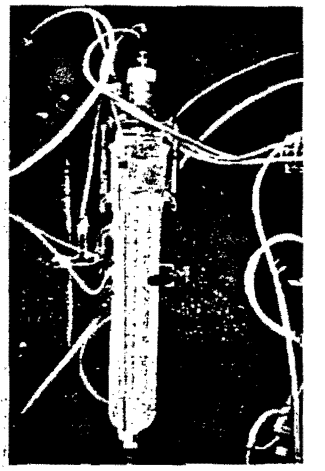
Laboratories



Quality Assurance Management Plan

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EA Manual EAL-001
Revision No. 7
July 1997

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EA Laboratories

EA LABORATORIES

QUALITY ASSURANCE MANAGEMENT PLAN

REVIEW AND APPROVAL

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1. INTRODUCTION

1.1 THE QUALITY POLICY

EA Laboratories, a business unit within EA Engineering, Science, and Technology, Inc., is dedicated to providing its clients with on-time, error free analytical hardcopy and electronic data which are technically valid and legally defensible, and which meet the Data Quality Objectives (DQOs) identified for data use. Accordingly, to ensure achievement of these goals, EA Laboratories has established a formal Quality Assurance (QA) program with the following objectives:

- Define personal and technical performance expectations,
- Train laboratory staff in order to achieve personal and technical excellence,
- Implement procedures to monitor the analytical process,
- Establish a records management system for documentation of process activities, and
- Assess laboratory compliance with specified requirements.

The human factor in any system plays a pivotal role in the ultimate quality of the data. Many times, it is human intervention and interpretation which is seemingly excluded in the design and management of quality systems. Yet the laboratory should not be viewed as a "black box" for the generation of data points, and it is the expressed goal of the EA Laboratories Quality Program that each member of the laboratory staff is a fully participating TEAM player in the analytical effort. It is expected that each member of the laboratory is more than a conduit for sample analysis, providing the consultative skills associated with individual areas of expertise, and exercising those skills to the benefit of our clients. It is further expected that the management of EA Laboratories encourages excellence in analytical testing and provides the necessary resources and environment to develop analytical assessment skills to identify problems with all processes associated with their function, and to take appropriate corrective actions.

EA Laboratories QA program objective is to maximize the use of expertise available to the program through collaboration. With this as an introduction, it should be clear that each individual has a vital role to play in the overall attainment of quality analytical results.

1.2 THE QUALITY SYSTEM

The total "quality" of any analytical result is the sum of all the system parts that generate and report the value. The system is not static and, by its very nature, should be constantly evolving to reflect current quality improvements and technology changes. All parts of the system must be open to review and modification if significant quality improvement is to occur over time. The Quality System is comprised of related activities covering quality assurance, quality control, and quality assessment (U.S. EPA 1976, Taylor 1985; ASTM 1988).

1.2.1 Quality Assurance

Quality Assurance refers to the system of activities whose purpose is to provide to the producer or user of a product or a service the assurance that it meets defined standards of quality with a stated level of confidence. The Quality Assurance program elements include the policies, organization, objectives, functional activities, and specific activities designed to achieve the desired quality goals for laboratory operations.

1.2.2 Quality Control

Quality Control refers to the overall system of activities whose purpose is to control the quality of a product or service so that it meets the needs of the users. Quality control includes the routine activities and checks, such as periodic calibrations, duplicate analyses, use of spiked samples, etc., included in normal internal procedures to control the accuracy and precision of the measurement process (U.S. EPA 1976). Also incorporated in this are those activities conducted on an occasional basis by persons outside of the data generation process, such as supervisory review of data, updating control limits, training, etc.

1.2.3 Quality Assessment

Quality Assessment refers to the system of activities whose purpose is to provide assurance that the overall quality control job is being done effectively. Quality assessment involves a continuing evaluation of the performance of the data generation system and the data produced with a view to having corrective measures initiated where necessary. Quality assessment includes those activities that are performed on an occasional basis, usually initiated and performed by persons outside of normal routine operations, such as on-site system audits, submission of performance evaluation samples, deliverables review, etc., to assess the capability and performance of the data generation process (U.S. EPA 1976).

1.3 QUALITY SYSTEM DOCUMENTATION

The foundation for the Quality System is a tiered documentation hierarchy. The first tier consists of the EA Corporate manuals which describe the Corporate Quality Assurance program, and administrative policies and procedures. The second tier documentation consists of the laboratory Quality Assurance Management Plan, laboratory standard operating procedures (SOPs), analytical methods, training records, and laboratory notebooks. The third tier includes all Quality Assurance Project Plans which describe how the QA program is implemented to meet project specific data quality goals.

1.3.1 EA Corporate Quality Assurance Program Manual (EA QAPM)

EA's Corporate Quality Assurance Program Manual provides the policies, procedures, and action taken by corporate management to assure the results of studies, analyses, and other work meet internal EA minimum requirements, and are acceptable to both clients and regulatory agencies. The manual is maintained by the Chief Administrative Officer (CAO) who is responsible for establishing the corporate total quality program which is implemented by management staff.

1.3.2 EA Laboratories Quality System Documentation

1.3.2.1 Quality Assurance Management Plan (QAMP)

The purpose of the QAMP is to describe EA Laboratories' Analytical Chemistry quality assurance program. The QAMP derives elements from the EA QAPM, and specifically provides the baseline from which the quality program associated with environmental sample analysis can continually improve quality and productivity. By its very nature, the QAMP is constantly evolving to reflect current quality improvements and technology changes.

The manual is organized around the elements required by the U.S. EPA (1980a,b) for QA program and project plans. These basic elements include:

- personnel and training;
- facilities and equipment;
- sample custody;
- calibration and preventive maintenance;
- analytical methods;
- quality control procedures;
- data reduction, validation, and reporting;
- audits;
- corrective action;
- assessment

A glossary of terms used throughout the plan is included in Appendix A.

The Quality Services Manager (QSM) is responsible for the production, distribution, and updating of the QAMP. The QAMP is distributed as a controlled document with an active distribution list maintained by the QSM. At a minimum, the QAMP is reviewed and updated on an annual basis.

1.3.2.2 Laboratory Standard Operating Procedures (SOPs)

SOPs for laboratory operations are approved and issued by the QSM. The laboratory SOP Manual (EAL-002) is distributed as a controlled document, and assigned to a designated person within each area. As SOPs are issued, revised or withdrawn, a new table of contents is distributed with the

updated SOPs. A circulation list is given to each SOP manual holder so that each member of the laboratory can read the update and acknowledge understanding by signing the list. The circulation list and the outdated SOPs are returned to the QSM.

The QSM is responsible for verifying that outdated SOPs are not in use in the laboratory during internal audits. A list of laboratory SOPs can be found in the Table of Contents of EAL-002, EA Laboratories Standard Operating Procedures.

1.3.2.3 Analytical Methods SOPs

EA Laboratories uses laboratory specific Methods for all preparation, cleanup and determinative analyses. The laboratory Methods Manual (EAL-005) is distributed as a controlled document, and assigned to a designated person within each area. These protocols are derived from standard environmental methods, and include additional information, including instrument configuration, calibration standard concentrations, quality control sample acceptance criteria and corrective actions. Methods are approved by the Section Chief and the QSM.

The QSM is responsible for verifying that outdated method SOPs are not in use in the laboratory during internal audits. A list of method SOPs can be found in the Table of Contents of EAL-005, EA Laboratories Analytical Methods.

1.3.2.4 Training Records

Training records are maintained for each temporary and permanent employee and include documentation on employee orientation, technical training, safety training, LIMS training, quality improvement training, and proficiency certifications. The training records are the primary responsibility of the employee and his/her immediate supervisor. The QSM is responsible for auditing training records to verify that the information is complete and current.

1.3.2.5 Laboratory Notebooks

Laboratory notebooks include logbooks for standards preparation, instrument/analysis runs, and instrument maintenance. Copies of the standards preparation and instrument/analysis logs are included in all full data packages (EAL-SOP-304) and are reviewed by the analyst and QC Chemist as part of the technical data review process. Instument maintenance notebooks are reviewed by the area Section Chief and QC chemist on a on a routine basis. The QSM is responsible for verifying that notebooks are properly maintained during internal audits.

1.3.3 Quality Assurance Project Plan (QAPP)

A QAPP is usually required for environmental data operations performed under U.S. EPA oversight. The U.S. EPA has established guidance for the QAPP format and content (EPA 1994). The QSM is responsible for coordinating laboratory input for all QAPPs as requested by the client. This ensures that the analytical approach specified for the project will meet the Data Quality Objectives identified in the QAPP.

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2.0 LABORATORY ORGANIZATION

2.1 STRUCTURE

EA Laboratories is a business unit within EA Engineering, Science, and Technology, Inc. (Figure 2-1) providing comprehensive environmental analytical services to internal and external clients. The organizational structure of EA Laboratories is shown in Figure 2-2.

EA's Corporate Quality Function is the responsibility of the Project Risk Executive who reports directly to the Chief Executive Office of EA Engineering, Science, and Technology, Inc. and is responsible for providing oversight for EA Laboratories QA program. Specific responsibilities to EA Laboratories include:

- Reviews and approves EA Laboratories Quality Assurance Management Plan
- Reviews Quality Assurance Project Plans prepared by the laboratory.
- Acts in a role of advise and consent to QSM.
- Coordinates quality system improvement initiatives among Operations, Branches and the laboratory.

The responsibilities of the EA Laboratories staff can be outlined as follows (Taylor 1984):

Upper management	establishes policy; provides resources to implement program; oversight.
Supervisory staff	implementation of the program; general supervision.
Technical staff	technical competence to carry out daily operations; strict adherence to methods and procedures; identification of any technical defects in program and recommendation of corrections.
Quality Services Staff	oversight of QA program; advisory assistance to supervisory staff; evaluation of effectiveness of QA program; recommendation of policy.

2.2 LABORATORY STAFF POSITION ROLES AND RESPONSIBILITIES

All laboratory personnel are involved with the QA program. The extent of their involvement depends on their assignment in the laboratory.

2.2.1 Laboratory Director

The Laboratory Director reports directly to the Chief Executive Officer of EA Engineering, Science, and Technology, Inc. The Director has the following responsibilities:

- Provide sufficient and appropriate resources essential to the implementation of quality policies and the achievement of quality objectives. Resources may include:

  - Laboratory facility with sufficient space and engineering controls to facilitate proper performance of all operational and support activities.
  - Laboratory equipment and reference materials for the correct performance of analyses.
  - Personnel staff with sufficient training and background to ensure implementation of the quality system.

- Identifies quality factors affecting market position and objectives relative to new products, processes, or services in order to allocate resources on a planned and timely basis.
- Determines the level of competence, experience, and training necessary to ensure the capability of personnel.
- Provides the managerial staff with the authority and resources necessary to discharge their duties.
- Ensures personnel are free from any commercial, financial, and other pressures which might adversely affect the quality of their work.
- Establishes and documents the responsibility, authority, and interrelation of all personnel who manage, perform, or verify work affecting the quality of the client deliverable.

2.2.2 Quality Services Manager

The Quality Services Manager reports directly to the Laboratory Director, and is responsible for the quality system and its implementation. The Quality Services Manager is responsible for the following:

- Develops EA Laboratories QA program.
- Establishes documentation requirements for all procedures, quality documents and records.
- Directs assessment of the QA program through internal performance, systems and data audits with follow-up to verify implementation of corrective actions
- Manages laboratory certifications.
- May exercise authority to shut down any instrument, method or operational group if an out-of-control situation persists without corrective action.
- Provides oversight for all external inspections, coordinates written response to findings, and maintains audit records.

- Establishes personnel training program, assists with training activities, maintains training records, and provides feedback to management with status of staff training activities.
- Manages the Laboratory Health and Safety Program.

2.2.3 Business Manager

The Business Manager reports directly to the Laboratory Director, and is responsible for the following:

- Oversees all financial activities within the laboratory, including budgets, procurement, invoicing, cost accounting.
- Has a reporting responsibility to the Chief Financial Officer of EA Engineering, Science, and Technology, Inc.
- Manages all administrative activities within the laboratory.
- Ensures staff are qualified and trained, and documents qualifications and training according to the laboratory policies and procedures.

2.2.4 Client Services Manager

The Client Services Manager reports directly to the Laboratory Director, and is responsible for the following:

- Coordinates all client services group activities to ensure quality of services provided to clients.
- Ensures staff are qualified and trained, and documents qualifications and training according to the laboratory policies and procedures.
- Establishes and maintains a documented communication mechanism between Client Services and the laboratory to ensure that all contractual and operational reporting requirements are met.
- Establishes and maintains a mechanism to determine client satisfaction.

2.2.5 Laboratory Project Manager

Each Laboratory Project Manager reports to the Client Services Manager, and is responsible for the following:

- Serves as point source for client-laboratory contact through project duration.
- Responsible for identifying project specific QA/QC requirements.
- Coordinates projects for the duration of their life cycle within the laboratory.
- Ensures coordination of production efforts, on-time delivery of data packages which meet all client specifications for parameters, methods, quality control, and report format.

2.2.6 Sample Management Officer

The Sample Management Officer reports to the Client Services Manager, and is responsible for the following:

- Receives, logs, and assigns control numbers to incoming samples.
- Follows standard operating procedures and QA/QC requirements for all analyses performed and assignment of samples for analysis and storage by other analysts.
- Responsible for sample storage facilities. Maintains a log record on these facilities, including temperature of storage rooms, and procedures for sample storage area.
- May assist in training and supervising technicians in analyses and quality control procedures for sample tracking.
- Follows all laboratory safety rules.

2.2.7 Information Systems Manager

The Information Systems Manager reports directly to the Business Manager, and is responsible for the following:

- Responsible for development of the laboratory QA program for automated systems, and for staff training.
- Responsible for the site preparation, and onsite configuration of hardware and software for EA Laboratories' Laboratory Management Information System (LIMS).
- Identifies custom programming needs, and prepares protocols for system operation.
- Responsible for routine system maintenance.

2.2.8 Operations Manager

The Operations Manager reports directly to the Laboratory Director, and is responsible for the following:

- Is responsible for all operational and support activities within EA Laboratories to ensure client satisfaction with laboratory performance in all areas of concern, including service, deliverables, on-time delivery.
- Ensures staff are qualified and trained, and documents qualifications and training according to the laboratory policies and procedures.
- Develops cross-functional training program to increase efficiency within the laboratory and to improve production operations.
- Evaluates equipment and personnel needs within operations groups.

- Ensures all operations groups follow the QA program and works closely with the QSM to maintain compliance.

2.2.9 Section Chief

Each Section Chief reports to the Operations Manager, and is responsible for the following:

- Participates in planning laboratory programs on the basis of specialized knowledge of problems and methods and probable value of results.
- Assist the Operations Manager in one or more areas of overall management of the analytical laboratory, including personnel, physical plant, and financial budgeting and planning.
- Troubleshoots problems regarding analytical procedures and equipment performance.
- Performs quantitative and qualitative analyses using manual or specialized and complex instrumental methods.
- Fully competent and proficient in the operation of sophisticated scientific equipment.
- Interprets results, prepares reports, and provides technical advice in specialized area.
- Supervises and trains staff in methods of analyses, standard operating procedures, and QA/QC requirements.
- Provides advice to the Operations Manager in budgetary and personnel matters.

2.2.10 Quality Control Chemist

Each Section Quality Control (QC) Chemist reports to the Operations Manager, and is responsible for the following:

- The QC Chemist initiates and coordinates all quality control measures for the section.
- Assists the Section Chief in implementing all quality control measures and to provide leadership in producing the data and data package quality which meets client specifications.
- Monitors and verifies the status and quality of analytical data within the section.
- Responsible for coordinating and facilitating the section(s) interaction with the Quality Services and Client Services departments to ensure that clients' expectations and requirements are met and the section's performance meets and exceeds such criteria.
- Reviews training documentation for section staff to ensure analysts have the qualifications and training to perform quality work and generate acceptable packages.
- Responsible for 100% technical review of all data packages and preparation of the narrative.
- Responsible for compliance review against all applicable documents including some or all of the following: Methods, Project Summaries, Quality Assurance Project Plans, and Quality Assurance Program Plans.
- Determines methodologies and instrumentation required to meet project requirements.
- Ensures section staff implementation of and compliance with all applicable Standard Operating Procedures (SOPs) and Method SOPs.

2.2.11 Chemist

Each Chemist reports to the appropriate Section Chief. They are responsible for the on-schedule performance and documentation of all analyses assigned. Other responsibilities include:

- Compliance with all applicable Standard Operating Procedures (SOPs) and Method SOPs.
- Understands and follows all aspects of the QA program and project specific QA/QC requirements as they pertain to work assignments.
- Evaluates and documents results of all quality control parameters.
- Remains alert to any conditions that may affect data quality and reports them immediately to his/her immediate supervisor.
- Initiates, signs, and submits Nonconformance Records to his/her section QC Chemist within 24 hours of occurrence.
- Maintains his/her assigned work area in a neat, clean and orderly fashion.

2.2.12 Technician

Each Technician reports to the appropriate Section Chief, and is responsible for the following:

- Compliance with all applicable Standard Operating Procedures (SOPs) and Method SOPs.
- Understands and follows all aspects of the QA program and project specific QA/QC requirements as they pertain to work assignments.
- Maintains complete records of activities as required.
- Follows all sample disposal and documentation procedures, in compliance with EA Laboratories Chemical Hygiene Plan and SOPs
- Maintains his/her assigned work area in a neat, clean and orderly fashion.

2.3 FUNCTIONAL ROLES

In addition to positions within the organizational structure of EA Laboratories, there are functional roles which can be assigned to qualified personnel at the discretion of the Laboratory Director regardless of their position within the organization.

2.3.1 QC Chemist Function

This function is performed by a senior level Chemist who is responsible for the following:

- Works according to priorities set by the Operations Manager.

- Reviews assigned data packages for compliance with all applicable documents including some or all of the following: methods, Project Summaries, Quality Assurance Project Plans, and Quality Assurance Program Plans.
- Interacts with LPMs, the Operations Manager, and Quality Services staff when sample nonconformance is identified in order to determine client options.
- Provides guidance to analysts on analytical requirements for assigned analyses.
- Maintains current copies of EA Laboratories Methods.

2.3.2 Group Leader

This function is performed by a senior level Chemist who is responsible for the following:

- Directs the coordination of work assignments for laboratory technicians and chemists, monitoring workload to ensure completion of analyses within required time frame and SOPs.
- Determines methodologies and instrumentation required to meet project requirements.
- Reviews data requirements and evaluates data deliverables for deficiencies in contractual compliance or internal quality control standards.
- Assists in planning for expansions or purchases to increase the efficiency and output of the laboratory.
- Ensures section data quality is compliant with contractual requirements or internal quality control requirements.
- Prepares from experience and skills new procedures specifications, SOPs, methods development and other documentation necessary for section performance.
- Troubleshoots instrumentation and keeps abreast of changing technology. Provides technical support for chemists, technicians, project managers and clients.

2.4 DEPUTIES FOR KEY PERSONNEL

In the event of the absence of key laboratory personnel, EA Laboratories has designated persons who are authorized to act in his or her behalf (Table 2-1).

2.5 PERSONNEL QUALIFICATIONS

Each position within EA Laboratories is described in an official EA Position Description, which describes general duties, responsibilities, authority, etc. In addition, functional descriptions are used to describe specific or unique duties. Functional descriptions are approved by the Laboratory Director. The EA Corporate Human Resources Policies and Procedures Manual and the EA Employee Manual (May, 1996) contains general policy and instructions. Minimum requirements of education and experience have been established for each position and are included in each Position Description.

Educational backgrounds and documented work experience of all degreed EA Laboratories employees are summarized in their respective Professional Profiles, which are prepared by each employee at time of employment and updated annually at time of performance evaluation. Complete resumes and company application are contained in confidential personnel files maintained by EA Human Resources.

2.6 TRAINING

EA Laboratories uses a formal program for the training of employees. A defined sequence of events for familiarization and training is followed to prevent placing the employee in a situation that is unfamiliar. EA Laboratories employees are provided on-the-job training and certification, which cover the following areas: employee orientation, safety training, LIMS training, and technical training.

2.6.1 Employee Orientation

Employee orientation is conducted for each new staff member to familiarize them with the laboratory staff, company policies and benefits, LIMS, safety protocols, and the QA program. Orientation is documented in a checklist (Figure 2-3) which is added to the employee's Training Record upon completion.

2.6.2 Technical Training

Technical training is the responsibility of the employee's immediate supervisor. The training and certification procedures are documented in the checklists shown in Figures 2-4 and 2.5. No employee is permitted to perform any technical procedure unless proficiency has been demonstrated (EAL-SOP-180).

2.6.3 Training Records

Training documentation is maintained in a Training Record (EAL-SOP-295) which is initiated at orientation and maintained by the QSM. Training records are audited on a regular basis as part of EA Laboratories internal audit program. It is the responsibility of the employee and their supervisor to document all technical training and to submit proficiency documentation to the QSM. It is the responsibility of the QSM to verify that training records are current and complete.

TABLE 2-1 AUTHORIZED DEPUTIES FOR KEY PERSONNEL

KEY PERSONNEL	DEPUTIES
Laboratory Director	Business Manager Operations Manager Quality Services Manager
Business Manager	Operations Manager Client Services Manager
Quality Services Manager	Laboratory Director Designated Quality Control Specialists
Operations Manager	Business Manager Section Chiefs
Client Services Manager	Business Manager Operations Manager
Laboratory Project Manager	Client Services Manager Alternate Designated Laboratory Project Manager

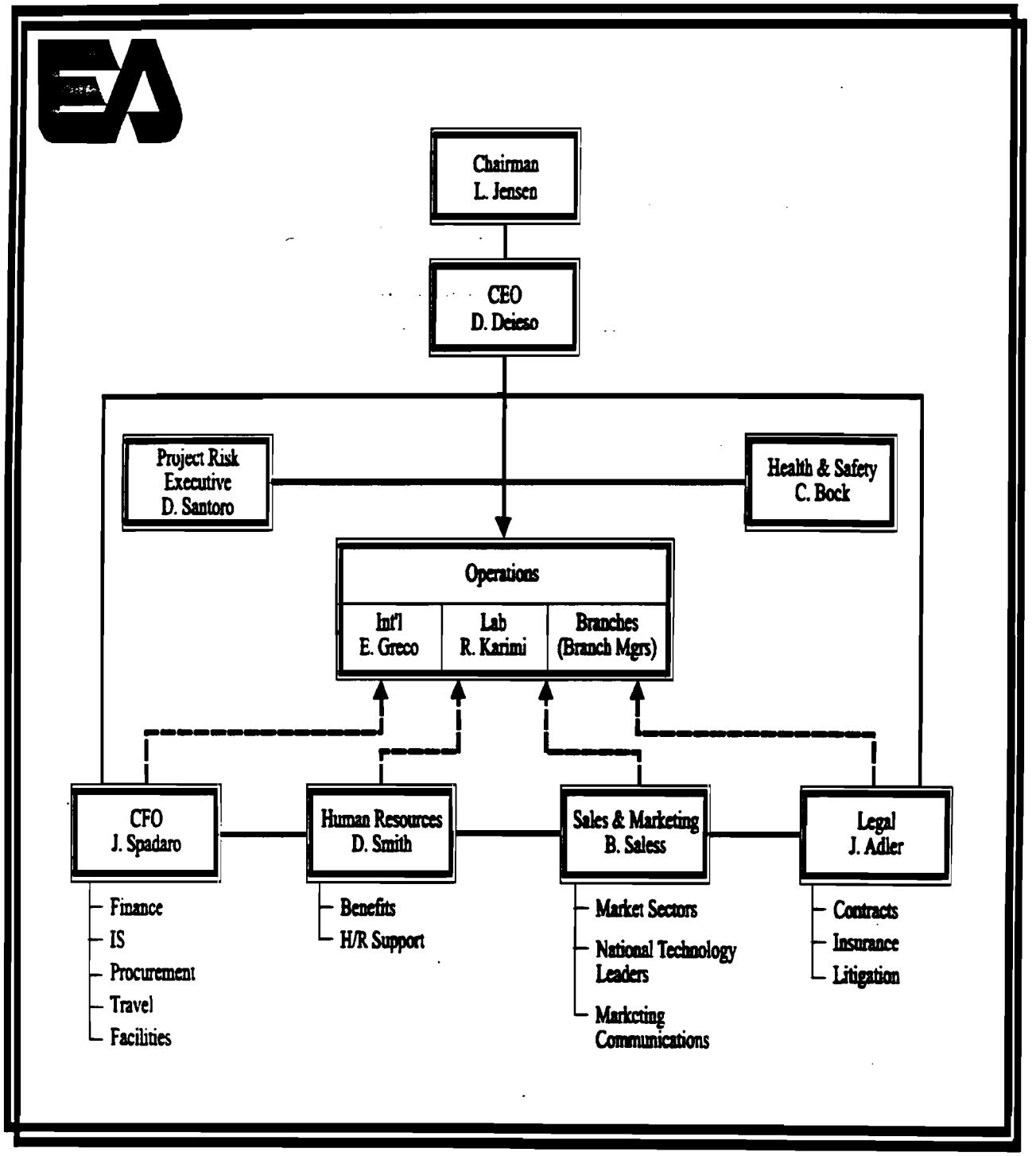

Figure 2-1. Organizational Structure of EA Engineering, Science, and Technology, Inc.

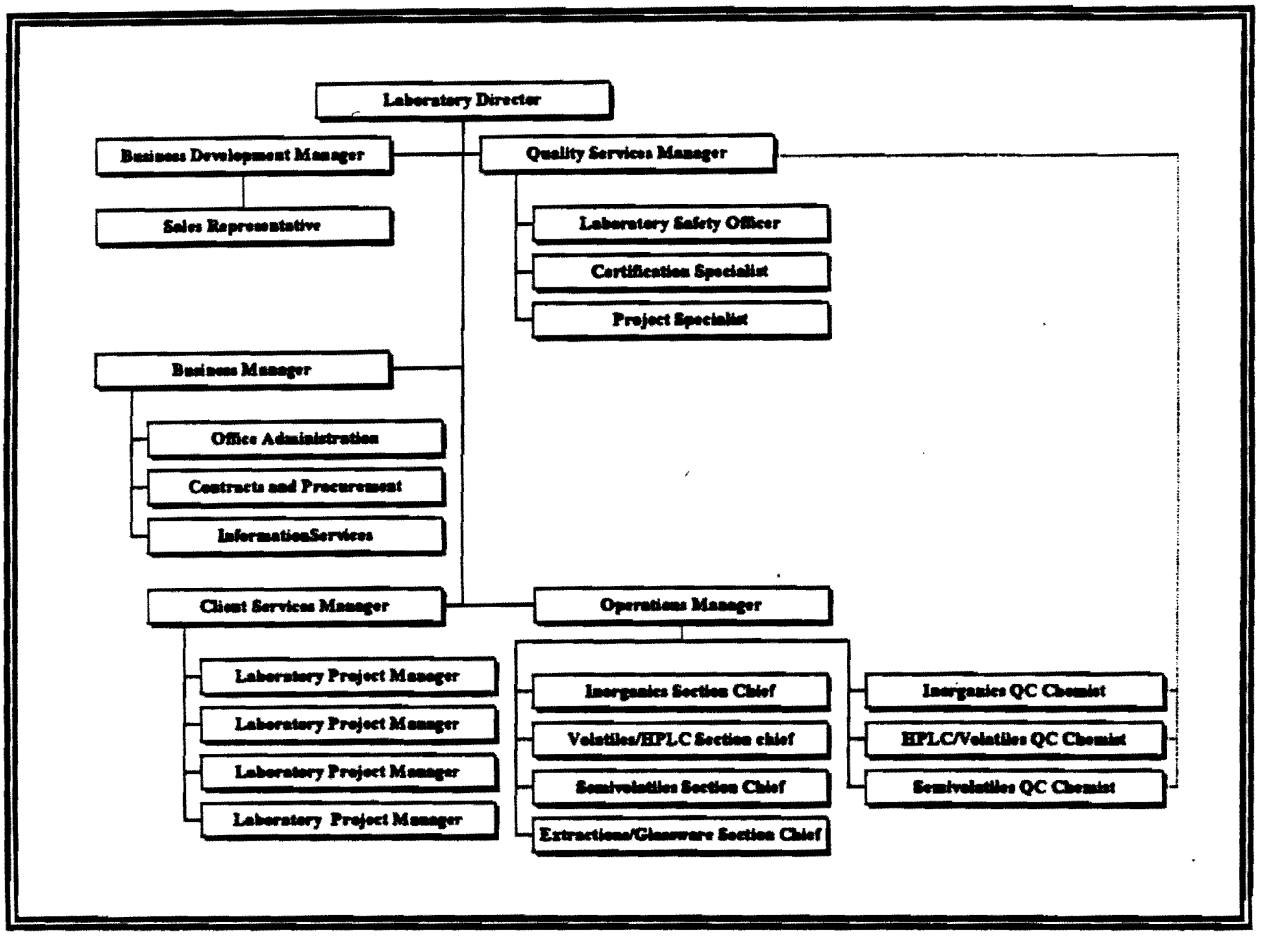

Figure 2-2. Organizational Structure of EA Laboratories

EA LABORATORIES
ORIENTATION AND GENERAL TRAINING RECORD

Employee _____ Job Title _____

Date of Hire _____ Budget Group _____

Check each item as it is covered with the employee. After each session, the trainer signs and dates the form.

1. Human Resources - *Trainer* _____ *Date* _____
   - ____ Job description provided and discussed with the employee.
   - ____ EA corporate and laboratory organization discussed.
   - ____ EEO policies discussed.
   - ____ Employee signed Signature Logbook.
   - ____ Facility toured and functions within areas explained.

2. Information Systems - *Trainer* _____ *Date* _____
   - ____ Overview of systems and capabilities provided.
   - ____ Training needs assessed and scheduled.
   - ____ Passcard issued.
   - ____ LAN and LIMS accounts opened.

3. Health and Safety - *Trainer* _____ *Date* _____
   - ____ Overview of Chemical Hygiene program provided, issued copy of Chemical Hygiene Plan.
   - ____ Training needs assessed and scheduled.
   - ____ Respirator fitting required and scheduled.
   - ____ Physical and inoculations required and scheduled.
   - ____ Employee "Right to Know Training " completed

4. Quality Assurance - *Trainer* _____ *Date* _____
   - ____ EA Laboratories Quality Assurance Program training completed.
   - ____ Required EA Laboratories General SOPs List distributed and completion dates agreed upon.
   - ____ Uncontrolled copy of EA Laboratories Quality Assurance Manual (EAL-001) provided.

5. Supervisor - *Trainer* _____ *Date* _____
   - ____ Explained how work assignments will be given.
   - ____ Discussed reporting relationship, how feedback occurs, formal and informal.
   - ____ Identified tools and resources available to the employee.
   - ____ Explained need for confidentiality and sense of professionalism.
   - ____ Introduced the employee to the assigned Sponsor.
   - ____ Provided training schedule and tasks to employee.

