.

10.13 Prepare a drying column by adding approximately 10 cm of prepared sodium sulfate into a chromatographic column with fritted disc. Alternatively, a funnel/filter paper assembly may be used. Rinse with 30 ml of methylene chloride and collect in a waste beaker and discard in chlorinated solvent waste bottle. Place the cleanup column under hood along with a prepared K-D apparatus to collect the "dried" extract **(alternatively, see section 10.19 for use of Zymark Turbovap® concentrator).** Make sure all apparatus is labeled accordingly. Take the sample extract contained in the 150 ml beaker and pour it into the top of the drying column. If the solvent layer is emulsified, it may have to be broken up by centrifuging. Collect the extract in the Kuderna Danish.

10.14 After all of the extract has passed through the sodium sulfate column, rinse down the inside of the column with an excess of 25ml solvent. Add 2-3 glass boiling beads to the K-D flask and attach a three ball Snyder column (wrapped in aluminum foil). Prime the Snyder column by adding 1ml of methylene chloride to the top.

10.15 Position the K-D concentrator setup on the hot water bath (temperature specified by the method) with only the 10 ml concentrator tube submersed in water. Evaporate the extract to below 10 ml not allowing it to go to dryness. Remove the K-D apparatus from the bath and allow the solvent condensed in the Snyder column to drip back down into the concentrator tube. Allow the solvent to cool for approximately 10 minutes.

10.16 If solvent exchange to hexane is necessary, add the appropriate amount of solvent specific to the extraction method to the cooled methylene chloride and reconcenetrate the extract to fewer than 5 ml. Aluminum foil may be placed around the lower rounded surface of the K-D flask to aid evaporation. Allow the solvent to cool and remove the Snyder column and K-D flask, while rinsing the connector joints with approximately 1 ml of hexane into the receiver. Bring the extract to the volume appropriate for the specific extraction method and mix well.

10.17 Bring the extract to a volume appropriate for the specific extraction method and mix well. Transfer to 2 GC auto sampler vials with PTFE-lined crimp caps and label with sample ID and batch number. Store at 4°C ±2°C until analysis.

10.18 If further concentration is necessary, the nitrogen blowdown technique (N-Evap®) may be used.

10.18.1 Place the concentrator tube in a warm bath (35°C) and evaporate the solvent to the final volume as specified be the specific extraction method using a gentle stream of clean, dry nitrogen (filtered through a column of activated carbon).

10.18.2 The internal wall of the tube must be rinsed several times with methylene chloride or appropriate solvent during the operation. During evaporation, the tube must be positioned to avoid water condensation (i.e., the solvent level should be below the level of the water bath). The extract must not be allowed to become dry.

Note: When the volume of solvent is reduce below 1ml, semivolatile analytes may be lost.

10.19 Concentration using Zymark. Sample extracts may be concentrated by using the Zymark Turbovap® concentrator **(section 10.13 – from drying column fill concentrator tubes instead of K-D apparatus).**

10.19.1 Prepare the Zymark by:

10.19.1.1 Check incoming gas supply (sufficient reserve and turned on).
10.19.1.2 Check water bath level (> sample level).
10.19.1.3 With cover down, turn on unit and set BATH TEMP.
10.19.1.4 Select the TEMPERATURE display (35°C - 40°C).
10.19.1.5 Allow bath to come to temperature (display stops blinking)
10.19.1.6 Fill concentrator tubes with samples.
10.19.1.7 Open cover and place tubes in water bath.
10.19.1.8 Close cover

10.19.2 Start Concentration

10.19.2.1 Press ENDPOINT SELECT until desired mode is selected (Sensor)
10.19.2.2 Set ENDPOINT TIME (NA).
10.19.2.3 Press SELECT DISPLAYED CONDITION until pressure is selected. Pull out regulator knob, adjust gas pressure, push in to lock.
10.19.2.4 Press the START/STOP button for each cell position used.

10.19.3 Stop Concentration

10.19.3.1 To pause concentration for all tubes, raise the cover. To continue operation, lower the cover.
10.19.3.2 To stop concentration process for an individual cell (running manually or need to stop and choose one of the other operation modes), press the

START/STOP key for the corresponding cell. **CAUTION: This clears out the TIME ELAPSED.**

10.19.3.3 Otherwise, when a cell reaches its selected endpoint, its corresponding light blinks and the beeper sounds briefly every 30 seconds.

10.19.3.4 Raise the cover to silence the beeper.

Rapid beeping during concentration indicates low gas pressure – check your gas supply.

10.19.3.5 For extracts that require solvent exchange – add the appropriate volume of method-specific solvent.

10.19.3.6 Concentrate to < 10 mls > 1.0 ml.

10.19.3.7 Remove the sample

Prompt removal of the completed tube is important since highly volatile compounds may be lost of allowed to sit for an extended period of time, and steam from a water bath of over 50°C can condense on the cover and possibly drip into sample tubes if they are left in place.

10.19.3.8 Add the appropriate solvent to volume and mix thoroughly.

10.19.4 Shut down

10.19.4.1 Replace the plastic closures in each cell position.

10.19.4.2 Leave the cover open to keep moisture from accumulating on the inside cover.

10.19.4.3 Clean the concentrator tubes according to procedures described in “The Procedures for Washing Glassware” SOP.

10.19.4.4 Turn the power and gas supply off (optional).

10.19.5 Proceed to sample cleanup, if necessary (see the specific extraction method), or transfer the extract to 2 GC auto sampler vials with PTFE-lined crimp caps. Store at 4°C ±2°C until analysis. Note: for 8270C analyses, the extracts must be stored at -10°C to -20°C. Extracts for 625 analysis must be stored at 4°C ±2°C.

11.0 QUALITY ASSURANCE

11.1 Refer to the specific determinative or extraction method

11.2 A method blank, blank spike and MS/MSD are required with each extraction batch.

11.3 Prior to running samples, the laboratory must demonstrate initial proficiency by generating data of acceptable accuracy and precision (P&A study) for target analytes in a clean matrix. A P&A study is performed as an initial determination of capability, on an annual basis (continued demonstration of capability – a successful PT result may be used in place of a P&A for continued DOC), and if any significant changes have been made to the instrument. In general, 4 replicates or blank spikes are analyzed using the same procedures and conditions for sample analysis. The percent recoveries are compared to default limits or in-house control limits once established. The standard deviation of

the 4 replicate percent recoveries are compared to either ±20 or to in-house limits once established. If percent recovery or standard deviation criteria are not met, corrective action must be taken to bring the system back into control. Refer to specific analytical method SOP for acceptance limits.

12.0 DOCUMENTATION

12.1 All the information required by the extraction logbooks must be thoroughly completed. Any problems associated with the extraction must be clearly noted in the logbook.

12.2 All standards preparation must be documented in the standards preparation logbook.

12.3 Equipment maintenance logs must be maintained.

12.4 All laboratory logbooks must be routinely reviewed and initialed or signed by the lab manager.

12.5 Any corrections to laboratory data must be done using a single line through the error. The initials of the person and date of correction must appear next to the correction.

13.0 DATA REVIEW & REPORTING

13.1 Standard, reagent, and instrument logbooks) must be reviewed by the laboratory supervisor on a monthly basis. Preparation logbooks must be reviewed with each data package by the department Technical Manager.

13.2 Copies of preparation logbook pages may be requested for Tier packages.

14.0 POLLUTION PREVENTION & WASTE MANAGEMENT

14.1 Pollution Prevention. Users of this method must perform all procedural steps that control the creation and/or escape of wastes of hazardous materials to the environment. The amounts of standards, reagents, and solvents must be limited to the amounts specified in this SOP. All safety practices designed to limit the escape of vapors, liquids, or solids to the environment must be followed. All method users must be familiar with the waste management practices described in section 14.2

14.2 Waste Management. Individuals performing this method must follow established waste management procedures as described in the Sample and Waste Disposal SOP. This document describes the proper disposal of all waste materials generated during the testing of samples as follows:

14.2.1 Non-hazardous aqueous wastes
14.2.2 Hazardous aqueous wastes
14.2.3 Chlorinated organic solvents
14.2.4 Non-chlorinated organic solvents
14.2.5 Hazardous solid wastes
14.2.6 Non-hazardous solid wastes
14.2.7 Microbiological wastes

15.0 METHOD PERFORMANCE

15.1 Refer to the specific analytical SOP for method performance.

16.0 ADDITIONAL REFERENCES

16.1 None

17.0 ANNUAL REVIEW

1. _____ _____
Reviewed by: **Date:**

2. _____ _____
Reviewed by: **Date:**

3. _____ _____
Reviewed by: **Date:**

4. _____ _____
Reviewed by: **Date:**

5. _____ _____
Reviewed by: **Date:**

Lab Manager: Douglas Yargeau

QA Officer: Robert Treggiari

TITLE: **SOXHLET EXTRACTION**

TEST METHOD: SW846 3540C, Revision 3, December 1996

REVISED SECTIONS: 10.4

1.0 SCOPE & APPLICATION

1.1 Method 3540C is a procedure for extracting nonvolatile and semivolatile organic compounds from solids such as soils, sludges, and wastes. The Soxhlet extraction process ensures intimate contact of the sample matrix with the extraction solvent.

1.2 This method is applicable to the isolation and concentration of water-insoluble and slightly water soluble organics in preparation for a variety of chromatographic procedures.

1.3 This method is restricted to use by or under the supervision of trained analysts. Each analyst must demonstrate the ability to generate acceptable results with this method.

2.0 SUMMARY

2.1 The solid sample is mixed with anhydrous sodium sulfate, placed in an extraction thimble or between two plugs of glass wool, and extracted using an appropriate solvent in a Soxhlet extractor.

2.2 The extract is then dried, concentrated (if necessary), and, as necessary, exchanged into a solvent compatible with the cleanup or determinative step being employed.

3.0 METHOD DETECTION AND REPORTING LIMIT

3.1.1 See the appropriate determinative method for method detection limit data.

4.0 DEFINITIONS

4.1 ALIQUOT - a measured portion of a sample, or solution, taken for sample preparation and/or analysis.

4.2 BATCH – A group of samples which behave similarly with respect to the sampling or the testing procedures being employed and which are processed as a unit. For QC purposes, if the number of samples in a group is greater than 20 (or 10 for certain methods), then each group of 20 samples (or 10 samples for certain methods) or less will all be handled as a separate batch.

4.3 DRY WEIGHT – the weight of a sample based on percent solids. The weight after drying in an oven.

4.4 CONTAMINATION - a component of a sample or an extract that is not representative of the environmental source of the sample. Contamination may stem from other samples, sampling equipment, while in transit, from laboratory reagents, laboratory environment, or analytical instruments. EXTRACTABLE - a compound that can be partitioned into an organic solvent from the sample matrix and is amenable to gas chromatography. Extractables include semivolatile (BNA), semi-volatile hydrocarbons (C9-C40) and pesticide/Aroclor compounds.

4.5 FIELD SAMPLE - a portion of material to be analyzed that is contained in single or multiple containers and identified by a unique sample number. HOLDING TIME – the elapsed time expressed in days from the date of sampling until the date of its extraction and analysis.

4.6 INSUFFICIENT QUANTITY - when there is not enough volume (water sample) or weight (soil/sediment) to perform any of the required operations: sample analysis or extraction, percent moisture, MS/MSD, etc.

4.7 SOIL - used herein synonymously with soil/sediment and sediment.

4.8 METHOD BLANK - an analytical control consisting of all reagents, internal standards, and surrogate standards, that is carried throughout the entire analytical procedure. The method blank is used to define the level of laboratory, background, and reagent contamination.

4.9 MATRIX SPIKE - aliquot of a matrix (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.

4.10 MATRIX SPIKE DUPLICATE - a second aliquot of the same matrix as the matrix spike (above) that is spiked in order to determine the precision of the method.

4.11 MATRIX DUPLICATE – a second aliquot of the original sample in order to determine the precision of the method.

4.12 REAGENT WATER - water in which an interferant is not observed at or above the minimum quantitation limit of the parameters of interest. Accutest uses deionized water (municipal water which passes through Accutest's DI treatment system).

4.13 SURROGATES (Surrogate Standard) - Compounds added to every blank, sample, matrix spike, matrix spike duplicate, and standard; used to evaluate analytical efficiency by measuring recovery. Surrogates are brominated, fluorinated, or isotopically labeled compounds not expected to be detected in environmental media.

5.0 HEALTH & SAFETY

5.1 All safety practices must be followed as outlined in the Accutest Laboratories Employee Safety Handbook and Chemical Hygiene Plan. Safety glasses, gloves, and lab coats must be worn. All samples, solutions, and extracts must be treated as unknown and potentially hazardous.

5.1 The toxicity or carcinogenicity of each reagent used in this method has not been precisely determined; however, each chemical should be treated as a potential health hazard. Exposure to these reagents should be reduced to the lowest possible level.

5.2 All applicable extraction procedures must be performed within a properly functioning fume hood.

6.0 COLLECTION, PRESERVATION & HOLDING TIMES

6.1 See the specific determinative method for collection, preservation, and holding times requirements.

7.0 APPARATUS & MATERIALS

7.1 Soxhlet extractor – 40 mm ID, with 500 ml round bottom flask

7.2 Drying column – 20 mm ID Pyrex chromatographic column with Pyrex glass wool at bottom.

7.3 KD-apparatus

7.4 Boiling Chips

7.5 Water bath – heated

7.6 Vials – glass

7.7 Glass or paper thimble or glass wool – contaminant-free

7.8 Heating mantle – Rheostat controlled.

7.9 Disposable glass Pasteur pipet and bulb

8.0 STANDARDS & REAGENTS

8.1 Reagent Grade Acetone

8.2 Reagent Grade Methylene Chloride

8.3 Sodium sulfate (granular, anhydrous), N2SO4. Purify by heating at 400 Deg. C for 4 hours in a shallow tray.

8.4 Surrogates/spiking solutions (see specific determinative method)

8.5 All applicable standard/reagent preparation information, including vendor, lot number, date of preparation, calculations, and initials must be entered in the appropriate standard/reagent preparation logbook. Vendors typically used by Accutest include Fisher Scientific, VWR, Accustandard, Supelco, Chemservices, Ultra, and ERA. Additional vendors may be utilized as necessary.

9.0 INTERFERENCES

9.1 See the specific determinative method for interferences. Generally, method interference can be reduced by appropriate glassware cleaning (see "The Procedure for Washing Glassware" SOP) and the removal of any plastics contact from sampling, prep or analysis.

10.0 PROCEDURE

10.1 Decant and discard any water layer on a sediment sample. Homogenize the sample thoroughly (refer to the SOP for the Procedure for Obtaining Representative Aliquots – MQA247 for details) and discard any foreign objects such as sticks, leaves, and rocks, and weigh out appropriate amount of sample (according to the specific determinative method) and record in the extraction logbook. ***DO NOT TARGET WEIGH (i.e. attempt to obtain exactly the required amount).***

10.2 Determine percent solids (refer to SOP MGN007).

10.3 Blend 15g of the solid sample with approximately 15 g of anhydrous sodium sulfate (additional sodium sulfate may be used if required) and place in extraction thimble. 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.

10.4 Add 1.0 ml of the surrogate standard spiking solution onto the samples, method blank, and quality control samples (see below).

NOTE: Spike Witnesses are required for all spiking procedures. The spike witness is required to verify that the addition of the correct spiking solution, volume, and concentration has occurred. The initials of the spike witness must be documented in the logbook within the spike witness field. For occasions where a spike witness is not present (weekends and/or evening shifts) the spike witness field must be given an "NA" (Not Applicable) designation and verified by a supervisor or manager when available.

10.5 Add 1.0 ml of spiking solution to the matrix spike/matrix spike duplicate and blank spike.

10.6 Place approximately 300 ml of the extraction solvent (1:1 Acetone:Methylene chloride) into a 500-ml round bottom flask containing one or two clean boiling chips. Attach the flask to the extractor and extract the sample for 16-24 hours at 4-6 cycles/hour.

10.7 Allow the extract to cool after the extraction is complete. If concentration by KD is to be performed refer to the next sections. If concentration by Turbovap is to be performed refer to SOP MOP214.

10.8 Assemble the KD concentrator.

10.9 Prerinse a funnel and filter paper with methylene chloride. Add sodium sulfate. Attach funnel to a KD concentrator tube with glass boiling bead.

10.9.1 Decant extract into funnel. Rinse funnel with methylene chloride.

10.9.2 Attach a three-ball Snyder column, prime the Snyder column by adding 1 ml of methylene chloride to the top.

10.9.3 Position the K-D concentrator setup on the hot water bath with only the 10 ml concentrator tube submersed in the water. Evaporate the extract to below 5 ml not allowing it to go to dryness. Remove the K-D apparatus from the bath and allow the solvents condensed in the Snyder column to drip back down into the concentrator tube. Allow the solvent to cool for approximately 10 minutes, then remove the Snyder column and K-D flask while rinsing the connector joints with 1 ml of methylene chloride. Since the volume is now greater than 1 ml, concentrate by directing a gentle stream of nitrogen gas over the extract surface to 1ml (or according to the applicable method requirements).

10.9.4 Transfer the extract to a 2 ml teflon capped crimp vial using a Pasteur pipette. .

10.9.5 Label the vial with a sample number and batch number. Store the vials by batch in the freezer located in the semivolatiles laboratory (must transfer custody to GC analyst).

11.0 QUALITY ASSURANCE

11.1 See the specific determinative method for quality assurance criteria.

12.0 DOCUMENTATION

12.1 The soil preparation logbook must be completed daily (including the ASE position of the sample). If samples require re-extraction, a brief explanation of the reason should be documented in this log. Any observations (such as a low recovery of extract) must be documented in this logbook.

12.2 The standard preparation logbook must be completed for all standard preparations. All information requested must be completed; the page must be signed and dated by the respective person. Submit the log to the supervisor for verification.

12.3 The Accutest lot number must be cross-referenced on the standard vial.

12.4 The instrument Maintenance logbook must be completed when any type of maintenance is performed on the instrument. Each instrument will have a separate log.

12.5 All laboratory logbooks must be routinely reviewed and initialed or signed by the lab manager or quality assurance.

12.6 Any corrections to laboratory data must be done using a single line through the error. The initials of the person and date of correction must appear next to the correction.

13.0 DATA REVIEW AND REPORTING

13.1 Refer to the determinative method.

13.2 Procedures for handling non-conforming data.

13.2.1 Refer to the determinative method.

14.0 POLLUTION PREVENTION & WASTE MANAGEMENT

14.1 Pollution Prevention. Users of this method must perform all procedural steps in a manner that controls the creation an/or escape of wastes or hazardous materials to the environment. The amounts of standards, reagents, and solvents must be limited to the amounts specified in this SOP. All safety practices designed to limit the escape of vapors, liquids, or solids to the environment must be followed. All method users must be familiar with the waste management practices described in section 14.2 .

14.2 Waste Management. Individuals performing this method must follow established waste management procedures as described in the Sample and Laboratory Waste Disposition SOP MSM036-02. This document describes the proper disposal of all waste materials generated during the testing of samples as follows:

14.2.1 Non-hazardous aqueous wastes
14.2.2 Hazardous aqueous wastes
14.2.3 Chlorinated organic solvents
14.2.4 Non-chlorinated organic solvents
14.2.5 Hazardous solid wastes
14.2.6 Non-hazardous solid wastes

15.0 ADDITIONAL REFERENCES

15.1 None.

Lab Manager: Brad Madadian

QA Officer: Robert Treggiari

TITLE: MANUAL INTEGRATION OF CHROMATOGRAPHIC PEAKS

REFERENCES: Accutest Laboratories of New England, NELAC Quality Systems, Chapter 5, 2009

REVISED SECTIONS: Header/Footer

1.0 SCOPE & APPLICATION

1.1 This SOP is intended for use in all methods that employ chromatographic processes to separate chemical species. Specific instructions are provided for properly performing manual integration of chromatographic peaks. These procedures are designed to insure that manual integration is appropriately performed and documented.

2.0 SUMMARY

2.1 This SOP describes the requirements and criteria to determine when manual integration of chromatographic peaks is appropriate, acceptable procedures for performing manual integration, supervisory approval and documentation of manual integration and situations where manual integration is prohibited.

3.0 METHOD DETECTION LIMIT

3.1 Not applicable

4.0 DEFINITIONS

Background. Constant signal from a gas chromatograph detector resulting from detectable substances in the carrier gas, septum bleed, or chromatographic column bleed. These materials produce a constant signal noise, which establishes the chromatographic baseline.

Baseline. The ambient signal from a gas chromatograph detector that consists of background noise only.

Chromatographic Peak. The baseline deflection resulting from a detector response to a compound which has eluted from a chromatographic column.

Machine Integration. Determination of chromatographic peak area using an established computer algorithm such as an integrator.

Manual Peak Integration. Manual selection of the baseline criteria for the computer algorithm used for chromatographic peak integration.

Normal (Gaussian) Peak Shape. Symmetric chromatographic peaks with the highest concentration of detected material at the midpoint of the peak retention time range.

Overlapping Peaks. Chromatographic peaks with overlapping retention time ranges whose leading or trailing edge does not return to baseline before another compound is detected resulting in a positive peak inflection.

Retention Time. The time for any compound to move through the chromatographic column and be detected. The retention time is usually established at the apex of a chromatographic peak. The peak retention range begins at the time of positive baseline inflection and ends when the slope of the negative inflection returns to baseline.

Split Peaks. Chromatographic peaks with multiple peak apexes, giving the appearance of overlapping chromatographic peaks.

5.0 HEALTH & SAFETY

5.1 Not applicable

6.0 PROCEDURE

6.1 Recognizing Unacceptable Machine Integration:

6.1.1 Poor Machine Integration. Poor machine integration typically occurs on poorly defined chromatographic peaks, low concentration peaks, or peaks that are masked or overlap with other compounds present in the sample.

Poor integration is distinguished by baselines that take too much or too little peak area. The machine drawn baseline used for the integration cuts portions of the peak, picks up noise ahead of or beyond the peak or establishes a baseline above or below the background baseline.

6.1.1.1 Before performing manual integration, the analysts must attempt to perform corrective action by optimizing the peak integration parameters. Manual peak integration can be performed if the peak qualifies and the chromatographic peak integrator parameters have been optimized.

6.1.2 Corrective Action For Poor Machine Integration.

6.1.2.1 The analyst is required to note if excessive manual integration is needed on an instrument. If the need for manual integration becomes excessive, check the peak integration parameters with the supervisor before attempting to perform manual integration of chromatographic peaks. The supervisor will check the following parameters:

6.1.2.1.1 Peak Area Reject. Establishes the detection threshold in area units for detecting and integrating a chromatographic peak.

6.1.2.1.2 Peak Width. Establishes a minimum half height peak width (in scans or minutes) for detecting a chromatographic peak. Establishing the width at too large a value filters out narrow peaks.

6.1.2.1.3 Initial threshold. The slope value selected that indicates the beginning (start threshold) or end (stop threshold) of a chromatographic peak. Selecting too low of a threshold value results in integration of background noise.

6.1.2.1.4 Shoulder Detection. In off mode, does not drop baseline tangents for unresolved chromatographic peaks. Manual integration will always be required.

6.1.2.1.5 Data Point Sampling. The number of data points per sampling used to perform any integration related determination. Peaks from capillary columns usually employ one data point per sampling.

6.1.2.1.6 Smoothing. The smoothing option prevents the splitting of bumpy chromatographic peaks and eliminates the integration of noisy baselines. Smoothing is always turned on.

6.1.2.2 Upon confirmation that the peak integration parameters are appropriately set, the analysts may perform manual integration for qualifying peaks. Peak integration parameters may be reviewed by the manager.

6.2 Ground Rules for Manual Integration.

6.2.1 Prohibited Cases. Manual integration is prohibited in certain situations where the peak is well defined and appropriately integrated.

6.2.1.1 Manual integration is disallowed for peaks that are symmetrical in shape and exhibit acceptable baseline to baseline machine integration.

6.2.1.2 Manual integration is disallowed for the sole purpose of adding area to meet QC acceptance criteria. Inappropriate integration in this scenario would exhibit either a manual baseline below the normal chromatographic baseline, or a manual baseline that extends beyond the peak retention range.

6.2.1.3 Manual integration is disallowed for the sole purpose of subtracting area to meet QC acceptance criteria. Inappropriate integration in this scenario would exhibit either a manual baseline above the normal chromatographic baseline, or a manual baseline that crops a well defined chromatographic peak extends before the end of the peak retention range.

6.2.2 Acceptable Cases. The machine integrator sometimes does not integrate appropriately in situations where peak integrity is marginal.

6.2.2.1 Overlapping Chromatographic Peaks. The retention times of two compounds are close to each other causing an overlap of the two. The peaks may require manual integration to select the area appropriately.