Figure 2-3. Employee Orientation Checklist

TECHNICAL TRAINING RECORD

Employee _____ Job Title _____

Date of Hire _____ Procedure _____

The Trainer initials each applicable item as it is covered with the employee. After completion of training, the employee, Section Chief, and Quality Services Manager sign and date the form.

_____ Discussed the training SOP with the trainee.

_____ Distributed a uncontrolled copy of the analytical SOP or reference procedure to the trainee. This copy is to be turned into the supervisor/trainer at the end of the training period.

_____ Had the trainee read the applicable procedure.

_____ Discussed the procedure and clarify sections that the trainee does not understand

_____ Discussed safety concerns related to the procedure.

_____ Demonstrated the procedure including technique and quality control.

_____ Had the trainee conduct the procedure while the trainer observes and discusses the procedure with the trainee.

_____ Evaluated performance against QC criteria for procedures and discuss any out of control. situations.

_____ Explained routine instrument maintenance specified in the appropriate instrument manual and have trainee observe when needed.

_____ Had the trainee perform required review and documentation.

_____ Reviewed trouble shooting and repair protocol.

_____ **ALL PERFORMANCE DOCUMENTATION IS ATTACHED TO THIS FORM, including precision and accuracy study (must be completed for methods which require a P&A study), or blind performance evaluation analysis, or four compliant laboratory control samples.**

Employee *Date*

Section Chief *Date*

Quality Services Manager *Date*

Figure 2-4. Technical Training Form

3. FACILITIES AND EQUIPMENT

3.1 LABORATORY PHYSICAL LAYOUT

The building occupied by EA Laboratories Analytical Chemistry Laboratory at 19 Loveton Circle, Sparks, Maryland is a 17,600 square-foot, single-story brick building with a small mechanical penthouse on the second floor. The building is located on a five-acre site in an industrial park, north of Baltimore, Maryland. Figure 3-1 shows a floor plan of the laboratory, depicting major safety features. In addition, exterior structures provide space for storage of analytical samples, hazardous waste material, and compressed gases as well as mechanical systems.

3.2 LABORATORY UTILITIES

A variety of services are provided to the laboratory on a centralized basis, including potable water, ASTM Type I and Type II water, compressed air, vacuum, and general-use compressed gases (e.g., nitrogen and argon). Specialized gases (e.g., oxygen, helium, etc.) are provided on a localized basis to specific laboratories and/or instruments. The specialized gases are delivered in gas cylinders and chained in place as required.

The building is wired to provide 110- or 220-volt service at single or double phase as required. In addition, a 75-kW emergency generator provides emergency electrical service so that all fume hoods, certain supply fans, and dedicated outlets are automatically energized in the event of a power interruption by Baltimore Gas and Electric Company. Critical freezers, refrigerators, and instruments are connected to the emergency circuits. Instruments in the Metals and Wet Chemistry area are also fed from a centralized 150-kV Conditioned Power Transformer, which protects critical instruments from line surges.

3.3 VENTILATION AND HOOD SYSTEMS

General Practices: Exhaust hoods are used for all operations that may result in the release of toxic gases, vapors, fumes, or mists. Materials stored in hoods are kept to a minimum; these items must not block vents or air flow. The hoods are left on when not in use if toxic or volatile substances are stored in the hood or in a flammable storage unit below the hood.

System Design: The following are features of the HVAC systems designed to ensure the safety of occupants and the prevention of sample contamination:

- All fume hood exhaust fans are monitored by differential pressure switches such that, upon fan failure, an alarm condition is annunciated both audibly and visually at the hood location.

Operating policy provides for the proper actions of occupants upon the occurrence of an alarm condition at any hood. Upon alarm condition at a fume hood:

- If either sash is open above 3 in., the sash must be closed fully and the alarm reset. (If the alarm is cleared but re-alarms when the sash is above 3 in., it is not safe to operate the hood in the fully opened position.)
- If neither sash is open above 3 in., or if alarm does not clear as described above, operations in the fume hood must be stopped and the sash(es) closed.

- All exhaust fans for fume hoods are connected to the emergency generator.
- All supply and return fans associated with the air handlers that serve laboratories are supplied with emergency power when the main system fails.
- The air systems for laboratories were designed and balanced to provide proper pressurization for each room.
- The Organics VOA laboratory is maintained at positive pressure to surrounding areas and is supplied with 100 percent outdoor air to prevent contamination of samples or analytical procedures by means of return air from other laboratories.
- Air from the Extractions laboratory is completely exhausted to prevent contamination of samples in other laboratories.
- To further protect samples, the layout of return air paths to various air handlers was designed to prevent the possible cross-contamination of supply air by different laboratories.
- All fume hoods have the maximum amount of air exhausted from them whenever any sash is higher than 3 in. above the fully closed position, ensuring the safety of operating personnel.
- Exhaust air from fume hoods cannot be turned off, unless they are specifically designated as hoods that are not normally used; EA policy provides that such hoods should only be turned off when they are not in use.

Records are kept by the QS Manager to document that EA's facilities and precautions are compatible with current federal, state and local standards and regulations.

3.4 GLASSWARE

3.4.1 Selection

The laboratory glassware is specifically selected by EA Laboratories to meet the requirements of all inorganic and organic analyses conducted. Certain types of glassware are specified by methods or contract protocols; other types are selected by the specialists in each area. All volumetric glassware is Class A Pyrex or Kimax. Repair of damaged glassware is handled under an annual service contract by an outside vendor.

3.4.2 Cleaning

The laboratory is equipped with a Lancer Glassware Washer with automatic programmable wash and rinse cycles as follows:

Three washing levels
Detergent wash
Acid wash
Demineralized water rinse

The exact time in seconds can be varied for each of the above parameters as well as temperature of the wash or rinse cycles. Specific cycles are preprogrammed for Organics and Inorganics glassware.

The facility also includes several different drying ovens, as follows:

Lancer Drying Oven Model 14
- Three drying levels, 50-250 °C
- Two hot air filtered turbines
- Size: ~ 6 cu. ft.

Organic Oven
- Range: 50-550 °C
- Operating Temperature: 500 °C
- Size: 60 cu. ft.

In addition to the automatic washing procedures, manual washing of glassware is handled in specified sinks and fume hoods. The following SOPs describe specific cleaning procedures used for inorganic and organic glassware and sampling equipment washing and preparation:

EAL SOP No.	Title
062	Glassware cleaning procedure for inorganic and metals determinations
069	Cleaning procedures for bailers
033	Cleaning procedures for organic glassware
078	Operation of annealing oven controller

3.5 REAGENTS, SOLVENTS, GASES, AND STANDARDS

All reagents, solvents, gases, and standards used at EA Laboratories are purchased in accordance to specifications using EA purchasing policies. Each individual requiring any item shall prepare a standard Purchase Requisition (Form EA 0034, 7/27/84), identifying suggested vendor, address, and phone number and also including any special instructions, priority information, etc., obtain appropriate departmental approval, and forward the requisition to EA Laboratories Purchasing

Specialist. The Purchasing Specialist evaluates the suggested vendor, identifies alternative vendors, recommends final vendor, and obtains the required EA Laboratories approvals, up to and including Director, EA Laboratories. Once final approval has been obtained, an EA Purchase Order (Form EA 0420, 12/21/88) is prepared, signed, and issued to the vendor.

When a vendor delivers supplies, the Purchasing Specialist inspects the shipment and compares it with the original purchase requisition for item number, quantity, catalog number, etc. Supplies are distributed to requesting individual/department supervisor/manager, who stores the item in accordance with the type of supply. Specific storage cabinets are available for acids, solvents, refrigerated, and explosive samples. Original labels must be maintained as issued by manufacturing vendor and classification noted on container. The Section chief is responsible for maintaining records of reagent lot testing, date received, date opened, and expiration date.

Shipping information, bills of lading, etc., are forwarded to the Purchasing Specialist, EA Laboratories. The Material Safety Data Sheet (MSDS) is compared to existing MSDS sheets on file by the Purchasing Specialist and filed according to EAL-SOP-089. No container of chemicals (e.g., reagents, solvents, gases or standards) will be accepted that is not adequately labeled. The Purchasing Specialist is responsible for arranging return of any unacceptable shipment.

In certain instances, vendors will be required to furnish certificates of quality or other detailed testing information describing the quality of the shipment. This material will be reviewed by the Purchasing Specialist or Section Chief prior to acceptance of the supplies and release of these supplies to the laboratory areas. All traceability documentation is maintained by the appropriate Section Chief

Consumable supplies such as laboratory glassware, pipet tips, gloves, and sample containers are stored in the laboratory warehouse. The Operations Manager is responsible for maintaining and tracking the inventory.

3.6 ANALYTICAL INSTRUMENTATION

A current listing of the major analytical instrumentation used by EA Laboratories is maintained by the Business Manager. Service contracts are maintained for most of the instrumentation. In certain cases, it is impossible or impractical to have annual service contracts on older instruments. In these uses, service calls are made on an as-needed basis.

3.7 COMPUTER SYSTEMS

Laboratory Information Management System (LIMS): EA Laboratories' LIMS is a Perkin-Elmer LIMS 2000 running on a Concurrent Computer Corporation Model 3230 minicomputer platform and driven by Concurrent's proprietary Data Management System/32 data base engine. It was installed in August 1985 and since that time several additional in-house programs have been developed to

better tailor the original system to the needs of the laboratory. Each module is designed to handle a particular aspect of laboratory data management. Sample Management performs sample log-in and test assignment. Analytical data may be entered either by individual test or by sample using one of several Result Entry modules. Canned and ad hoc reports are produced via a handful of supplied or customized reporting utilities.

Local Area Network (LAN): EA Laboratories has installed a Novell NetWare 386 LAN in its newly renovated facility at 19 Loveton Circle, Sparks, MD. The network supports primarily administrative functions including word processing, spreadsheets, and E-mail and services over 60 workstations. The system has the potential for interfacing with the wide assortment of hardware (minicomputers, PCS, and instrumentation) and software (operating systems and technical applications) used in laboratories. As such, it plays a critical role in EA's on-going effort to improve product quality by allowing data processing, report production, and software development to become more centralized and efficient through the sharing of common data bases throughout the laboratory.

3.7.1. Description of Computer Security System.

Laboratory Information Management System (LIMS): The LIMS has several layers of security mechanisms built into it. The operating system, OS/32, requires that the system administrator assign a unique account number, password, and privilege mask to each user. During sign-on the system prompts for a username, a valid account number, and a valid password. The user can (and should) change their password later to one of his/her own choosing. The account number can only be changed by a privileged user, this privilege being controlled by the assigned privilege mask. System and application programs are protected from tampering by segregating them on accounts, or disk volumes, or both separate from user accounts. The LIMS software features additional security mechanisms. Prior to allowing access to a program, it checks a user's username against a data base maintained by the system administrator to verify that a user is on the correct account. If not, access is denied; otherwise, access is allowed and the username is recorded in all audit trail transactions. Finally, the system console is protected against tampering by using a Virtual Console Facility (VCF), generally negating the need for a physical console device. The privilege mask assigned to all users controls access to the VCF.

Local Area Network: Novell has build extensive security features into its Netware 386 operating system. These features comprise log-in, rights, attribute, and file server security. The system requires a valid username and password. The username and password required during log-in controls access to the network. Trustee assignments (made by the system administrator) establish rights controlling which users have access to which directories and files and they are allowed to do with them. Attributes (also made by the system administrator) further control available access to files and directories. The file server and console is physically secured in a computer closet.

3.7.2. Data Backup Methods, Frequency, and Storage

Laboratory Information Management System (LIMS): EA Laboratories maintains a stringent magnetic tape backup policy. The system administrator backs up all new or modified files on a daily and weekly basis, except for the LIMS data base files which are backed up only once a week. The LIMS has a transaction log file that is backed up with each daily and this transaction log allows the data base to be reconstructed (rolled forward) from a weekly security copy. In addition, the system administrator performs monthly backups of the entire system (minus the LIMS data base). All tapes are stored in a secure tape cabinet on the premises.

Local Area Network: The network is backed up similarly to the LIMS in that the system administrator regularly performs daily, weekly, and monthly backups. Unlike the LIMS the entire system is copied for each type of backup. All tapes are kept in a secured computer closet.

3.7.3. Arrangements for Emergency Backup Computer Equipment and/or Services.

Laboratory Information Management System (LIMS): EA Laboratories outsources to an independent contractor its preventative maintenance and emergency hardware repairs. Parts and repairs are generally available with 48 hours of an event.

Local Area Network: Because of the wide availability of PC and network parts and services EA Laboratories maintains it LAN internally.

3.8 FACILITY SECURITY

The EA Laboratories building has two separate security systems: an exterior penetration system with motion detectors that is tied into a central station; and a computerized card access system that controls access to critical areas inside and outside the building. The exterior system controls all exterior doors and windows with motion detectors and central alarm. All interior laboratory areas where samples are maintained, samples are analyzed, or where raw data is accessible are controlled by a card reader access system. This system records all attempts to access any door whether or not the card used has proper authorization or not. Information is automatically recorded in a centralized data base, from which appropriate reports are obtained. Each employee is issued unique computer card numbers, which can be electronically blocked out of any area instantly. All samples are maintained in refrigerated (4 ± 2 °C 2 - 4.5°C for North Carolina samples) storage units, which are in an exterior locked fenced area adjacent to the main building, accessible via card access; each storage unit has its own unique lock on the door to the unit.

3.9 PERMANENT CLOSE OF LABORATORY

In the event, for any reason, it becomes necessary to permanently close this facility, it is EA Laboratories policy that clients will be notified of the date on which no further work will be accepted. All work underway will be completed to the client's specifications.

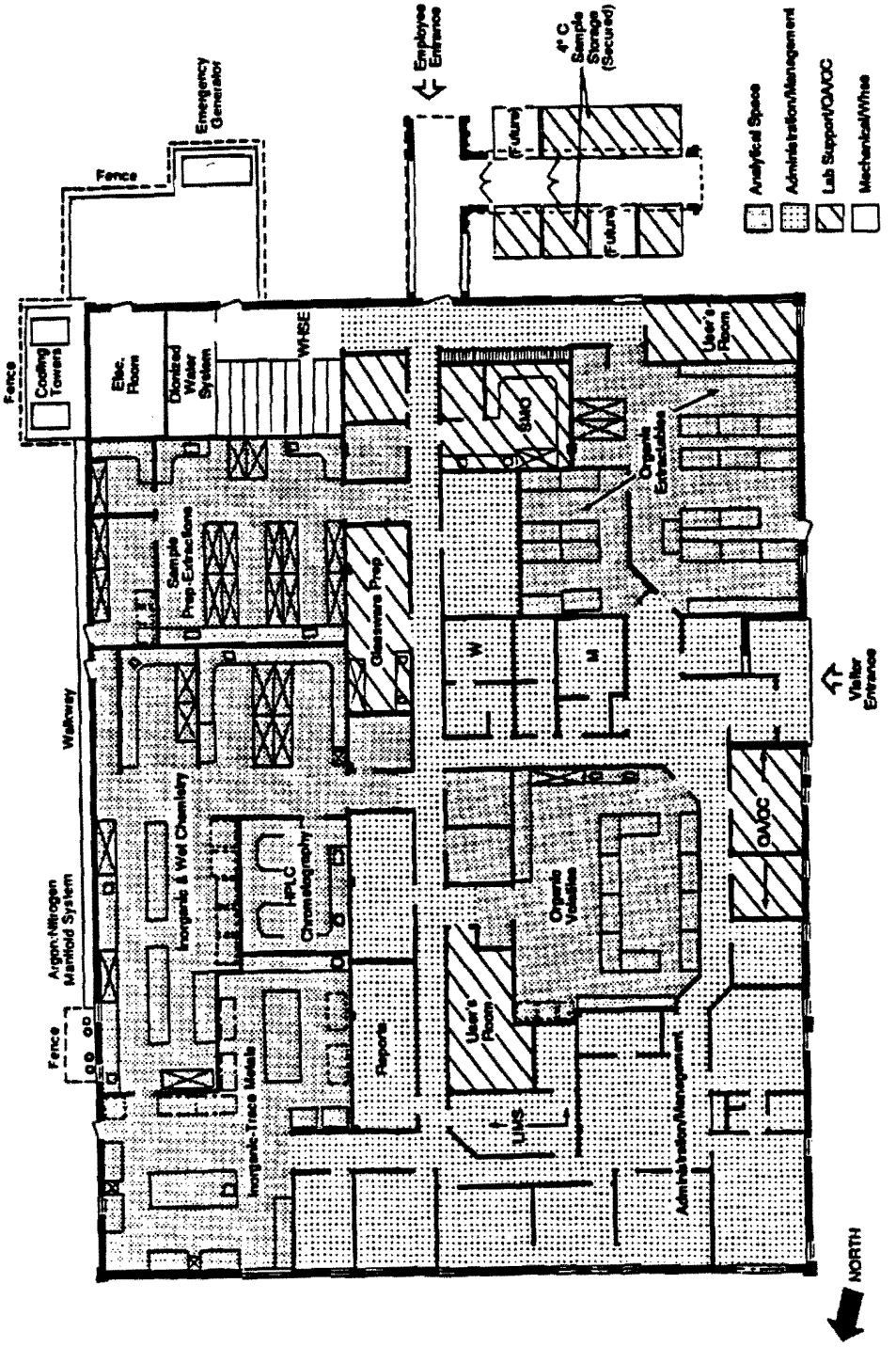

Figure 3-1. EA Laboratories Floor Plan.

4. SAMPLE COLLECTION AND HANDLING

4.1 SAMPLE BOTTLE PREPARATION

The chain-of-custody procedure begins with the preparation of the sample containers and preservatives to be used in sample collection. Unless superseded by specific project requirements, EA Laboratories purchases and distributes pre-cleaned sample containers. Vendors are required to provide documentation of analysis for each lot of containers, and the documentation is kept on file in the Sample Management Office. Contaminant levels in each lot are also evaluated by the laboratory through analysis of randomly selected containers in each vendor lot.

In the event that precleaned containers are not available, sample containers are cleaned in the laboratory according to the procedures given in Table 4-1, which are specific for the parameters to be determined. These procedures are documented in laboratory standard operating procedures (EAL-SOP-033 and EAL-SOP-062).

Tables 4-2 and 4-3 define the type of container required for specific analyses and matrix, preservation techniques and holding times for aqueous and solid samples. Preservatives are supplied with the sample containers to be added in the field.

Sample kits, which are coolers containing chain-of-custody forms, custody seals, sample containers, preservatives, ice and packing material, are prepared by the Sample Management Office in response to receipt of the Analytical Task Order (ATO) described in EAL-SOP-208 (Figure 4-1).

4.2 SAMPLING

Samples are not usually collected by EA Laboratories staff; although trained, experienced and certified laboratory personnel are available to provide this service. Samplers are alerted to any special considerations necessary to ensure collection of representative samples. After the samples are collected, they are split as necessary among containers and preservatives appropriate to the parameters to be determined. Each container is provided with a sample label that is filled out at the time of collection. At this time, a chain-of-custody form (Figure 4-2) is initiated. The collected samples are cooled, if necessary, and returned to the laboratory by the most expedient means to ensure that holding times will be met. The chain-of-custody form is signed and dated as necessary as the samples pass from the collectors to those persons responsible for their transportation.

4.3 SAMPLE LABELING

The importance of sample labeling cannot be overstated. Improperly or inadequately labeled samples are of little value in any analytical procedure. Improperly labeled samples lead to

questions with regard to location, project, sampling station, date sampled, and sampler. All of this information is essential for proper sample handling.

The following information, at a minimum, is required on each sample label:

Client	Date collected
Project number	Time collected
Location	Collected by
Station	Analysis/Preservative(s)

After the label has been completed in the field and has been affixed to the sample container, the label is covered with clear tape. Pre-printed pressure-sensitive labels are available from the laboratory.

Failure to provide the requested information may result in wasted time and resources if it is necessary to discard samples because of inadequate information.

TABLE 4-1 CLEANING PROCEDURES FOR SAMPLE CONTAINERS

Parameter Group	Material	Cleaning
Unpreserved inorganics	Plastic	Detergent & hot water wash Deionized water rinse
Nutrients	Plastic	Non-phosphate detergent & hot water wash HCl soak Deionized water rinse
Pesticides	Glass	Detergent & hot water wash Acetone and deionized water rinse Dry at 400 C
Metals	Plastic	Detergent & hot water wash HNO_3 soak Deionized water rinse
Cyanide	Plastic	Detergent & hot water wash HCl soak Deionized water rinse
Sulfide	Plastic	Detergent & hot water wash Deionized water rinse
Volatile organics	40-mL glass	Detergent & hot water wash Deionized water rinse Methanol rinse Bake at 400 C
Semivolatile organics	Amber glass	Detergent & hot water wash Acetone and deionized water rinse Dry at 400 C Methanol rinse

TABLE 4-2 REQUIRED CONTAINERS, PRESERVATION TECHNIQUE, AND HOLDING TIMES FOR AQUEOUS SAMPLES[a]

Parameter	Volume Required (mL)	Container[b]	Preservative	Recommended Holding Time[c]
Physical Properties				
Color	50	P, G	Cool, 4 C	48 hours
Specific conductance	100	P, G	Cool, 4 C	28 days[d]
Hardness	100	P, G	Cool, 4 C HNO_3 to pH <2	6 months [e]
Odor	1000	G only	Cool, 4 C	24 hours
pH	25	P, G	None required	Analyze immediately
Residue				
Filterable	100	P, G	Cool, 4 C	7 days
Nonfilterable	100	P, G	Cool, 4 C	7 days
Settleable	1,000	P, G	Cool, 4 C	48 hours
Total	100	P, G	Cool, 4 C	7 days
Volatile	100	P, G	Cool, 4 C	7 days
Temperature	1,000	P, G	None required	Analyze immediately
Turbidity	100	P, G	Cool, 4 C	48 hours
Metals				
Chromium (VI)	200	P, G	Cool, 4 C	24 hours
Mercury	250	P, G	HNO_3 to pH <2	28 days
Other Metals				
Dissolved	500	P, G	Filter onsite HNO_3 to pH <2	6 months[e]
Suspended	500		Filter onsite	6 months
Total	500	P, G	HNO_3 to pH <2	6 months[e]
Inorganics, Nonmetallic				
Acidity	100	P, G	Cool, 4 C	14 days
Alkalinity	100	P, G	Cool, 4 C	14 days
Bromide	100	P, G	None required	28 days
Chloride	50	P, G	None required	28 days
Chlorine, total residual	200	P, G	None required	Analyze immediately
Cyanides	500	P, G	Cool, 4 C NaOH to pH >12 Ascorbic acid	14 days 24 hours in presence of S^{2-}
Fluoride	300	P	None required	28 days

TABLE 4-2 REQUIRED CONTAINERS, PRESERVATION TECHNIQUE, AND HOLDING TIMES FOR AQUEOUS SAMPLES[a]

Parameter	Volume Required (mL)	Container[b]	Preservative	Recommended Holding Time[c]
Iodide	100	P, G	Cool, 4 C	24 hours
Nitrogen				
Ammonia	400	P, G	Cool, 4 C H_2SO_4 to pH <2	28 days
Kjeldahl, total	500	P, G	Cool, 4 C H_2SO_4 to pH <2	28 days
Nitrate plus nitrite	100	P, G	Cool, 4 C H_2SO_4 to pH <2	28 days
Nitrate	100	P, G	Cool, 4 C	48 hours
Nitrite	50	P, G	Cool, 4 C	48 hours
Dissolved oxygen				
Probe	300	G only	None required	Analyze immediately
Winkler	300	G only	Fix onsite and store in dark	8 hours
Phosphorus				
Orthophosphate, dissolved	50	P, G	Filter onsite Cool, 4 C	48 hours
Hydrolyzable	50	P, G	Cool, 4 C H_2SO_4 to pH <2	24 hours
Total	50	P, G	Cool, 4 C H_2SO_4 to pH <2	28 days
Total, dissolved	50	P, G	Filter onsite Cool, 4 C H_2SO_4 to pH <2	28 days
Silica	50	P only	Cool, 4 C	28 days
Sulfate	50	P, G	Cool, 4 C	28 days
Sulfide	500	P, G	Cool, 4 C Zinc acetate NaOH to pH >9	7 days
Organics				
Acrolein and acrylonitrile	80	G, teflon-lined septum	Cool, 4 C[f] pH 4 - 5	14 days
Biochemical oxygen demand	1,000	P, G	Cool, 4 C	48 hours
Chemical oxygen demand	50	P, G	H_2SO_4 to pH <2	28 days
Oil and grease	1,000	G only	Cool, 4 C H_2SO_4 to pH <2	28 days

TABLE 4-2 REQUIRED CONTAINERS, PRESERVATION TECHNIQUE, AND HOLDING TIMES FOR AQUEOUS SAMPLES[a]

Parameter	Volume Required (mL)	Container[b]	Preservative	Recommended Holding Time[c]
Organic carbon	50	P, G	Cool, 4 C H_2SO_4 or HCl to pH <2	28 days
Pesticides/PCBs	2,000	G, teflon-lined cap	Cool, 4 C pH 5-9	7 days until extraction 40 days after extraction
Phenolics (colorimetric)	500	G only	Cool, 4 C H_2SO_4 to pH <2	28 days
Purgeable aromatics	80	G, teflon-lined septum	Cool, 4 C[d] HCl to pH <2[e]	14 days
Purgeable halocarbons	80	G, teflon-lined septum	Cool, 4 C[d]	14 days
Semivolatile extractables[h]	4,000	G, teflon-lined cap	Cool, 4 C[d]	7 days until extraction 40 days after extraction
Surfactants	250	P, G	Cool, 4 C	48 hours
Total petroleum hydrocarbons (IR Spectrometric)	1,000	G only	Cool, 4 C H_2SO_4 to pH <2	28 days
Bacteria				
Coliform (fecal and total)	50	P, G	Cool, 4 C[d]	6 hours
Fecal streptococci	50	P, G	Cool, 4 C[d]	6 hours
Radiological				
Alpha, beta, and radium	1,000	P, G	HNO_3 to pH <2	6 months

(a) From time of sample collection (40 CFR Part 136.3).
(b) Polyethylene (P) or glass (G). For metals, polyethylene with a polypropylene cap (no liner) is preferred.
(c) Where shipping regulations prevent the use of the proper preservation technique or the holding time is exceeded, such as the case of a 24-hour composite, the final reported data for these samples should indicate the specific variance.
(d) If the sample is stabilized by cooling, it should be warmed to 25 C for reading, or temperature correction made and results reported at 25 C.
(e) Where HNO_3 cannot be used because of shipping restrictions, the sample may be initially preserved by icing and immediately shipped to the laboratory. Upon receipt in the laboratory, the sample must be acidified to a pH <2 with HNO_3 (normally 3 mL 1:1 HNO_3/L is sufficient).
(f) Use 0.008% $Na_2S_2O_3$ in the presence of residual chlorine.
(g) Samples receiving no pH adjustment must be analyzed within seven days of sampling.
(h) Includes phthalates, nitrosamines, organochlorine pesticides, PCBs, nitroaromatics, isophorone, polynuclear aromatic hydrocarbons, haloethers, chlorinated hydrocarbons, phenols, and TCDD.

TABLE 4-3 REQUIRED CONTAINERS, PRESERVATION TECHNIQUE, AND HOLDING TIMES FOR SOLID SAMPLES[a]

Parameter	Weight (g)	Container[b]	Preservative	Recommended Holding Time[c]
Physical Properties				
pH	25	P, G	None required	Analyze immediately
Solids				
Total	100	P, G	Cool, 4 C	7 days
Volatile	100	P, G	Cool, 4 C	7 days
Temperature	100	P, G	None required	Analyze immediately
Chromium (VI)	25	P, G	Cool, 4 C	24 hours
Mercury	5	P, G	Cool, 4 C	28 days
Other Metals	5	P, G	Cool, 4 C	6 months
Inorganics, Nonmetallic				
Bromide	10	P, G	None required	28 days
Chloride	10	P, G	None required	28 days
Cyanides	50	P, G	Cool, 4 C	14 days
Fluoride	10	P	None required	28 days
Iodide	10	P, G	Cool, 4 C	24 hours
Nitrogen				
Ammonia	10	P, G	Cool, 4 C	28 days
Kjeldahl, total	50	P, G	Cool, 4 C	28 days
Nitrate plus nitrite	10	P, G	Cool, 4 C	28 days
Nitrate	10	P, G	Cool, 4 C	48 hours
Nitrite	10	P, G	Cool, 4 C	48 hours
Phosphorus				
Orthophosphate	10	P, G	Cool, 4 C	48 hours
Hydrolyzable	10	P, G	Cool, 4 C	24 hours
Total	50	P, G	Cool, 4 C	28 days
Silica	50	P only	Cool, 4 C	28 days
Sulfate	10	P, G	Cool, 4 C	28 days

TABLE 4-3 REQUIRED CONTAINERS, PRESERVATION TECHNIQUE, AND HOLDING TIMES FOR SOLID SAMPLES[a]

Parameter	Weight (g)	Container[b]	Preservative	Recommended Holding Time[c]
Sulfide	10	P, G	Cool, 4 C	7 days
Organics				
Acrolein and acrylonitrile	80	G, teflon-lined septum	Cool, 4 C	14 days
Chemical oxygen demand	50	P, G	None Required	28 days
Oil and grease	50	G only	Cool, 4 C	28 days
Organic carbon	5	P, G	Cool, 4 C	28 days
Pesticides/PCBs	200	G, teflon-lined cap	Cool, 4 C	14 days until extraction 40 days after extraction
Phenolics (colorimetric)	50	G only	Cool, 4 C	28 days
Purgeable aromatics	200	G, teflon-lined septum	Cool, 4 C	14 days
Purgeable halocarbons	200	G, teflon-lined septum	Cool, 4 C	14 days
Semivolatile extractables[e]	200	G, teflon-lined cap	Cool, 4 C	14 days until extraction 40 days after extraction
Total petroleum hydrocarbons (IR/spectrometric)	50	G only	Cool, 4 C	28 days
Total petroleum hydrocarbons (as gasoline)	100	G only	Cool, 4 C	14 days
Total petroleum hydrocarbons (as diesel)	200	G only	Cool, 4 C	14 days until extraction 40 days after extraction

(a) From time of sample collection (40 CFR Part 136.3, 40 CFR Part 261).
(b) Polyethylene (P) or glass (G). For metals, polyethylene with a polypropylene cap (no liner) is preferred.
(c) Holding times are based on the date of sample collection. Where shipping regulations prevent the use of the proper preservation technique or the holding time is exceeded, such as the case of a 24-hour composite, the final reported data for these samples should indicate the specific variance.
(d) If the sample is stabilized by cooling, it should be warmed to 25 C for reading, or temperature correction made and results reported at 25 C.
(e) Includes phthalates, nitrosamines, organochlorine pesticides, PCBs, nitroaromatics, isophorone, polynuclear aromatic hydrocarbons, haloethers, chlorinated hydrocarbons, phenols, and TCDD.