6.2.2.2 Poorly defined baselines that result in too little or too much peak area being selected by the machine integrator.

6.2.2.3 Unacceptable machine integration. Integration baseline does not appropriately reflect the peak/baseline intersect point. The baseline may require re-drawing to assure a baseline to baseline integration that appropriately defines the peak.

6.3 Drawing A Manual Baseline. There are three integration procedures used for the integration of chromatographic peaks by Accutest. These procedures occur automatically during the machine integration of peaks. These same processes are used for manual peak integration and are the only techniques allowed.

6.3.2 Baseline to Baseline Integration. The peak integration baseline is a continuation of the existing baseline. Integration starts at the first positive deflection of the baseline, which signals the beginning of a chromatographic peak and end when the declining (negative) inflection returns to the original baseline, signaling the end of the peak.

6.3.3 Baseline to Valley Integration. This integration is used for two overlapping chromatographic peaks. Integration of the first peak begins using the baseline to baseline approach described in 6.3.2. A perpendicular is dropped from the valley of the two intersecting peaks to the baseline. Integration of the second peak of the overlapping pair ends when the declining (negative) inflection returns to the original baseline, signaling the end of the peak.

6.3.4 Valley to Valley Integration. This integration is used for multiple overlapping chromatographic peaks. Integration of the first peak begins using the baseline to baseline approach described in 6.3.2. A perpendicular is dropped from the valley of the two intersecting peaks to the baseline (valley to baseline). The valley to baseline approach continues for every successive overlapping peak. Integration of the last peak of the overlapping group ends when the declining (negative) inflection returns to the original baseline, signaling the end of the last peak.

6.3.5 For PCB and hydrocarbon range integration multiple peaks may be integrated. Follow procedures outlined under section 6.3.

6.4 Consistency of Execution. Manual integration can be a subjective process when chromatographic peaks are poorly defined. It is imperative that the analysts perform manual integration using a consistent approach. This is especially important on a batch level.

7.0 REVIEW, DOCUMENTATION, AND REPORTING

7.1 All integrations are reviewed and approved by the department manager or supervisor.

7.1.1 The supervisor reviews all automatic and manual integration and provides an electronic stamp on the quant report identifying the reviewer, date of review, and time of review Before and after manual integrations (that is, the instrument integration and the subsequent analyst manual integration) are maintained by the lab (and provided to the client upon request). A manual integration is also identified on the quant report by an “M” next to the compound area count.

Note: The “Before” integration is captured in LIMS by queuing that compound to “print” (a PDF is generated of the instrument integration). After the manual

integration is performed, the re-integrated compound is then queued to "print" and the manual integration is subsequently captured in the resulting PDF. <u>This procedure must be followed for all GC and GCMS analyses.</u>

Note: The audit trail for each GC and GCMS system must always be activated.

7.1.2 The extended report (includes quant report, chromatogram, and individual compound spectra report) is included in all full data packages. Quant reports, chromatograms, and spectra may be requested by the client.

7.1.3 Certain clients or regulatory programs may have specific requirements regarding reporting of manual integrations. For example, the Department of Defense (DoD) QSM requires that samples and analytes for which manual integration is performed are identified in the narrative. Special client requirements will be noted in the "Comments" field of the log in (on the daily work list). Refer to the applicable client technical specifications file on the QA server for details.

8.0 POLLUTION PREVENTION & WASTE MANAGEMENT

8.1 Not applicable

9.0 ADDITIONAL REFERENCES

9.1 Hewlett Packard, HP 3365 Series II Chemstation Operation Manual, 6/93.

9.2 Navy Installation Restoration Laboratory Quality Assurance Manual, 2/96, 9/99

9.3 DoD QSM, Version 4.2, Oct., 2010

Lab Manager: Brad Madadian

QA Officer: Robert Treggiari

TITLE: PROCEDURE FOR DEVELOPING METHOD DETECTION LIMITS.

REFERENCES: 40 CFR Part 136 Appendix B, NELAC Chapter 5 (Quality Systems), 2009, DoD QSM, October 2010, MA DEP 310CMR 42.00

REVISED SECTIONS: revised 2.1; Removed section 6.1.1; revised 6.1.4; 6.1.4.1; added 6.2.8

1.0 SCOPE AND APPLICATION

1.1 The procedure used for experimentally generating method detection limits is also described. This procedure applies to the majority of analytical methodology where an experimental demonstration of the ability to achieve detectability at a specified concentration is required.

1.2 Method detection limits (MDL) are determined annually for each instrument for every method/matrix type. Alternatively, the MDL may be verified at a frequency specified by the regulatory agency. Each calculated instrument MDL must meet the "1:10" rule which is the ratio of spike amount to calculated MDL.

1.3 Organic MDL studies are logged into the LIMS under a designated project, and tracked by Quality Assurance. Metals and general chemistry MDL studies are scheduled by the department manager.

1.4 MDLs must be performed on all new instruments and after major maintenance activities.

1.5 Annual MDL studies should be performed within the first quarter of the year. Additional studies may be performed throughout the year as necessary.

2.0 SUMMARY

2.1 Method Detection Limits. Method detection limits are determined by spiking seven replicates of the matrix to be measured with target analytes at 3-5 times the estimated detection limit concentration. The samples are analyzed and the values for each parameter are calculated. The mean and standard deviation of the data set is calculated. The method detection limit is defined as 3.143 times the standard deviation of the seven replicates**.** If more than 7 replicates are analyzed refer to 40 CFR, Part 136, Appendix B for the appropriate student's t value. MDLs are determined initially upon instrument startup (prior to analysis), on an annual basis, and after major maintenance to equipment. MDL data is archived with Quality Assurance.

2.2 MDLs are verified by the preparation and analysis of a standard spiked at 1-3 times the MDL (single-analyte tests) and 1-4 times the MDL (multiple analyte tests). However, if the MDL result is <3 or <4 times (depending on whether it is a single or multiple analyte test) the spike level it is considered "self-verified".

3.0 METHOD DETECTION LIMIT.

3.1 This procedure establishes the mechanism for experimentally determining method detection limits on an annual basis.

4.0 DEFINITIONS

4.1 METHOD DETECTION LIMIT - The method detection limit (MDL) is defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the analyte concentration is greater than zero and is determined from analysis of a sample in a given matrix containing the analyte. This definition is qualitative in nature and does not evaluate an acceptable quantitative limit for method performance.

4.2 REAGENT WATER - water in which an interferant is not observed at or above the minimum quantitation limit of the parameters of interest. Accutest uses deionized water (municipal water which passes through Accutest's DI treatment system).

4.3 MDL VERIFICATION STANDARD – A standard that is prepared and analyzed at a concentration of 1-4 times the level of the MDL as a verification of the study. If an extractable MDL, the verification standard must be extracted then analyzed.

4.4 1:10X Rule: The spike to MDL ratio must be between 1 and 10 for an MDL to be considered valid. If the ratio is less than 1 or greater than 10, the study must be reanalyzed for the affected compound.

4.5 Limit of Detection (LOD) – After each detection limit determination, the laboratory must immediately establish the LOD by spiking a quality system matrix at approximately 2 to 3 times the detection limit (for a single analyte standard) or 1 to 4 times the detection limit (for a multi-analyte standard). This spike concentration establishes the LOD.

4.6 Limit of Quantitation (LOQ) – The Limit of Quantitation is set per method and matrix at the laboratory reporting limit (RL). The LOQ is, at a minimum, verified quarterly and must demonstrate precision and bias at the LOQ.

5.0 HEALTH & SAFETY

5.1 This administrative procedure does not require specific health and safety precautions.

6.0 PROCEDURE

6.1 Determining Method Detection Limits Using 40CFR, Part 136, Appendix B Procedures.

6.1.1 Make an estimate of the detection limit using one of the following:

- The last calculated MDL

- The concentration value that corresponds to an instrument signal/noise ratio between 2 and 5.

- The concentration equivalent of three times the standard deviation of replicate instrumental measurements of the analyte in reagent water.

- The concentration corresponding to the region of the standard curve where there is a significant change in sensitivity, i.e., a break in the slope of the standard curve.

6.1.2 It is recognized that the experience of the analyst is important to this process. However, the analyst must include the above considerations in the initial estimate of the detection limit.

6.1.3 Prepare a laboratory standard using the applicable matrix at an analyte concentration corresponding to 1 to 5 times the estimated method detection limit. This matrix must be purified – and free of the analytes of interest (reagent water, sand (Ottawa sand), air, or glass beads or Teflon chips for metals).

Note: For methods certified by the MA DEP preparation must occur over at least 3 days.

6.1.4 Prepare a minimum of seven spiked replicates for the appropriate method detection limit analysis. Samples are processed using the appropriate method, making sure to follow all required steps (the replicates must be processed as a normal sample - through all preparation, clean up, and analytical procedures). Analyte concentration calculations are performed according to the method specifications, reporting the final results in matrix specific reporting units.

Note: For methods certified by the MA DEP analysis must occur over at least 3 days.

6.1.4.1 If more than seven spikes are analyzed, all must be included in the MDL determination (unless apparent instrument malfunction, obviously defective run – poor purge or similar judgment is observed).

6.1.5 Calculate the variance (S^2) and standard deviation (S) of the replicate measurements, as follows:

$$S^2 = \frac{1}{n-1}\left[\sum_{i=1}^{n} x_i^2 - \left(\sum_{i=1}^{n} x_i\right)2/n\right]$$

$$S = (S^2)^{1/2}$$

Where:

X_i; i=1 to n, = are the analytical results in the final method reporting units obtained from the n sample aliquot that are determined to be useable (minimum of 7) and $\sum$ refers to the sum of the X values from i=1 to n.

6.1.6 Compute the MDL as follows:

$MDL = t_{(n-1,\ 1-\mu\ =\ 0.99)}\ (S)$

Where:
MDL = the method detection limit

$t_{(n-1,1-\mu\ =\ 0.99)}$ = the students' t value appropriate for a 99% confidence level and a standard deviation estimate with n-1 degrees of freedom (See table).

S = standard deviation of the replicate analyses.

# reps	deg. Freedom	$t_{(n-1,\ 0.99)}$
7	6	3.143
8	7	2.998
9	8	2.896
10	9	2.821

6.1.7 The 95% confidence interval estimates for the MDL derived in 6.1.7 are computed according the following equations derived from percentiles of the chi square over degrees of freedom distribution (x^2/df).

LCL = 0.64 MDL
UCL = 2.20 MDL

Where: LCL and UCL are the lower and upper 95% confidence limits respectively based on seven aliquots.

6.2 MDL Study Log-In (Organics).

6.2.1 Provide Sample Management with COCs for each MDL study.

6.2.2 Organic MDL studies must be logged into LIMS under project ALNE3948.

6.2.3 Each study/matrix must be logged in under one job.

6.2.4 If a study fails, the re-run is simply added on the existing job.

6.2.5 Water MDLs must be logged in under the matrix code “STYW”.

6.2.6 Soil MDLs must be logged in under the matrix code “STYS”.

6.2.7 Air MDLs must be logged in under the matrix code “STYA”.

6.2.8 Drinking Water MDLs must be logged in under the matrix code “STYD”

6.3 MDL Study Processing (Organics)

6.3.1 Use the MDL Generation Tool to process each study. The tool is divided into 3 sections:

6.3.1.1 Bench Generation Tool (used to process an individual study)

6.3.1.2 Instrument Generation Tool (used to process all studies performed on the instrument)

6.3.1.3 Pooled Generation Tool (used to pool all studies per matrix from all instruments).

6.3.2 The MDL generation tool operates using several ground rules:

6.3.2.1 Instrument level pooling:

6.3.2.1.1 Pool lowest qualifying MDL that satisfies the 1:10 rule.

6.3.2.1.2 If 1:10 rule is not achieved following two studies at different concentrations, employ MDL value that is closest to satisfying the 10X rule. Accutest will accept a level of non-compliant MDLs.

6.3.2.2 Method level pooling

6.3.2.2.1 Report the highest MDL. Pooled at the instrument level, satisfying the 1:10 rule.

6.4 Inorganic MDL Management

6.4.1 Inorganic MDL data is managed using an Excel spreadsheet (Actual study procedures are the same as for Organics). All MDL Study data is entered by the Inorganics Department Manager into this spreadsheet, which is located in the annual MDL data on the QA (LINUXMA1) server.

6.5 Prior to exporting to LIMS the pooled MDL must be verified by the analysis of a standard at concentrations of 1-3 times (for single analyte tests) or 1-4 times (for multiple analyte tests) the MDL. The MDL verification must be prepared (as applicable) and analyzed on each instrument used to run the test. The verification standard must be processed as a normal sample (through all preparation and analytical procedures). Acceptance criteria are qualitative identification of the analyte.

6.6 Once a completed study is exported to the LIMS it can be approved for use.

6.7 If results are not reported to the MDL the reporting limit (RL) must be verified by the analysis of a standard at 1-2 times the RL. The acceptance criteria are recovery of the analyte within established method limits. The verification standard must be processed as a normal sample (through all preparation and analytical procedures).

6.8 LOD/LOQ verification (see Sec 4.5 and 4.6) must be completed at least quarterly for each analyte and matrix combination for which the laboratory holds DoD certification. If a laboratory uses multiple instruments for a given method the LOD/LOQ must be verified on each. If the LOD/LOQ verification fails, then the laboratory must repeat the detection limit determination and LOD determination at a higher concentration and set the LOD at this higher concentration. LOD/LOQ projects will be logged into the database LIMS system on a quarterly basis (Jan, April, July, and Oct.) and given a 30 day requirement for completion. Corrective Actions may follow if these limits cannot be met for all DoD certified parameters. The laboratory must maintain documentation for all MDL/LOD/LOQ determinations and verifications

7.0 DOCUMENTATION.

7.1 All method detection limits studies and precision and accuracy studies must be documented and retained for seven years.

7.2 The raw data from all applicable preparation and analysis, all calculations, and the final MDL sheets must be kept on file in the same fashion as client sample data, and must include all required documentation as specified in the analytical SOP for the method.

7.3 QA maintains an annual MDL study spreadsheet which includes tracking for instruments, matrices, and methods. Once a study is completed, QA updates the spreadsheet. Once all MDLs are performed on all applicable instruments, QA completes the processing of the data into the final pooled MDL. This information (individual MDL study, instrument report, pooled MDL, and verification data) is kept on file with QA.

7.4 MDL studies are also retained as electronic data on the local QA server (LINUXMA1). MDLs can be found in the “MDL” folder and are organized by year, method, matrix, sample I.D and Instrument. LOD/LOD studies can be found in the “LOD/LOQ” folder and are sorted by department, year, quarter, matrix and method.

8.0 QUALITY CONTROL

8.1 The MDL study must pass the “1:10” rule for at least 90% of the analytes (for multi-component tests), and 100% of the “critical” compounds (petroleum compounds – BTEXM, PAHs). ***Note: Specific clients may require that all compounds meet the 1:10 rule.***

8.2 The MDLs must be below the Reporting Limits.

8.3 For DoD projects, the RL concentration must be at least 3 times the MDL concentration.

8.4 All method-specific criteria for the MDL study should be met.

8.5 MDLs must be either "self-verified" or verified by preparation and analysis of a verification standard at about 1-4 or 1-3 times the MDL level (see section 6.5 for details). Criteria for the verification standard are positive identification of the analyte using method criteria. If verification is not achieved, prepare and analyze a new verification standard at a higher concentration. The MDL in the LIMS becomes the concentration of the first verification standard to meet identification criteria. Alternatively, a new MDL study may be performed. This verification is performed on every instrument used for the analysis.

8.5.1 Petroleum related compounds or analytes must be verified by preparation and analysis of a verification standard at 1-2 times the MDL level in order to meet client-specific technical specifications.

8.6 The minimum number of replicates for an MDL study is 7. If greater than 7 replicates are run, all replicates must be used for MDL determination unless a replicate can be shown to be an outlier (as determined by a statistical analysis such as the Grubb's Test for Outliers (see section 8.5.1) or by an obviously defective run – poor purge, etc.).

8.6.1 Grubbs Test:

8.6.1.1 Decide whether the smallest or highest number is suspected of being the outlier

8.6.1.2 Perform the following calculation

$$T = \frac{X - X_1}{s} \text{ or } \frac{X_n - X}{s}$$

Where: X = average of MDL results
X_n = highest result
X_1 = lowest results
S = standard deviation

8.6.1.3 Refer to Table 1. If T is larger than the tabulated table value, rejection may be made with the associated risk. The Risk of False Rejection may not exceed 10%.

9.0 DATA REVIEW AND REPORTING

9.1 Quality Assurance reviews all MDL data. MDL study data may be submitted to clients on request.

9.2 The MDLs for range tests (such as DRO, GRO, EPH Carbon ranges, etc.) are generally defaulted to 70% or 75% of the reporting limit. However, if the MDL is above 70% or 75% of the RL, the MDL concentration must be inserted into the LIMS.

9.3 Complete the verification spreadsheet, and revise final MDL concentrations as required.

10.0 POLLUTION PREVENTION & WASTE MANAGEMENT

10.1 Not applicable for administrative SOP.

Table One
Values for Use in the Grubbs Test for Outliers

Number of Data Points	Risk of False Rejection 0.1%	0.5%	1%	5%	10%
3	1.115	1.155	1.155	1.153	1.148
4	1.496	1.496	1.492	1.463	1.425
5	1.78	1.764	1.749	1.672	1.602
6	2.011	1.973	1.944	1.822	1.729
7	2.201	2.139	2.097	1.938	1.828
8	2.358	2.274	2.221	2.032	1.909
9	2.492	2.387	2.323	2.11	1.977
10	2.606	2.482	2.41	2.176	2.036
15	2.997	2.806	2.705	2.409	2.247
20	3.23	3.001	2.884	2.557	2.385
25	3.389	3.135	3.009	2.663	2.486
50	3.789	3.483	3.336	2.956	2.768
100	4.084	3.754	3.6	3.207	3.017

Sample Manager: Scott Parsick

QA Officer: Robert Treggiari

TITLE: **CHAIN OF CUSTODY AND LOG-IN PROCEDURES**

REFERENCES: ACCNE

REVISED SECTIONS: **Removed Raytek IR Gun and replaced with IR Gun; 7.1, 10.2.2,**

1.0 SCOPE & APPLICATION

1.1 To maintain documentation of custody of all bottle sets, samples, digestates, distillates, and extracts that fall under the responsibility of Accutest Laboratories. This procedure describes sample receipt, unique sample number assignment, electronic documentation, and chain of custody. Procedures for resolving non-conformances, with Company Sample Acceptance policy are also described.

2.0 SUMMARY

2.1 Samples are delivered to the Accutest laboratories by company courier, commercial shipper or client self-delivery.

2.2 Custody documentation is signed upon receipt at the laboratory. The sample management technician reviews the chain of custody (COC) received with the samples and verifies that the information on the form corresponds with the delivered samples. The temperature of samples are measured and documented, and the sample are then assigned a unique Accutest sample number and stored at 2 – 6°C or –10 to -20°C (for certain volatiles samples).

2.3 Non-compliant information and discrepancies are documented and relayed to the Accutest client services group via an email, memo, or the “Chronicle Access Application” and the client will be notified. Additionally, the Sample Manager may contact the client directly.

2.4 Completed custody documentation is forwarded to the login group for data entry into LIMS.

3.0 METHOD DETECTION LIMIT

3.1 Not applicable

4.0 DEFINITION

4.1 ALIQUOT - a measured portion of a sample, or solution, taken for sample preparation and/or analysis.

4.2 CHAIN OF CUSTODY – a custody documentation form which is provided with each client job detailing all of the required sample information (sample ID, date/time of collection, matrix, analyses requested, number of bottles, preservative added, sampler initials, client name, address, and telephone number, and any additional specific requests).

4.3 CALIBRATION BLANK - a volume of acidified deionized/distilled water.

4.4 CONTAMINATION - a component of a sample or an extract that is not representative of the environmental source of the sample. Contamination may stem from other samples, sampling equipment, while in transit, from laboratory reagents, laboratory environment, or analytical instruments.

4.5 FIELD SAMPLE - a portion of material to be analyzed that is contained in single or multiple containers and identified by a unique sample number.

4.6 FIELD BLANK – this is any sample that is submitted from the field and is identified as a blank. This includes rinsates and equipment blanks, etc.

4.7 FIELD REAGENT BLANK (FRB) – A laboratory prepared field blank sample bottle with reagent water and sample preservatives (including HCL and ascorbic acid), sealed, and shipped to the sampling site along with empty sample bottles and returned to the laboratory with filled sample bottles. The same batch of ascorbic acid and HCL should be used for the field reagent blanks as for the field samples.

4.8 HOLDING TIME – the elapsed time expressed in days from the date of sampling until the date of its analysis.

4.9 LIMS – the Laboratory Information Management System.

4.10 MATRIX - the predominant material of which the sample to be analyzed is composed. For the purpose of this SOP, a sample matrix is either water or soil/sediment. Matrix is not synonymous with phase (liquid or solid).

4.11 NIST CALIBRATED THERMOMETER – a thermometer which is compared with a Standard calibrated at the National Institute of Standards and Technology (NIST), and found to be within one scale division. These thermometers are checked annually against an in-house NIST calibrated thermometer (which is calibrated annually by the manufacturer using NIST calibrated primary reference thermometers).

4.12 REAGENT WATER - water in which an interferant is not observed at or above the minimum quantitation limit of the parameters of interest. Accutest uses deionized water (municipal water which passes through Accutest's DI treatment system).

4.13 SUBCONTRACT ANALYSIS – sending the samples to an outside laboratory either within the Accutest system or an entirely separate laboratory. Subcontract labs are typically used for analyses which are not performed by Accutest.

4.14 SAMPLE LOGIN – the process of entering all required information from the COC into the LIMS. This process translates all information related to collection time, turnaround time, sample analysis, and deliverables into a code which enables client requirements to be electronically distributed to the various departments within the laboratory for scheduling and execution.

4.15 TURNAROUND TIME – the period of time from receipt of the samples to reporting of the results.

5.0 HEALTH & SAFETY

5.1 All safety practices must be followed as outlined in the Accutest Laboratories Employee Safety Handbook and Chemical Hygiene Plan. Safety glasses, gloves, and lab coats must be worn. All samples, solutions, and extracts must be treated as unknown and potentially hazardous.

5.1 The toxicity or carcinogenicity of each reagent used in this method has not been precisely determined; however, each chemical should be treated as a potential health hazard. Exposure to these reagents should be reduced to the lowest possible level.

5.2 All acids are corrosive and should be handled with care. Flush spills with plenty of water. If acids contact any part of the body, flush with water and contact supervisor.

6.0 COLLECTION, PRESERVATION & HOLDING TIMES

6.1 Not applicable

7.0 APPARATUS

7.1 IR temperature gun

7.2 NIST Calibrated thermometer

7.3 pH paper (0-14)

7.4 disposable pipette

7.5 LaMotte Chlorine test kit (Model No. LP-26)

7.6 Disposable test tubes

8.0 STANDARDS & REAGENTS

NOTE: All chemicals listed are reagent grade unless otherwise specified. Distilled, deionized water should be used whenever water is required. All applicable standard/reagent preparation information, including vendor, lot number, date of preparation, expiration date, calculations, and initials must be entered in the appropriate standard/reagent preparation logbook. Vendors typically used by Accutest include Fisher Scientific, VWR, Accustandard, Supelco, Chemservices, CPI, Ultra, and ERA. Additional vendors may be utilized as necessary.

8.1 H2SO4

8.2 NAOH solution

8.3 Hydrochloric acid

8.4 Nitric acid

8.5 Ascorbic acid

8.6 Sodium bisulfate

8.7 Purge and trap methanol

8.8 Refer to the SOP for sample preservative preparation (MSM161).

9.0 INTERFERENCES

9.1 Not applicable.

10.0 PROCEDURE

10.1 **Acceptance of Samples Policy:**

It is the responsibility of the client to ensure proper sampling and to forward samples to Accutest in accordance with proper preservation and storage protocol. Accutest will be responsible for sample integrity in the field only if the sampling has been performed by its personnel.

Samples must be received with proper documentation. The client may use an Accutest COC or their own chain of custody form. A fully completed Chain of Custody (COC) must be received with the samples (indicating proper relinquishing of samples to laboratory). This COC must include the following information:

- Sample identification (point of collection)
- Sample Matrix
- Date and time of collection
- Name or initial of sampler
- Preservation type
- Important comments special instructions (e.g. “Sample was field filtered” or “lab to filter sample”)
- Requested analysis (must be specific)
- Signature, date, and time of relinquish of samples to laboratory.