EA®

ANALYTICAL TASK ORDER

No:

EA LABORATORIES
19 Loveton Circle
Sparks, MD 21152
Phone: (410) 771-4920
FAX (410) 771-4407

1. Turnaround requirement:(See Reverse Side)
   - ☐ Regular Status
   - ☐ Accelerated Status (additional charge)*
   - ☐ Rush Status (additional charge)*

* RESULTS REQUIRED BY: _____ (Date)

CONTACT EA LABS PRIOR TO SENDING SAMPLES

2. ☐ Original Order
   ☐ Amendment Order
   (Original ATO Document No _____)

3. QUOTATION NUMBER :_____

4. PROJECT NUMBER: _____
   Dept. No.: _____ Task No.: _____
   Project Name: _____
   Project Contact: _____

5. SHIPPING ADDRESS:
   Company Name: _____
   Address: _____
   _____
   Contact: _____
   Telephone: ( ) _____
   fax tELEPHONE: ( ) _____

6. Sample collection:
   Date for Bottles Due to Site: : _____
   Delivery Date Due to Lab: _____

7. REPORTING REQUIREMENTS:
   ☐ Report: ☐ EA Standard
   ☐ Other (specify) _____

   ☐ Electronic: Specify Format _____
   (Additional charge)

8. QC REQUIREMENTS MUST BE COMPLETED: See Terms and Conditions: QC samples billed at regular sample rate.
   - ☐ MATRIX DUPLICATE No. _____
   - ☐ MATRIX SPIKE No. _____
   - ☐ MATRIX SPIKE DUPLICATE No. _____
   - ☐ FIELD BLANKS No. _____
   - ☐ TRIP BLANKS No. _____
   - ☐ OTHER (as speciofied below)

   _____

9. PROGRAM REQUIREMENTS:
   - ☐ NPDES ☐ RCRA
   - ☐ SWDA ☐ Other: _____

10. PARAMETERS FOR ANALYSIS:

CATALOG NUMBER	ANALYSIS	NUMBER of SAMPLES	MATRIX**

** Specify: Air, Tissue, Sludge, Soil, Water, etc.

11. SUPPLIES:
   ☐ Deionized Water ☐ Chain-of-Custody Formsd

12. COMMENTS/SPECIAL INSTRUCTIONS: _____

_____

_____

13. REQUESTED BY: _____ Date: ___/___/___
Task Order must be completed, signed and dated prior to start of work.

14. ACCEPTED BY: _____ Date: ___/___/___
EA Laboratories

Figure 4-1 EA Laboratories Analytical Task Order Form

Company Name:	Project Manager or Contact: / Phone:	Parameters/Method Numbers for Analysis	Chain of Custody Record
Project No. / Dept.: Task:	Project Name:		EA Laboratories, 19 Loveton Circle, Sparks, MD 21152, Telephone: (410) 771-4920, Fax: (410) 771-4407
Sample Storage Location:	ATO Number:		Report Deliverables: 1 2 3 4 D E
Page of	Report #:		EDD: Yes/No
			DUE TO CLIENT: _____

Date	Time	Water	Soil	Sample Identification 19 Characters	No. of Containers	EA Labs Accession Number	Remarks
							LPM:
							Project Summary No.:

Sampled 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.

WHITE—EA Laboratories YELLOW—EA Laboratories PINK—Project Manager *Shaded Areas for Lab Use Only*

Figure 4-2 EA Laboratories Chain of Custody Form

5. SAMPLE CUSTODY

5.1 CHAIN-OF-CUSTODY PROCEDURES

Samples are physical evidence and are handled according to certain procedural safeguards. For the purposes of some types of legal proceedings, a showing to the court that the laboratory is a secure area may be all that is required for the analyzed evidence to be admitted. However, it is anticipated that in some cases, the court may require a showing of the hand-to-hand custody of the samples while they were at the laboratory. In the event that the court requires such a comprehensive chain-of-custody demonstration, the laboratory must be prepared to produce documentation that traces the in-house custody of the samples from the time of receipt to the completion of the analysis.

The National Enforcement Investigations Center (NEIC) of U.S. EPA defines custody of evidence in the following ways:

- it is in your actual possession; or
- it is in your view, after being in your physical possession; or
- it was in your possession and then you locked or sealed it up to prevent tampering; or
- it is in a secure area.

5.2 SAMPLE MANAGEMENT

EA Laboratories has a designated Sample Management Officer whose duties and responsibilities are described in Section 2.2.7.

5.3 SAMPLE RECEIPT AND LOGGING

After samples have been collected and labeled and the chain-of-custody forms initiated, the project manager completes the right side of the chain-of-custody form. This form provides sample-specific information and a listing of the parameters required on each sample, along with the required analytical sensitivity. The chain-of-custody and appropriate field data sheets (if required by client) are sealed in a water-tight plastic envelope and shipped with the samples to the laboratory.

Upon receipt at the laboratory, the Sample Management Officer or designee inspects the samples for integrity and checks the shipment against the chain-of-custody/analytical task order form. Cooler temperatures are checked and documented on the chain-of-custody. The pH of preserved

samples (except volatile organics) is measured and documented on the internal chain of custody which is maintained in the Sample Management Office (EAL-SOP-039). The pH of sample vials submitted for volatile organics determinations are checked by the analyst during analysis, and the pH is recorded in the instrument run logbook.

Discrepancies are addressed at this point and documented on the chain-of-custody form and must be resolved before samples are released to the laboratory for analysis. When the shipment and the chain-of-custody are in agreement, the custodian enters the samples into the Analytical Custody and Preservation Log (Figure 5-1) and assigns each sample a unique laboratory number. This number is affixed to each sample bottle. The custodian then enters the sample and analysis information into the laboratory computer system. The original of the chain-of-custody form is given to the data management group, with copies to the laboratory operations manager, each section chief, and the LPM. These log-in procedures are documented in EAL-SOP-035 and EAL-SOP-036.

5.4 SAMPLE STORAGE AND SECURITY

While in the laboratory, the samples and aliquots that require storage at approximately 4 °C are maintained in a locked refrigerator unless they are being used for analysis. Samples for purgeable organics determinations are stored in a separate locked refrigerator from other samples, sample extracts, and standards.

Similarly, there are refrigerators designated for extracts and standards. Samples that are required to be frozen are stored in a freezer. The sample storage areas are within the laboratory to which access is limited to laboratory chemists and controlled by doors with controlled access locks as described in Section 3.8.

So that the laboratory may satisfy sample chain-of-custody requirements, the following standard operating procedures for laboratory/sample security are implemented:

- Samples are stored in a secure area.
- Access to the laboratory is through a monitored area. Other outside-access doors to the laboratory are kept locked.
- Visitors sign a visitor's log and are escorted while in the laboratory.
- Refrigerators, freezers, and other sample storage areas are securely maintained or locked.
- Only the designated sample custodian and supervisory personnel have keys to locked sample storage area(s).
- Samples remain in secure sample storage until removed for sample preparation or analysis.
- All transfers of samples into and out of storage are documented on an internal chain-of-custody record(EAL-SOP-039).
- Internal custody records are maintained in the project files.

- After a sample has been removed from storage by the analyst, the analyst is responsible for the custody of the sample.

5.5 SAMPLE DISPOSAL

Unless otherwise specified by the client, samples are routinely retained at the laboratory for 60 days after the final analytical data report has been forwarded to the client so that any analytical problems can be addressed. Longer storage is arranged through the Sample Management Office. The samples are then discarded in accordance with guidance specified in EA Laboratories Laboratory Safety Plan and EAL-SOP-018. The laboratory reserves the right to return potential hazardous samples to the client for disposal.

EA Labs
19 Loveton Circle
Sparks, Maryland 21152
Note: Do not obliterate errors
Cross out with a single line only.

1997 Analytical Custody and Preservation

Page No. _____

Job No.	Client	Project Code	Matrix	Received		EA Sample No.		Locator Code	Aliquot Preservation											Custody Sheet		Loggers Initials	ATO No.
				Date	Time	Start	End		A	B	C	F	G	H	I	J	Pest	Soil	VOA	Other	Yes	No		

Effective Date 01/01/97

Figure 5-1. Analytical custody and preservation log.

6. CALIBRATION PROCEDURES

Instruments and equipment used in EA Laboratories are controlled by a formal calibration program. The program verifies that equipment is of the proper type, range, accuracy, and precision to provide data compatible with specified requirements. All instruments and equipment that measure a quantity, or whose performance is expected at a stated level, are subject to calibration. Calibration is performed by EA Laboratories personnel using reference standards or externally by calibration agencies or equipment manufacturers.

This section prescribes the practices used by EA Laboratories to implement a calibration program. Development and documentation of the laboratory calibration program is the responsibility of the laboratory managers. Implementation is the responsibility of the section chiefs and chemists. The Quality Services Manager (QSM) monitors the procedures. Specifics are not provided because the requirements for the calibration of instruments and equipment are dependent upon the type and expected performance of individual instruments and equipment. Therefore, EA Laboratories uses the guidelines provided herein to develop a calibration program.

Two types of calibration are discussed in this section:

- *operational calibration*, which is routinely performed as part of an analytical procedure or test method, such as the development of a standard curve for use with an atomic absorption spectrophotometer. Operation calibration is generally performed for instrument systems.
- *periodic calibration*, which is performed at prescribed intervals for equipment, such as balances and thermometers. In general, equipment which can be calibrated periodically is a distinct, singular purpose unit and is relatively stable in performance.

6.1 CALIBRATION SYSTEM

The following sections contain a discussion of the elements comprising the calibration system.

6.1.1 Calibration Procedures

Written procedures are used by EA Laboratories for all instruments and equipment subject to calibration. Whenever possible, recognized procedures, such as those published by ASTM or the U.S. EPA or procedures provided by manufacturers, are adopted. If established procedures are not available, a procedure is developed considering the type of equipment, stability characteristics of the equipment, required accuracy, and the effect of operational error on the quantities measured. As a minimum, the procedures include:

- Equipment to be calibrated
- Reference standards used for calibration
- Calibration technique and sequential actions
- Acceptable performance tolerances
- Frequency of calibration
- Calibration documentation format

6.1.2 Equipment Identification

Equipment that is subject to calibration are uniquely identified by a unique number assigned by EA Laboratories, and calibration records reference the specific instrument identification.

6.1.3 Calibration Frequency

Instruments and equipment are calibrated at prescribed intervals and/or as part of the operational use of the equipment. Frequency are based on the type of equipment, inherent stability, manufacturer's recommendations, values provided in recognized standards, intended data use, specified analytical methods, effect of error upon the measurement process, and prior experience.

6.1.4 Calibration Reference Standards

Two types of reference standards are used within EA Laboratories for calibration:

- Physical standards, such as weights for calibrating balances and certified thermometers for calibrating working thermometers, refrigerators and ovens, are generally used for periodic calibration.
- Chemical standards, such as Standard Reference Materials (SRMs) provided by the National Institute of Standards and Technology (NIST). These may include vendor-certified materials traceable to NIST SRMs. These are primarily used for operational calibration.

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

Physical reference standards are used only for calibration and are stored separately from equipment used in analyses. In general, physical reference standards are at least four to ten times as accurate as the requirements for the equipment which they are used to calibrate. In general, physical standards are recalibrated annually by a certified external agency, and documentation is maintained by the QSM.

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

6.1.5 Calibration Failure

Equipment that cannot be calibrated or becomes inoperable is labeled as such and removed from service. Such equipment must be repaired and satisfactorily recalibrated before reuse. For equipment that fails calibration, a nonconformance record (NCR) (Figure 11-1) is initiated to record the corrective action and to demonstrate satisfactory calibration.

Scheduled calibration of equipment does not relieve the laboratory staff of the responsibility for using properly functioning equipment. If an equipment malfunction is suspected, the equipment is tagged and removed from service and recalibrated. If it fails recalibration, the above process shall apply. The laboratory managers are responsible for the development and implementation of a contingency plan for major equipment failure. The plan includes guidelines on waiting for repairs, use of other instrumentation, subcontracting analyses, and evaluating scheduled priorities.

6.1.6 Calibration Records

Records are prepared and maintained for each piece of equipment subject to calibration. Records demonstrating accuracy of preparation, stability, and proof of continuity of reference standards is also maintained. Records for periodically calibrated equipment shall include, as appropriate:

- unique identification number of equipment and type of equipment
- calibration frequency and acceptable tolerances
- identification of calibration procedure used
- date calibration was performed
- identity of EA Laboratories personnel and/or external agencies performing calibration
- reference standards used for calibration
- calibration data
- certificates or statements of calibration provided by manufacturers and external agencies and traceability to national standards
- information regarding calibration acceptance or failure and any repair of failed equipment

Records for periodically calibrated equipment are maintained in the instrument log books, or in an equipment file maintained by the QSM, and physical reference standards are kept in a separate folder.

For instruments and equipment that are calibrated on an operational basis, calibration generally consists of determining instrumental response against compounds of known composition and

concentration or the preparation of a standard response curve of the same compound at different concentrations. Records of these calibrations are maintained in the instrument logbook, which provides an ongoing record of the calibration undertaken for a specific instrument. The logbook should be indexed in the laboratory operations records but should be maintained at the instrument by the chemist. All entries should be signed and dated by the chemist, and reviewed by the QC Chemist during technical review of the data package.

In addition to the instrument logbook, copies of the raw calibration data are kept with the analytical sample data. In this way results can be readily processed and verified because the raw data package is complete as a unit. If samples from several projects are processed together, the calibration data is copied and included with each group of data.

6.2 OPERATIONAL CALIBRATION

Operational calibration is generally performed as part of the analytical procedure and refers to those operations in which instrument response (in its broadest interpretation) is related to analyte concentration. Included is the preparation of a standard response (calibration) curve and often the analysis of blanks.

6.2.1 Preparation of Calibration Curve

Preparation of a standard calibration curve is accomplished by using calibration standards. The process is summarized as:

- Preparation of a standard calibration curve is accomplished by the analysis of calibration standards that are prepared by adding the analyte(s) of interest to the solvent that is introduced into the instrument.
- The concentrations of the calibration standards are chosen to cover the working range of the instrument or method.
- All sample measurements are made within this working range.
- The calibration curve is prepared by plotting or regressing the instrument responses versus the analyte concentrations.
- The concentrations of the analyzed samples are back-calculated from the calibration curve.

6.2.2 Instrument Calibration Procedures

Unless superseded by specific method or project requirements, operational calibration requirements are specified in the determinative Method SOP.

6.3 PERIODIC CALIBRATION

Periodic calibrations are performed for equipment, such as balances and thermometers, that are required in analytical methods but that are not routinely calibrated as part of the analytical procedure. Table 6-1 lists the periodic calibration requirements used by EA Laboratories.

TABLE 6-1 SUMMARY OF PERIODIC CALIBRATION REQUIREMENTS

Instrument	Calibration Frequency		Acceptance Limits	Corrective Actions
Analytical Balances	Daily:	Sensitivity (Class P weight)	0.001 g	Adjust sensitivity
	Monthly:	Checked with Class S weights	Std. dev. less than 0.1 mg	Service balance
	Annually:	Calibrated by outside vendor against certified Class S weights	Class S tolerances	Service balance
Thermometers	Annually:	Calibrated against certified NIST thermometers by outside vendor	±0.5 C	Tag and remove from service
Automatic Pipettors	Monthly:	Gravimetric check	High volume (>100 mL): ≤1.0% relative error as RSD Low volume (<100 mL): ≤2.0% relative error as RSD	Service or replacement

7. LABORATORY PROCEDURES

7.1 ANALYTICAL METHODS

7.1.1 Standard Methods

Unless superseded by client or project requirements, analysis of samples is performed using EPA or EPA-approved methods, where such methods exist. For those analyses that do not have EPA methods, the analytical methods used are taken from standard sources. The analytical methods performed by EA Laboratories or its approved subcontractors is maintained by the QS Manager.

7.1.2 Initial Demonstration of Method Performance

All analytical methods used by EA Laboratories, including EPA or an EPA-approved method, without published performance criteria. Verification studies for nonstandard analytical methods are performed and evaluated using current applicable EPA program guidelines. Documentation is maintained by the QSM, and includes the following:

- Method SOP in EA Laboratories format (EAL-SOP-302)
- Calibration procedure and method range
- Initial method precision and accuracy from the analysis of Laboratory Control Samples
- Method Detection Limit (MDL) study

7.1.3 Method References

American Public Health Association, American Water Works Association, Water Pollution Control Federation. 1992. Standard Methods for the Examination of Water and Wastewater, 18th edition. APHA, Washington, D.C.

United States Environmental Protection Agency. 1983. Methods for Chemical Analysis of Water and Wastes. EPA-600/4-79-020. U.S. EPA, Cincinnati, Ohio.

United States Environmental Protection Agency. 1995. Test Methods for Evaluating Solid Waste. Physical/Chemical Methods. EPA SW-846, 3rd edition, including Update IIB. U.S. EPA, Washington, D.C.

United States Environmental Protection Agency. 1988. Methods for the Determination of Organic Compounds in Drinking Water. EPA-600/4-88/039. U.S. EPA, Cincinnati, Ohio.

United States Environmental Protection Agency. March 1990. U.S. EPA Contract Laboratory Program. Statement of Work for Inorganics Analysis. ILMO3.0. U.S. EPA, Washington, D.C.

United States Environmental Protection Agency. 1993 U.S. EPA Contract Laboratory Program. Statement of Work for Organics Analysis. OLMO3.1. U.S. EPA, Washington, D.C.

United States Environmental Protection Agency. 1988. Methods for Organic Chemical Analysis of Municipal and Industrial Wastes. 40 CFR Part 136, Appendix A.

United States Environmental Protection Agency. 1993. Mtehods for the Determination of Inorganic Substances in Environmental Samples. EPA/600/R-93/100. U.S. EPA, Washington, D.C.

7.2 STANDARD OPERATING PROCEDURES

A standard operating procedure (SOP) is a written step-by-step description of laboratory operating procedures. EA Laboratories documents all procedures in formal, approved SOPs, which are issued in a document-controlled manual (EA Manual EAL-002). All SOPs are submitted in draft to the QS Manager who is responsible for initiating the review and approval process and for distributing and controlling the final SOPs (EAL-SOP-088).

The SOPs address the following areas:

- Storage containers and sample preservatives
- Sample receipt and logging
- Sample custody
- Sample handling procedures
- Sample transportation
- Glassware cleaning
- Laboratory security
- Equipment calibration and maintenance
- Documentation
- Safety
- Data handling procedures
- Document control
- Personnel training and documentation
- Sample and extract storage
- Traceability of standards
- Maintaining instrument records and logbooks

- Corrective Action Process
- Records management

The QSM is responsible for maintaining the original copies of all SOPs, as well as a historical file of all versions.

7.3 DETECTION AND QUANTITATION LEVELS

7.3.1 Detection Levels

A detection level, or limit, has been defined by the Committee on Environmental Improvement of the American Chemical Society (ACS) (Anal. Chem. 55:2210-2218 (1983)) as "the lowest concentration that can be determined to be statistically different from a blank." Various methods are available for determining detection limits; most of them are based on the standard deviation of measurements in the region near the blank responses. The following detection limits are determined routinely in the laboratory:

Instrument detection limits (IDL) are determined using the protocols given in the inorganic and organic statements of work for the U.S. EPA Contract Laboratory Program; the procedures and calculations are detailed in EAL-SOP-047 and -048. A standard deviation is calculated from replicate measurements of a low-level standard and multiplied by three to give the IDL. IDLs are used as an index of instrument performance that does not include sample effects and, therefore, represent the lowest detection limit achievable. IDLs can vary between instruments of the same type and can change when redetermined.

Method detection limits (MDL) are determined using the U.S. EPA procedure published in 40 CFR 136 Appendix B (EAL-SOP-049). The 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 procedure requires that *"all sample processing steps of the analytical method be included in the determination of the method detection limit."* MDLs therefore are influenced by the sample matrix and sample preparation process as well as the analytical instrumentation. A minimum of seven replicates spiked at one to five times the expected MDL are analyzed. The MDL is calculated by multiplying the standard deviation of the measurements by the Student t-value for a 99% confidence level. Because of the wide variety of matrix types analyzed by the laboratory, MDLs are routinely determined only in reagent water. These MDLs represent, therefore, the optimum values, and the MDLs for actual sample matrices are likely to be higher. MDLs can be determined for specific matrices when requested by the client.

Unless superseded by other program, project, or client requirements, IDLs and MDLs are determined annually. In addition, IDLs and MDLs are redetermined after an instrument is moved or modified and MDLs after a method has been significantly changed. Where more than one instrument is used in sample analyses by a given technique (e.g., GC/MS, GC, GFAA, or ICP), detection limit studies are performed for each instrument. A standard laboratory reporting limit is established for each analyte based on the highest detection limit determined. Data for all instruments are maintained for use in reporting data when project-specific requirements dictate lower detection limits.

A detection limit measured at any one time is only an estimate of the 'true' detection limit because the measured standard deviation used to calculate the detection limit is subject to random error and is only an estimate of the population standard deviation. The confidence limits on the standard deviation, and hence the detection limit, can be determined using the chi-square distribution (40 CFR 136 Appendix B). The 95% confidence limits for an MDL determined from seven replicates are 0.64 MDL and 2.20 MDL. A redetermination of the detection limit could produce any value between the chi-square limits, even if all the conditions remain the same. Day-to-day changes in instrument performance can further produce changes in the measured detection limit.

When interpreting data and detection limits it is important to remember that, when a measured concentration is greater than the detection limit, the analyte has the specified probability of actually being present (i.e., of having a true concentration greater than zero); however, the detection limit cannot be used to say anything about the presence or absence of an analyte that has a measured concentration less than the detection limit. From the definition of the MDL there is only a 1% chance that a sample with no analyte will produce a concentration greater than or equal to the MDL (false positive). The probability is 50%, however, that a sample with a concentration at the MDL will be measured as less than the MDL (false negative).

It is also important when interpreting low-level data to consider the precision of measurements close to the detection limit. The relative standard deviation (s/x) of a value at the MDL is 32%, and the 3σ limits are the MDL ± MDL. For a sample with an analyte concentration at the MDL, 50% of the time the measured value will be less than the MDL and 50% of the time between the MDL and 3 × MDL.

7.3.2 Quantitation Level

To ensure better precision in low-level data and to reduce the false negative error rate, quantitation limits have been proposed as the minimum concentration at which an analyte can be quantified with an acceptable degree of confidence. The ACS Committee on Environmental Improvement has recommended that quantitation limits be calculated by multiplying 10 times the standard deviation, giving a relative standard deviation of 10%. The Committee further advised that

quantitative interpretation, decision-making, and regulatory actions should be limited to data at or above the limit of quantitation.

EA Laboratories has established *Reporting Limits* (RLs) as laboratory quantitation levels. These are the minimum concentrations to be reported for routine laboratory analyses in a variety of environmental matrices at the stated precision and accuracy of the method. RLs are derived from the MDLs and from consideration of analyte sensitivity for a given analytical technique. RLs are verified for each analytical run through analysis of a low level standard at a concentration near the RL. Unless superseded by client, project or program requirements, RLs are used to report analytical results.

7.3.3 Sample Quantitation Level

Quantitation levels are established in clean standard matrices and are optimum values. The sample quantitation level (SQL) are influenced by the complexity of the sample matrix which may result in SQLs that are higher than the laboratory RLs. due to the necessity to minimize interferences by diluting the sample or extract, by performing additional cleanup steps, or by modifying the method procedures.

7.4 DOCUMENTATION

EA Laboratories maintains extensive records to ensure that all aspects of the analytical process are adequately documented. Because the keeping of laboratory records is a legal requirement, it is important to consider the format and quality of the records. These records should convey:

- What was done.
- When it was done.
- Who did it.
- What was found.

7.4.1 Recordkeeping

The requirements for laboratory recordkeeping are given in EAL-SOP-065. All data entries are made in indelible, water-resistant ink. The date of the entry and the observer is clear on each entry. The observer uses his/her full name or initials. An initial and signature log is maintained so that the recorder of every entry can be identified.

All information is recorded in a notebook or on other records at the time the observations are made. ***Recording information on loose pieces of paper is not allowed.*** If a portion of a page is

intentionally left blank it is crossed with an "X", and initialed and dated by the person making the entry.

When a mistake is made, the wrong entry is crossed out with a single line, initialed and dated by the person making the entry, and the correct information recorded. Obliteration of an incorrect entry or writing over it is not allowed; neither is the use of correction tape or fluid on any laboratory records.

7.4.2 Laboratory Records

The following are some of the records that are used to document activities in the laboratory. These are in addition to those discussed elsewhere in this manual, such as chain-of-custody (COC) forms, log-in sheets, internal COCs, maintenance records (Section 9), and nonconformance forms (Section 11).

Reagent and Titrant Preparation Records: The procedure for each analysis includes the procedures for reagent/ titrant preparation, including concentration, storage, and discard information. After a reagent/titrant is prepared, the following information is entered on a label affixed to the storage bottle: (1) its identity, (2) intended use, (3) titer/concentration, (4) preparation date, (5) storage requirement, (6) discard date, and (7) preparer. For titrimetric analyses, the procedure includes directions for standardizing the titrant, and the laboratory data sheets include space for titrant standardization data.

Standards Preparation Logs: The preparation of stock, intermediate, and working standard solutions is recorded in standards preparation logbooks, which are specific to the requirements of each operational group. Each standard is assigned a number that is used to trace the preparation from stock to working standards and to reference the analysis of the standards. The logbooks are completed by the appropriate analysts as they prepare the standards and are reviewed by the section QC Chemist as part of the data package review process.

Sample Preparation Logs: Sample preparation operations, such as digestions and extractions, are documented in sample preparation logs, which are specific to the operations involved. The information in these logs can include: date, analyst, sample identification, weight or volume of sample used, reagents used, final volume, and volume of spiking, surrogate, or internal standard solution. The logbooks are completed by the Chemists or Technicians as they prepare the standards and are reviewed by the section QC Chemist as part of the data package review process.

Bench Data Sheets: Laboratory bench data sheets are used for those analyses in which instrument responses are manually transcribed from instrument readout or from recorder tracings. The data sheets are preprinted to reflect the requirements of the analysis and are used to ensure that the

information is recorded in a complete and organized manner. The logbooks are completed by the appropriate analysts as they perform the analyses, and are reviewed by the section QC Chemist as part of the data package review process.

Instrument Run Logs: When instrumentation is involved in the data generation, the sequence of the introduction of standards, field samples, and QC samples is recorded in an instrument run log. The following information is recorded when applicable: instrument identification, date, time, analyst, sample identifications, dilutions, filenames for disk storage. In addition, instrument malfunctions, repairs, and maintenance activities are recorded in the run logs. The logbooks are reviewed by the section QC Chemist as part of the data package review process.

Strip Chart Recordings/Chromatograms/Computer Output: All strip chart recordings, chromatograms, computer output, and other instrument-generated records are clearly labeled with the following information: instrument identification, date, analyst, sample identifications, and operational conditions, if applicable.

8. ANALYTICAL QUALITY CONTROL

A quality control program is a systematic process that controls the validity of analytical results by measuring the accuracy and precision of each method and matrix, developing expected control limits, using these to detect anomalous events, and requiring corrective action techniques to prevent or minimize the recurrence of these events.

8.1 CONTROL SAMPLES

An important part of analytical quality control are quality control samples, which are samples that are introduced in the measurement process to monitor various aspects of the analytical procedures. Control samples can be prepared from environmental samples or be generated from standard materials in the laboratory. Figure 8-1 illustrates some of the possible field and laboratory control samples and where they are introduced in the sample collection and analysis process. These QC samples are discussed below. Not all of these control samples are required or used for all projects.

8.2 LABORATORY QUALITY CONTROL SAMPLES

The frequency and control limits specified below for the QC samples are only general requirements. Any requirements given in a project, program, or sampling plan, EA Laboratories SOP or method, or client request take precedence over those given here.

8.2.1 Blanks

Blanks are materials that are as free of analyte as possible and that are introduced at various stages during sample processing to monitor possible contamination introduced by the various operations.

8.2.1.1 Calibration Blanks

A calibration blank is an organic or aqueous solution that contains all the reagents and solvents in the same proportions as those used to prepare the calibration standards. The use of the calibration blank is described in Section 6.2.2.

8.2.1.2 Method Blanks

A method blank is a volume of deionized laboratory water for water samples or a purified solid matrix for soil/sediment samples that is carried through the entire sample preparation and analysis scheme as if it were an environmental sample. The method blank volume or weight will be approximately equal to the sample volumes or sample weights being processed. Method blanks are used to monitor interferences caused by contaminants in solvents, reagents, glassware, and

other sample processing hardware. They are also called reagent blanks and preparation blanks. A method blank is always prepared and analyzed for each batch of samples prepared.

The method blank must contain less than the method detection limit for the compounds of interest for any sample concentration less than 10× MDL. If this criteria is not met, then all sample processing is halted until corrective measures are taken and documented. All samples processed with the out-of-control method blank will be reprocessed and reanalyzed.

For methods, such as the determination of nitrate + nitrite in aqueous samples by automated colorimetry, in which the samples are not subjected to any processing steps that are not done to the standards, the method and calibration blanks are the same and are not separately prepared.

8.2.1.3 Volatiles Storage Blanks

A holding blank is laboratory pure water that is stored alongside a set of samples in the same kind of sample container; normally used only for samples destined for determination of volatile organics. It is used to monitor possible contamination introduced into the samples during storage (EAL-SOP-317).

8.2.2 Calibration Verification (ICV, CCV)

An Initial Calibration Verification (ICV) standard is prepared independently of the calibration standards, and is analyzed immediately following initial calibration to verify calibration. Acceptance criteria are specified in the determinative method.

The calibration curve is verified periodically throughout the analytical sequence with a Continuing Calibration Verification (CCV) standard which is the midlevel standard from the initial calibration. Acceptance criteria are specified in the determinative method.

8.2.3 Laboratory Control Samples

A laboratory control sample (LCS) is an aqueous or solid control sample of known composition, which is analyzed using the same sample preparation, reagents, and analytical methods employed for field samples. An LCS is obtained from an outside source or is prepared in the laboratory by spiking reagent water or a clean solid matrix from a stock solution that is different than that used for the calibration standards. The LCSs contain the analytes of interest for single-analyte methods and selected analytes for multianalyte methods according to the appropriate analytical method.

Laboratory control samples are used to demonstrate whether the sample preparation and analysis steps are in control, apart from sample matrix effects. LCSs are also called quality control reference sample and method blank spike.

Normally, laboratory control samples are analyzed with each batch of twenty (20) or fewer samples. The percent recovery is calculated and plotted on control charts and compared against control limits (see Section 8.5.2 below). If the recovery is outside the limits, corrective action is taken as described in Section 8.5.4.

For methods, such as the determination of nitrate + nitrite in aqueous samples by automated colorimetry, in which the samples are not subjected to any processing steps that are not done to the standards, the laboratory control sample and verification standard are the same and are not separately prepared.