For Air Sample analysis; the Accutest Air Sampling Field Data Sheet should be thoroughly filled out with additional air specific information such as:

- Air type
- Canister serial number
- Canister size
- Flow Controller serial number
- Canister pressures at start and stop of sampling
- Weather, Atmospheric and Temperature information

Samples must be received in good condition, in the proper sample containers, properly preserved (including temperature - **see note below 10.2.2**), with sufficient volume to perform sample analysis, and adhering to method-specified holding times. The COC must be filled out in ink, and sample jars must be properly labeled in ink.

If any of the above criteria have not been met the client must be notified immediately, and provide a written request to proceed with analysis.

Accutest reserves the right to refuse acceptance of any sample which in its sole and absolute discretion and judgment is hazardous/toxic and poses or may pose a health, safety or environmental risk in handling or processing.

10.2 **External Chain of Custody Procedure** -Samples may be received via a carrier or by walk in. Upon receipt, sample management will review the custody document for the following information

10.2.1 If dictated by the project protocol, document the custody seal identification number used on the sample shipping container, and whether the seal was intact or broken. If the seal is broken upon receipt, contact the lab manager immediately.

10.2.2 The temperature of the samples in the cooler will be documented on the COC. Move packing material and ice away from the sample container or temperature blank if included. Insert an NIST calibrated thermometer into the temperature blank (if provided – refer to the Temperature Maintenance of Shipping Coolers SOP (MSM083) for details) or take the temperature of the sample container with the IR temperature gun (if no temperature blank provided) . If using the IR temperature gun, do not remove the sample from the cooler. Hold the gun a few inches from the sample container. Take the temperature of the outside of the sample container and record on the COC. Record temperature of a representative bottle (from the center of the cooler). Apply the applicable correction factor to the final temperature (refer to the tag on the thermometer or IR gun for the current correction factors). If the temperature is outside the control limits of 2-6°C. client needs to be notified and this must be documented. Client decisions of whether to perform or forgo analysis must be documented. If Non-Conformances occur it is noted in the comments section of the Accutest Laboratories Sample Receipt Summary Form (Note – temperatures less than 2°C do not have to be communicated to the client). The method of temperature reading must be recorded in the sample receipt application.

NOTE: If the samples have been collected within one hour of delivery, and the temperature of the samples have not yet reached 4°C ± 2°C – make a note of this on the COC. In this situation, sample temperature is considered acceptable since there has not been enough time for cooling to take place.

10.2.3 Verify the matrix of samples: WW-waste water, GW-ground water, DW-drinking water, SO-soil, SOL-other solid, LIQ-liquid, AIR-air, FB-field blank, TB-trip blank, etc.

10.2.4 Specific location from which the sample was taken.

10.2.5 Initials of the person who collected the samples.

10.2.6 Date and time of sample collection.

10.2.7 The volume and preservation of each bottle.

10.2.8 Signature of whom the sample was relinquished by, and to whom it was relinquished.

10.2.9 The analysis to be performed.

10.2.10 The client's name, address, and phone number.

10.2.11 Any special requirements, such as turnaround, or reporting deliverables.

10.2.12 For Army Corps Projects – the Cooler Receipt form must be completed.

10.2.13 For applicable clients, a sample receipt checklist must be completed (form number QA101).

10.3 The sample technician must document any inconsistencies between the samples received and what is stated on the COC. Any issues with the samples such as insufficient volume, incorrect or no preservatives, dates incorrect or missing, and insufficient holding times must be discussed and approved by the client before sample login can be completed. Additionally, if samples show any signs of damage or contamination, the client must be notified immediately. This notification (including any client decisions of whether to perform or forgo analysis) must be documented and attached to the project file. If, the analysis requested section of the custody is missing or ambiguous, the sample custodian may accept the samples if the needed information can be obtained before the end of the next working day. The sample custodian must make all reasonable efforts to contact the client and resolve any problems encountered on the chain of custody or with the samples. Any unclear project instructions must be clarified by the client, and this information must be documented in a telephone conversation log or a Sample Management internal memo that must be archived in the project folder. Any changes or additions must be verified in writing by the client. This verification is considered part of the chain of custody and will follow the original COC through the remainder of the login process.

10.4 Once the sample custodian is satisfied with the information on the chain of custody document, custody is transferred to the sample custodian by signing and dating the chain of custody. From this point the custody of the sample is the responsibility of the sample custodian.

10.5 **Refer to SOP MQA250-02 for instructions on how to enter samples into the LIMS.**

10.6 At this point, sample job numbers are assigned with the next available sample ID. Sample job ID numbers are prefixed with a 'M' followed by a 4-5-digit number assigned by the LIMS system (i.e., M4387). This sequence can be followed by an optional alpha suffix, to represent a relog. Sample numbers consist of a job number plus a second series of numbers beginning at one and continuing chronologically for each point of collection. The assigned sample ID must be written on the chain of custody form, in the Accutest ID section in black ink. If there are special circumstances regarding a sample (soluble), a Suffix may be included in the sample ID, for example, M4387-1A. The final portion of the sample ID documents the number of bottles received for each sample. For example, if there were 3 bottles provided for sample M4387-1, then the sample bottle labels on each bottle will be in the following order: M4387-1 Bot#1 , M4387-1 Bot#2 , M4387-1 Bot#3.

10.7 After custody has been accepted, the bottle set must be split. The Sample Receipt Log is completed electronically and bar code labels generated. The information required must be documented. This includes the client, date of receipt, the sample numbers, when assigned, the number of bottles, location, type of preservation, volume of each bottle, and the pH of each **bottle (Note: the pH of volatile samples is not checked during log-in. Sample pH for these analyses are checked at the instrument level, and documented in the analysis logbook).** To determine the pH an aliquot of sample is removed using a clean disposable pipette and applied to wide-range pH paper. The preservative is noted on the electronic log-in form by documenting it with the preservative type (that is, if the pH result is correct, the appropriate preservative is checked off). If preservation or volume is incorrect for the tests required, the client must be contacted immediately and informed that the test cannot be done until additional sample can be obtained (if preservative is added to the sample – it is also noted on the COC, and may be also noted in the Chronicle). Any comments should be noted on the COC or Chronicle, such as headspace in VOA vials, leaking bottles, or broken bottles.

Note: If preservative is added to a sample to achieve the proper pH, the same amount of

preservative must be added to the associated field blank. The amount of preservative added should be documented in the sample receipt program.

Note: Water samples for volatile analysis must be checked for headspace. The client must be notified of any samples exhibiting headspace greater than "pea size" (>6mm or 1/4 inch). The client must provide permission to analyze any samples with headspace greater than "pea-size", and this permission must be documented with the project.

10.8 Samples collected for cyanide analysis must be screened for residual chlorine using the LaMotte Chlorine test kit (refer to the SOP MSM143). Follow the directions attached to the test kit. Record the results in the residual chlorine log. If a sample is determined to contain residual chlorine, the client must be notified if samples for organic analyses are included with the project. For cyanide samples, add ascorbic acid (approximately 0.5 grams). Re-test the sample to determine that no residual chlorine is present. Add additional ascorbic acid as necessary. Care must be taken to prevent contamination. An aliquot of the sample is poured into the residual chlorine test tube.

10.8.1 Samples analyzed by EPA Method 608 (Pesticide/PCBs) that will not be extracted for 72 hours should be adjusted to a pH range of 5.0-9.0 with H_2SO_4 or a NAOH solution. Record the initial pH and adjusted pH and volume of acid or base used (if adjustment is necessary) in the "Chronicle Access Application".

10.9 The lab must wait 24 hours prior to digestion for aqueous metals samples that are preserved in the lab.

10.10 All bottles must be labeled with sample number, preservation and department which will need the bottle, and the shelf on which the sample is located.

10.11 Fast-track results. Samples that require an accelerated turnaround are handled in the following manner. The original custody must be clearly marked - red for 1 week or less. Copies must be made for each laboratory group that will be doing prep or analysis on the samples and marked the same as the original. The copies must be distributed to the lab groups as soon as possible, and no later than the end of the day in which the samples came in.

10.12 Composite Request. If the sample is to be composited, then the composite logbook must be used to document the procedure.

10.13 The original chain of custody and any other communications are then given to the administrative assistant for computer log in to the LIMS. The original COC and any other communications with the client are kept on file and available in Report Generation for reference.

10.14 **Internal Chain of Custody Procedure**

10.14.1 The purpose of an internal chain of custody is to document each date, time a sample changes hands. When a sample changes hands the recipient is responsible for the security of the sample.

10.14.2 Accutest uses an electronic internal chain of custody to document changes of custody in the laboratory.

10.14.3 When a sample(s) is received at Accutest, the external chain of custody is closed and the internal chain of custody is started for each sample bottle.

10.14.3.1 Sample management inputs the information from the COC into the LIMS, and assigns the bottles with a job and bottle number.

10.14.3.2 Administration logs the jobs and analysis information into the LIMS.

10.14.3.3 Once the analyst is ready to retrieve the sample from its proper location (as designated in the work list), the samples are removed from the refrigerator. The analyst enters his/her LIMS user name and password and scans each bottle label bar code. Once finished, the analyst scans the bottles back in, and returns them to the proper location.

10.15 Procedure for obtaining representative sub samples.

10.15.1 If it is necessary to obtain a sample to be sub-contracted to another facility or outside laboratory refer to the SOP for the Procedure for Obtaining Representative Sample Aliquot (MQA247) for details on the techniques used to obtain an appropriate sample aliquot.

10.15.2 In general, the sample is homogenized using metal spatulas (for non-metal analyses) or other Teflon-coated or wood mixing devices - or vigorous shaking if it is an aqueous sample - prior to collecting the sub sample. Once the sample is homogenized, the sub sample may be aliquoted into the applicable sample container. Sufficient sample must be aliquoted to perform the analysis.

10.16 Treatment and handling of foreign soils.

10.16.1 Foreign soils labeling. Foreign soils must be labeled with a special label available from Quality Assurance. This label clearly indicates that the soil is from a foreign source.

10.16.2 Sample bottles, shipping containers, and applicable instrument waste must be treated prior to disposal. All soil residues or samples must be dry-heated, incinerated, hydroclaved or autoclaved and marked as disposed. Soils in this facility are generally oven heated at 193-220°C for 4 minutes. Time starts when the entire sample has reached the required temperature. See the Compliance Agreement for other disposal type requirements. Large containers and shipping materials that can't be treated in the oven or autoclave must be thoroughly cleaned with a dilute bleach solution.

10.16.3 Upon soils receipt; sample management will login the samples and supply a LIMS generated number identifier. This must be recorded in the Foreign Soils Receipt Logbook, found in QA, along with the samples origin and date received. Sample Management personnel who handle the sample must read and sign off on the current USDA Compliance Agreement.

10.16.4 Any non-treated effluent from foreign sample preparation must be boiled for at least 2 minutes prior to disposal.

10.16.5 Samples shipped in non-standard sample containers (such as 5 gallon plastic buckets) must be enclosed in 2 heavy-duty plastic bags. The client must be informed of this requirement prior to shipment to the laboratory.

10.16.6 Prior to handling foreign soils, the analyst must read and sign off on the current USDA Compliance Agreement.

11.0 QUALITY ASSURANCE

11.1 Refer to the documentation section (12.0).

11.2 Internal Quality System audits of the Sample Management department will be scheduled annually. Refer to the Internal Audit SOP (MQA220) for details. Additional audits may be performed as necessary.

12.0 DOCUMENTATION

12.1 The sample receipt program must be reviewed by the Sample Manager and Quality Assurance on a periodic basis to ensure proper documentation of sample log in.

12.2 Once the job is logged into LIMS with the proper test codes, etc. they are double-checked by administration and the Laboratory Director or Laboratory Manager.

12.3 All client requests or revisions must be documented; preferably with a fax or printed email (telephone conversation log may be sufficient in certain situations). This data will be inserted in the project file as applicable. The “Chronicle Access Program” may also serve as documentation of special instruction.

12.4 External and internal sample chain of custody must be completed as described in the above sections (the internal COC may be generated directly from the LIMS).

12.7 Any hand corrections made on any laboratory document must be done using a single line through the error. The initials of the person and date of correction must appear next to the correction.

12.8 For subcontracted work - the lab must communicate in writing any samples that need special reports or handling. For example, the any samples requiring MCL exceedence reporting must be communicated (preferably on the COC) to the subcontract laboratory.

12.9 The lab must document the reason for all sample rejections. All samples should be provided with a job number to allow for proper rejection documentation.