8.2.4 Natural Matrix Spikes

A spike is a sample to which is added a known amount of analyte(s) before analysis. From the concentrations of the analyte in the spiked and unspiked samples a percent recovery is calculated:

$$\text{Percent recovery} = \frac{A - B}{C} \times 100$$

where:

A = sample concentration of the spiked sample (ppm)
B = sample concentration of the unspiked sample (ppm)
C = concentration of the spike (ppm)

A matrix spike is prepared by adding analyte to a subsample of a field sample before sample preparation and analysis. Matrix spikes indicate the performance of the entire method in the given matrix. Generally, for multianalyte methods a representative suite of the analytes is used in the matrix spike. Department of Defense (DoD) programs, such as Naval Facilities Engineering Service Center (NFESC) and Air Force Center for Environmental Excellence (AFCEE), require spiking of all target analytes in the matrix spike. Matrix spikes are performed for every batch of twenty (20) or fewer samples for organic analyses and for most inorganic analyses.

The percent recovery of a matrix spike is compared against the LCS limits (or method project limits, as appropriate), and the results are qualified when the percent recovery is outside the limits.

8.2.5 Analytical Spikes

An analytical spike is prepared by adding analyte to an aliquot of a processed sample prior to analysis. They are used to determined whether the analysis system is in control when a matrix

spike is outside its limits. Analytical spikes are used at the discretion of the analyst as a diagnostic tool or when required by a specific program (e.g., the post digestion spike in furnace AAS under CLP).

8.2.6 Replicates

Replicate samples are samples that have been divided into two or more portions at some step in the measurement process. Each portion is then carried through the remaining steps of the process. Replicate samples provide information on the precision of the operations involved.

The mean and relative percent difference (RPD) of the duplicates are calculated as follows:

$$\text{Mean} = \frac{X_1 + X_2}{2}$$

$$\text{RPD} = \frac{|X_1 - X_2|}{\text{Mean}} \times 100$$

where:

X_1 = concentration of first replicate
X_2 = concentration of second replicate

8.2.6.1 Natural Matrix Duplicates

Method duplicates are a pair of subsamples from a field sample that are taken through the entire preparation and analysis scheme. They indicate the precision of the entire method in the given matrix. For chromatographic organic methods the matrix spike (8.2.4) is duplicated, providing a matrix spike duplicate. Matrix spike duplicates are prepared for every batch of twenty (20) or fewer samples for organic analyses. For metallic and inorganic analytes a field sample is duplicated. Method duplicates are prepared for every batch of twenty (20) or fewer samples for inorganic analyses.

The RPD of a duplicate determination analyzed under a program or method that has limits established is compared against the limits, and the results are flagged when the RPD is outside the limits. For samples that do not have limits, the RPD is recorded and reported when requested by the client.

8.2.6.2 Analytical Duplicates

Analytical duplicates are prepared by taking two aliquots of a processed sample and analyzing them in the same manner. They are used to monitor the precision of the analysis system for the processed matrix. Analytical duplicates are used at the discretion of the analyst as a diagnostic tool or when required by a specific program (e.g., furnace AAS under CLP).

8.2.7 Surrogates

Surrogates are organic compounds that are similar to the analytes of interest in chemical composition, extraction, and chromatography, but are not normally found in environmental samples. These compounds are spiked into all blank, standards, samples, and spiked samples prior to purging or extraction in order to monitor preparation and analysis of samples. Generally, surrogates are used in chromatographic organic analyses, but not in inorganic analyses. Surrogate spike recoveries must fall within the control limits specified in the method or program. Surrogate recoveries are not calculated if sample dilution causes the surrogate concentration to fall below the quantitation limit.

8.2.8 Performance Evaluation Samples

Performance evaluation (PE) samples are standard reference materials or QC samples of known concentration obtained from EPA, NIST, or a commercial source. The analysis of PE samples monitors method accuracy.

8.2.8.1 Blind Performance Samples

Blind performance samples are PE samples for which the analyte concentrations are unknown to the analysts; they are used as part of external and internal performance audits. The laboratory analyzes blind PE samples as part of its laboratory certification efforts. Samples from the U.S. EPA water supply and water pollution studies are analyzed twice yearly for the Maryland and other state certification programs. In addition, other states and agencies require the periodic analysis of their own PE samples. Blind performance samples are also introduced into the laboratory by the QS Manager on an as needed basis or as specified by client QA requirements. The results of the analysis of PE samples are maintained by The QS Manager. The laboratory section chiefs are required to investigate and respond to any results that are outside the control limits.

8.2.8.2 Double Blind Performance Samples

Double blinds are performance samples which are not identified to the laboratory as performance samples as well as for which the analyte concentrations are unknown. EA Laboratories participates in a quarterly double blind study through a vendor program. In addition, double blinds can be submitted with project samples as part of the client's QA program.

8.2.8.3 Reference Samples

Reference samples are obtained from the same source as those described in 8.2.7, but are used by the analyst to check the accuracy of an analytical procedure prior to analysis of samples. These are particularly applicable when a minor revision has been made to an analytical procedure or instrument.

8.3 FIELD QUALITY CONTROL SAMPLES

These samples are not included in the laboratory quality control program but are logged in as unique samples and analyzed when submitted with project samples. Data for these QC samples are reported with associated samples.

8.3.1 Field Blanks

Blanks that are collected in the field are an important link in the quality control data chain for a set of samples. The analytical data derived from these blanks are necessary to assess field operations: contaminant-free sample containers, preserving reagents and equipment; potential on-site environmental contamination; personnel expertise in sample collection; and problems that may occur in sample storage and transport.

The field quality control blanks should not be isolated from actual samples. They must be considered as samples and must be treated identically: preserved with the same reagents; stored and transported in the same containers as the samples; etc.

The types of blanks required and their frequencies are included in sampling section of all site-specific plans.

8.3.1.1 Trip Blanks

Trip blanks are to be used when sampling for volatile organics. The purpose is to determine if contamination has occurred as a result of improper sample container cleaning, contaminated blank source water, sample contamination during storage and transportation due to exposure to volatile organics (e.g., gasoline fumes) and other environmental conditions during the sampling event.

Trip blanks are prepared prior to the sampling events either by the laboratory providing sample containers or by field team personnel who are responsible for the initial preparation of sample container and field equipment. The water must be free of volatile organic contaminants. Any appropriate preservatives must be added at the time that the blanks are prepared. The sample containers are sealed, labeled appropriately, and transported to the field in the sample containers as the sample vials. These blanks are not opened in the field. They are transferred to the ice

chest designated for volatile sample storage and transport and accompany the samples to the analyzing laboratory. Subsequent blanks for volatile organics (field and equipment) should use the same source water as the trip blanks, unless the water used for field and equipment blanks can be proven equivalent.

8.3.1.2 Field Blanks

Field blanks are used to evaluate the effects of on-site environmental contaminants, the purity of reagents used as preservatives or additives and the general sample containers filling/collection techniques.

Field blanks are prepared on site by filling the sample container(s) with analyte-free water, adding appropriate preservatives or additives, sealing the containers and completing the appropriate documentation. The field blanks must be handled in the same manner as the sample group for which it was intended (i.e., blanks are stored and transported with the sample group).

8.3.1.3 Sampling Equipment Blanks

Equipment blanks (also called rinsate blanks) are required if sampling equipment must be cleaned in the field and reused for subsequent sample collection. These blanks are used to determine the effectiveness of field cleaning procedures are well as those sources of contamination that may be found in a trip blank.

The final rinse of analyte-free water is passed over or through the sampling equipment, collected in appropriate sample containers, and preserved. These blanks must be included in the same storage and transport containers as the samples.

8.3.2 Replicate Field Samples

To help assess the precision of the measurement system field replicate samples are collected or prepared. These are of two types: collocated samples and split samples. The types of replicate field samples and their frequencies will be specified in the site-specific plan.

8.3.2.1 Collocated Samples

Collocated samples or collection replicates are two or more separate samples collected independently in such a manner that they equally represent a medium at a given time and location (e.g., side-by-side soil core sample; two water samples collected at essentially same time from the same point in a lake). Collocated samples provide intralaboratory precision information for the entire measurement system, including sample collection, homogeneity, handling, shipping, storage, preparation, and analysis.

8.3.2.2 Split Samples

A split sample is a single collected sample that is divided into two or more portions in the field; each portion is then carried through the remaining steps in the measurement process as a separate sample. The splits are either sent to the same laboratory or divided among several laboratories. Split samples, when analyzed by the same organization, provide precision information on homogeneity, handling, shipping, storage, preparation, and analysis. When analyzed by different organizations, they serve an oversight function in assessing the analytical portion of the measurement system.

8.3.3 Blind Samples

Blind samples are performance evaluation samples (Section 8.2.8) that are introduced into the laboratory disguised as field samples. They are double-blind samples, i.e., both the concentration and identity are unknown to the analyst.

8.4 DATA QUALITY OBJECTIVES

Data Quality Objectives (DQOs) are the chemical data specifications in terms of precision, accuracy, completeness, representativeness, and comparability which are developed to ensure defensible environmental decision making. DQOs are developed as part of a planning process for environmental data collection operations which was developed by EPA. In this section, the characteristics of DQOs are described. DQOs are defined for and are specific to each project and documented in a Quality Assurance Project Plan (QAPP) or Laboratory Project Summary.

8.4.1 Precision

Precision is the mutual agreement among individual measurements of the same property and is a measure of the random error component of the data collection process. The overall precision of the data is the sum of that due to the sampling and analysis. The sampling precision is assessed by collecting field duplicates. The analytical precision is determined by preparing and analyzing duplicate subsamples. Precision can be expressed in several different ways, each of which has its uses; for multiple measurements these include the standard deviation, the relative standard deviation, and the range, and for duplicates the relative percent difference.

8.4.2 Accuracy

Accuracy is the degree of agreement of a measured value with the true or expected value of the measured quantity. It is a measure of the bias or systematic error of the entire data collection process. Sources of these errors include the sampling process, field and laboratory contamination,

sample preservation and handling, sample matrix, sample preparation methods, and calibration and analysis procedures. Sampling accuracy is assessed by evaluating the results of field/trip blanks, analytical accuracy through the use of calibration and method blanks, calibration verification samples, laboratory control samples, and matrix spikes.

8.4.3 Representativeness

Data representativeness is the degree to which data accurately and precisely represent a characteristic of a population, parameter variations at a sampling point, or an environmental condition. Representativeness is a quantitative parameter that is most concerned with the proper design of the sampling program. The sampling program has been designed so that the samples collected are as representative as possible of the medium being sampled and that a sufficient number of samples will be collected. Representativeness is addressed by the description of the sampling techniques and the rationale used to select the sampling locations.

8.4.4 Completeness

Completeness is defined as the percentage of measurements made that are judged to be valid data. To achieve this objective, every effort is made to avoid sample loss through accidents or inadvertence. Accidents during sample transport or lab activities which cause the loss of the original sample will result in irreparable loss of data. Collection of sufficient sample allows reanalysis in the event of an accident involving a sample aliquot. The assignment of a set of continuous laboratory numbers to a batch of samples which have undergone chain-of-custody inspection makes it more difficult for the analyst to overlook samples when setting up a batch of samples for analysis. The continuous laboratory numbers also make it easy during the data compilation stage to pick out the samples which have not been analyzed and to order their analysis before the data are reported and before holding times have been exceeded. The completeness of each batch of samples can be calculated by dividing the total number of analyses completed by the number that should have been performed on that batch times 100.

8.4.5 Comparability

Data comparability is a measure of the confidence with which one data set can be compared to another. It cannot be described in quantitative terms, but must be considered in designing the sampling plans, analytical methodology, quality control, and data reporting. The use of standard sampling techniques and validated, EPA-approved analytical methods assures that the parameters being measured are comparable with data generated from other sources. Reporting of data in units used by other organizations also assures comparability.

8.5 CONTROL CHARTS

Quality control charts are graphical plots that are used to determine whether a process is in a state of statistical control. The vertical axis of the control chart is the value of the parameter being measured, and the horizontal axis is the time or sequence of the measurements. A control chart is characterized by a central line, warning limits, and control limits. The central line is the mean, theoretical, or most probable value for the measured parameter. The limits are values on either side of the central line with which are associated probabilities that an observed value will be within the limits. The warning limits are the 2σ or 95% limits; that is, if the process is operating correctly and only random scatter is being observed, nineteen out of twenty points should fall inside the warning limits. The control limits are the 3σ or 99% limits; only one point in a hundred should fall outside these limits by chance alone.

8.5.1 Accuracy and Precision Charts

The control charts used in the laboratory are generated from the analysis of laboratory control samples (LCS), which are used to demonstrate that a method is in control, apart from sample matrix effects. The data from the LCS measurements are plotted on two Shewhart control charts. One is for the accuracy of the method (Figure 8-2), which determines whether bias is developing in the parameter being monitoring. The other is for the precision (Figure 8-3), which demonstrates whether the variability of the method is within acceptable limits. The parameter that is plotted on the accuracy chart is the percent recovery of the LCS measurement, calculated from:

$$\text{percent recovery (\%R)} = \frac{\text{found concentration}}{\text{expected concentration}} \times 100$$

The moving ranges between each successive pair of percent recoveries are calculated and plotted on the precision chart:

$$\text{moving range } (R_i) = |\%R_{i+1} - \%R_i| \quad \text{for } i = 1,2,3...(n-1)$$

8.5.2 Calculation of Chart Limits

To calculate the warning and control limits for the charts, 20-30 values of the percent recoveries are collected. From these data the mean percent recovery, $\overline{\%R}$, and the mean moving range, $\overline{R}$, are calculated. The central line of the accuracy chart is the mean percent recovery, $\overline{\%R}$. For control charts based on the moving range of two measurements, the upper and lower warning and control limits of the accuracy chart are given by:

$$\text{Upper control limit (UCL)} \quad = \overline{\%R} + 2.660\overline{R}$$

Upper warning limit (UWL) $= \overline{\%R} + 1.773\overline{R}$

Lower warning limit (LWL) $= \overline{\%R} - 1.773\overline{R}$

Lower control limit (LCL) $= \overline{\%R} - 2.660\overline{R}$

The central line of the precision chart is the mean moving range, $\overline{R}$. The warning and control limits for the precision chart are given by:

$UCL = 3.267\overline{R}$
$UWL = 2.511\overline{R}$
$LWL = 0$
$LCL = 0$

The limits are updated at least quarterly or when the method is changed significantly.

8.5.3 How the Charts are Used

As the value for the control sample is calculated it is compared against the established limits. If the value is within the limits, the analysis is in control and data generated can be used. The percent recovery and the associated run information are entered into the LIMS data base from which they can be retrieved to plot control charts and to update the limits.

8.5.4 Out-of-Control Situations

The following three conditions are used with the control charts to indicate that a possible out-of-control situation exists:

1) any point outside the control limits;
2) any two consecutive points between the warning and control limits; or
3) seven successive points on the same side of the central line.

When one of these conditions exists, the method and the calculations must be investigated to determine if a cause for the condition can be found. When an analyst observes that an out-of-control situation has occurred, the analyst's supervisor is notified, and the appropriate corrective action procedures are initiated. The situation is monitored closely to determine whether control has been reestablished. Analytical data generated during an out-of-control situation are evaluated and reanalyses are performed as necessary where data quality is impacted. If the problem cannot be identified or corrected, the QC Chemist is notified and corrective action is initiated. An

out-of-control event and the corrective action taken are documented on a nonconformance record form (Section 11).

8.5.5 References

American Society for Testing and Materials. 1976. ASTM Manual on Presentation of Data and Control Chart Analysis. STP 15D. ASTM, Philadelphia.

Duncan, A.J. 1974. Quality Control and Industrial Statistics, 4th edition. Irwin, Homewood, Ill.

Naval Facilities Engineering Service Center. February 1996. Navy Installation Restoration Laboratory Quality Assurance Guide. Interim Guidance Document. NFESC, Port Hueneme, California.

United States Environmental Protection Agency. 1979. Handbook for Analytical Quality Control in Water and Wastewater Laboratory. EPA-600/4-79-019. U.S. EPA, Cincinnati, Ohio.

FIELD QA/QC SAMPLE DEFINITIONS

Trip blanks are used to determine if contamination resulting from glassware cleaning, contaminated source water, sample contamination during storage or transport or other exposure during sampling may impact sample results. Trip blanks are for VOC analyses only.

Field blanks are used to determine if onsite water sources used for decontamination have impacted sample results.

Rinsates are used to ascertain the quality of equipment decontamination between uses.

Field duplicate samples are utilized to determine the reproducibility of sampling technique and laboratory precision.

Split samples are utilized as a quality control check on laboratory performance (analyzed by an outside laboratory or regulatory agency).

Field SOPs are written procedures for sample collection.

Field audits are independent assessments of field crew adherence to project-specific work plans and the QAPP and corporate QA program.

LABORATORY QA/QC SAMPLE DEFINITIONS

Method blanks are samples of laboratory or DI water or sample solvents which are treated like samples. Method blanks identify interferents or contaminants which are introduced by the analytical procedure.

Calibration samples are certified standards containing measurable amounts of the analytes to be identified and quantified.

Laboratory control samples consist of laboratory pure water or solvent and a measured concentration of the analyte(s) being tested. Laboratory control samples are treated like samples and subjected to all steps of the analytical procedure.

Surrogate spiking compounds are compounds similar in performance to the compounds of interest which are introduced into each sample, standard, and blank during organic analyses. They are utilized to determine the performance of the analytes.

Matrix spikes are field samples to which a measured amount of analyte(s) of interest are added. Recovery of the matrix spike is utilized to determine the effects of sample matrix on the recovery of the analyte(s) of interest and may be indicative of bias.

Matrix duplicates, matrix spike duplicates are second aliquots of field samples treated identically as samples or sample matrix spikes. These are utilized to determine sample matrix effects on analytical precision.

Figure 8-1 Quality Control Samples

9. PREVENTIVE MAINTENANCE

9.1 PROGRAM REQUIREMENTS

Within EA Laboratories, preventive maintenance is an organized program of actions (such as equipment cleaning, lubricating, reconditioning, adjustment, and/or testing) taken to maintain proper instrument and equipment performance and to prevent instruments and equipment from failing during use. An adequate preventive maintenance program increases reliability of a measurement system. A preventive maintenance program considers the following:

- instruments, equipment, and parts thereof that are subject to wear, deterioration, or other change in operational characteristics without periodic maintenance
- spare parts that should be available within the laboratory to minimize downtime
- frequency that maintenance is required

The implementation of a preventive maintenance program is dependent upon the specific instruments and equipment used. EA Laboratories Managers are responsible for preparation and documentation of the program. Section Chiefs implement the program, and the Quality Services Manager (QSM) reviews implementation to verify compliance. For each operational group, at a minimum the preventive maintenance program includes the following:

- a listing of the instruments and equipment included in the program
- the frequency of maintenance considering manufacturer's recommendations and/or previous experience with the equipment
- a file for each instrument in the program containing the following information:

    - a list of spare parts maintained by the laboratory
    - external service contracts
    - items to be checked and/or serviced during maintenance and directions for performing maintenance (if external service is not provided or if not stated in manufacturer's instrument manuals).

Unless superseded by specific project requirements, the preventive maintenance activities performed by EA Laboratories are given in Table 9-1.

9.2 DOCUMENTATION

Preventive maintenance will be conducted by qualified laboratory personnel or outside vendors and documented in instrument logbooks. Date of service, person performing service, type of service performed, reason for service, and replacement parts are recorded. Copies of service records from outside vendors are maintained by the Section Chief with the instrument file.

TABLE 9-1 PREVENTIVE MAINTENANCE REQUIREMENTS

Instrument	Item Checked/Serviced	Frequency
Gas Chromatograph	EC (Ni-63) wipe test	Semiannually
	Change column	As needed
	Change gas wool plug	As needed
	Replace septum	As needed
	Change fuses	As needed
	Clean and silanize or replace glass liners or injectors	As needed
	Clean FID/NPD detectors	As needed or annually
	Clean purge vessel	Daily
	Bake trap	Between each analysis
	Replace trap	As needed
	Replace carrying gas filters	As needed
GC/MS	GC/MS maintenance is the same as GC with the following additions:	
	Mechanical pump oil	Quarterly
	Vacuum chaff filter	Semiannually
	Turbo pump oil	Annually
	Computer air filter	Semiannually
	Card cage air filter	Semiannually
	Source-clean ceramics, polish lenses	As needed
	Clean poles and ceramics	As needed
	Clean contacts on the component boards	As needed
	Vacuum the component boards	As needed
	Replace quartz injection port insert	As needed
	Replace septum	As needed
	Injection port liner checked	Daily
	Column maintenance	As needed
	Disk drive	Semiannually (service engineer), or as needed
	Printer	Quarterly
HPLC	Pressure	Daily
	Plunger Scale	Annually
	High Pressure Pump	Annually
	Low Pressure Pump	Annually
	Check valves	Annually or as needed
	Lamps / detector	As needed
	Column	As needed

TABLE 9-1 PREVENTIVE MAINTENANCE REQUIREMENTS

Instrument	Item Checked/Serviced	Frequency
Atomic Absorption Spectrophotometer	5-point calibration performed	Daily, with each run
	Electrical	Each shift
	Lamps	Each parameter
	Optics	Annually
	Clean windows	Daily, with each parameter
	Replace graphite tube	At beginning of each run
	Replace contact rings	Quarterly, or as needed
	Replace quartz windows	As needed
	Clean furnace windows	At beginning of new run
	Align background lamp (3F)	When serviced by repairman
	Check lamp intensity	Each parameter
Inductively Coupled Plasma Spectrophotometer	Sample introduction system	Daily
	Check pumps	Daily
	Check electronics	Daily
	Clean, realign torch	As needed
	Change nebulizer	As needed
	Clean mixing chamber	As needed
	Check nebulizer press	As needed
	Replace pump tubing	Daily, or as needed
	Clean air filters (7P) and water filter	Semiannually
Ion Chromatograph	Plunger seals	As needed
	Plumbing	
	Oil pumps	
	Check valves	
	Column	
	Change fuses	
	Prime pump head	
	Check pressure	Daily
Infrared Spectrophotometer	Clean cells	Daily
Total Organic Carbon Instrument	Check oxygen purity	Each new cylinder
	Check heater	Daily when used
	Add acid	Monthly
UV/Vis Spectrophotometer	Clean cells and windows	Daily
	Lamp	As needed
	Wavelength checked	Annually
	Serviced	As needed
Auto-Analyzer	Pump oiled	Monthly
	Tubing	As needed
	Lamps	As needed

TABLE 9-1 PREVENTIVE MAINTENANCE REQUIREMENTS

Instrument	Item Checked/Serviced	Frequency
pH Meter	Electronics checked Electrolyte changed	Daily Checked weekly; changed when low
Refrigerators/Freezers	Temperature checked and logged Compartment cleaned	Daily on each work day Quarterly
Walk-in Coolers	Temperature checked and logged Unit cleaned	Daily on each work day Quarterly
Balances	Service representative calibration Internal weight train, gears, electronics Calibration Checked	Annually Annual service Daily with class "P" weights Analytical: Weekly with class "S" weights Toploading: Monthly with class "S" weights
Thermometers	Calibrated	Annually for mercury in glass thermometers Quarterly for all other thermometers
Class S Weights	Calibrated	Annually
Deionized/Organopure Water	Conductivity check Ion-exchange bed changed Replace filters	Daily Weekly As needed
Vacuum Pumps and Air Compressor	Check performance Lubrication, belts, etc.	Weekly As needed
Water Baths	Water level Bath cleaned	Added as needed Semiannually

10. DATA HANDLING

The procedures for data handling followed in the laboratory are an important part of the laboratory quality assurance program. The overall flow of data through the laboratory from project start through sample analysis and data generation and reporting is shown in Figure 10-1. EA Laboratories treats all records and project data as client confidential; no information will be shown to anyone outside EA without client approval.

10.1 DATA COLLECTION

For inorganic and general organic analyses where the instruments are not directly coupled to computerized data systems, the raw data are instrument responses in the form of meter, recorder, or printer output. The chemist performing the analysis enters the bench-generated data into a bound laboratory workbook specific for each parameter. All entries are made in ink. These data consist of instrumental responses (absorbances, percent transmittances, etc.), standard and spike concentrations, sample numbers, and any other pertinent information. The workbooks are under the control of the group supervisor who is responsible for their security. For computerized instruments the output is in the form of printer output and files on magnetic disks, which are filed by sample batch.

For chromatographic organic analyses, the raw data are instrument responses in the form of chromatograms, integrator output, or computer- generated data files. The chromatograms and printer output are stored in project-specific files. The data files are archived on magnetic tape or disks.

10.2 DATA REDUCTION

Data reduction includes all processes that change either the values or numbers of data items. The data reduction processes used in the laboratory include establishment of calibration curves, calculation of sample concentrations from instrument responses, and computation of quality control parameters.

10.2.1 Calibration

The calibration or standard curve is the curve that plots instrument response against analyte concentration. The curve is prepared by measuring the responses of a series of solutions of the analyte (calibration standards) with known concentrations. Least-squares regression can be used to fit a curve through the standard concentration- response data. The regression analysis also provides parameters, such as the correlation coefficient, that can be used to assess the condition of the analysis.

For many chromatographic methods the calibration factor (CF) is used to establish the linearity of the instrument response over the calibration range. If the calibration curve satisfies the specified linearity

criteria, a single-point standard can be used for sample calculations. Use of the internal standard method with GC/MS analyses requires the determination of response factors (RF).

Formulas used in calibration are in Table 10.1.

10.2.2 Sample Calculations

The reduction of instrument responses to sample concentrations takes different forms for different types of methods. The discussion below deals with non-chromatographic and chromatographic methods and solid sample calculations. Formulas used to calculate sample concentrations are in Tables 10.2.

Non-chromatographic Methods - For most spectrophotometric analyses, the sample concentrations are calculated from the measured instrument responses using a calibration curve. The sample concentrations can be back-calculated from a regression equation fitted to calibration data. For gravimetric and titrimetric analyses, the calculations are performed according to equations given in the method.

Chromatographic Methods - For chromatographic analyses, the unknown concentrations are determined using response factors with either internal or external standardization. When the compound has been identified, the quantitation of that compound is based on the integrated abundance of the primary ion(s). If the sample produces an interference for the primary ion, a secondary ion is used to quantify. The concentration in the sample is calculated using the response factor (RF).

Quantitation by the external standard technique for GC analyses involves calculation of the concentrations of the target compound from the sample response and the response of a standard solution of the compound. These calculations are generally performed by the associated computerized data systems.

Solid Samples - The dry-weight concentration of a solid sample is calculated from the analytical concentration according to the formula in Table 10.2.

Significant Figures and Units - The number of significant figures in the reported data is consistent with the limits of uncertainty inherent in the analytical method. The data reporting conventions are set forth in EAL-SOP-172. The units used in reporting data are those commonly used for the analyses performed. Concentrations in liquid samples are expressed in terms of weight per unit volume (e.g., milligrams per liter or micrograms per liter). Concentrations in solid or semisolid matrices are expressed in terms of weight per unit weight (e.g., milligrams per kilograms). In addition solid concentrations are converted to a dry-weight basis, using the percent solids of the sample.

10.2.3 Quality Control Parameters

Important data reduction methods for quality control samples are the expression of the precision and accuracy of the measurements. Formulas used in these processes are in Table 10.3.

Precision is the mutual agreement among individual measurements of the same property, usually under similar conditions. Precision can be expressed in several different ways, such as relative percent difference and relative standard deviation. The relative percent difference (RPD) is used for duplicate measurements. The relative standard deviation (RSD) is used for replicate measurements.
Accuracy is the degree of agreement of a measured value with the true or expected value of the measured quantity. The accuracy of control sample measurements is generally expressed as a percent recovery. For samples without a background level of the analyte, such as reference materials, laboratory control samples, and performance evaluation samples, the percent recovery (%R) is used to estimate accuracy. The percent recovery for measurements in which a known amount of analyte (a spike) is added to an environmental sample can also be used to evaluate accuracy.

10.3 LABORATORY DATA VERIFICATION

Data verification is a systematic process of reviewing data against a set of criteria to identify outliers or errors and to delete suspect values or to flag them for the user. There are at least two types of data validation which are used for environmental analytical data: verification of reported results, and validation of data for useability using guidelines developed by the U. S. EPA for Contract Laboratory Program protocols. While there is overlap in the review requirements between the two, evaluation criteria are different due to distinct objectives between the two processes: data verification is performed to assess the ability of the laboratory to analyze samples according to a prescribed analytical method; data validation is designed to verify the usability of a body of data as defined by project specific Data Quality Objectives.

10.3.1 Laboratory Verification of Reported Results

This validation is referred to as *data verification* to avoid confusion with other types. Data verification is performed by the laboratory prior to release of results, and to evaluate the data against acceptance criteria specified for Quality Control samples either in the laboratory QAMP or in a Quality Assurance Project Plan (QAPP). The quality control data produced during analysis are reviewed by the analyst and QC Chemist during the analytical process to validate data integrity during collection and reporting of analytical data. Figure 10-1 shows the review process. Technical Review Checklists (Figure 10-2) are used to document the performance and review of the quality control and analytical data.

Where spreadsheets or databases are developed by laboratory staff for the purpose of manipulating data, these software applications must be verified and documented prior to use and must be reverified if changes are made. The documentation will include:

- A description of the program
- A copy of the test data, and the resulting output
- A copy of the manual calculations used to verify the software.

All documentation for initial verification or modifications must be reviewed and approved by the Section Chief prior to implementation, and maintained on file by the Section Chief or QC Chemist to be reviewed by the QSM during routine internal audits.

10.3.1.1 Laboratory Verification Process and Responsible Personnel

Initial review of analytical and quality control data is the responsibility of the analyst. Data are checked for errors in transcription, calculations, and dilution factors and for compliance with quality control requirements. Failure to meet method performance quality control criteria results in reanalysis of the sample or lot if data usability is affected. After the initial review is completed, the data are collected from summary sheets, workbooks, or computer files and assembled into a data package. The second level of data review is the prime responsibility of the QC Chemist , who verifies the data is compliant with method and project requirements.

The Section Chief is responsible for development and implementation of data review checklists for use in their respective groups. An example of this technical review checklist is shown in Figure 10-2. The areas addressed in the checklist include the following:

- proper chain-of-custody and sample handling procedures followed
- parametric holding times met
- samples prepared and analyzed according to specified methods
- instrumentation calibrated according to specified methods
- spike (surrogate or standard) recoveries within specified ranges
- blanks prepared and analyzed as required
- calculations performed correctly and verified
- transcription of raw and final data correct
- detection limits determined correctly and within required limits

The checklist is completed and signed by the analyst and QC Chemist.

The third level of review is performed by the Laboratory Project Manager who certifies the data by signing the Analytical Narrative in the final report.

Finally, the QS staff is responsible for a minimum 10% audit of all final reports. The reports are chosen randomly for review.

10.3.1.2 Identification and Treatment of Outliers

There are no absolute guarantees against nonrepresentative data points. Therefore, all personnel involved in sample handling, analysis, and data management must be alert to potential contamination and procedural errors. However, if nonrepresentative data points appear in the final stages of analysis, there is a mechanism for identifying apparently or obviously erroneous or nonrepresentative data (outliers). The following procedures are primary methods for outlier identification and represent the type of logic to be applied to situations or parameters not specifically dealt with here.

Interrelated Data Cross Checks

- Inorganic carbon species and pH. The carbonate equilibrium dictates that (1) below a pH of 8.2-8.3, bicarbonate is completely dominant with only undetectable amounts of carbonate (and hydroxide) present, and (2) below a pH of 4.2-4.5, only CO_2 should exist in detectable amounts. These interactions are used as cross-checks for alkalinity determinations involving speciation.

- Phase change speciations. Any suite of analyses involving total, particulate, or dissolved speciation will generally be subject to comparisons between parameters (e.g., total vs. dissolved metals concentrations). Obviously, dissolved concentrations should not exceed total concentrations (disregarding combined precision effects when true total and dissolved concentrations are the same or very similar).

  All such speciation analyses must be checked for such impossible situations before final data sign-off. For all parameters with short holding times, these determinations must be made as soon as possible. For this reason, it is often advisable for the analyst(s) to perform certain analyses concurrently (e.g., ammonia and total Kjeldahl nitrogen, total and dissolved phosphorus, and total oxidized nitrogen and nitrate). Frequently, similarity of variances is increased, thus improving the reliability of comparisons (and differences).

- Residue-analyses. Analyses for total dissolved residue and similar analyses are also a type of speciation and are therefore subject to comparisons similar to those mentioned above. For instance, total residue should exceed all other species values, at least within the combined effects of individual analysis precisions.

- Compound list duplication. When requested analyses result in duplication of reported analytes, the results should be checked to ensure reasonable agreement between the reported values.

Correction/Elimination-Procedures: If simple errors (e.g., miscalculations) cannot be identified, the analysis must be performed again (with the project manager's knowledge). Obvious corrections due to miscalculation may be made with the knowledge of the laboratory manager.

10.3.2 EPA Contract Laboratory Program Data Validation

Data validation procedures described in Section 10.3.1 should not be confused with those of the U.S. EPA Contract Laboratory Program as specified in *Functional Guidelines for Evaluating Organic/Inorganic Analyses* (U.S. EPA, June 1988). This process is used to validate the usability of a body of data as defined by project specific Data Quality Objectives. The data review is performed from the perspective of the end-users, and requires their input on the intended use of the data. There is some overlap in the review requirements between laboratory data validation and CLP Data Validation; however, the data review criteria are different, and includes areas not covered by the CLP Statements of Work (SOWs) and some over which the laboratory has no control, such as field and trip blanks and holding times from sampling.

EA Laboratories does not perform CLP Data Validation which is not a part of the Quality Assurance or reporting requirements of the SOWs. When required, it should be done by a group not associated with the laboratory performing the analysis. Both field and laboratory activities are evaluated in CLP Data Validation to ensure that all possible sources of error associated with data collection are reviewed.

10.4 DATA REPORTING

10.4.1 Data Release

It is the policy of EA Laboratories that ***no data is released to a client unless it is final*** and has been through a tiered review process, including the analyst, QC Chemist, and Laboratory Project Manager (LPM) responsible for certification of the results.

In the event that results are required by the client on a fast turnaround prior to release of the entire report, ***final data*** may be released ***with knowledge and approval of the client and LPM*** after review by the analyst and certifying Manager. A signed cover sheet must be used for all data released prior to the final report, and must be signed and dated by the analyst or QC Chemist and the LPM to indicate review, certification, and approval.

Furthermore, the LPM must include any data released prior to the final report in the report file as a facsimile or as a copy of the signed cover sheet and results sent to the client.

This policy ensures that only accurate data will be released to the client. In the past the laboratory has released data as preliminary results which changed upon further review. Although such data was clearly stamped "Preliminary Data Subject to Change", there have been instances where the data was used by the client, the results changed, and the laboratory was judged to be at fault. In order to avoid such occurrences, it is EA Laboratories position that no result will be released unless it is supportable and final.

10.4.2 Hardcopy Reports

After a data package has been reviewed and signed off on, it is given to the laboratory Reports group. The Reports Group Leader is responsible for collecting the data packages and other report-related information, such as copies of chain-of-custody, communication record, and nonconformance forms, and for tracking analysis status and the date the data are due to the client. The sample data and any required backup information are assembled into a draft data report, which is formatted according to project/client requirements (EAL-SOP-303). An analytical narrative, method table, and tables of data qualifiers are generated. The report preparation is tracked using the Report Review Checklist (EAL-SOP-187). Each report is assigned a unique report number that is used in tracking, problem resolution, and archiving. The draft report is reviewed by the Reports staff using the Report Review Checklist (Figure 10-4). Each completed report is reviewed by the Reports Group Leader or by someone other than the preparer for compliance with all report deliverables requirements

After completion of the review of the data report, the Laboratory Project Manager or his deputy signs off on the data report, and the report is forwarded to the project manager/ client. The laboratory copy of the report is filed by the Reports Group in the Central Project File according to report number.

10.4.3 Electronic Reports

Electronic reports are prepared by the Information Systems (IS) group to the client specifications which are determined at the time of project start-up. It is the responsibility of the IS Manager to oversee the preparation of the electronic deliverable and to reconcile it with the hardcopy report. Electronic data files are maintained on the laboratory computer network or in electronic archive by the IS Network Administrator.

10.5 DATA STORAGE

All client data are maintained in a secure manner either within EA Laboratories or in a secure off-site location. Project-related hardcopy information is maintained in the Central Project Files, which are the responsibility of the QS Manager and the Laboratory Project Manager. The procedures used to organize, maintain, and archive the Central Project Files are detailed in EAL-SOP-196. The central files include project statements of work or proposals, correspondence to and from the client, chain-of-custody records, quality assurance plans, final data reports, and references as to where laboratory backup data can be located in the laboratory or in archives. The analytical data reports are filed by report number in a separate section of the central files and contain all documentation sent to the project manager/client as well as all backup information on the analyses. The central files do not include laboratory notebooks, stripcharts, instrument logbooks, or computer disks/tapes, which are stored within the responsible operational groups. When a project is complete, the project files and other related data are checked, inventoried, and put into the archive system at EA's Hunt Valley warehouse. A unique box number is assigned to each archive box and entered into the archive file on the LAN system; the contents of each box are also listed in the file.

Unless superseded by program, project, or client specific requirements, the disposal date of archived HARDCOPY files is TEN YEARS from the archive date., and the disposal date of ELECTRONIC data is TWO YEARS from the archive date.

This policy for electronic data is consistent with Good Laboratory Practices prescribed in 21 CFR 58, and Good Manufacturing Practices designated in 21 CFR 820. The Laboratory Managers are responsible for ensuring that all electronic data is stored to prevent deterioration and that records are maintained identifying the tape/disk, archive date, and discard date.

TABLE 10.1 CALIBRATION FORMULAS

Application	Formula	Symbols
Linear regression calibration curves	$R = C\,a_1 + a_0$	C = concentration of the calibration standard R = instrument response a_0 = intercept of regression curve (instrument response when concentration is zero) a_1 = slope of regression curve (change in response per change in concentration)
Calibration factors [1]	$CF = \frac{A_x V_f}{C V_i}$	C = concentration of the calibration standard CF = calibration factor A_x = peak size of target compound in sample extract V_f = final volume of extracted sample (mL) V_i = initial volume of sample extracted (mL)
Response factors [2]	$RF = \frac{C_{is} A_x V_f}{C A_{is} V_i}$	C = concentration of the calibration standard RF = internal standard response factor C_{is} = concentration of the internal standard (ug/L) A_x = area of the characteristic ion for the target compound V_f = final volume of extracted sample (mL) A_{is} = area of the characteristic ion for the internal standard V_i = initial volume of sample extracted (mL)

1. Used for quantitation by the external standard technique.
2. Used for quantitation by the internal standard technique.

TABLE 10.2 SAMPLE CONCENTRATION FORMULAS

Application	Formula	Symbols
Concentrations calculated from linear regression calibration curves	$C = (R - a_0)/a_1$	C = analytical concentration R = instrument response a_0 = intercept of regression curve (instrument response when concentration is zero) a_1 = slope of regression curve (change in response per change in concentration)
Concentrations calculated from calibration factors [1]	$C = \frac{A_x V_f}{CF\ V_i}$	C = concentration (ug/L) CF = calibration factor A_x = peak size of target compound in sample extract V_f = final volume of extracted sample (mL) V_i = initial volume of sample extracted (mL)
Concentrations calculated from response factors [2]	$C = \frac{C_{is} A_x V_f}{RF\ A_{is} V_i}$	C = concentration (ug/L) RF = internal standard response factor C_{is} = concentration of the internal standard (ug/L) A_x = area of the characteristic ion for the target compound V_f = final volume of extracted sample (mL) A_{is} = area of the characteristic ion for the internal standard V_i = initial volume of sample extracted (mL)
Solid samples [3]	$K = \frac{C\ V\ D}{W\ (\%S/100)}$	K = dry-weight concentration (mg/kg) C = analytical concentration (mg/L) V = final volume (mL) of processed sample solution D = dilution factor W = wet weight (g) of as-received sample taken for analysis %S = percent solids of as-received sample

1. Used for quantitation by the external standard technique.
2. Used for quantitation by the internal standard technique.
3. Used to calculate the dry-weight concentration of a solid sample from the analytical concentration of the processed sample.

TABLE 10.3 QUALITY CONTROL FORMULAS

CHARACTERISTIC	FORMULA	SYMBOLS
Precision (as relative percent difference, %RPD)	$RPD = \frac{\lvert x_1 - x_2 \rvert}{(x_1 + x_2)/2} \times 100 = \frac{\lvert x_1 - x_2 \rvert}{(x_1 + x_2)} \times 200$	x1, x2 = duplicate values.
Precision (as relative standard deviation, %RSD)	$RSD(\%) = \frac{s}{\bar{x}} \times 100$	s = standard deviation $\bar{x}$ = mean of the measurements.
Accuracy (as percent recovery (%R) for samples without a background level of the analyte, such as reference materials, laboratory control samples, and performance evaluation samples)	$\%R = \frac{X}{T} \times 100$	X = found concentration T = true or assumed concentration
Accuracy (as percent recovery (%R) for measurements in which a known amount of analyte (a spike) is added to an environmental sample)	$\%R = \frac{X - B}{T} \times 100$	X = found concentration B = background concentration T = true or assumed concentration

FIGURE 10-1. LABORATORY INFORMATION FLOW

STEP	PERSONNEL	RESPONSIBILITIES
1	LABORATORY PROJECT MANAGER	a. Through client contact determines the project-specific data package/client report deliverables, and any modifications to the standard. Distributes the information to the laboratory staff.
2	ANALYST	a. Generates complete data package ("D" or "E") according to requirements specified for project. b. Performs initial quality control review.
3	QC CHEMIST *EDD SUBDIRECTORY/FILE NAME* *	a. Evaluates data package for compliance with deliverable order according to "D" or "E" specifications. b. Performs technical data review for compliance with analytical method, QAPjP, and client QA program, data validation criteria, or other client specifications. c. Develops analytical narrative. d. Transfers intermediate files to network project subdirectory specified by IS group, and identifies subdirectory and file names on Initial Report Review Form. e. Certifies data quality meets all specified requirements before release to Laboratory Supervisor.
4	LABORATORY SUPERVISOR	a. Checks data package against COC: samples, methods, package level. b. Certifies data quality meets all specified requirements before release to Division Manager.
5	REPORTS GROUP LEADER	a. Checks the data package against COC for EDD, Summary Table, and control chart requirements. b. Routes to Quality Services for control charts, and Information Services for EDDs/Summary Tables.
6	QS MANAGER - *CONTROL CHARTS* *	a. Generates control charts, writes narrative section on method control.
7	IS MANAGER - *EDDS, SUMMARY TABLES* *	a. Prepares EDDs and/or summary tables according to client specifications.
8	REPORTS GROUP LEADER	a. Assembles draft report from all data packages. b. Organizes and completes narrative. c. Reviews report for completeness, spelling, grammar before release. d. Routes report to Laboratory Project Manager for final review and signature to certify results. e. Sends completed report to client, files laboratory copy in Central Project Files.
9	LABORATORY PROJECT MANAGER	a. Reviews final report for completeness, signs narrative certifying results, signs cover letter, and reviews invoice.

* These are options which depend upon client requirements, and are generated by the specified group, except USAEC control charts which are generated by the analyst and submitted with the lot data package.

DATA REVIEW CHECKLIST

All questions should be answered with a "Y" for yes, "N" for no or "NA" for not applicable. All "N" answers require an explanation in the comments section.

PROJECT INFORMATION *REPORT NO:* *SHIP DATE:*

Client: Test: Instrument: Analyst: Method:

EA Nos: Matrix: Compound List: TICs:

COMMUNICATION

NCR

	Primary Analyst	QC Chemist	#
Has the Project Summary been reviewed?			
Have all associated memos or E-mail been included?			
Have the chain-of-custodies been reviewed for errors?			

HOLDING TIMES

Were TCLP/DI WET extraction holding times met?			
Were extraction holding times met?			
Were samples properly preserved?			
Were samples initially analyzed within holding time?			
Were dilutions/reanalyses completed within holding time?			
Were re-extractions initiated within holding time?			

EXTRACTIONS

Was the extraction sheet reviewed for errors/completeness?			
Were extracts concentrated to the appropriate final volume?	NA	NA	
Were extracts cleaned by appropriate methods?	NA	NA	
Was an MS/MSD extracted with this batch?			
If not, was an LCS duplicate extracted?			

CALIBRATIONS

Did DFTPP/BFB tune meet specified criteria?			
Did the resolution check meet specified criteria?	NA	NA	
Did the degradation check meet specified criteria?	NA	NA	
Did the initial calibration meet specified criteria?			
Did the daily/continuing calibrations meet specified criteria?			
Has an RF/%RSD/%D been hand-calculated?			

QUALITY CONTROL ANALYSIS

Did the method blank and LCS meet surrogate criteria?			
Did the method blank and LCS meet internal standard criteria?			
Did the LCS meet specified target analyte criteria?			
Did the LCS duplicate meet specified target analyte criteria?			
Was the method blank free of target analytes?			

SAMPLE ANALYSIS

Did all samples meet surrogate criteria?			
Did all samples meet internal standard criteria?			
Were all samples analyzed within appropriate cal/tune time?			
Did the MS/MSD meet specified criteria?			
Were all target analytes within calibration range?			
Were the appropriate dilutions performed?			
Were the appropriate reanalyses performed?			
Have all reported results been confirmed?			
Have the proper reporting limits/analyte lists been used?			

Figure 10-2. Example Technical; Review Checklist, Page 1

PACKAGE ORGANIZATION

Have all samples been included in the data package?	____	____
Have all forms been checked for correct information?	____	____
Are all required forms present in the data package?	____	____
Has the electronic file been generated?	____	____
If corrections were made, has a new EDD been generated?	____	____
Chain-of-custodies	____	____
TCLP/DI WET extraction sheet	____	____
Extraction sheets	_NA_	_NA_
Dry weight logs	____	____
Example calculation worksheet	____	____
Injection logs	____	____
Standards logs	____	____
GPC logs and UV trace chromatographs	_NA_	_NA_
Form IIs (Surrogate Recovery Forms)	____	____
Form IIIs (MS/MSD Recovery Forms)	____	____
Form IIIs (LCS Recovery Forms)	____	____
Form IVs (Method Blank Forms)	____	____
Form Vs (Tune Forms)	____	____
Form VIIIs (Internal Standard Forms)	____	____
Form Is (Sample Data Forms)	____	____
Are spectra included for all reported target analytes?	____	____
Are spectra included for all reported TICs?	____	____
Form Xs (Pest/PCB Identification Forms)	_NA_	_NA_
Form VIs (Initial Calibration Forms)	____	____
Are all initial calibrations included?	____	____
Are all ICV calibrations included?	____	____
Form VIIs (Cont. Calibration Forms)	____	____
Are all daily calibrations included?	____	____
Are all tune calibrations included?	____	____
Form VIIIs (Sequence Summary Forms)	_NA_	_NA_
Form IXs (GPC/Florisil Forms)	_NA_	_NA_

ERM Directory:_____

Forms Filename:_____ Generated by:_____

Intermediate Filename:_____

NARRATIVE NOTES

_____
_____
_____
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_____

Primary Analyst:_____ QC Chemist:_____

Figure 10-2. Example Technical; Review Checklist, Page 2

11. THE CORRECTIVE ACTION PROCESS

The Corrective Action Process is the laboratory's mechanism for identifying and solving nonconformance problems. The objective of the Corrective Action Process is to ensure that recognized nonconformances in performance of any activity associated with laboratory processes lead to effective remedial measures, and the steps taken to correct an existing condition are documented to provide assurance that any deficiencies are recognized in later interpretation and are not recurrent.

The steps comprising the Corrective Action Process and an example are listed in the table below:

Corrective Action Steps	Example
1. Define the problem.	LCS recoveries are outside control limits.
2. Investigate.	Analyst reviews run data, including calibration and quantitation.
2. Determine the cause.	1. Analyst finds LCS spike solution is 8 months older than the standard used to prepare the calibration standards. 2. Analyst analyzes LCS standard and finds recoveries agree with processed LCS.
3. Develop a corrective action plan.	1. Immediate Corrective Action is to prepare and verify a fresh LCS spike standard for laboratory use, and discard any remaining old standard. 2. Laboratory Section Chief/Group leader reviews shelf life requirements and revises SOP to ensure that LCS standard is made at the same frequency as the calibration stocks.
3. Implement and document the corrective action.	Staff advised of change in procedure and implementation.
4. Follow-up to verify that the corrective action has eliminated the problem.	Section Chief verifies that staff is following new procedure, and that the problem has not reoccurred.
5. Document process.	All documentation leading to problem resolution is maintained by the areas Section Chief. Revised SOP documents changed procedure.

In the example given above, the solution was straightforward; however, in many cases the solution takes some time to find. In cases where an investigation is underway, documentation of the noncomformance and the Corrective Action contains a discussion of the status of the investigation. In addition, there may be instances where a nonconformance is investigated and there is no assignable cause after all quality control checks have been evaluated. In this case, the Corrective Action documentation should state and indicate that the laboratory will continue to observe the process to determine if the nonconformance was isolated or reoccurs.

11.1 NONCONFORMANCES

11.1.1 Definition of Nonconformance

A nonconforming item or situation (EAL-SOP-072) is one that has the potential to affect the quality or quantity of data generated by the laboratory or the interpretation or use of the data by the client. These include:

- Deviations or variances from the prescribed requirements in the QAMP, SOP, or Method SOP
- Deviations or variances from the prescribed requirements in Quality Assurance Project Plan (QAPP) or Project Summary
- Out-of-control laboratory performance quality control samples
- Malfunctions of equipment or instruments; or any unusual occurrences or circumstances.

Nonconformances may be identified at any point along the flow of samples and data through the laboratory.

11.1.2 Classifications of Nonconformances

Nonconformances are designated as *CRITICAL, MAJOR* or *MINOR*, and are differentiated with respect to the impact on the quality of the sample data for its intended use.

CRITICAL nonconformances are those that prevent the laboratory from initiating sample analysis, so that no results can be reported to the client. Generally, but not always, critical nonconformances are identified during sample receipt.

MAJOR nonconformances are those that occur in the course of analysis and report generation which would affect the quality of reported results. Generally, but not always, major nonconformances are identified during sample analysis .

MINOR nonconformances are those associated with failed laboratory method performance, but which do not affect the reportability or quality of client sample results. Minor nonconformances are addressed through the corrective action process; however, *no client contact is required.*

11.2 NONCONFORMANCE AND CORRECTIVE ACTION DOCUMENTATION

CRITICAL, MAJOR, and *MINOR* nonconformances are recorded and reported using EA Laboratories Laboratory Nonconformance Record (NCR). Each NCR has a unique control

number that is used to cross-reference the nonconformance and its resolution in instrument logs, Technical Data Review Forms, on control charts, etc.

The NCR is a three-part form. After all the sections have been completed and signed, the top (white) copy is retained by the Quality Services Manager, the second (yellow) copy is given to the QC Chemist and put in the data package after the narrative has been written, and the third (pink) copy is maintained by the LPM in the project file.

The Nonconformance Record (NCR) form is divided into four sections: Nonconformance, LPM Review/Client Contact, Corrective Action, and Verification of Nonconformance and Corrective Action.

Nonconformance: The person who identifies the nonconformance (the Originator) initiates the NCR by completing this section. ***ALL ITEMS MUST BE COMPLETED.*** A space for the date of the nonconformance is also provided. A checklist of typical nonconforming situations and their classification is given, followed by space for a detailed description of the occurrence. The section is signed and dated by the originator and the QC Chemist and forwarded to the assigned Laboratory Project Manager (LPM) within 8 hours of the occurrence.

NONCONFORMANCE (EAL-SOP-072)

Client/Site BOOM AAP		Project # 123456.78	Sample Number(s) 970023 - 970030	
Parameter BNA	Method 3540/8270	Matrix SOIL	EAL Report Number 970001	Date of Occurrence 1/3/97

SAMPLE RECEIPT - CRITICAL/MAJOR	SAMPLE PREPARATION/ANALYSIS - MAJOR/MINOR	QC SAMPLE - MAJOR/MINOR	EQUIPMENT - MAJOR/MINOR
___ Broken/incomplete Chain-of-custody	___ Sample Preparation Outside Holding Time	___ Method Blank	___ Refrigerator/freezer
___ Broken or Missing Aliquot	___ Sample Re-Preparation Outside Holding Time	X LCS	___ Balance
___ Receive / Outside Holding Time	___ Insufficient Sample Amount for MS/MSD	___ Sample Surrogates	___ Thermometer
___ Aliquot Preservation	___ Analysis Outside Holding Time	___ MS/MSD	___ Weights
___ Cooler Temperature Check	___ Instrument Tune	___ Duplicate	___ Instrument Failure
___ Insufficient Sample Amount	___ Initial/Continuing Calibration		

Description: LCS recoveries below QC limits, see attached summary. Surrogates in LCS and method blank are acceptable.

Immediate Corrective Action

___ Sample Re-preparation and Re-analysis Inside Holding Time - MINOR	X Sample Re-preparation and Re-analysis Outside Holding Time - MAJOR
___ Sample Re-analysis inside Holding Time - MINOR	___ Sample Re-analysis Outside Holding Time - MAJOR
	___ Batch Re-preparation and Re-analysis Outside Holding Time - MAJOR
	___ Batch Re-analysis Outside Holding Time - MAJOR

Originator Hugh N. Crier	QC Chemist Sue P. Erman	Date 1/3/97

FORWARD FORM TO LPM WITHIN THE 8 HOUR SHIFT IN WHICH THE NONCONFORMANCE OCCURS.

LPM Review/Client Contact: Upon receipt of the NCR, the LPM reviews the nonconformance to determine if client contact is required.

ALL CRITICAL AND MAJOR NONCONFORMANCES REQUIRE CLIENT CONTACT WITHIN 24 HOURS OF THE OCCURRENCE.

This turnaround is required in the event that the client decides on disposition of the affected samples which makes the initiated corrective action identified by the originator unnecessary. An exception to this requirement is where several analytical fractions are required for a set of samples. In that case, the LPM may choose to delay client contact until all NCRs have been submitted in order to deal with disposition of the samples efficiently.

LPM REVIEW /CLIENT CONTACT

_X_ ***CLIENT CONTACT REQUIRED WITHIN 24 HOURS - CRITICAL/ MAJOR NONCONFORMANCE (EAL-SOP-072)***
___ ***CLIENT CONTACT NOT REQUIRED - MINOR NONCONFORMANCE (EAL-SOP-072)***

Impact of Nonconformance on Sample Results

SAMPLE RECEIPT - CRITICAL	IMPACT	POSSIBLE OPTIONS
___ Broken/incomplete COC	Legal Defensibility of Data	Sample Custody Affidavit to Document custody transfers
___ Broken/Missing Aliquot, Insufficient Sample	Completeness of Data Set	Omit Sample Form Data Set; Resample
___ Cooler Temperature, Aliquot Preservation	Regulatory or Program Compliance	Omit Sample Form Data Set; Resample
SAMPLE PREPARATION/ANALYSIS - MAJOR		
___ Sample Prepared Outside Holding Time	Regulatory or Program Compliance	Analyze and Report Data with Qualification; Resample
___ Sample Re-Prepared Outside Holding Time	Regulatory or Program Compliance	Analyze and Report Data with Qualification; Resample
___ Insufficient Sample Amount MS/MSD	Evaluation of sample measurement bias	Evaluate Surrogates or Report Data with Qualification
___ Analysis Outside Holding Time	Regulatory or Program Compliance	Analyze and Report Data with Qualification; Resample
QC SAMPLE - MAJOR		
___ Method Blank	Data Accuracy due to laboratory contamination	Re-Prepare/Re-Analyze Batch; Report data with qualification
_X_ LCS	Accuracy/precision of data due to lab performance	Re-Prepare/Re-Analyze Batch; Report data with qualification

Resolution per Client	Discussed with Mr. Tom Thumb. It was decided to report the results without re-extracting since the failed compounds are not chemicals of concern at the site, and the low recoveries do not impact the detection limits. No re-extraction is required, and the narrative will indicate that this is at the client's direction.
Laboratory Project Manager	Happy N. Pleasant — Date 1/3/97

FORWARD FORM TO SUPERVISOR IMMEDIATELY FOLLOWING LPM REVIEW OR CLIENT RESOLUTION.

Corrective Action: The LPM is required to forward the NCR to the section QC Chemist immediately following review or client resolution on the disposition of the sample results. The QC Chemist is responsible for initiating the Corrective Action Process. The NCR form lists the commonly observed general corrective actions, and the appropriate one is checked. The Laboratory Group Leader/Section Chief then completes the description of the corrective action taken. Documentation must be attached to show what actions have been taken to resolve the problem (For example, data for verification of a standard, communications records if discussion with vendor are required, temperature logs if required for process control, etc.).

CORRECTIVE ACTION

___ All potential sources of error evaluated. No assignable cause, monitoring process for any reoccurrence.
_X_ Root cause of nonconformance found, corrective action implemented.
___ Nonconformance previously identified, corrective action process has been initiated and an investigation is ongoing.

Description	LCS standard analyzed to verify accuracy. Concentrations of those compounds which were found to be below acceptance limits was below 80%. A new stock standard has been ordered from the vendor, and the shelf life criteria for the BNA standard used for LCS preparation has been reduced from 6 months to five. A revision to EAL-SOP-299 has been submitted to QS for processing.	
QC Chemist Sue P. Erman	Section Chief Juana B. Chief	Date 1/6/97

Verification of Nonconformance and Corrective Action: This section is signed and dated by the QSM to acknowledge the receipt of the Nonconformance Record, and verification of the status of the Corrective Action.

VERIFICATION OF NONCONFORMANCE AND CORRECTIVE ACTION

Quality Services Manager Harry D. Trainer	Date 1/6/97

11.3 NONCONFORMANCES AND DATA USEABILITY

EA Laboratories stated policy (EAL-SOP-305) appears in every Laboratory Analytical Data Report narrative (EAL-SOP-304): *All quality control criteria for method performance must be met for all target analytes for data to be reported. These criteria generally apply to instrument tune, calibration, method blanks, and Laboratory Control Samples (LCS). In some instances where method criteria fail, useable data can be obtained and are reported with client approval. The narrative will then include a thorough discussion of the impact on data quality.*

The NCR must document the steps of the Corrective Action Process as described above. The end user of the data, not the laboratory, will evaluate useability for the decision process. Laboratory input is important to assist the client in making a decision regarding the disposition of the affected data; however, that is a separate situation from the nonconformance and input can be given to the assigned LPM at the time of the occurrence. Such input is attached to the NCR.

11.4 CORRECTIVE ACTIONS REPORTS TO MANAGEMENT

The Quality Services Manager provides the Laboratory Director, Section Chiefs and QC Chemists with monthly summary reports detailing QA activities during the month. The purpose of the monthly summary is to provide management with a tool for the identification of problem areas so that decisions can be made on ways to improve the Quality system and to prevent reoccurrences. These summary reports include:

- Nonconformance Report Summary
- SOP Status Summary
- Method SOP Status Summary
- Laboratory Data Report Audit Summary

- Method Detection Limits Status Summary
- Certifications/Proficiency Expenditures Summary

12. PERFORMANCE AND SYSTEMS AUDITS

Audits are systematic checks to determine the quality of operation of some activity or function. Audits are of two types: performance and system.

12.1 PERFORMANCE AUDITS

Performance audits are independent sample checks made by a section chief or auditor to arrive at a quantitative measure of the quality of the data produced by one section or the entire measurement process. Performance audits are conducted by introducing control samples, in addition to those used routinely, into the data production process. These control samples may include: performance evaluation samples of known concentrations; field samples spiked with known amounts of analyte; and split field samples that are analyzed by two or more analysts within or without the organization.

The laboratory regularly participates in external performance audits as part of its laboratory certification efforts. Performance evaluation (PE) samples from the U.S. EPA water supply and water pollution studies are analyzed twice yearly for Maryland and any other state certification program which requires participation. In addition, other states and agencies require the periodic analysis of their own PE samples.

The results of performance audits are summarized and maintained by the laboratory QS manager and distributed to the section chiefs and QC Chemists who must investigate and respond to any results that are outside the control limits.

12.2 SYSTEM AUDITS

System audits are on-site qualitative inspections and reviews of the quality assurance system used by some part of or the entire measurement system. System audits are conducted by the corporate QA group with the assistance and involvement of field, laboratory, and project personnel. The audits are performed against a set of requirements, which may be a QA project or program plan, a standard method, or a project statement of work. A checklist is generally generated from the requirements and becomes the basis for the audit. The results of any deficiencies noted during the audit are summarized in a Audit Finding Report. Examples of an audit checklist and Audit Finding Report form are shown in Figures 12-1 and 12-2, respectively.

12.3 RESPONSIBILITY, AUTHORITY, AND TIMING

The Quality Services Manager (QSM) is responsible for the conduct of all internal audits, which include system, performance, and data audits. Audits will be conducted at appropriate intervals for each operational and support, but at a minimum on an annual basis. Audits may be conducted

more frequently for a specific task or activity. The QSM will keep on record a tentative schedule that details the number and types of audits, both scheduled and unscheduled, for the current year and a current list of the dates of completed audits.

All audits may be conducted by teams that will consist of, at a minimum, an audit team leader and an auditor. The audit team leader will be responsible for all the activities of a specific audit including organization, implementation, completion, and reporting. The audit team may also include a technical assistant who will provide technical expertise and assistance to the team on a specific task or function. Members of the auditing team will have auditing experience or will be trained in the use of the auditing procedures.

Specific audits will be planned, organized, and clearly defined before they are initiated. Procedures for the auditing activities will be identified prior to implementation of the audit, and will be designed to meet all requirements for the specific audit. In general, auditors will identify nonconformances or deficiencies, report and document them, initiate corrective action through appropriate channels, and follow up with a compliance review.