13.0 DATA REVIEW

13.1 The COCs are reviewed by Administration and the Lab Director/Manager. Quality Assurance and/or Report Generation review the COCs during data package assembly or review.

14.0 DATA REPORTING

14.1 External and internal chain of custodies and/or the chronicle may be included in the deliverables on client request.

15.0 POLLUTION PREVENTION & WASTE MANAGEMENT

15.1 Pollution Prevention. Users of this method must perform all procedural steps that control the creation and/or escape of wastes of hazardous materials to the environment. The amounts of standards, reagents, and solvents must be limited to the amounts specified in this SOP. All

safety practices designed to limit the escape of vapors, liquids, or solids to the environment must be followed. All method users must be familiar with the waste management practices described in section 15.2.

15.2 Waste Management. Individuals performing this method must follow established waste management procedures as described in the Sample and Waste Disposal SOP. This document describes the proper disposal of all waste materials generated during the testing of samples as follows:

15.2.1 Non-Hazardous aqueous wastes
15.2.2 Hazardous aqueous wastes
15.2.3 Chlorinated organic solvents
15.2.4 Non-chlorinated organic solvents
15.2.5 Hazardous solid wastes
15.2.6 Non-hazardous solid wastes
15.2.7 Microbiological wastes

APPENDIX C
Health and Safety Plan Addendum

Health and Safety Plan Addendum
for
Pre-Design Investigation
Niagara Falls Air Reserve Station
Niagara Falls, New York

Prepared for

United States Department of the Air Force
Air Force Reserve Command, 914th Airlift Wing
Niagara Falls, New York
and
Air Force Center for Engineering and the Environment
Lackland Air Force Base, Texas

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January 2014
Version: FINAL
EA Project No. 62654.01

Health and Safety Plan Addendum
for
Pre-Design Investigation
Niagara Falls Air Reserve Station
Niagara Falls, New York

Prepared for

United States Department of the Air Force
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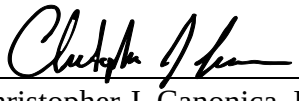


24 January 2014

Christopher J. Canonica, P.E., Program Manager — Date
EA Engineering, P.C.

24 January 2014

Peter Garger CIH, CSP — Date
Corporate Safety and Health Director

January 2014
Version: FINAL
EA Project No. 62654.01

CONTENTS

Page

LIST OF FIGURES

LIST OF ACRONYMS

CFR	Code of Federal Regulations
HASP	Health and Safety Plan
NYANG	New York Air National Guard
NYSDEC	New York State Department of Environmental Conservation
OSHA	Occupational Safety and Health Administration

1. INTRODUCTION

1.1 GENERAL

This Health and Safety Plan (HASP) Addendum is an addendum to the HASP associated with the 2010 Work Plan (EA 2010)[1] for the Installation-Wide Groundwater Monitoring Project for Niagara Falls Air Reserve Station in Niagara Falls, New York. The HASP Addendum contains site-specific information to protect the health and safety of personnel while performing work related to the Pre-Design Investigations at sites FT005, LF008, ST010, SS014, DS002, and DS004. Activities to be conducted at these sites include soil, groundwater sampling, and well installation.

This HASP Addendum describes the safety organization, procedures, and protective equipment that have been established based on an analysis of potential physical, chemical, and biological hazards. Specific hazard control methodologies have been evaluated and selected to minimize the potential for accidents or injuries to occur. One copy of the HASP and this HASP Addendum will be maintained for use during the scheduled field sampling efforts. The copies will be made available for site use and employee review at all times.

This HASP Addendum addresses regulations and guidance practices set forth in the Occupational Safety and Health Administration (OSHA) Standards for Construction Industry, 29 Code of Federal Regulations (CFR) 1926, including 29 CFR 1926.65, *Hazardous Waste Operations and Emergency Response* and 29 CFR 1926.59, *Hazardous Communications*.

The following are provided as attachments:

- **Attachment A**—Worker Training and Physical Examination Record
- **Attachment B**—Health and Safety Plan Review Record
- **Attachment C**—Site Entry and Exit Log
- **Attachment D**—Accident Investigation Report. If an accident occurs, 914th Remedial Program Manager will be contacted and provided this report.
- **Attachment E**—Emergency Telephone Numbers and Hospital Directions
- **Attachment F**—Emergency Equipment Available Onsite
- **Attachment G**—Map to Hospital
- **Attachment H**—Personal Protective Equipment Activity Record.

NOTE: This site-specific HASP Addendum should be left open to display Attachment E (Emergency Telephone Numbers and Hospital Directions) and made available to site personnel in a conspicuous location in the event of an emergency.

[1] EA. 2010. Work Plan for Installation-Wide Groundwater Monitoring Project for Niagara Falls Air Reserve Station, Niagara Falls, New York. August.

1.2 SITE AND FACILITY DESCRIPTION

The Niagara Falls Air Reserve Station is located in Niagara County, New York, approximately 15 mi north of the City of Buffalo, and 6 mi east of the City of Niagara Falls (Figure 1). The base covers approximately 547 acres in the towns of Wheatfield to the east and Niagara to the west (Figure 1). The 914th Airlift Wing has the primary installation mission, and trains reserve officers and airmen for combat-ready status for any national emergency. Current activities include airlifting troops and supplies, providing front line troops with personnel and logistical support, and providing medical evacuations.

1.3 SITE HISTORY

Niagara Falls Air Reserve Station was established as Niagara Falls Air Force Reserve Facility in November 1942. The federal government leased 468 acres of municipal airport land for use by the Army Air Corps. In 1946, 132.2 acres of the leased land were returned to the City of Niagara Falls. The 136th Fighter Squadron of the New York Air National Guard (NYANG) was established on 8 December 1948 and occupied Old Camp Bell near the Bell Aircraft Plant on the installation. The 76th Air Base Squadron was activated on 1 February 1952 as the installation host unit.

On 16 February 1953, the 518th Air Defense Group replaced the 76th Air Base Squadron as the host unit and the NYANG 47th Fighter Interceptor Squadron replaced the 136th Fighter Interceptor Squadron. In August 1955, the U.S. Air Force reactivated the 15th Fighter Group to replace the 518th Air Defense Group. On 1 July 1960, the 15th Fighter Group was deactivated and the 4621st Support Group began operations as the installation host unit. The 4621st Support Group was redesignated as the 4621st Air Base Group on 1 July 1964.

The North American Defense Command Defense System CIM-10B Boeing Michigan Aeronautical Research Center missile was deployed in the western portion of the installation in 1959. The 35th Air Defense Missile Squadron and the missiles were deactivated in the late-1960s, and the NYANG 107th Tactical Fighter Group became the tenant organization occupying the western portion of the installation.

The 49th Fighter Interceptor Squadron, 1 Detachment, assumed responsibility for the installation from the 4621st Air Base Group in March 1970. On 1 January 1971, the installation was transferred from the Aerospace Defense Command to the Air Force Reserve Command and the 914th Tactical Airlift Group became the host unit. The main tenant organization, NYANG 107th Tactical Fighter, was re-designated as the 107th Fighter Interceptor Group. In early-1992, the Niagara Falls Air Force Reserve Facility was renamed the Niagara Falls Air Reserve Station. In late 1993, the 107th Fighter Interceptor Group was re-designated as the 107th Air Refueling Wing and the 914th Tactical Airlift Wing was re-designated as the 914th Airlift Wing.

1.4 POLICY STATEMENT

EA will take every reasonable step to provide a safe and healthy work environment, and to eliminate or control hazards in order to minimize the possibility of injuries, illnesses, or accidents to site personnel. EA and EA subcontractor employees will be familiar with the HASP and this HASP Addendum for each of the project activities they perform. Prior to entering the site, the HASP and this HASP Addendum will be reviewed and an agreement to comply with the requirements will be signed by EA personnel, subcontractors, and visitors (Attachment B).

Operational changes that could affect the health and safety of the site personnel, community, or environment will not be made without approval from EA's Project Manager and Program Health and Safety Officer. This document will be periodically reviewed to ensure that it is current and technically correct. Any changes in site conditions and/or the scope of work will require a review and modification to the HASP Addendum. Such changes will be documented in the form of a revision to this addendum.

2. KEY PERSONNEL

The following table contains information on key project personnel:

Title	Name	Telephone No.
Officer-in-Charge	Gordy Porter	410-584-7000
Program Health and Safety Officer	Peter Garger, CIH	410-527-2425
Regional Program Manager/Consulting Engineer	Chris Canonica, P.E.	315-431-4610
Project Manager	Benjamin Young	770-789-5736
Site Manager	Frank DeSantis Jr.	315-395-7689
Field Team Leader	Lynette Mokry, P.G.	315-430-1786
Site Safety Health Officer	James Peterson	315-727-3308
Quality Assurance Officer	Jen Bouchard, P.G.	315-431-4610
Installation Restoration Program Project Manager	Kim Powell	716-236-3123

3. SCOPE OF WORK

This HASP Addendum was developed to designate and define site-specific health and safety protocols applicable to project activities to be implemented and followed during field activities and consulting work at the Niagara Falls Air Reserve Station, Niagara County, New York. The scope of work covered by this HASP Addendum includes:

- Pre-Design Investigation activities at the following sites:
  - FT005 - Site 10, Former Fire Training Area No. 1
  - LF008 - Site 3, Landfill
  - ST010 - Site 13, Closed 4,000 Gal UST
  - SS014 - Site 7, JP-4 Tank Truck Spill
  - DS002 - Site 8, Former Building 202 Drum Storage Yard
  - DS004 - Site 5, Former BOMARC Missile Site

Each of these activities is summarized below and additional detail for each activity is provided in the Installation-Wide Groundwater Monitoring Project Work Plan (EA 2010)[2].

3.1 MONITORING WELL INSTALLATION

Monitoring wells will be installed using hollow-stem and/or fliud rotary drilling methods. Newly installed monitoring wells will be developed using surge and purge techniques with a submersible pump.

3.2 SOIL AND GROUNDWATER SAMPLING

Subsurface soil samples will be collected using 4-ft acetate sleeves deployed using direct-push drilling techniques and sent to an offsite laboratory for analysis.

Groundwater samples will also be collected using direct-push sample techniques (e.g., the Geoprobe® SP-22 groundwater sampler or similar) or low-flow sampling techniques and sent to an offsite laboratory for analysis.

3.3 STORAGE AND DISPOSAL OF WASTE

EA is responsible for the proper storage, handling, and disposal of investigative-derived waste including personal protective equipment, and solids and liquids generated during field activities. Investigative-derived waste will be managed in accordance with New York State Department of Environmental Conservation (NYSDEC) Division of Environmental Remediation Technical and

2 EA. 2010. Work Plan for Installation-Wide Groundwater Monitoring Project. Niagara Falls Air Reserve Station, Niagara Falls, New York. August.

Administrative Guidance Memorandum 4032 (NYSDEC, 1989)[3] and in cooperation with personnel from the 914th MSG/CEV.

[3] NYSDEC. 1989. Technical and Administrative Guidance Memorandum #4032, Disposal of Drill Cuttings. 21 November.

4. POTENTIAL HAZARD ANALYSIS

Based upon the above field activities, the following potential hazard conditions may be anticipated:

- The use of mechanical equipment such as drill rigs, powered augers, and hammer drills can create a potential for crushing and pinching hazards due to movement and positioning of the equipment, movement of lever arms and hydraulics, entanglement of clothing and appendages in exposed drives and augers, and impact of steel tools, masts, and cables should equipment rigging fail or other structural failures occur during hydraulic equipment operation and drilling mast extension and operation. Heavy equipment work must be conducted only by trained, experienced personnel. If possible, personnel must remain outside the turning radius of large, moving equipment. At a minimum, personnel must maintain visual contact with the equipment operator. When not operational, equipment must be set and locked so that it cannot be activated, released, dropped, etc.

- Equipment can be energized due to contact with overhead or underground electrical lines, utilities impaired by excavation of communication or potable/wastewater lines, or a potential for fire or explosion may occur due to excavation of below ground propane/ natural gas lines. Prior to commencement of invasive operations, a drilling/excavation permit will be obtained and the area will be inspected and flagged. Personnel should be aware that although an area may be cleared, it does not mean that unanticipated hazards will not appear. Safe distances will be maintained from live electrical equipment as specified in the HASP (EA 2010)[1]. Workers should always be alert for unanticipated events such as snapping cables, digging into unmarked underground utilities, etc. Such occurrences should prompt involved individuals to halt work immediately and take appropriate corrective measures to gain control of the situation.