Records will be kept of all auditing tasks and findings.

12.4 DOCUMENTATION

To ensure that the previously defined scope of the individual audits is accomplished and that the audits follow established procedures, a checklist will be completed during each audit. The checklist will detail the activities to be executed and ensure that the auditing plan is accurate. Audit checklists will be prepared in advance and will be available for review. At a minimum, the checklist will allow space for the following information:

- date and type of audit
- name and title of auditor
- description of group, task or facility being audited
- names of lead technical personnel present at audit
- checklist of audit items according to scope of audit
- deficiencies or nonconformances

Following each system, performance and data audit, the QSM will prepare a report to document the findings of the specific audit. The report is submitted to the Director, EA Laboratories; Director, Corporate Quality Assurance; and the Section Chief and QC Chemist of the audited group to ensure that objectives of the QA program are met. In general, the format of the audit quality assurance reports will consist, at a minimum, of the following:

- description and date of audit;

- name of auditor;
- copies of completed, signed, and dated audit form and/or checklist;
- summary of findings of the audit including any nonconformance or deficiencies;
- date of report and appropriate signatures; and
- description of corrective actions

A copy of the signed and dated report for each audit will be maintained by the QSM, and will also be place in project files, as necessary.

EA LABORATORIES INSPECTION CHECKLIST

DIVISION/AREA:_____ PROJECT/PROGRAM:_____

AUDIT NUMBER:_____ AUDIT FINDING REPORT: _____

Q.A. AUDITOR:_____ DATE:_____

INSPECTION PARAMETERS	YES	NO	N/A	* COMMENTS
1. Standard Operating Procedures				
a. Date/Revision No. of applicable SOPs				
b. Copy of SOP(s) present in work area				
c. SOP(s) have proper signatures and are current				
d. Personnel awareness of SOP(s) requirements				
2. Protocol				
a. Personnel awareness of protocol requirements				
b. Are specific protocol requirements being followed?				
3. Equipment Records				
a. Are equipment/instrument SOP(s) available for inspection?				
b. Is user aware of SOP(s)?				
c. Is the equipment/instrument manual in the work area?				
d. Is there an instrument Use Log in the area?				
e. Where are maintenance/service records with part(s) replacement numbers kept? Are they current?				
f. Where are calibration, standardization, inspection and cleaning records kept? Are they current?				
4. Protective Clothing				
a. Is the protective clothing required for the observed procedure being properly worn?				
5. Data Records				
a. Are supervisor's signatures present where required?				
b. Are data properly recorded in ink? On the computer?				
c. Are error corrections made by a single line in ink with date, initials and explanation?				
d. Are all blanks filled in?				

* Attach a separate sheet for comments.

Figure 12-1. Example of an Audit Checklist.

EA Laboratories
19 Loveton Circle

Audit

Finding Report

Sparks, Maryland 21152
(410) 771-4920

AUDITOR SIGNATURE DATEE	AUDIT NUMBER	AUDIT FINDING NUMBER	AUDIT DATE
INDIVIDUAL CONTACTED	AREA	PROJECT/PROGRAM NUMBER	

COMPLETED BY AUDITOR

REQUIREMENTS
FINDING
RECOMMENDED CORRECTIVE ACTION

SCHEDULED RESPONSE DATE	RESPONSIBLE FOR CORRECTIVE ACTION

COMPLETED BY AUDITED ORGANIZATION

CORRECTIVE ACTION

DATE	SUBMITTED BY	MANAGEMENT APPROVAL

COMPLETED BY AUDITOR

DATE RESPONSE RECEIVED	AUDITOR	RESPONSEACCEPTABLE YES	NO
REASON FOR REJECTION			
AFR VERIFICATION			
DATE VERIFIED	AUDITOR SIGNATURE	DOCUMENT USED FOR CLOSE OUT	CLOSED DATE

Figure 12-2. Audit Finding Report Form

APPENDIX A

GLOSSARY

GLOSSARY

Analytical Duplicate: Two aliquots of a processed sample that are analyzed in the same manner. Used to monitor the precision of the analysis system for the processed matrix.

Analytical Spike: An aliquot of a processed sample to which a known amount of analyte is added prior to analysis. Used to determined whether the analysis system is in control when a matrix spike is outside its limits.

Batch: A group of samples that are prepared or analyzed together with the same method sequence and the same lots of reagents and with the manipulations common to each sample within the same time period or in continuous sequential time periods.

Calibration Blank: An organic or aqueous solution that contains all the reagents and solvents in the same proportions as those used to prepare the calibration standards.

Calibration Check Compounds (CCC): Target compounds used to evaluate the calibration stability of a GC/MS system.

Calibration Standard: A material of known concentration or composition used to establish the concentration-response relationship. It is usually not processed through the whole analytical scheme.

Calibration Verification Solution: A standard solution, prepared independently of the calibration standards, used to verify calibration. Also called reference standard, check standard, and quality control sample.

Continuing Calibration Verification (CCV): The periodic analysis of the calibration verification solution during the analytical run.

Data Validation: Data validation is a systematic process of reviewing data against a set of criteria to identify outliers or errors and to delete suspect values or to flag them for the user.

Digestion Blank: A specific type of method blank (q.v.); sometimes used instead of method blank when the sample preparation procedure involves a digestion.

Extraction Blank: A specific type of method blank (q.v.); sometimes used instead of method blank when the sample preparation procedure involves an extraction.

Field Sample: A representative sample of any material collected from any source and submitted to the laboratory for analysis. Also called an environmental sample.

GLOSSARY

Holding Blank: Laboratory pure water stored alongside a set of samples in the same kind of sample container; normally used only for samples destined for determination of volatile organics.

Initial Calibration Verification (ICV): The analysis of the calibration verification solution immediately after calibration.

Laboratory Control Sample: An aqueous or solid control sample of known composition, which is analyzed using the same sample preparation, reagents, and analytical methods employed for field samples. Used to demonstrate whether the sample preparation and analysis steps are in control, apart from sample matrix effects. Also called spiked method blank and laboratory fortified blank.

Matrix Spike: A subsample of a field sample to which is added a known amount of analyte(s) before sample preparation and analysis. Indicates the performance of the entire method in the given matrix by measuring recovery. Also called a method spike.

Matrix Spike Duplicate: A second aliquot of the same sample as that used for the matrix spike that is also spiked.

Method Blank: A volume of deionized laboratory water for water samples or a purified solid matrix for soil/sediment samples that is carried through the entire sample preparation and analysis scheme as if it were a field sample. Used to monitor interferences caused by contaminants in solvents, reagents, glassware, and other sample processing hardware. Also called reagent blank and preparation blank.

Method Duplicate: A pair of subsamples from a field sample taken through the entire preparation and analysis scheme. Indicates the performance of the entire method in the given matrix by measuring the precision.

Performance Evaluation Sample: A reference material, the analyte concentrations of which are unknown to the analysts, that is used as part of an external or internal performance audit. Also called single blind sample.

Processed Sample: A sample that has been carried through the sample preparation steps of a method.

System Performance Check Compounds (SPCC): Target compounds designated to monitor chromatographic performance, sensitivity, and compound instability or degradation.

Surrogate: An organic compound that is similar to the analytes of interest in chemical

GLOSSARY

composition, extraction, and chromatography, but not normally found in environmental samples. Used to monitor preparation and analysis of samples.

APPENDIX B

CURRENT REPORTING LIMITS (RLs)

EA LABORATORIES

REPORTING LIMITS FOR WATER SAMPLES [a]

Parameter	Units	Reporting Limit
Explosives - nitroaromatics (SW846 8330 revision 1/salting out, Dec 1990)		
HMX	ug/L	0.50
RDX	ug/L	0.50
135TNB	ug/L	0.25
13DNB	ug/L	0.25
NB	ug/L	0.25
TETRYL	ug/L	0.75
246TNT	ug/L	0.50
26DNT	ug/L	0.50
24DNT	ug/L	0.25
4amDNT	ug/L	0.50
2amDNT	ug/L	0.50
2NT	ug/L	0.50
4NT	ug/L	0.50
3NT	ug/L	0.50
PETN	ug/L	2.0
NG	ug/L	2.0
Herbicides GC/ECD - chlorinated compounds (SW846 8150)		
Dicamba	ug/L	2.7
Dalapon	ug/L	58
MCPP	ug/L	1900
MCPA	ug/L	2500
Dichloroprop	ug/L	6.5
2,4-D	ug/L	12
2,4,5-TP	ug/L	1.7
2,4,5-T	ug/L	2.0
2,4-DB	ug/L	9.1
Dinoseb	ug/L	2.0
Herbicides GC/ECD - chlorinated compounds (SW846 8150) - Appendix IX compounds		
2,4-D	ug/L	12
2,4,5-TP	ug/L	1.7
2,4,5-T	ug/L	2.0
Herbicides GC/ECD - chlorinated compounds (SW846 8150) - Appendix II compounds		
2,4-D	ug/L	12
2,4,5-TP	ug/L	1.7
2,4,5-T	ug/L	2.0
Pesticides and PCBs GC/ECD - organochlorine compounds (SW846 8080, EPA 608)		
Aldrin	ug/L	0.05
α-BHC	ug/L	0.05
β-BHC	ug/L	0.05
δ-BHC	ug/L	0.05
γ-BHC (Lindane)	ug/L	0.05
α-Chlordane	ug/L	0.05
γ-Chlordane	ug/L	0.05
Chlordane-Technical	ug/L	1.0

(a) EA Laboratories has established *Reporting Limits* (RLs) as laboratory quantitation levels. These are the minimum concentrations to be reported for routine laboratory analyses in clean environmental matrices. The RLs are values believed to provide greater than 50% probability of avoiding a false negative.

Updated: April 1, 1996/HJG
Supercedes: February 6, 1996/mmu

EA LABORATORIES

REPORTING LIMITS FOR WATER SAMPLES [a]

Parameter	Units	Reporting Limit
4,4'-DDD	ug/L	0.10
4,4'-DDE	ug/L	0.10
4,4'-DDT	ug/L	0.10
Dieldrin	ug/L	0.10
Endosulfan I	ug/L	0.05
Endosulfan II	ug/L	0.10
Endosulfan sulfate	ug/L	0.10
Endrin	ug/L	0.10
Endrin aldehyde	ug/L	0.10
Endrin ketone	ug/L	0.10
Heptachlor	ug/L	0.05
Heptachlor epoxide	ug/L	0.05
Methoxychlor	ug/L	0.5
Toxaphene	ug/L	5.0
Aroclor 1016	ug/L	1.0
Aroclor 1221	ug/L	2.0
Aroclor 1232	ug/L	1.0
Aroclor 1242	ug/L	1.0
Aroclor 1248	ug/L	1.0
Aroclor 1254	ug/L	1.0
Aroclor 1260	ug/L	1.0
Pesticides and PCBs GC/ECD - organochlorine compounds (SW846 8080) - Appendix IX compounds		
Aldrin	ug/L	0.05
α-BHC	ug/L	0.05
β-BHC	ug/L	0.05
δ-BHC	ug/L	0.05
γ-BHC (Lindane)	ug/L	0.05
Chlordane-Technical	ug/L	1.0
4,4'-DDD	ug/L	0.10
4,4'-DDE	ug/L	0.10
4,4'-DDT	ug/L	0.10
Dieldrin	ug/L	0.10
Endosulfan I	ug/L	0.05
Endosulfan II	ug/L	0.10
Endosulfan sulfate	ug/L	0.10
Endrin	ug/L	0.10
Endrin aldehyde	ug/L	0.10
Heptachlor	ug/L	0.05
Heptachlor epoxide	ug/L	0.05
Methoxychlor	ug/L	0.5
Toxaphene	ug/L	5.0
Aroclor 1016	ug/L	1.0
Aroclor 1221	ug/L	2.0
Aroclor 1232	ug/L	1.0
Aroclor 1242	ug/L	1.0
Aroclor 1248	ug/L	1.0
Aroclor 1254	ug/L	1.0
Aroclor 1260	ug/L	1.0

(a) EA Laboratories has established *Reporting Limits* (RLs) as laboratory quantitation levels. These are the minimum concentrations to be reported for routine laboratory analyses in clean environmental matrices. The RLs are values believed to provide greater than 50% probability of avoiding a false negative.

Updated: April 1, 1996/HJG
Supercedes: February 6, 1996/mmu

EA LABORATORIES

REPORTING LIMITS FOR WATER SAMPLES [a]

Parameter	Units	Reporting Limit
Pesticides and PCBs GC/ECD - organochlorine compounds (SW846 8080) - Appendix II compounds		
Aldrin	ug/L	0.05
α-BHC	ug/L	0.05
β-BHC	ug/L	0.05
δ-BHC	ug/L	0.05
γ-BHC (Lindane)	ug/L	0.05
Chlordane-Technical	ug/L	1.0
4,4'-DDD	ug/L	0.10
4,4'-DDE	ug/L	0.10
4,4'-DDT	ug/L	0.10
Dieldrin	ug/L	0.10
Endosulfan I	ug/L	0.05
Endosulfan II	ug/L	0.10
Endosulfan sulfate	ug/L	0.10
Endrin	ug/L	0.10
Endrin aldehyde	ug/L	0.10
Heptachlor	ug/L	0.05
Heptachlor epoxide	ug/L	0.05
Methoxychlor	ug/L	0.5
Toxaphene	ug/L	5.0
Aroclor 1016	ug/L	1.0
Aroclor 1221	ug/L	2.0
Aroclor 1232	ug/L	1.0
Aroclor 1242	ug/L	1.0
Aroclor 1248	ug/L	1.0
Aroclor 1254	ug/L	1.0
Aroclor 1260	ug/L	1.0
Pesticides GC/NPD - organophosphorus compounds (SW846 8140)		
Azinphos methyl	ug/L	1.0
Bolstar	ug/L	1.0
Chlorpyrifos (Dursban)	ug/L	1.0
Coumaphos	ug/L	1.0
Demeton (-O & -S)	ug/L	1.0
Diazinon	ug/L	1.0
Dichlorvos	ug/L	1.0
Disulfoton	ug/L	1.0
Ethoprop	ug/L	1.0
Fensulfothion	ug/L	1.0
Fenthion	ug/L	1.0
Merphos	ug/L	1.0
Mevinphos	ug/L	1.0
Naled	ug/L	1.0
Methyl Parathion	ug/L	1.0
Phorate	ug/L	1.0
Ronnel	ug/L	1.0
Stirphos	ug/L	1.0
Tokuthion	ug/L	1.0
Trichloronate	ug/L	1.0

(a) EA Laboratories has established *Reporting Limits* (RLs) as laboratory quantitation levels. These are the minimum concentrations to be reported for routine laboratory analyses in clean environmental matrices. The RLs are values believed to provide greater than 50% probability of avoiding a false negative.

Updated: April 1, 1996/HJG
Supercedes: February 6, 1996/mmu

EA LABORATORIES

REPORTING LIMITS FOR WATER SAMPLES [a]

Parameter	Units	Reporting Limit
Pesticides GC - thiocarbamate (EPA 634)		
EPTC	ug/L	1.0
Butylate	ug/L	1.0
Vernolate	ug/L	2.0
Pebulate	ug/L	1.0
Molinate	ug/L	1.0
Cycloate	ug/L	2.0
Semivolatile organics GC/ECD - phthalate esters (SW846 8060, EPA 606)		
Bis (2-ethylhexyl) phthalate	ug/L	5.0
Butyl benzyl phthalate	ug/L	0.5
Diethyl phthalate	ug/L	0.5
Dimethyl phthalate	ug/L	0.5
Di-n-butyl phthalate	ug/L	0.5
Di-n-octyl phthalate	ug/L	5.0
Semivolatile organics GC/ECD - chlorinated compounds (SW846 8120, EPA 612)		
2-Chloronaphthalene	ug/L	2.0
1,2-Dichlorobenzene	ug/L	2.0
1,3-Dichlorobenzene	ug/L	2.0
1,4-Dichlorobenzene	ug/L	2.0
Hexachlorobenzene	ug/L	2.0
Hexachlorobutadiene	ug/L	2.0
Hexachlorocyclopentadiene	ug/L	2.0
Hexachloroethane	ug/L	2.0
1,2,4-Trichlorobenzene	ug/L	2.0
Semivolatile organics GC/FID - PAHs (SW846 8100, EPA 610)		
Acenaphthene	ug/L	1.0
Acenaphthylene	ug/L	1.0
Anthracene	ug/L	1.0
Benzo[a]anthracene	ug/L	1.0
Benzo[b]fluoranthene	ug/L	1.0
Benzo[k]fluoranthene	ug/L	1.0
Benzo[a]pyrene	ug/L	1.0
Benzo[ghi]perylene	ug/L	1.0
Chrysene	ug/L	1.0
Dibenzo[a,h]anthracene	ug/L	1.0
Fluoranthene	ug/L	1.0
Fluorene	ug/L	1.0
Indeno[1,2,3-cd]pyrene	ug/L	1.0
Naphthalene	ug/L	1.0
Phenanthrene	ug/L	1.0
Pyrene	ug/L	1.0
Semivolatile organics GC/MS - (SW846 8270, EPA 625)		
Acenaphthene	ug/L	10
Acenaphthylene	ug/L	10
Aniline	ug/L	10

(a) EA Laboratories has established *Reporting Limits* (RLs) as laboratory quantitation levels. These are the minimum concentrations to be reported for routine laboratory analyses in clean environmental matrices. The RLs are values believed to provide greater than 50% probability of avoiding a false negative.

Updated: April 1, 1996/HJG
Supercedes: February 6, 1996/mmu

EA LABORATORIES

REPORTING LIMITS FOR WATER SAMPLES [a]

Parameter	Units	Reporting Limit
Anthracene	ug/L	10
Benzidine	ug/L	50
Benzo[a]anthracene	ug/L	10
Benzo[b]fluoranthene	ug/L	10
Benzo[k]fluoranthene	ug/L	10
Benzo[a]pyrene	ug/L	10
Benzo[ghi]perylene	ug/L	10
Benzoic acid	ug/L	50
Benzyl alcohol	ug/L	10
Bis(2-chloroethyl) ether	ug/L	10
Bis(2-chloroethoxy)methane	ug/L	10
Bis(2-ethylhexyl) phthalate	ug/L	10
Bis(2-chloroisopropyl) ether	ug/L	10
4-Bromophenyl phenyl ether	ug/L	10
Butylbenzylphthalate	ug/L	10
Carbazole	ug/L	10
4-Chloroaniline	ug/L	10
4-Chloro-3-methylphenol	ug/L	10
2-Chloronaphthalene	ug/L	10
2-Chlorophenol	ug/L	10
4-Chlorophenyl phenyl ether	ug/L	10
Chrysene	ug/L	10
Cyclohexanone	ug/L	10
Dibenz[a,h]anthracene	ug/L	10
Dibenzofuran	ug/L	10
Di-n-butyl phthalate	ug/L	10
1,2-Dichlorobenzene	ug/L	10
1,3-Dichlorobenzene	ug/L	10
1,4-Dichlorobenzene	ug/L	10
3,3'-Dichlorobenzidine	ug/L	10
2,4-Dichlorophenol	ug/L	10
Diethyl phthalate	ug/L	10
2,4-Dimethylphenol	ug/L	10
Dimethyl phthalate	ug/L	10
2,4-Dinitrophenol	ug/L	50
2,4-Dinitrotoluene	ug/L	10
2,6-Dinitrotoluene	ug/L	10
1,2-Diphenylhydrazine	ug/L	10
Di-n-octyl phthalate	ug/L	10
Fluoranthene	ug/L	10
Fluorene	ug/L	10
Hexachlorobenzene	ug/L	10
Hexachlorobutadiene	ug/L	10
Hexachlorocyclopentadiene	ug/L	10
Hexachloroethane	ug/L	10
Indeno[1,2,3-cd]pyrene	ug/L	10
Isophorone	ug/L	10
2-Methyl-4,6-dinitrophenol	ug/L	50
2-Methylnaphthalene	ug/L	10

(a) EA Laboratories has established *Reporting Limits* (RLs) as laboratory quantitation levels. These are the minimum concentrations to be reported for routine laboratory analyses in clean environmental matrices. The RLs are values believed to provide greater than 50% probability of avoiding a false negative.

Updated: April 1, 1996/HJG
Supercedes: February 6, 1996/mmu

REPORTING LIMITS FOR WATER SAMPLES [a]

Parameter	Units	Reporting Limit
2-Methylphenol	ug/L	10
4-Methylphenol	ug/L	10
Naphthalene	ug/L	10
2-Nitroaniline	ug/L	50
3-Nitroaniline	ug/L	50
4-Nitroaniline	ug/L	50
Nitrobenzene	ug/L	10
2-Nitrophenol	ug/L	10
4-Nitrophenol	ug/L	50
N-Nitrosodiphenylamine	ug/L	10
N-Nitrosodimethylamine	ug/L	10
N-Nitroso-di-n-propylamine	ug/L	10
Pentachlorophenol	ug/L	50
Phenanthrene	ug/L	10
Phenol	ug/L	10
Pyrdine	ug/L	10
Pyrene	ug/L	10
1,2,4-Trichlorobenzene	ug/L	10
2,4,5-Trichlorophenol	ug/L	50
2,4,6-Trichlorophenol	ug/L	10
Semivolatile organics GC/MS - (SW846 8270) -Appendix IX compounds		
Acenaphthene	ug/L	10
Acenaphthylene	ug/L	10
Acetophenone	ug/L	10
2-Acetylaminofluorene	ug/L	20
4-Aminobiphenyl	ug/L	20
Aniline	ug/L	10
Anthracene	ug/L	10
Aramite	ug/L	20
Benzo[a]anthracene	ug/L	10
Benzo[b]fluoranthene	ug/L	10
Benzo[k]fluoranthene	ug/L	10
Benzo[a]pyrene	ug/L	10
Benzo[ghi]perylene	ug/L	10
Benzyl alcohol	ug/L	10
Bis(2-chloroethyl) ether	ug/L	10
Bis(2-chloroethoxy)methane	ug/L	10
Bis(2-chloro-1-methylethyl)ether	ug/L	10
Bis(2-ethylhexyl) phthalate	ug/L	10
4-Bromophenyl phenyl ether	ug/L	10
Butylbenzylphthalate	ug/L	10
4-Chloroaniline	ug/L	10
Chlorobenzilate	ug/L	10
4-Chloro-3-methylphenol	ug/L	10
2-Chloronaphthalene	ug/L	10
2-Chlorophenol	ug/L	10
4-Chlorophenyl phenyl ether	ug/L	10
Chrysene	ug/L	10

(a) EA Laboratories has established *Reporting Limits* (RLs) as laboratory quantitation levels. These are the minimum concentrations to be reported for routine laboratory analyses in clean environmental matrices. The RLs are values believed to provide greater than 50% probability of avoiding a false negative.

Updated: April 1, 1996/HJG
Supercedes: February 6, 1996/mmu

EA LABORATORIES

REPORTING LIMITS FOR WATER SAMPLES [a]

Parameter	Units	Reporting Limit
m-Cresol	ug/L	10
o-Cresol	ug/L	10
p-Cresol	ug/L	10
Diallate	ug/L	10
Dibenz[a,h]anthracene	ug/L	10
Dibenzofuran	ug/L	10
Di-n-butyl phthalate	ug/L	10
1,2-Dichlorobenzene	ug/L	10
1,3-Dichlorobenzene	ug/L	10
1,4-Dichlorobenzene	ug/L	10
3,3'-Dichlorobenzidine	ug/L	10
2,4-Dichlorophenol	ug/L	10
2,6-Dichlorophenol	ug/L	10
Diethyl phthalate	ug/L	10
Dimethoate	ug/L	20
p-(Dimethylamino)azobenzene	ug/L	10
7,12-Dimethylbenz(a)anthracene	ug/L	10
3,3'-Dimethylbenzidine	ug/L	10
a,a-Dimethylphenethylamine	ug/L	10
2,4-Dimethylphenol	ug/L	10
Dimethyl phthalate	ug/L	10
m-Dinitrobenzene	ug/L	20
4,6-Dinitro-o-cresol	ug/L	50
2,4-Dinitrophenol	ug/L	50
2,4-Dinitrotoluene	ug/L	10
2,6-Dinitrotoluene	ug/L	10
Di-n-octyl phthalate	ug/L	10
Dinoseb	ug/L	20
1,4-Dioxane	ug/L	20
Diphenylamine	ug/L	10
Disulfoton	ug/L	10
Ethyl methanesulfonate	ug/L	20
Famphur	ug/L	20
Fluoranthene	ug/L	10
Fluorene	ug/L	10
Hexachlorobenzene	ug/L	10
Hexachlorobutadiene	ug/L	10
Hexachlorocyclopentadiene	ug/L	10
Hexachloroethane	ug/L	10
Hexachlorophene	ug/L	50
Hexachloropropene	ug/L	10
Indeno[1,2,3-cd]pyrene	ug/L	10
Isodrin	ug/L	20
Isophorone	ug/L	10
Isosafrole	ug/L	10
Kepone	ug/L	20
Methapyrilene	ug/L	100
3-Methylcholanthrene	ug/L	10
Methyl methanesulfonate	ug/L	10

(a) EA Laboratories has established *Reporting Limits* (RLs) as laboratory quantitation levels. These are the minimum concentrations to be reported for routine laboratory analyses in clean environmental matrices. The RLs are values believed to provide greater than 50% probability of avoiding a false negative.

Updated: April 1, 1996/HJG
Supercedes: February 6, 1996/mmu

REPORTING LIMITS FOR WATER SAMPLES [a]

Parameter	Units	Reporting Limit
2-Methylnaphthalene	ug/L	10
Methyl parathion	ug/L	10
Naphthalene	ug/L	10
1,4-Naphthoquinone	ug/L	10
1-Naphthylamine	ug/L	10
2-Naphthylamine	ug/L	10
2-Nitroaniline	ug/L	50
3-Nitroaniline	ug/L	50
4-Nitroaniline	ug/L	50
Nitrobenzene	ug/L	10
2-Nitrophenol	ug/L	10
4-Nitrophenol	ug/L	50
4-Nitroquinoline 1-oxide	ug/L	40
N-Nitrosodi-n-butylamine	ug/L	10
N-Nitrosodiethylamine	ug/L	20
N-Nitrosodiphenylamine	ug/L	10
N-Nitrosodimethylamine	ug/L	10
N-Nitroso-di-n-propylamine	ug/L	10
N-Nitrosomethylethylamine	ug/L	10
N-Nitrososmorpholine	ug/L	10
N-Nitrosopiperidine	ug/L	20
N-Nitrosopyrrolidine	ug/L	40
N-Nitro-o-toluidine	ug/L	10
Parathion	ug/L	10
Pentachlorobenzene	ug/L	10
Pentachloronitrobenzene	ug/L	20
Pentachlorophenol	ug/L	50
Phenacetin	ug/L	20
Phenanthrene	ug/L	10
Phenol	ug/L	10
p-Phenylenediamine	ug/L	10
Phorate	ug/L	10
2-Picoline	ug/L	10
Pronamide	ug/L	10
Pyrdine	ug/L	10
Pyrene	ug/L	10
Safrole	ug/L	10
Sulfotepp	ug/L	40
1,2,4,5-Tetrachlorobenzene	ug/L	10
2,3,4,6-Tetrachlorophenol	ug/L	10
Thionazin	ug/L	20
o-Toluidine	ug/L	10
1,2,4-Trichlorobenzene	ug/L	10
2,4,5-Trichlorophenol	ug/L	50
2,4,6-Trichlorophenol	ug/L	10
o,o,o-Triethylphosphorothioate	ug/L	10
1,3,5-Trinitrobenzene	ug/L	10

Semivolatile organics GC/MS - (SW846 8270) -Appendix II compounds

(a) EA Laboratories has established *Reporting Limits* (RLs) as laboratory quantitation levels. These are the minimum concentrations to be reported for routine laboratory analyses in clean environmental matrices. The RLs are values believed to provide greater than 50% probability of avoiding a false negative.

Updated: April 1, 1996/HJG
Supercedes: February 6, 1996/mmu

EA LABORATORIES

REPORTING LIMITS FOR WATER SAMPLES [a]

Parameter	Units	Reporting Limit
Acenaphthene	ug/L	10
Acenaphthylene	ug/L	10
Acetophenone	ug/L	10
2-Acetylaminofluorene	ug/L	20
4-Aminobiphenyl	ug/L	20
Anthracene	ug/L	10
Benzo[a]anthracene	ug/L	10
Benzo[b]fluoranthene	ug/L	10
Benzo[k]fluoranthene	ug/L	10
Benzo[a]pyrene	ug/L	10
Benzo[ghi]perylene	ug/L	10
Benzyl alcohol	ug/L	10
Bis(2-chloroethyl) ether	ug/L	10
Bis(2-chloroethoxy)methane	ug/L	10
Bis(2-chloro-1-methylethyl)ether	ug/L	10
Bis(2-ethylhexyl) phthalate	ug/L	10
4-Bromophenyl phenyl ether	ug/L	10
Butylbenzylphthalate	ug/L	10
4-Chloroaniline	ug/L	10
Chlorobenzilate	ug/L	10
4-Chloro-3-methylphenol	ug/L	10
2-Chloronaphthalene	ug/L	10
2-Chlorophenol	ug/L	10
4-Chlorophenyl phenyl ether	ug/L	10
Chrysene	ug/L	10
m-Cresol	ug/L	10
o-Cresol	ug/L	10
p-Cresol	ug/L	10
Diallate	ug/L	10
Dibenz[a,h]anthracene	ug/L	10
Dibenzofuran	ug/L	10
Di-n-butyl phthalate	ug/L	10
1,2-Dichlorobenzene	ug/L	10
1,3-Dichlorobenzene	ug/L	10
1,4-Dichlorobenzene	ug/L	10
3,3'-Dichlorobenzidine	ug/L	10
2,4-Dichlorophenol	ug/L	10
2,6-Dichlorophenol	ug/L	10
Diethyl phthalate	ug/L	10
Dimethoate	ug/L	20
p-(Dimethylamino)azobenzene	ug/L	10
7,12-Dimethylbenz(a)anthracene	ug/L	10
3,3'-Dimethylbenzidine	ug/L	10
2,4-Dimethylphenol	ug/L	10
Dimethyl phthalate	ug/L	10
m-Dinitrobenzene	ug/L	20
4,6-Dinitro-o-cresol	ug/L	50
2,4-Dinitrophenol	ug/L	50
2,4-Dinitrotoluene	ug/L	10

(a) EA Laboratories has established *Reporting Limits* (RLs) as laboratory quantitation levels. These are the minimum concentrations to be reported for routine laboratory analyses in clean environmental matrices. The RLs are values believed to provide greater than 50% probability of avoiding a false negative.