- Work around large equipment often creates excessive noise. Noise can cause workers to be startled, annoyed, or distracted; it can cause physical damage to the ear, pain, and temporary and/or permanent hearing loss; and it can interfere with communication. If workers are subjected to noise exceeding an 8-hour time-weighted average sound level of 85 dBA, hearing protection will be selected with an appropriate noise reduction rating to comply with 29 CFR 1910.95 and to reduce noise below levels of concern.

- Personnel may be injured during physical lifting and handling of heavy equipment, construction materials, or containers. Additionally, personnel may encounter slip, trip, and fall hazards associated with excavations, manways, and construction debris and materials. Precautionary measures should be taken in accordance with the HASP (EA 2010)[1]and this HASP Addendum.

- Field operations conducted during the winter months can impose excessive heat loss to personnel conducting strenuous activities during unseasonably cold weather days and can

impose cold-related illness symptoms during unseasonably cold weather days or when wind chill is high. In addition, heavy rains, electrical storms, and high winds may create extremely dangerous situations for employees.

- Entry into a confined space in support of this project is forbidden.

- System operations and field investigation activities intended to define potential sources of environmental contamination often require employees to be in direct proximity or contact with hazardous substances. Employees may be exposed through inhalation of toxic dusts, vapors, or gases. Normal dust particulates from surficial soil may have absorbed or absorbed toxic solvents, petroleum compounds, or toxic metal salts or metal particulates. Air monitoring equipment will be used to monitor airborne organic vapors and particulates. Real-time air monitoring for volatile organic compounds and/or particulate levels at the perimeter of the work area may be necessary. Monitoring activities will consist of a combination of continuous and periodic monitoring, which will be performed dependent upon the type of activity being conducted at the site. Water collected during well development and groundwater sampling activities may also contain toxic vapors, liquids, and gases and be inhaled during normal operations, or may be splashed onto the skin or eyes. Ingestion of toxic materials contained in dusts or particulates can be ingested if eating, smoking, drinking, and gum chewing are permitted prior to personnel washing their hands and face or removing contaminated work clothing and personal protective equipment. Some chemicals may be absorbed directly through the skin. Personal protective equipment, properly designed for the chemicals of concern, will always be provided and worn when a potential for skin contact is present.

The potential constituents of concern that may be present at the sites include volatile organic compounds and metals.

5. PERSONAL PROTECTIVE EQUIPMENT

Based upon currently available information, it is anticipated that Level D protection will be required for currently anticipated conditions and activities. If at any time the sustained level of total organic vapors in the worker breathing zone exceeds 5 parts per million above background, site workers will evacuate the area and the condition will be brought to the attention of the Site Health and Safety Officer. Efforts will then be undertaken to mitigate the source of the vapors. Once the sustained level of total organic vapors has decreased to below 5 parts per million above background, site workers will be allowed to continue activities at the direction of the Site Health and Safety Officer.

The personal protective equipment components for use during this project are detailed in the Work Plan and HASP (EA 2010)[1]. The components of Level D personal protective equipment are summarized below.

5.1 LEVEL D PERSONAL PROTECTIVE EQUIPMENT

Level D will be worn for initial entry onsite and initially for all activities and will consist of the following:

- Coveralls or appropriate work clothing
- Steel-toe, steel-shank safety boots/shoes
- Hard hats (when overhead hazards are present or as required by the Site Health and Safety Officer)
- Chemical resistant gloves (nitrile/neoprene) when contact with potentially contaminated soil or water is expected
- Safety glasses with side shields
- Hearing protectors (during drilling or other operations producing excessive noise)
- Boot covers (optional unless in contact with potentially contaminated soil or water)
- Polycoated coveralls (when contact with contaminated soil and water is anticipated, e.g., when surging/pumping wells and pressure-washing equipment)
- Insulated clothing, hats, etc. must be worn when temperatures or wind chill fall below 40°F.

6. SITE CONTROL AND SECURITY

Only authorized personnel will be permitted to conduct field activities. Authorized personnel include those who have completed hazardous waste operations initial training, as defined under OSHA Regulation 29 CFR 1910.120/29 CFR 1926.65, have completed their training or refresher training within the past 12 months, and have been certified by a physician as fit for hazardous waste operations.

6.1 SAFE WORK PRACTICES

Safe work practices that will be followed by site workers include, but are not limited to, the following rules:

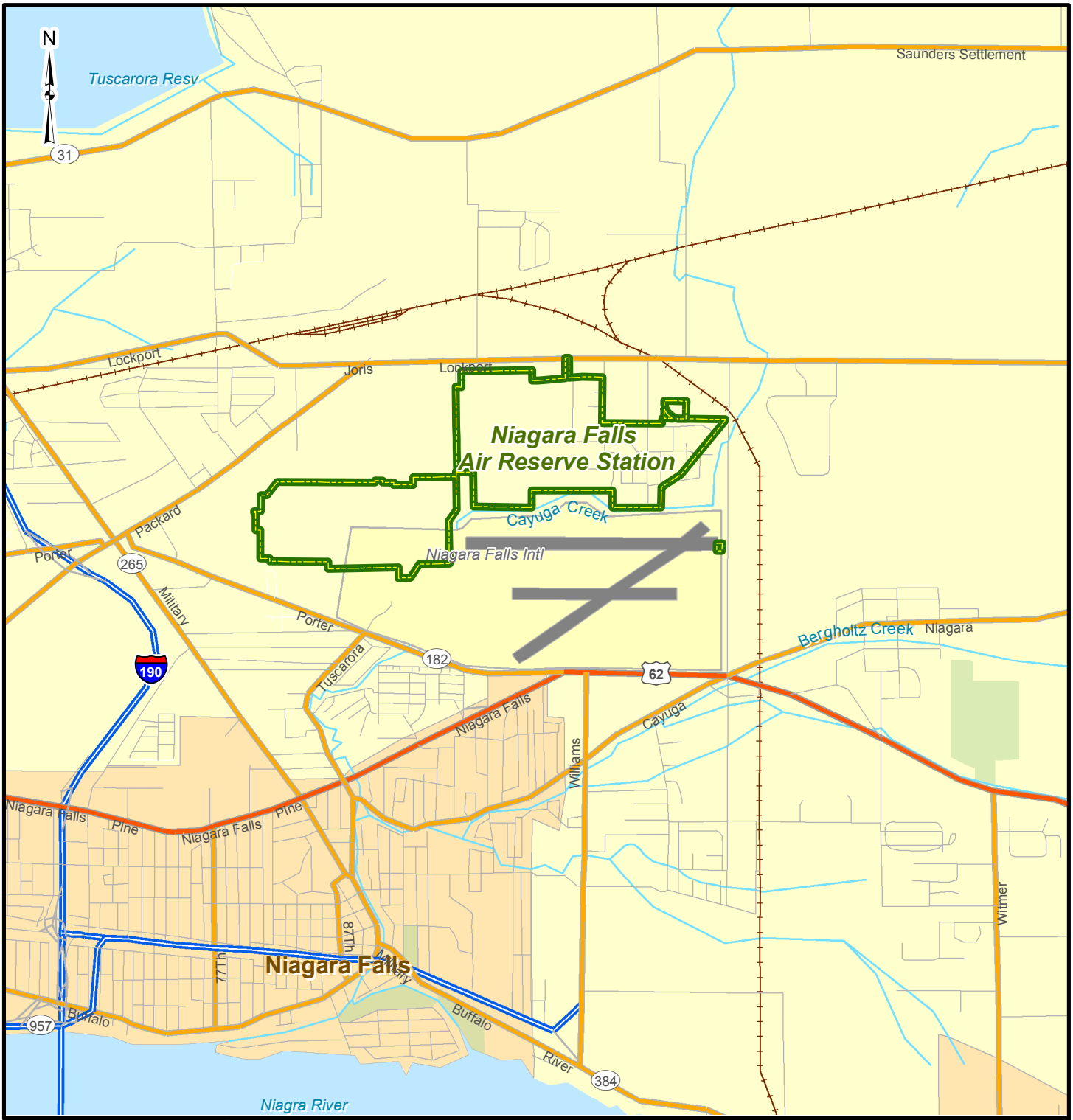
- The work day is from 7:00 a.m. to 4:00 p.m.
- Do not enter restricted or posted areas without permission and escort from base operations.
- Smoking is limited to designated areas.
- Possessing, using, purchasing, distributing, or having controlled substances in their system throughout the day or during meal breaks is prohibited.
- Consuming or possessing alcoholic beverages is prohibited.
- Good housekeeping – employees will be instructed about housekeeping throughout field activities.
- Sitting or kneeling in areas of obvious contamination is prohibited.
- Avoid to the extent possible overgrown vegetation and tall grass areas.
- Entry into flightline areas will only occur when escorted by Niagara Falls Air Reserve Station flightline trained personnel.

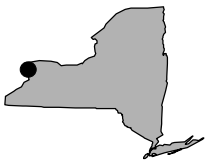
6.2 DAILY STARTUP AND SHUTDOWN PROCEDURES

The following protocols will be followed daily prior to start of work activities:

- The Site Health and Safety Officer will review site conditions to determine if modification of work and safety plans is needed.

- Personnel will be briefed and updated on new safety procedures as appropriate.
- Safety equipment will be checked for proper function.
- The Site Health and Safety Officer will ensure that the first aid kit is adequately stocked and readily available.
- The Contractor is responsible for the security of its own equipment. All onsite equipment and supplies will be locked and secure.





0 0.4 0.8 1.2 1.6 2

Miles

EA®	Health and Safety Plan Addendum Niagara Falls Air Reserve Station Niagara Falls, New York	FIGURE 1 Site Location Map

PROJECT MGR:	DESIGNED BY:	CREATED BY:	CHECKED BY:	SCALE:	DATE:	PROJECT NO:	FILE NO:
BY	FDJR	FDJR	BY	AS SHOWN	AUG 2013	6242107	G:\Projects\Federal\DOD\6265401

Attachment A

Worker Training and Physical Examination Record

ATTACHMENT A

WORKER TRAINING AND PHYSICAL EXAMINATION RECORD

SITE: Niagara Falls Air Reserve Station, Niagara Falls, New York

Name	OSHA 40-Hour Hazardous Waste Operations Training		OSHA Hazardous Waste Supervisor Training	Confined Space Training	CPR (date of expiration)	First Aid (date of expiration)	Date of Last Physical Examination
	Initial	Annual					
EA PERSONNEL							
DeSantis, Frank, --	3/26/07	3/19/13	1/9/12	7/6/10	1/15/2015	1/15/2015	10/21/2011
Miller, Megan, E.	7/12/07	6/5/12	8/28/08	---	1/15/2015	1/15/2015	10/17/2012
Mokry, Lynette, A.	2/23/90	11/21/12	6/94		1/15/2015	1/15/2015	10/13/2011
Peterson, Robert, J.	9/15/08	6/5/12	---	7/6/10	1/08/2013	1/08/2013	9/2009
Williams, Hilary, A.	8/21/09	6/15/12	---	---	1/15/2015	1/15/2015	8/28/2012
Yarrington, Charles, S.	8/31/12	N/A	---	8/31/12	8/30/2014	8/30/2014	5/2012
Young, Benjamin, A.	5/89	6/5/12	6/92	---	1/15/2015	1/15/2015	12/6/2011
SUBCONTRACTOR OR ADDITIONAL PERSONNEL							
---	---	---	---		---	---	---
---	---	---	---		---	---	---

NOTE: Prior to performing work at the site, this Health and Safety Plan must be reviewed and an agreement to comply with the requirements must be signed by all personnel, including contractors, subcontractors, and visitors. Contractors and subcontractors are ultimately responsible for ensuring that their own personnel are adequately protected. All personnel onsite shall be informed of the site emergency response procedures and any potential safety or health hazards of the operations.

Attachment B

Health and Safety Plan
Review Record

ATTACHMENT B

HEALTH AND SAFETY PLAN REVIEW RECORD

I have read the Health and Safety Plan for this site and have been briefed on the nature, level, and degree of exposure likely as a result of participation in this project. I agree to conform to all the requirements of this Plan.

SITE: Niagara Falls Air Reserve Station, Niagara Falls, New York			
Name	Signature	Affiliation	Date

Attachment C

Site Entry and Exit Log

ATTACHMENT C

SITE ENTRY AND EXIT LOG

SITE: Niagara Falls Air Reserve Station, Niagara County, New York				
Name	Date	Time of Entry	Time of Exit	Initials

Attachment D

Accident Investigation Report

ACCIDENT/LOSS REPORT

THIS REPORT MUST BE COMPLETED BY THE INJURED EMPLOYEE OR SUPERVISOR AND FAXED TO EA CORPORATE HUMAN RESOURCES WITHIN 24 HOURS OF ANY ACCIDENT. THE FAX NUMBER IS **(410) 771-1780**.