Updated: April 1, 1996/HJG
Supercedes: February 6, 1996/mmu

EA LABORATORIES

REPORTING LIMITS FOR WATER SAMPLES [a]

Parameter	Units	Reporting Limit
2,6-Dinitrotoluene	ug/L	10
Di-n-octyl phthalate	ug/L	10
Dinoseb	ug/L	20
Diphenylamine	ug/L	10
Disulfoton	ug/L	10
Ethyl methanesulfonate	ug/L	20
Famphur	ug/L	20
Fluoranthene	ug/L	10
Fluorene	ug/L	10
Hexachlorobenzene	ug/L	10
Hexachlorobutadiene	ug/L	10
Hexachlorocyclopentadiene	ug/L	10
Hexachloroethane	ug/L	10
Hexachloropropene	ug/L	10
Indeno[1,2,3-cd]pyrene	ug/L	10
Isodrin	ug/L	20
Isophorone	ug/L	10
Isosafrole	ug/L	10
Kepone	ug/L	20
Methapyrilene	ug/L	100
3-Methylcholanthrene	ug/L	10
Methyl methanesulfonate	ug/L	10
2-Methylnaphthalene	ug/L	10
Methyl parathion	ug/L	10
Naphthalene	ug/L	10
1,4-Naphthoquinone	ug/L	10
1-Naphthylamine	ug/L	10
2-Naphthylamine	ug/L	10
2-Nitroaniline	ug/L	50
3-Nitroaniline	ug/L	50
4-Nitroaniline	ug/L	50
Nitrobenzene	ug/L	10
2-Nitrophenol	ug/L	10
4-Nitrophenol	ug/L	50
N-Nitrosodi-n-butylamine	ug/L	10
N-Nitrosodiethylamine	ug/L	20
N-Nitrosodiphenylamine	ug/L	10
N-Nitrosodimethylamine	ug/L	10
N-Nitroso-di-n-propylamine	ug/L	10
N-Nitrosomethylethylamine	ug/L	10
N-Nitrosopiperidine	ug/L	20
N-Nitrosopyrrolidine	ug/L	40
N-Nitro-o-toluidine	ug/L	10
Parathion	ug/L	10
Pentachlorobenzene	ug/L	10
Pentachloronitrobenzene	ug/L	20
Pentachlorophenol	ug/L	50
Phenacetin	ug/L	20
Phenanthrene	ug/L	10

(a) EA Laboratories has established *Reporting Limits* (RLs) as laboratory quantitation levels. These are the minimum concentrations to be reported for routine laboratory analyses in clean environmental matrices. The RLs are values believed to provide greater than 50% probability of avoiding a false negative.

Updated: April 1, 1996/HJG
Supercedes: February 6, 1996/mmu

REPORTING LIMITS FOR WATER SAMPLES [a]

Parameter	Units	Reporting Limit
Phenol	ug/L	10
p-Phenylenediamine	ug/L	10
Phorate	ug/L	10
Pronamide	ug/L	10
Pyrene	ug/L	10
Safrole	ug/L	10
1,2,4,5-Tetrachlorobenzene	ug/L	10
2,3,4,6-Tetrachlorophenol	ug/L	10
Thionazin	ug/L	20
o-Toluidine	ug/L	10
1,2,4-Trichlorobenzene	ug/L	10
2,4,5-Trichlorophenol	ug/L	50
2,4,6-Trichlorophenol	ug/L	10
o,o,o-Triethylphosphorothioate	ug/L	10
1,3,5-Trinitrobenzene	ug/L	10
Semivolatile organics HPLC - PAHs (SW846 8310, EPA 610)		
Acenaphthene	ug/L	1.0
Acenaphthylene	ug/L	2.0
Anthracene	ug/L	0.20
Benzo[a]anthracene	ug/L	0.10
Benzo[b]fluoranthene	ug/L	0.15
Benzo[k]fluoranthene	ug/L	0.10
Benzo[a]pyrene	ug/L	0.10
Benzo[ghi]perylene	ug/L	0.20
Chrysene	ug/L	0.10
Dibenzo[a,h]anthracene	ug/L	0.20
Fluoranthene	ug/L	0.20
Fluorene	ug/L	0.20
Indeno[1,2,3-cd]pyrene	ug/L	0.10
Naphthalene	ug/L	1.0
Phenanthrene	ug/L	0.20
Pyrene	ug/L	0.20
Total petroleum hydrocarbons GC (EAL-M-8015-GRO)		
TPH as gasoline (GC)	ug/L	100
Total petroleum hydrocarbon GC/FID (EALL-M-8015-DRO)		
TPH as JP4 (extractable)	ug/L	2000
TPH as diesel (GC)	ug/L	500
TPH as JP8 (extractable)	ug/L	2000
Total petroleum hydrocarbons GC (Mississippi)		
TPH as diesel (GC)	ug/L	160
Volatile organics GC/ELCD - halogenated compounds (SW 846 5030/8010, EPA 601)		
Bromodichloromethane	ug/L	1
Bromoform	ug/L	1
Bromomethane	ug/L	1

(a) EA Laboratories has established *Reporting Limits* (RLs) as laboratory quantitation levels. These are the minimum concentrations to be reported for routine laboratory analyses in clean environmental matrices. The RLs are values believed to provide greater than 50% probability of avoiding a false negative.

Updated: April 1, 1996/HJG
Supercedes: February 6, 1996/mmu

REPORTING LIMITS FOR WATER SAMPLES [a]

Parameter	Units	Reporting Limit
Carbon tetrachloride	ug/L	1
Chlorobenzene	ug/L	1
Chloroethane	ug/L	1
2-Chloroethylvinyl ether	ug/L	1
Chloroform	ug/L	1
Chloromethane	ug/L	1
Dibromochloromethane	ug/L	1
1,2-Dichlorobenzene	ug/L	1
1,3-Dichlorobenzene	ug/L	1
1,4-Dichlorobenzene	ug/L	1
1,1-Dichloroethane	ug/L	1
1,2-Dichloroethane	ug/L	1
1,1-Dichloroethene	ug/L	1
cis-1,2-Dichloroethene	ug/L	1
trans-1,2-Dichloroethene	ug/L	1
Dichlorofluoromethane	ug/L	1
Dichloromethane	ug/L	1
1,2-Dichloropropane	ug/L	1
cis-1,3-Dichloropropene	ug/L	1
trans-1,3-Dichloropropene	ug/L	1
1,1,2,2-Tetrachloroethane	ug/L	1
Tetrachloroethene	ug/L	1
1,1,1-Trichloroethane	ug/L	1
1,1,2-Trichloroethane	ug/L	1
Trichloroethene	ug/L	1
Trichlorofluoromethane	ug/L	1
Vinyl chloride	ug/L	1
Volatile organics GC/FID - acrolein, acrylonitrile (SW846 8030, EPA 603)		
Acrolein	ug/L	5
Acrylonitrile	ug/L	5
Volatile organics GC/FID - non-halogenated compounds (SW846 8015)		
Diethyl ether	ug/L	5
Methyl ethyl ketone (MEK)	ug/L	5
Methyl isobutyl ketone (MIBK)	ug/L	5
Volatile organics GC/PID - aromatic compounds (SW846 5030/8020, EPA 602)		
Benzene	ug/L	1
Chlorobenzene	ug/L	1
1,2-Dichlorobenzene	ug/L	1
1,3-Dichlorobenzene	ug/L	1
1,4-Dichlorobenzene	ug/L	1
Ethylbenzene	ug/L	1
Toluene	ug/L	1
m&p-Xylene	ug/L	1
o-Xylene	ug/L	1
Volatile organics GC/MS - 5 mL purge (SW846 5030/8240, EPA 624)		

(a) EA Laboratories has established *Reporting Limits* (RLs) as laboratory quantitation levels. These are the minimum concentrations to be reported for routine laboratory analyses in clean environmental matrices. The RLs are values believed to provide greater than 50% probability of avoiding a false negative.

Updated: April 1, 1996/HJG
Supercedes: February 6, 1996/mmu

EA LABORATORIES

REPORTING LIMITS FOR WATER SAMPLES [a]

Parameter	Units	Reporting Limit
Acetone	ug/L	10
Acrolein	ug/L	50
Acrylonitrile	ug/L	50
Benzene	ug/L	5
Bromodichloromethane	ug/L	5
Bromoform	ug/L	5
Bromomethane	ug/L	10
2-Butanone	ug/L	10
n-Butylbenzene	ug/L	5
sec-Butylbenzene	ug/L	5
tert-Butylbenzene	ug/L	5
tert-Butyl methyl ether (MTBE)	ug/L	5
Carbon disulfide	ug/L	5
Carbon tetrachloride	ug/L	5
Chlorobenzene	ug/L	5
Chloroethane	ug/L	10
2-Chloroethyl vinyl ether	ug/L	10
Chloroform	ug/L	5
Chloromethane	ug/L	10
2-Chlorotoluene	ug/L	5
4-Chlorotoluene	ug/L	5
3-Chloro-1-propene	ug/L	5
Dibromochloromethane	ug/L	5
1,2-Dibromo-3-chloropropane	ug/L	10
1,2-Dibromoethane	ug/L	5
Dibromomethane	ug/L	5
1,2-Dichlorobenzene	ug/L	5
1,3-Dichlorobenzene	ug/L	5
1,4-Dichlorobenzene	ug/L	5
Dichlorodifluoromethane	ug/L	10
1,1-Dichloroethane	ug/L	5
1,2-Dichloroethane	ug/L	5
1,1-Dichloroethene	ug/L	5
1,2-Dichloroethene (total)	ug/L	5
1,2-Dichloropropane	ug/L	5
2,2-Dichloropropane	ug/L	5
1,1-Dichloropropene	ug/L	5
cis-1,3-Dichloropropene	ug/L	5
trans-1,3-Dichloropropene	ug/L	5
Diisopropyl ether	ug/L	5
Ethylbenzene	ug/L	5
Hexachlorobutadiene	ug/L	5
2-Hexanone	ug/L	10
Isopropylbenzene	ug/L	5
4-Isopropyltoluene	ug/L	5
4-Methyl-2-pentanone (MIBK)	ug/L	10
Methylene chloride	ug/L	5
Naphthalene	ug/L	5
n-Propylbenzene	ug/L	5

(a) EA Laboratories has established *Reporting Limits* (RLs) as laboratory quantitation levels. These are the minimum concentrations to be reported for routine laboratory analyses in clean environmental matrices. The RLs are values believed to provide greater than 50% probability of avoiding a false negative.

Updated: April 1, 1996/HJG
Supercedes: February 6, 1996/mmu

EA LABORATORIES

REPORTING LIMITS FOR WATER SAMPLES [a]

Parameter	Units	Reporting Limit
Styrene	ug/L	5
1,1,1,2-Tetrachloroethane	ug/L	5
1,1,2,2-Tetrachloroethane	ug/L	5
Tetrachloroethene	ug/L	5
Toluene	ug/L	5
1,2,3-Trichlorobenzene	ug/L	5
1,2,4-Trichlorobenzene	ug/L	5
1,1,1-Trichloroethane	ug/L	5
1,1,2-Trichloroethane	ug/L	5
Trichloroethene	ug/L	5
Trichlorofluoromethane	ug/L	5
1,2,3-Trichloropropane	ug/L	5
1,2,4-Trimethylbenzene	ug/L	5
1,3,5-Trimethylbenzene	ug/L	5
Vinyl acetate	ug/L	10
Vinyl chloride	ug/L	10
m-Xylene	ug/L	5
m&p-Xylene	ug/L	5
o&p-Xylene	ug/L	5
o-Xylene	ug/L	5
Volatile organics GC/ECD and PID in series (SW846 5030/8021)		
Benzene	ug/L	1
Bromobenzene	ug/L	1
Bromochloromethane	ug/L	1
Bromodichloromethane	ug/L	1
Bromoform	ug/L	1
Bromomethane	ug/L	1
n-Butylbenzene	ug/L	1
sec-Butylbenzene	ug/L	1
tert-Butylbenzene	ug/L	1
Carbon tetrachloride	ug/L	1
Chlorobenzene	ug/L	1
Chlorodibromomethane	ug/L	1
Chloroethane	ug/L	1
Chloroform	ug/L	1
Chloromethane	ug/L	1
2-Chlorotoluene	ug/L	1
4-Chlorotoluene	ug/L	1
1,2-Dibromo-3-chloropropane	ug/L	1
1,2-Dibromoethane	ug/L	1
Dibromomethane	ug/L	1
1,2-Dichlorobenzene	ug/L	1
1,3-Dichlorobenzsene	ug/L	1
1,4-Dichlorobenzene	ug/L	1
Dichlorodifluormethane	ug/L	1
1,1-Dichloroethane	ug/L	1
1,2-Dichloroethane	ug/L	1
1,1-Dichloroethene	ug/L	1

(a) EA Laboratories has established *Reporting Limits* (RLs) as laboratory quantitation levels. These are the minimum concentrations to be reported for routine laboratory analyses in clean environmental matrices. The RLs are values believed to provide greater than 50% probability of avoiding a false negative.

Updated: April 1, 1996/HJG
Supercedes: February 6, 1996/mmu

REPORTING LIMITS FOR WATER SAMPLES [a]

Parameter	Units	Reporting Limit
cis-1,2-Dichloroethene	ug/L	1
trans-1,2-Dichloroethene	ug/L	1
1,2-Dichloropropane	ug/L	1
1,3-Dichloropropane	ug/L	1
2,2-Dichloropropane	ug/L	1
1,1-Dichloropropene	ug/L	1
cis-1,3-Dichloropropene	ug/L	1
trans-1,3-Dichloropropene	ug/L	1
Ethylbenzene	ug/L	1
Hexachlorobutadiene	ug/L	1
Isopropylbenzene	ug/L	1
p-Isopropyltoluene	ug/L	1
Methylene Chloride	ug/L	1
Napthalene	ug/L	1
n-Propylbenzene	ug/L	1
Styrene	ug/L	1
1,1,1,2-Tetrachloroethane	ug/L	1
1,1,2,2-Tetrachloroethane	ug/L	1
Tetrachloroethene	ug/L	1
Toluene	ug/L	1
1,2,3-Trichlorobenzene	ug/L	1
1,2,4-Trichlorobenzene	ug/L	1
1,1,1-Trichloroethane	ug/L	1
1,1,2-Trichloroethane	ug/L	1
Trichloroethene	ug/L	1
Trichlorofluoromethane	ug/L	1
1,2,3-Trichloropropane	ug/L	1
1,2,4-Trimethylbenzene	ug/L	1
1,3,5-Trimethylbenzene	ug/L	1
Vinyl chloride	ug/L	1
o-Xylene	ug/L	1
m-Xylene	ug/L	1
p-Xylene	ug/L	1
Volatile organics GC/MS - 5 mL purge (SW846 5030/8260) - including Appendix IX compounds		
Acetone	ug/L	10
Acetonitrile	ug/L	100
Acrolein	ug/L	50
Acrylonitrile	ug/L	50
Allyl chloride	ug/L	5
Benzene	ug/L	5
Bromodichloromethane	ug/L	5
Bromobenzene	ug/L	5
Bromochloromethane	ug/L	5
Bromoform	ug/L	5
Bromomethane	ug/L	5
n-Butylbenzene	ug/L	5
sec-Butylbenzene	ug/L	5
tert-Butylbenzene	ug/L	5

(a) EA Laboratories has established *Reporting Limits* (RLs) as laboratory quantitation levels. These are the minimum concentrations to be reported for routine laboratory analyses in clean environmental matrices. The RLs are values believed to provide greater than 50% probability of avoiding a false negative.

Updated: April 1, 1996/HJG
Supercedes: February 6, 1996/mmu

EA LABORATORIES

REPORTING LIMITS FOR WATER SAMPLES [a]

Parameter	Units	Reporting Limit
2-Butanone	ug/L	10
Carbon disulfide	ug/L	5
Carbon tetrachloride	ug/L	5
Chlorobenzene	ug/L	5
Chloroethane	ug/L	5
2-Chloroethylvinyl ether	ug/L	10
Chloroform	ug/L	5
Chloromethane	ug/L	5
Chloroprene	ug/L	10
2-Chlorotoluene	ug/L	5
4-Chlorotoluene	ug/L	5
Dibromochloromethane	ug/L	5
1,2-Dibromo-3-chloropropane	ug/L	5
1,2-Dibromoethane	ug/L	5
Dibromomethane	ug/L	5
1,2-Dichlorobenzene	ug/L	5
1,3-Dichlorobenzene	ug/L	5
1,4-Dichlorobenzene	ug/L	5
trans-1,4-dichloro-2-butene	ug/L	100
Dichlorodifluoromethane	ug/L	5
1,1-Dichloroethane	ug/L	5
1,2-Dichloroethane	ug/L	5
1,1-Dichloroethene	ug/L	5
cis-1,2-Dichloroethene	ug/L	5
trans-1,2-Dichloroethene	ug/L	5
1,2-Dichloropropane	ug/L	5
1,3-Dichloropropane	ug/L	5
2,2-Dichloropropane	ug/L	5
1,1-Dichloropropene	ug/L	5
cis-1,3-Dichloropropene	ug/L	5
trans-1,3-Dichloropropene	ug/L	5
Diisopropyl ether	ug/L	5
Ethylbenzene	ug/L	5
Ethyl methacrylate	ug/L	5
Hexachlorobutadiene	ug/L	5
2-Hexanone	ug/L	10
Isobutyl alcohol	ug/L	100
Isopropylbenzene	ug/L	5
4-Isopropyltoluene	ug/L	5
Methacrylonitrile	ug/L	100
Methyl bromide	ug/L	5
Methyl chloride	ug/L	5
Methylene bromide	ug/L	5
Methylene chloride	ug/L	5
Methyl iodide	ug/L	5
Methyl methylacrylate	ug/L	5
Methyl tert-butyl ether (MTBE)	ug/L	5
4-Methyl-2-pentanone	ug/L	10
Naphthalene	ug/L	5

(a) EA Laboratories has established *Reporting Limits* (RLs) as laboratory quantitation levels. These are the minimum concentrations to be reported for routine laboratory analyses in clean environmental matrices. The RLs are values believed to provide greater than 50% probability of avoiding a false negative.

Updated: April 1, 1996/HJG
Supercedes: February 6, 1996/mmu

REPORTING LIMITS FOR WATER SAMPLES [a]

Parameter	Units	Reporting Limit
Pentachloroethane	ug/L	10
Propionitrile	ug/L	100
n-Propylbenzene	ug/L	5
Styrene	ug/L	5
1,1,1,2-Tetrachloroethane	ug/L	5
1,1,2,2-Tetrachloroethane	ug/L	5
Tetrachloroethene	ug/L	5
Toluene	ug/L	5
1,2,3-Trichlorobenzene	ug/L	5
1,2,4-Trichlorobenzene	ug/L	5
1,1,1-Trichloroethane	ug/L	5
1,1,2-Trichloroethane	ug/L	5
Trichloroethene	ug/L	5
Trichlorofluoromethane	ug/L	5
1,2,3-Trichloropropane	ug/L	5
1,2,4-Trimethylbenzene	ug/L	5
1,3,5-Trimethylbenzene	ug/L	5
Vinyl acetate	ug/L	10
Vinyl chloride	ug/L	5
m-Xylene	ug/L	5
m&p-Xylene	ug/L	5
o&p-Xylene	ug/L	5
o-Xylene	ug/L	5
Volatile organics GC/MS - 5 mL purge (SW846 5030/8260) - Appendix II compounds		
Acetone	ug/L	10
Acetonitrile	ug/L	100
Acrolein	ug/L	50
Acrylonitrile	ug/L	50
Allyl chloride	ug/L	5
Benzene	ug/L	5
Bromochloromethane	ug/L	5
Bromodichloromethane	ug/L	5
Bromoform	ug/L	5
2-Butanone	ug/L	10
Carbon disulfide	ug/L	5
Carbon tetrachloride	ug/L	5
Chlorobenzene	ug/L	5
Chloroethane	ug/L	5
Chloroform	ug/L	5
Chloroprene	ug/L	10
Dibromochloromethane	ug/L	5
1,2-Dibromo-3-chloropropane	ug/L	5
1,2-Dibromoethane	ug/L	5
trans-1,4-dichloro-2-butene	ug/L	100
Dichlorodifluoromethane	ug/L	5
1,1-Dichloroethane	ug/L	5
1,2-Dichloroethane	ug/L	5
1,1-Dichloroethene	ug/L	5

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Updated: April 1, 1996/HJG
Supercedes: February 6, 1996/mmu

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REPORTING LIMITS FOR WATER SAMPLES [a]

Parameter	Units	Reporting Limit
cis-1,2-Dichloroethene	ug/L	5
trans-1,2-Dichloroethene	ug/L	5
1,2-Dichloropropane	ug/L	5
1,3-Dichloropropane	ug/L	5
2,2-Dichloropropane	ug/L	5
1,1-Dichloropropene	ug/L	5
cis-1,3-Dichloropropene	ug/L	5
trans-1,3-Dichloropropene	ug/L	5
Ethylbenzene	ug/L	5
Ethyl methacrylate	ug/L	5
2-Hexanone	ug/L	10
Isobutyl alcohol	ug/L	100
Methacrylonitrile	ug/L	100
Methyl bromide	ug/L	5
Methyl chloride	ug/L	5
Methylene bromide	ug/L	5
Methylene chloride	ug/L	5
Methyl iodide	ug/L	5
Methyl methylacrylate	ug/L	5
4-Methyl-2-pentanone	ug/L	10
Propionitrile	ug/L	100
Styrene	ug/L	5
1,1,1,2-Tetrachloroethane	ug/L	5
1,1,2,2-Tetrachloroethane	ug/L	5
Tetrachloroethene	ug/L	5
Toluene	ug/L	5
1,1,1-Trichloroethane	ug/L	5
1,1,2-Trichloroethane	ug/L	5
Trichloroethene	ug/L	5
Trichlorofluoromethane	ug/L	5
1,2,3-Trichloropropane	ug/L	5
Vinyl acetate	ug/L	10
Vinyl chloride	ug/L	5
Xylenes (total)	ug/L	5
Volatile organics GC/MS - 25 mL purge (EPA 524.2, SW846 5030/8260, CLP OLC01, SW846 5030/8240)		
Acetone	ug/L	5
Benzene	ug/L	1
Bromodichloromethane	ug/L	1
Bromobenzene	ug/L	1
Bromochloromethane	ug/L	1
Bromoform	ug/L	1
Bromomethane	ug/L	1
n-Butylbenzene	ug/L	1
sec-Butylbenzene	ug/L	1
tert-Butylbenzene	ug/L	1
2-Butanone	ug/L	5
Carbon disulfide	ug/L	1
Carbon tetrachloride	ug/L	1

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REPORTING LIMITS FOR WATER SAMPLES [a]

Parameter	Units	Reporting Limit
Chlorobenzene	ug/L	1
Chloroethane	ug/L	1
2-Chloroethylvinyl ether	ug/L	10
Chloroform	ug/L	1
Chloromethane	ug/L	1
2-Chlorotoluene	ug/L	1
4-Chlorotoluene	ug/L	1
Dibromochloromethane	ug/L	1
1,2-Dibromo-3-chloropropane	ug/L	1
1,2-Dibromoethane	ug/L	1
Dibromomethane	ug/L	1
1,2-Dichlorobenzene	ug/L	1
1,3-Dichlorobenzene	ug/L	1
1,4-Dichlorobenzene	ug/L	1
1 Dichlorodifluoromethane	ug/L	1
1,1-Dichloroethane	ug/L	1
1,2-Dichloroethane	ug/L	1
1,1-Dichloroethene	ug/L	1
cis-1,2-Dichloroethene	ug/L	1
trans-1,2-Dichloroethene	ug/L	1
1,2-Dichloropropane	ug/L	1
1,3-Dichloropropane	ug/L	1
2,2-Dichloropropane	ug/L	1
1,1-Dichloropropene	ug/L	1
cis-1,3-Dichloropropene	ug/L	1
trans-1,3-Dichloropropene	ug/L	1
Diisopropyl ether	ug/L	1
Ethylbenzene	ug/L	1
Hexachlorobutadiene	ug/L	1
2-Hexanone	ug/L	5
Isopropylbenzene	ug/L	1
4-Isopropyltoluene	ug/L	1
Methylene chloride	ug/L	1
Methyl tert-butyl ether (MTBE)	ug/L	1
4-Methyl-2-pentanone	ug/L	5
Naphthalene	ug/L	1
n-Propylbenzene	ug/L	1
Styrene	ug/L	1
1,1,1,2-Tetrachloroethane	ug/L	1
1,1,2,2-Tetrachloroethane	ug/L	1
Tetrachloroethene	ug/L	1
Toluene	ug/L	1
1,2,3-Trichlorobenzene	ug/L	1
1,2,4-Trichlorobenzene	ug/L	1
1,1,1-Trichloroethane	ug/L	1
1,1,2-Trichloroethane	ug/L	1
Trichloroethene	ug/L	1
Trichlorofluoromethane	ug/L	1
1,2,3-Trichloropropane	ug/L	1

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REPORTING LIMITS FOR WATER SAMPLES [a]

Parameter	Units	Reporting Limit
1,2,4-Trimethylbenzene	ug/L	1
1,3,5-Trimethylbenzene	ug/L	1
Vinyl acetate	ug/L	5
Vinyl chloride	ug/L	1
m-Xylene	ug/L	1
m&p-Xylene	ug/L	1
o&p-Xylene	ug/L	1
o-Xylene	ug/L	1
Inorganic Nonmetals/General Organics		
BOD (EPA 405.1)	mg/L	1.0
Chloride (EPA 300.0)	mg/L	0.10
Chloride (EPA 325.2)	mg/L	1.0
Total Residual Chlorine (EPA 330.4)	mg/L	0.10
Chromium, hexavalent (SW846 7196)	mg/L	0.01
COD (EPA 410.4)	mg/L	10
Conductivity (EPA 120.1)	umhos/cm	1.0
Cyanide (EPA 335.2)	mg/L	0.01
Fluoride (EPA 300.0)	mg/L	0.10
Fluoride (EPA 340.2)	mg/L	0.20
Hardness (EPA 130.2)	mg/L	1.0
Hydrocarbons, total petroleum (USEPA 418.1)	mg/L	1.0
Nitrogen, ammonia (EPA 350.1)	mg/L	0.10
nitrate (EPA 300.0)	mg/L	0.10
nitrate (EPA 353.2)	mg/L	0.05
nitrate+nitrite (EPA 353.2)	mg/L	0.05
nitrite (EPA 300.0)	mg/L	0.10
nitrite (USGS 4540-84)	mg/L	0.05
total Kjeldahl(351.2)	mg/L	0.25
Oil & Grease (EPA 413.1)	mg/L	1.0
Phenols (EPA 420.1)	mg/L	0.01
Phosphorus, orthophosphate (EPA 300.0)	mg/L	0.10
Phosphorus, orthophosphate (EPA 365.1)	mg/L	0.05
total (EPA 365.3)	mg/L	0.05
Sulfate (EPA 300.0)	mg/L	0.10
Sulfate (EPA 375.4)	mg/L	2.0
Surfactants (EPA 425.1)	mg/L	0.1
Sulfide (EPA 376.1)	mg/L	1.0
Sulfite (EPA 377.1)	mg/L	3.0
TOC (EPA 415.2)	mg/L	1.0
TFR (EPA 160.1)	mg/L	10.0
TNFR (EPA 160.2)	mg/L	5.0
TR (EPA 160.3)	mg/L	10.0
Alkalinity (310.1)	mg/L	1.0
Color (110.2)	color units	1.0
Silicia (EPA 370.1)	mg/L	2.0
Turbidity (EPA 180.1)	NTU	0

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REPORTING LIMITS FOR WATER SAMPLES [a]

Parameter	Units	Reporting Limit
Bromide (EPA 300.0)	mg/L	0.1
Metals - Cold Vapor (SW846 7470, EPA 245.1, EPA CLP SOW 3/90)		
Mercury	ug/L	0.20
Metals - Furnace (SW846 7000 series, EPA 200 series, EPA CLP SOW 3/90)		
Antimony	ug/L	6.0
Arsenic	ug/L	10.0
Cadmium	ug/L	5.0
Chromium	ug/L	10.0
Copper	ug/L	10.0
Lead	ug/L	3.0
Selenium	ug/L	5.0
Silver	ug/L	10.0
Thallium	ug/L	10.0
Metals - ICP (SW846 6010, EPA 200.7, EPA CLP ILMO3.0)		
Aluminum	ug/L	200
Antimony	ug/L	60.0
Arsenic	ug/L	100
Barium	ug/L	200
Beryllium	ug/L	5.0
Boron	ug/L	100
Cadmium	ug/L	5.0
Calcium	ug/L	1000
Chromium	ug/L	10.0
Cobalt	ug/L	50.0
Copper	ug/L	10.0
Iron	ug/L	100
Lead	ug/L	100
Lithium	ug/L	2.0
Magnesium	ug/L	1000
Manganese	ug/L	15.0
Molybdenum	ug/L	50.0
Nickel	ug/L	40.0
Phosphorus	ug/L	100
Potassium	ug/L	1000
Selenium	ug/L	100
Silicon	ug/L	200
Silver	ug/L	10.0
Sodium	ug/L	1000
Strontium	ug/L	100
Thallium	ug/L	100
Tin	ug/L	25.0
Titanium	ug/L	10.0
Vanadium	ug/L	50.0
Zinc	ug/L	20.0
Metals - TRACE ICP (SW846 6010, EPA 200.7, EPA CLP ILMO3.0)		

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REPORTING LIMITS FOR WATER SAMPLES [a]

Parameter	Units	Reporting Limit
Antimony	ug/L	6.0
Arsenic	ug/L	10.0
Cadmium	ug/L	5.0
Lead	ug/L	3.0
Selenium	ug/L	5.0
Thallium	ug/L	10.0
Total organic lead - GC (California LUFT,1988)		
Total organic lead (ICP)	mg/L	0.33

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REPORTING LIMITS FOR SOIL SAMPLES [a]

Parameter	Units	Reporting Limit
Explosives - nitroaromatics (SW846 8330 revision 1)		
HMX	mg/kg	0.50
RDX	mg/kg	0.50
135TNB	mg/kg	0.25
13DNB	mg/kg	0.25
NB	mg/kg	0.25
TETRYL	mg/kg	0.65
246TNT	mg/kg	0.25
26DNT	mg/kg	0.25
24DNT	mg/kg	0.25
4amDNT	mg/kg	0.50
2amDNT	mg/kg	0.50
2NT	mg/kg	0.25
4NT	mg/kg	0.25
3NT	mg/kg	0.25
PETN	mg/kg	1.0
NG	mg/kg	1.0
Herbicides GC/ECD - chlorinated compounds (SW846 8150)		
Dicamba	ug/kg	54
Dalapon	ug/kg	1200
MCPP	ug/kg	38000
MCPA	ug/kg	50000
Dichloroprop	ug/kg	130
2,4-D	ug/kg	240
2,4,5-TP	ug/kg	34
2,4,5-T	ug/kg	40
2,4-DB	ug/kg	180
Dinoseb	ug/kg	40
Pesticides and PCBs GC/ECD - organochlorine compounds (SW846 8080)		
Aldrin	ug/kg	1.7
α-BHC	ug/kg	1.7
β-BHC	ug/kg	1.7
δ-BHC	ug/kg	1.7
γ-BHC (Lindane)	ug/kg	1.7
α-Chlordane	ug/kg	1.7
γ-Chlordane	ug/kg	1.7
Chlordane-Technical	ug/kg	33
4,4'-DDD	ug/kg	3.3
4,4'-DDE	ug/kg	3.3
4,4'-DDT	ug/kg	3.3
Dieldrin	ug/kg	3.3
Endosulfan I	ug/kg	1.7
Endosulfan II	ug/kg	3.3