NOTE WHENEVER AN EMPLOYEE IS SENT FOR MEDICAL TREATMENT FOR A WORK RELATED INJURY OR ILLNESS, PAGE 4 OF THIS REPORT MUST ACCOMPANY THAT INDIVIDUAL TO ENSURE THAT ALL INVOICES/BILLS/CORRESPONDENCE ARE SENT TO HUMAN RESOURCES FOR TIMELY RESPONSE.

A. DEMOGRAPHIC INFORMATION:

NAME OF INJURED EMPLOYEE:_____
HOME ADDRESS:_____
HOME PHONE:_____ DATE OF BIRTH:_____
AGE:_____ SEX: M F
MARITAL STATUS:_____ NAME OF SPOUSE (if applicable)_____
SOCIAL SECURITY NUMBER:_____ DATE OF HIRE:_____
NUMBER OF DEPENDENTS:_____
EMPLOYEES JOB TITLE:_____
DEPT. REGULARLY EMPLOYED:_____
WAS THE EMPLOYEE INJURED ON THE JOB: Y N
PRIMARY LANGUAGE OF THE EMPLOYEE: _____

B. ACCIDENT/INCIDENT INFORMATION:

DATE OF ACCIDENT:_____ TIME OF ACCIDENT:_____
REPORTED TO WHOM: _____ NAME OF SUPERVISOR_____
EXACT LOCATION WHERE ACCIDENT OCCURRED (**including street, city, state and County**):
_____
_____
EXPLAIN WHAT HAPPENED (include what the employee was doing at the time of the accident and how the accident occurred): _____
_____
_____
_____

DESCRIBE THE INJURY AND THE SPECIFIC PART OF THE BODY AFFECTED (i.e., laceration, right hand, third finger):
_____
_____

OBJECT OR SUBSTANCE THAT DIRECTLY INJURED EMPLOYEE:_____
_____
NUMBER OF DAYS AND HOURS EMPLOYEE USUALLY WORKS PER WEEK:_____
IS THE EMPLOYEE EXPECTED TO LOSE AT LEAST ONE FULL DAY OF WORK?_____
DOES THE EMPLOYEE HAVE A PREVIOUS CLAIM? Y N if yes, STATUS Open Closed
WAS THE EMPLOYEE ASSIGNED TO RESTRICTED DUTY? _____

C. ACCIDENT INVESTIGATION INFORMATION

WAS SAFETY EQUIPMENT PROVIDED? Y N If yes, was it used? Y N
WAS AN UNSAFE ACT BEING FORMED ? Y N If yes, describe_____
WAS A MACHINE PART INVOLVED? Y N If yes, describe _____
WAS THE MACHINE PART DEFECTIVE? Y N If yes, in what way _____
WAS A 3RD PARTY RESPONSIBLE FOR THE ACCIDENT/INCIDENT? Y N
If yes, list Name, address and phone number_____
_____
WAS THE ACCIDENT/INCIDENT WITNESSED? Y N
If yes, list Name, address and phone number: _____
_____

D. PROVIDER INFORMATION

WAS FIRST AID GIVEN ON SITE? Y N
If yes, what type of medical treatment was given _____
PHYSICIAN INFORMATION (if medical attention was administered)
NAME:_____
ADDRESS (incl. City, state and zip):_____
PHONE:_____

HOSPITAL ADDRESS (incl. Name, address, city, state, zip code & phone)
_____
_____

WAS THE EMPLOYEE HOSPITALIZED? Y N If yes, on what date_____
WAS THE EMPLOYEE TREATED AS AN OUTPATIENT, RECEIVE EMERGENCY TREATMENT OR AMBULANCE SERVICE? _____

PLEASE ATTACH THE PHYSICIANS WRITTEN RETURN TO WORK SLIP

***NOTE* A PHYSICIANS RETURN TO WORK SLIP IS REQUIRED PRIOR TO ALLOWING THE WORKER TO RETURN TO WORK**

E. AUTOMOBILE ACCIDENT INFORMATION (complete if applicable)

AUTHORITY CONTACTED AND REPORT #_____
EA EMPLOYEE VEHICLE YEAR, MAKE AND MODEL_____

V.I.N._____ PLATE/TAG #_____
OWNER'S NAME AND ADDRESS:_____
_____
DRIVER'S NAME AND ADDRESS: _____
_____
RELATION TO INSURED:_____DRIVER'S LICENSE #_____
DESCRIBE DAMAGE TO YOUR PROPERTY:_____
_____
_____
DESCRIBE DAMAGE TO OTHER VEHICLE OR PROPERTY:_____
_____
_____
OTHER DRIVER'S NAME AND ADDRESS: _____
_____
OTHER DRIVER'S PHONE:_____
OTHER DRIVER'S INSURANCE COMPANY AND PHONE:_____
_____
LOCATION OF OTHER VEHICLE:_____
NAME, ADDRESS AND PHONE OF OTHER INJURED PARTIES:_____
_____
_____
WITNESSES

NAME:_____ PHONE:_____
ADDRESS:_____
STATEMENT:_____
_____
SIGNATURE: _____

NAME:_____ PHONE:_____
ADDRESS:_____
STATEMENT:_____
_____
SIGNATURE: _____

F. ACKNOWLEDGEMENT

NAME OF SUPERVISOR:_____
DATE OF THIS REPORT: _____ REPORT PREPARED BY:_____

I have read this report and the contents as to how the accident/loss occurred is accurate to the best of my knowledge.

Signature: _____ Date: _____
Injured Employee

I am seeking medical treatment for a work related injury/illness.

Please forward all bills/invoices/correspondence to:

EA ENGINEERING, SCIENCE, AND TECHNOLOGY, INC.

11019 McCORMICK ROAD

HUNT VALLEY, MD 21031

ATTENTION: Michele Bailey
HUMAN RESOURCES

(410) 584-7000

INCIDENT REPORT

THIS REPORT IS TO BE COMPLETED WHEN A NEAR MISS OCCURS THAT COULD HAVE POTENTIALLY RESULTED IN SERIOUS PHYSICAL HARM. PLEASE FAX THIS FORM TO EA HUMAN RESOURCES DEPARTMENT AT **(410) 771-1780**.

EXPLAIN WHAT HAPPENED (include what the employee was doing at the time the near miss and how it occurred:) _____

REPORT PREPARED BY: _____ DATE:_____

Attachment E

Emergency Telephone Numbers and Hospital Directions

ATTACHMENT E

EMERGENCY TELEPHONE NUMBERS AND HOSPITAL DIRECTIONS

SITE: Niagara Falls Air Reserve Station, 2405 Franklin Drive, Niagara Falls, New York	
Police: Niagara Falls Air Reserve Station Security	(716)-236-2280
Fire: Niagara Falls Air Reserve Station Fire Department	(716)-236-2117
Ambulance:	(716)-236-2117
Hospital: Memorial Hospital, Niagara Falls, New York	(716) 278-4000
New York Regional Poison Control Center: 750 East Adams Street, Syracuse, New York	(315) 723-7000 800-222-1222
Directions to Memorial Hospital, 621 10th Street, Niagara Falls, New York Exit the base at the Lockport Road gate (main gate), turn left on Lockport Road continue 1.4 miles to Packard Road. Continue on Packard Road approximately 3 miles turn right on Pine Avenue continue approximately 0.2 miles, bear right onto Maple Avenue for 1.8 miles. Turn right on 10th Street. Head east on Lafayette Street approximately 1.5 miles. Arrive at Memorial Hospital.	
EA Program Safety and Health Officer ***Peter Garger, CIH***	(410) 527-2425
Program Manager: ***Christopher Canonica, P.E.***	(315) 431-4610
Niagara Falls ARS Project Manager: **Kim Powell/Ellen Marien**	(716) 236-3123/(716) 236-3126
Versar Project Manager: ***Nathan Mullens***	(843) 338-1851
EA Project Manager: ***Ben Young***	(770) 789-5736
EA Medical Services All One Health ***Contact: Dr. Jerry Berke***	800-350-4511
Site Manager/Site Health and Safety Officer ***Frank DeSantis Jr.***	(315) 395-7689
In case of accident or exposure incident, contact EA Corporate Health and Safety Officer ***Peter Garger, CIH***	(410) 527-2425

Attachment F

Emergency Equipment Available Onsite

ATTACHMENT F

EMERGENCY EQUIPMENT AVAILABLE ONSITE

Type of Equipment	Location
Communications Equipment	
Mobile Telephone	In EA vehicle
Medical Support Equipment	
First Aid Kits	In EA vehicle/Site 10 Trailer
Eye Wash Station	In EA vehicle/Site 10 Trailer
Fire Fighting Equipment	
Fire Extinguishers	In EA vehicle/Site 10 Trailer

Attachment G

Map to Hospital

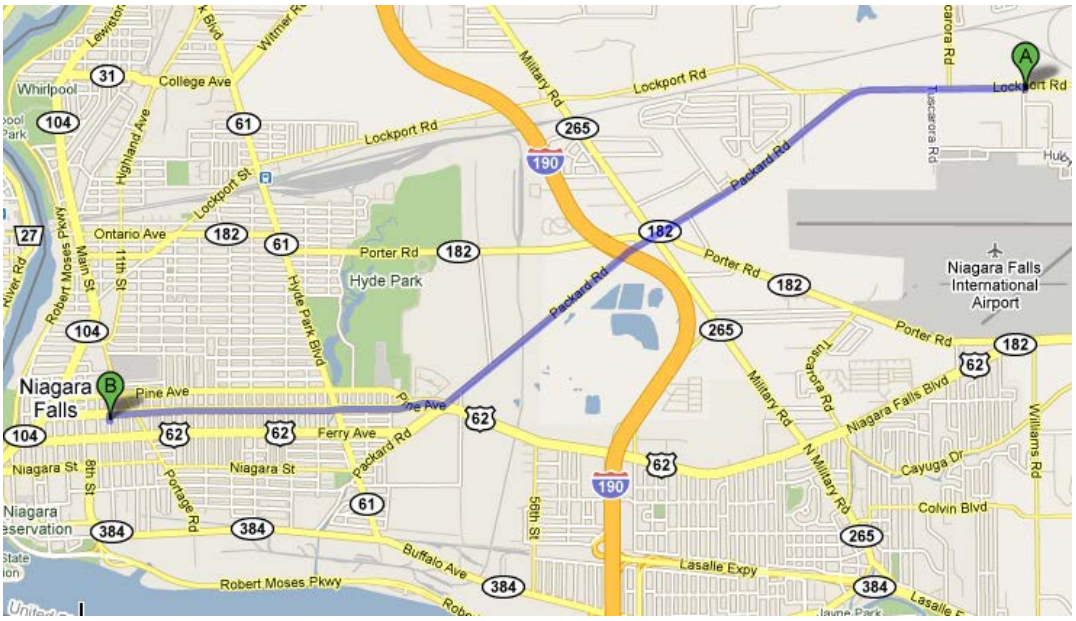
ATTACHMENT G

MAP TO HOSPITAL

Directions to Niagara Falls Memorial Medical Center, 621 10th St Niagara Falls, NY 14301

Exit the base at the Lockport Road gate (main gate), turn left on Lockport Road continue 1.4 miles to Packard Road. Continue on Packard Road approximately 3 miles turn right on Pine Avenue continue approximately 0.2 miles, bear right onto Maple Avenue for 1.8 miles. Turn right on 10th Street. Head east on Lafayette Street approximately 1.5 miles. Arrive at Memorial Hospital.

Total trip is 6.2 miles; travel time is approximately 15 minutes.



Attachment H

Personal Protective Equipment Activity Record

ATTACHMENT B

PERSONAL PROTECTIVE EQUIPMENT ACTIVITY RECORD

SITE: Niagara Falls Air Reserve Station, Niagara Falls, New York		
Weather Condition:		Onsite Hours: From To
Changes in Personal Protective Equipment Levels[(a)]	Work Operations	Reasons for Change
Site Health and Safety Plan Violations	Corrective Action Specified	Corrective Action Taken (yes/no)
Observations and Comments:		
Completed by: _____ Site Health and Safety Officer		Date
(a) Only the Site Health and Safety Officer may change personal protective equipment levels, using only criteria specified in the Health and Safety Plan.		