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REPORTING LIMITS FOR SOIL SAMPLES [a]

Parameter	Units	Reporting Limit
Endosulfan sulfate	ug/kg	3.3
Endrin	ug/kg	3.3
Endrin aldehyde	ug/kg	3.3
Endrin ketone	ug/kg	3.3
Heptachlor	ug/kg	1.7
Heptachlor epoxide	ug/kg	1.7
Methoxychlor	ug/kg	17
Toxaphene	ug/kg	170
Aroclor 1016	ug/kg	33
Aroclor 1221	ug/kg	67
Aroclor 1232	ug/kg	33
Aroclor 1242	ug/kg	33
Aroclor 1248	ug/kg	33
Aroclor 1254	ug/kg	33
Aroclor 1260	ug/kg	33
Pesticides GC/NPD - organophosphorus compounds (SW846 8140)		
Azinphos methyl	ug/kg	33
Bolstar	ug/kg	33
Chlorpyrifos (Dursban)	ug/kg	33
Coumaphos	ug/kg	33
Demeton (-O & -S)	ug/kg	33
Diazinon	ug/kg	33
Dichlorvos	ug/kg	33
Disulfoton	ug/kg	33
Ethoprop	ug/kg	33
Fensulfothion	ug/kg	33
Fenthion	ug/kg	33
Merphos	ug/kg	33
Mevinphos	ug/kg	33
Naled	ug/kg	33
Methyl Parathion	ug/kg	33
Phorate	ug/kg	33
Ronnel	ug/kg	33
Stirphos	ug/kg	33
Tokuthion	ug/kg	33
Trichloronate	ug/kg	33
Pesticides-GC-thiocarbamates (EPA 634)		
EPTC	ug/kg	30
Butylate	ug/kg	30
Vernolate	ug/kg	60
Pebulate	ug/kg	30
Molinate	ug/kg	30
Cycloate	ug/kg	60

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REPORTING LIMITS FOR SOIL SAMPLES [a]

Parameter	Units	Reporting Limit
Semivolatile organics GC/ECD - phthalate esters (SW846 8060)		
Bis (2-ethylhexyl) phthalate	ug/kg	170
Butyl benzyl phthalate	ug/kg	17
Diethyl phthalate	ug/kg	17
Dimethyl phthalate	ug/kg	17
Di-n-butyl phthalate	ug/kg	170
Semivolatile organics GC/ECD - chlorinated compounds (SW846 8120)		
2-Chloronaphthalene	ug/kg	67
1,2-Dichlorobenzene	ug/kg	67
1,3-Dichlorobenzene	ug/kg	67
1,4-Dichlorobenzene	ug/kg	67
Hexachlorobenzene	ug/kg	67
Hexachlorobutadiene	ug/kg	67
Hexachlorocyclopentadiene	ug/kg	67
Hexachloroethane	ug/kg	67
1,2,4-Trichlorobenzene	ug/kg	67
Semivolatile organics GC/FID- PAHs - GC (SW846 8100)		
Acenaphthene	ug/kg	33
Acenaphthylene	ug/kg	33
Anthracene	ug/kg	33
Benzo[a]anthracene	ug/kg	33
Benzo[b]fluoranthene	ug/kg	33
Benzo[k]fluoranthene	ug/kg	33
Benzo[a]pyrene	ug/kg	33
Benzo[ghi]perylene	ug/kg	33
Chrysene	ug/kg	33
Fluoranthene	ug/kg	33
Fluorene	ug/kg	33
Indeno[1,2,3-cd]pyrene	ug/kg	33
Naphthalene	ug/kg	33
Phenanthrene	ug/kg	33
Pyrene	ug/kg	33
Semivolatile organics GC/MS - (SW846 3450/8270)		
Acenaphthene	ug/kg	330
Acenaphthylene	ug/kg	330
Aniline	ug/kg	1700
Anthracene	ug/kg	330
Benzidine	ug/kg	1700
Benzo[a]anthracene	ug/kg	330
Benzo[b]fluoranthene	ug/kg	330
Benzo[k]fluoranthene	ug/kg	330
Benzo[a]pyrene	ug/kg	330

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REPORTING LIMITS FOR SOIL SAMPLES [a]

Parameter	Units	Reporting Limit
Benzo[ghi]perylene	ug/kg	330
Benzoic acid	ug/kg	1700
Benzyl alcohol	ug/kg	330
Benzyl butyl phthalate	ug/kg	330
Bis(2-chloroethyl) ether	ug/kg	330
Bis(2-chloroethoxy)methane	ug/kg	330
Bis(2-ethylhexyl) phthalate	ug/kg	330
Bis(2-chloroisopropyl) ether	ug/kg	330
4-Bromophenyl phenyl ether	ug/kg	330
Carbazole	ug/kg	330
4-Chloroaniline	ug/kg	330
4-Chloro-3-methylphenol	ug/kg	330
2-Chloronaphthalene	ug/kg	330
2-Chlorophenol	ug/kg	330
4-Chlorophenyl phenyl ether	ug/kg	330
Chrysene	ug/kg	330
Dibenzo[a,h]anthracene	ug/kg	330
Dibenzofuran	ug/kg	330
Di-n-butyl phthalate	ug/kg	330
1,2-Dichlorobenzene	ug/kg	330
1,3-Dichlorobenzene	ug/kg	330
1,4-Dichlorobenzene	ug/kg	330
3,3'-Dichlorobenzidine	ug/kg	330
2,4-Dichlorophenol	ug/kg	330
Diethyl phthalate	ug/kg	330
2,4-Dimethylphenol	ug/kg	330
Dimethyl phthalate	ug/kg	330
2,4-Dinitrophenol	ug/kg	1700
2,4-Dinitrotoluene	ug/kg	330
2,6-Dinitrotoluene	ug/kg	330
1,2-Diphenylhydrazine	ug/kg	330
Di-n-octyl phthalate	ug/kg	330
Fluoranthene	ug/kg	330
Fluorene	ug/kg	330
Hexachlorobenzene	ug/kg	330
Hexachlorobutadiene	ug/kg	330
Hexachlorocyclopentadiene	ug/kg	330
Hexachloroethane	ug/kg	330
Indeno[1,2,3-cd]pyrene	ug/kg	330
Isophorone	ug/kg	330
2-Methyl-4,6-dinitrophenol	ug/kg	1700
2-Methylnaphthalene	ug/kg	330
2-Methylphenol	ug/kg	330
4-Methylphenol	ug/kg	330
Naphthalene	ug/kg	330

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REPORTING LIMITS FOR SOIL SAMPLES [a]

Parameter	Units	Reporting Limit
2-Nitroaniline	ug/kg	1700
3-Nitroaniline	ug/kg	1700
4-Nitroaniline	ug/kg	1700
Nitrobenzene	ug/kg	330
2-Nitrophenol	ug/kg	330
4-Nitrophenol	ug/kg	1700
N-Nitrosodiphenylamine	ug/kg	330
N-Nitrosodimethylamine	ug/kg	330
N-Nitroso-di-n-propylamine	ug/kg	330
Pentachlorophenol	ug/kg	1700
Phenanthrene	ug/kg	330
Phenol	ug/kg	330
Pyrene	ug/kg	330
1,2,4-Trichlorobenzene	ug/kg	330
2,4,5-Trichlorophenol	ug/kg	1700
2,4,6-Trichlorophenol	ug/kg	330
Semivolatile organics HPLC - PAHs (SW846 8310)		
Acenaphthene	ug/kg	40
Acenaphthylene	ug/kg	70
Anthracene	ug/kg	5.0
Benzo[a]anthracene	ug/kg	2.0
Benzo[b]fluoranthene	ug/kg	2.0
Benzo[k]fluoranthene	ug/kg	2.0
Benzo[a]pyrene	ug/kg	2.0
Benzo[ghi]perylene	ug/kg	2.0
Chrysene	ug/kg	5.0
Dibenzo[a,h]anthracene	ug/kg	2.0
Fluoranthene	ug/kg	7.0
Fluorene	ug/kg	7.0
Indeno[1,2,3-cd]pyrene	ug/kg	2.0
Naphthalene	ug/kg	40
Phenanthrene	ug/kg	5.0
Pyrene	ug/kg	9.0
Total petroleum hydrocarbons GC (EAL-M-8015-GRO)		
TPH as gasoline (GC)	ug/kg	100
Total petroleum hydrocarbon GC/FID (EALL-M-8015-DRO)		
TPH as JP4 (extractable)	mg/kg	100
TPH as diesel (GC)	mg/kg	25
TPH as JP8 (extractable)	mg/kg	100
Total petroleum hydrocarbons GC (Mississippi)		

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REPORTING LIMITS FOR SOIL SAMPLES [a]

Parameter	Units	Reporting Limit
TPH as diesel (GC)	mg/kg	6.4
Volatile organics GC/ELCD - halogenated compounds (SW 846 8010)		
Bromodichloromethane	ug/kg	1
Bromoform	ug/kg	1
Bromomethane	ug/kg	1
Carbon tetrachloride	ug/kg	1
Chlorobenzene	ug/kg	1
Chloroethane	ug/kg	1
2-Chloroethylvinyl ether	ug/kg	1
Chloroform	ug/kg	1
Chloromethane	ug/kg	1
Dibromochloromethane	ug/kg	1
Dibromomethane	ug/kg	1
1,2-Dichlorobenzene	ug/kg	1
1,3-Dichlorobenzene	ug/kg	1
1,4-Dichlorobenzene	ug/kg	1
Dichlorodifluoromethane	ug/kg	1
1,1-Dichloroethane	ug/kg	1
1,2-Dichloroethane	ug/kg	1
1,1-Dichloroethene	ug/kg	1
trans-1,2-Dichloroethene	ug/kg	1
1,2-Dichloropropane	ug/kg	1
cis-1,3-Dichloropropene	ug/kg	1
trans-1,3-Dichloropropene	ug/kg	1
Methylene chloride	ug/kg	1
1,1,1,2-Tetrachloroethane	ug/kg	1
1,1,2,2-Tetrachloroethane	ug/kg	1
Tetrachloroethene	ug/kg	1
1,1,1-Trichloroethane	ug/kg	1
1,1,2-Trichloroethane	ug/kg	1
Trichloroethene	ug/kg	1
Trichlorofluoromethane	ug/kg	1
1,2,3-Trichloropropane	ug/kg	1
Vinyl chloride	ug/kg	1
Volatile organics GC/FID - acrolein, acrylonitrile (SW846 8030)		
Acrolein	ug/kg	5
Acrylonitrile	ug/kg	5
Volatile organics GC/FID - non-halogenated compounds (SW846 8015)		
Diethyl ether	ug/kg	5
Methyl ethyl ketone (MEK)	ug/kg	5
Methyl isobutyl ketone (MIBK)	ug/kg	5

(a) EA Laboratories has established *Reporting Limits* (RLs) as laboratory quantitation levels. These are the minimum concentrations to be reported for routine laboratory analyses in specified environmental matrices. The RLs are values believed to provide greater than 50% probability of avoiding a false negative.

Updated: April 1, 1996/HJG
Supercedes: June 1995

EA LABORATORIES

REPORTING LIMITS FOR SOIL SAMPLES [a]

Parameter	Units	Reporting Limit
Volatile organics GC/PID - aromatic compounds (SW846 5030/8020)		
Benzene	ug/kg	1
Chlorobenzene	ug/kg	1
1,2-Dichlorobenzene	ug/kg	1
1,3-Dichlorobenzene	ug/kg	1
1,4-Dichlorobenzene	ug/kg	1
Ethylbenzene	ug/kg	1
Toluene	ug/kg	1
m&p-Xylene	ug/kg	1
o-Xylene	ug/kg	1
Volatile organics GC/ECD and PID in series (SW846 5030/8021)		
Benzene	ug/kg	1
Bromobenzene	ug/kg	1
Bromochloromethane	ug/kg	1
Bromodichloromethane	ug/kg	1
Bromoform	ug/kg	1
Bromomethane	ug/kg	1
n-Butylbenzene	ug/kg	1
sec-Butylbenzene	ug/kg	1
tert-Butylbenzene	ug/kg	1
Carbon tetrachloride	ug/kg	1
Chlorobenzene	ug/kg	1
Chlorodibromomethane	ug/kg	1
Chloroethane	ug/kg	1
Chloroform	ug/kg	1
Chloromethane	ug/kg	1
2-Chlorotoluene	ug/kg	1
4-Chlorotoluene	ug/kg	1
1,2-Dibromo-3-chloropropane	ug/kg	1
1,2-Dibromoethane	ug/kg	1
Dibromomethane	ug/kg	1
1,2-Dichlorobenzene	ug/kg	1
1,3-Dichlorobenzsene	ug/kg	1
1,4-Dichlorobenzene	ug/kg	1
Dichlorodifluormethane	ug/kg	1
1,1-Dichloroethane	ug/kg	1
1,2-Dichloroethane	ug/kg	1
1,1-Dichloroethene	ug/kg	1
cis-1,2-Dichloroethene	ug/kg	1
trans-1,2-Dichloroethene	ug/kg	1
1,2-Dichloropropane	ug/kg	1
1,3-Dichloropropane	ug/kg	1
2,2-Dichloropropane	ug/kg	1
1,1-Dichloropropene	ug/kg	1

(a) EA Laboratories has established *Reporting Limits* (RLs) as laboratory quantitation levels. These are the minimum concentrations to be reported for routine laboratory analyses in specified environmental matrices. The RLs are values believed to provide greater than 50% probability of avoiding a false negative.

Updated: April 1, 1996/HJG
Supercedes: June 1995

REPORTING LIMITS FOR SOIL SAMPLES [a]

Parameter	Units	Reporting Limit
cis-1,3-Dichloropropene	ug/kg	1
trans-1,3-Dichloropropene	ug/kg	1
Ethylbenzene	ug/kg	1
Hexachlorobutadiene	ug/kg	1
Isopropylbenzene	ug/kg	1
p-Isopropyltoluene	ug/kg	1
Methylene Chloride	ug/kg	1
Napthalene	ug/kg	1
n-Propylbenzene	ug/kg	1
Styrene	ug/kg	1
1,1,1,2-Tetrachloroethane	ug/kg	1
1,1,2,2-Tetrachloroethane	ug/kg	1
Tetrachloroethene	ug/kg	1
Toluene	ug/kg	1
1,2,3-Trichlorobenzene	ug/kg	1
1,2,4-Trichlorobenzene	ug/kg	1
1,1,1-Trichloroethane	ug/kg	1
1,1,2-Trichloroethane	ug/kg	1
Trichloroethene	ug/kg	1
Trichlorofluoromethane	ug/kg	1
1,2,3-Trichloropropane	ug/kg	1
1,2,4-Trimethylbenzene	ug/kg	1
1,3,5-Trimethylbenzene	ug/kg	1
Vinyl chloride	ug/kg	1
o-Xylene	ug/kg	1
m-Xylene	ug/kg	1
p-Xylene	ug/kg	1
Volatile organics GC/MS - (SW846 5030/8240)		
Acetone	ug/kg	10
Acrolein	ug/kg	50
Acrylonitrile	ug/kg	50
Benzene	ug/kg	5
Bromodichloromethane	ug/kg	5
Bromoform	ug/kg	5
Bromomethane	ug/kg	10
2-Butanone	ug/kg	10
n-Butylbenzene	ug/kg	5
sec-Butylbenzene	ug/kg	5
tert-Butylbenzene	ug/kg	5
tert-Butyl methyl ether (MTBE)	ug/kg	5
Carbon disulfide	ug/kg	5
Carbon tetrachloride	ug/kg	5
Chlorobenzene	ug/kg	5
Chloroethane	ug/kg	10

(a) EA Laboratories has established *Reporting Limits* (RLs) as laboratory quantitation levels. These are the minimum concentrations to be reported for routine laboratory analyses in specified environmental matrices. The RLs are values believed to provide greater than 50% probability of avoiding a false negative.

EA LABORATORIES

REPORTING LIMITS FOR SOIL SAMPLES [a]

Parameter	Units	Reporting Limit
2-Chloroethyl vinyl ether	ug/kg	10
Chloroform	ug/kg	5
Chloromethane	ug/kg	10
2-Chlorotoluene	ug/kg	5
4-Chlorotoluene	ug/kg	5
3-Chloro-1-propene	ug/kg	5
Dibromochloromethane	ug/kg	5
1,2-Dibromo-3-chloropropane	ug/kg	10
1,2-Dibromoethane	ug/kg	5
Dibromomethane	ug/kg	5
1,2-Dichlorobenzene	ug/kg	5
1,3-Dichlorobenzene	ug/kg	5
1,4-Dichlorobenzene	ug/kg	5
Dichlorodifluoromethane	ug/kg	10
1,1-Dichloroethane	ug/kg	5
1,2-Dichloroethane	ug/kg	5
1,1-Dichloroethene	ug/kg	5
1,2-Dichloroethene (total)	ug/kg	5
1,2-Dichloropropane	ug/kg	5
2,2-Dichloropropane	ug/kg	5
1,1-Dichloropropene	ug/kg	5
cis-1,3-Dichloropropene	ug/kg	5
trans-1,3-Dichloropropene	ug/kg	5
Diisopropyl ether	ug/kg	5
Ethylbenzene	ug/kg	5
Hexachlorobutadiene	ug/kg	5
2-Hexanone	ug/kg	10
Isopropylbenzene	ug/kg	5
4-Isopropyltoluene	ug/kg	5
4-Methyl-2-pentanone (MIBK)	ug/kg	10
Methylene chloride	ug/kg	5
Naphthalene	ug/kg	5
n-Propylbenzene	ug/kg	5
Styrene	ug/kg	5
1,1,1,2-Tetrachloroethane	ug/kg	5
1,1,2,2-Tetrachloroethane	ug/kg	5
Tetrachloroethene	ug/kg	5
Toluene	ug/kg	5
1,2,3-Trichlorobenzene	ug/kg	5
1,2,4-Trichlorobenzene	ug/kg	5
1,1,1-Trichloroethane	ug/kg	5
1,1,2-Trichloroethane	ug/kg	5
Trichloroethene	ug/kg	5
Trichlorofluoromethane	ug/kg	5
1,2,3-Trichloropropane	ug/kg	5

(a) EA Laboratories has established *Reporting Limits* (RLs) as laboratory quantitation levels. These are the minimum concentrations to be reported for routine laboratory analyses in specified environmental matrices. The RLs are values believed to provide greater than 50% probability of avoiding a false negative.

Updated: April 1, 1996/HJG
Supercedes: June 1995

EA LABORATORIES

REPORTING LIMITS FOR SOIL SAMPLES [a]

Parameter	Units	Reporting Limit
1,2,4-Trimethylbenzene	ug/kg	5
1,3,5-Trimethylbenzene	ug/kg	5
Vinyl acetate	ug/kg	10
Vinyl chloride	ug/kg	10
m-Xylene	ug/kg	5
m&p-Xylene	ug/kg	5
o&p-Xylene	ug/kg	5
o-Xylene	ug/kg	5
Volatile organics GC/MS - (SW846 5030/8260)		
Acetone	ug/kg	10
Benzene	ug/kg	5
Bromodichloromethane	ug/kg	5
Bromobenzene	ug/kg	5
Bromochloromethane	ug/kg	5
Bromoform	ug/kg	5
Bromomethane	ug/kg	5
n-Butylbenzene	ug/kg	5
sec-Butylbenzene	ug/kg	5
tert-Butylbenzene	ug/kg	5
2-Butanone	ug/kg	10
Carbon disulfide	ug/kg	5
Carbon tetrachloride	ug/kg	5
Chlorobenzene	ug/kg	5
Chloroethane	ug/kg	5
2-Chloroethylvinyl ether	ug/kg	10
Chloroform	ug/kg	5
Chloromethane	ug/kg	5
2-Chlorotoluene	ug/kg	5
4-Chlorotoluene	ug/kg	5
Dibromochloromethane	ug/kg	5
1,2-Dibromo-3-chloropropane	ug/kg	5
1,2-Dibromoethane	ug/kg	5
Dibromomethane	ug/kg	5
1,2-Dichlorobenzene	ug/kg	5
1,3-Dichlorobenzene	ug/kg	5
1,4-Dichlorobenzene	ug/kg	5
1 Dichlorodifluoromethane	ug/kg	5
1,1-Dichloroethane	ug/kg	5
1,2-Dichloroethane	ug/kg	5
1,1-Dichloroethene	ug/kg	5
cis-1,2-Dichloroethene	ug/kg	5
trans-1,2-Dichloroethene	ug/kg	5
1,2-Dichloropropane	ug/kg	5
1,3-Dichloropropane	ug/kg	5

(a) EA Laboratories has established *Reporting Limits* (RLs) as laboratory quantitation levels. These are the minimum concentrations to be reported for routine laboratory analyses in specified environmental matrices. The RLs are values believed to provide greater than 50% probability of avoiding a false negative.

Updated: April 1, 1996/HJG
Supercedes: June 1995

EA LABORATORIES

REPORTING LIMITS FOR SOIL SAMPLES [a]

Parameter	Units	Reporting Limit
2,2-Dichloropropane	ug/kg	5
1,1-Dichloropropene	ug/kg	5
cis-1,3-Dichloropropene	ug/kg	5
trans-1,3-Dichloropropene	ug/kg	5
Diisopropyl ether	ug/kg	5
Ethylbenzene	ug/kg	5
Hexachlorobutadiene	ug/kg	5
2-Hexanone	ug/kg	10
Isopropylbenzene	ug/kg	5
4-Isopropyltoluene	ug/kg	5
Methylene chloride	ug/kg	5
Methyl tert-butyl ether (MTBE)	ug/kg	5
4-Methyl-2-pentanone	ug/kg	10
Naphthalene	ug/kg	5
n-Propylbenzene	ug/kg	5
Styrene	ug/kg	5
1,1,1,2-Tetrachloroethane	ug/kg	5
1,1,2,2-Tetrachloroethane	ug/kg	5
Tetrachloroethene	ug/kg	5
Toluene	ug/kg	5
1,2,3-Trichlorobenzene	ug/kg	5
1,2,4-Trichlorobenzene	ug/kg	5
1,1,1-Trichloroethane	ug/kg	5
1,1,2-Trichloroethane	ug/kg	5
Trichloroethene	ug/kg	5
Trichlorofluoromethane	ug/kg	5
1,2,3-Trichloropropane	ug/kg	5
1,2,4-Trimethylbenzene	ug/kg	5
1,3,5-Trimethylbenzene	ug/kg	5
Vinyl acetate	ug/kg	10
Vinyl chloride	ug/kg	5
m-Xylene	ug/kg	5
m&p-Xylene	ug/kg	5
o&p-Xylene	ug/kg	5
o-Xylene	ug/kg	5
Inorganic nonmetals		
Bromide-IC	mg/kg	2.0
Chloride - IC	mg/kg	2.0
Chloride - Automated colorimetry	mg/kg	10
Cyanide	mg/kg	0.2
Fluoride - IC	mg/kg	2.0
Hydrocarbons, total petroleum (USEPA 9071/418.1)	mg/kg	25.0
Nitrogen,		

(a) EA Laboratories has established *Reporting Limits* (RLs) as laboratory quantitation levels. These are the minimum concentrations to be reported for routine laboratory analyses in specified environmental matrices. The RLs are values believed to provide greater than 50% probability of avoiding a false negative.

Updated: April 1, 1996/HJG
Supercedes: June 1995

EA LABORATORIES

REPORTING LIMITS FOR SOIL SAMPLES [a]

Parameter	Units	Reporting Limit
ammonia	mg/kg	25.0
nitrate - IC	mg/kg	2.0
nitrite - IC	mg/kg	2.0
total Kjeldahl	mg/kg	10
Phenols	mg/kg	0.13
Phosphorus, orthophosphate - IC	mg/kg	2.0
Sulfate - IC	mg/kg	2.0
Sulfide	mg/kg	25
TOC (SW846 9060)	mg/kg	4000
Metals - Cold Vapor		
Mercury	mg/kg	0.10
Metals - Furnace		
Antimony	mg/kg	0.60
Arsenic	mg/kg	1.0
Cadmium	mg/kg	0.50
Chromium	mg/kg	1.0
Copper	mg/kg	1.0
Lead	mg/kg	0.30
Selenium	mg/kg	0.50
Silver	mg/kg	1.0
Thallium	mg/kg	1.0
Metals - ICP		
Aluminum	mg/kg	20.0
Antimony	mg/kg	6.0
Arsenic	mg/kg	10.0
Barium	mg/kg	20.0
Beryllium	mg/kg	0.50
Boron	mg/kg	10.0
Cadmium	mg/kg	0.50
Calcium	mg/kg	100
Chromium	mg/kg	1.0
Cobalt	mg/kg	5.0
Copper	mg/kg	1.0
Iron	mg/kg	10.0
Lead	mg/kg	10.0
Magnesium	mg/kg	100.0
Manganese	mg/kg	1.5
Molybdenum	mg/kg	5.0
Nickel	mg/kg	4.0
Potassium	mg/kg	100
Selenium	mg/kg	10.0

(a) EA Laboratories has established *Reporting Limits* (RLs) as laboratory quantitation levels. These are the minimum concentrations to be reported for routine laboratory analyses in specified environmental matrices. The RLs are values believed to provide greater than 50% probability of avoiding a false negative.

Updated: April 1, 1996/HJG
Supercedes: June 1995

EA LABORATORIES

REPORTING LIMITS FOR SOIL SAMPLES [a]

Parameter	Units	Reporting Limit
Silicon	mg/kg	20.0
Silver	mg/kg	1.0
Sodium	mg/kg	100
Thallium	mg/kg	10.0
Tin	mg/kg	2.5
Titanium	mg/kg	1.0
Vanadium	mg/kg	5.0
Zinc	mg/kg	2.0
Total organic lead		
Lead (Flame AA)	mg/kg	0.33

(a) EA Laboratories has established *Reporting Limits* (RLs) as laboratory quantitation levels. These are the minimum concentrations to be reported for routine laboratory analyses in specified environmental matrices. The RLs are values believed to provide greater than 50% probability of avoiding a false negative.

Updated: April 1, 1996/HJG
Supercedes: June 1995

EA LABORATORIES REPORTING LIMITS FOR TCLP LEACHATES (a)

Parameter	Units	Reporting Limit (b)	TC Rule Regulatory Level
Herbicides GC/ECD - chlorinated compounds (SW846 8150)			
2,4-D	ug/L	120	10,000
2,4,5-TP	ug/L	17	1,000
Pesticides and PCBs GC/ECD - organochlorine compounds (SW846 3520/8080)			
γ-BHC (Lindane)	ug/L	0.25	400
Chlordane-Technical	ug/L	5.0	30
Endrin	ug/L	0.50	20
Heptachlor (and its oxides)	ug/L	0.25	8
Methoxychlor	ug/L	2.5	10,000
Toxaphene	ug/L	25	500
Semivolatile organics GC/MS - (SW846 3520/8270)			
1,4-Dichlorobenzene	ug/L	50	7,500
2,4-Dinitrotoluene	ug/L	50	130
Hexachlorobenzene	ug/L	50	130
Hexachlorobutadiene	ug/L	50	50
Hexachloroethane	ug/L	50	3,000
2-Methylphenol	ug/L	50	200,000
3-Methylphenol	ug/L	50	200,000
4-Methylphenol	ug/L	50	200,000
Nitrobenzene	ug/L	50	2,000
Pentachlorophenol	ug/L	250	100,000
Pyrdine	ug/L	50	5,000
2,4,5-Trichlorophenol	ug/L	250	400,000
2,4,6-Trichlorophenol	ug/L	50	2,000
Volatile organics GC/MS - 5 mL purge (SW846 5030/8240)			
Benzene	ug/L	5	500
2-Butanone (MEK)	ug/L	10	200,000
Carbon tetrachloride	ug/L	5	500
Chlorobenzene	ug/L	5	100,000
Chloroform	ug/L	5	6,000
1,2-Dichloroethane	ug/L	5	500
1,1-Dichloroethene	ug/L	5	700
Tetrachloroethene	ug/L	5	700
Trichloroethene	ug/L	5	500
Vinyl chloride	ug/L	10	200
Metals - Cold Vapor (SW846 7470)			
Mercury	ug/L	0.20	200
Metals - ICP (SW846 3010/6010)			
Arsenic	ug/L	100	5,000
Barium	ug/L	200	100,000
Cadmium	ug/L	5.0	1,000

(a) 40 CFR 264

(b) EA Laboratories has established *Reporting Limits* (RLs) as laboratory quantitation levels. These are the minimum concentrations to be reported for routine laboratory analyses in clean environmental matrices. The RLs are values believed to provide greater than 50% probability of avoiding a false negative.

EA LABORATORIES REPORTING LIMITS FOR TCLP LEACHATES (a)

Parameter	Units	Reporting Limit (b)	TC Rule Regulatory Level
Chromium	ug/L	10.0	5,000
Lead	ug/L	100	5,000
Selenium	ug/L	100	1,000
Silver	ug/L	10.0	5,000

(a) 40 CFR 264

(b) EA Laboratories has established *Reporting Limits* (RLs) as laboratory quantitation levels. These are the minimum concentrations to be reported for routine laboratory analyses in clean environmental matrices. The RLs are values believed to provide greater than 50% probability of avoiding a false negative.

EA LABORATORIES

REPORTING LIMITS FOR TISSUE SAMPLES [a]

Parameter	Units	Reporting Limit
Pesticides and PCBs - GC/ECD organochlorine compounds (SW846 8080) - Clam tissue		
Aldrin	mg/kg	0.0017
α-BHC	mg/kg	0.0017
β-BHC	mg/kg	0.0017
δ-BHC	mg/kg	0.0017
γ-BHC (Lindane)	mg/kg	0.0017
α-Chlordane	mg/kg	0.0017
γ-Chlordane	mg/kg	0.0017
4,4'-DDD	mg/kg	0.0033
4,4'-DDE	mg/kg	0.0033
4,4'-DDT	mg/kg	0.0033
Dieldrin	mg/kg	0.0033
Endosulfan I	mg/kg	0.0017
Endosulfan II	mg/kg	0.0033
Endosulfan sulfate	mg/kg	0.0033
Endrin	mg/kg	0.0033
Endrin aldehyde	mg/kg	0.0033
Endrin ketone	mg/kg	0.0033
Heptachlor	mg/kg	0.0017
Heptachlor epoxide	mg/kg	0.0017
Methoxychlor	mg/kg	0.017
Toxaphene	mg/kg	0.170
Aroclor 1016	mg/kg	0.033
Aroclor 1221	mg/kg	0.067
Aroclor 1232	mg/kg	0.075
Aroclor 1242	mg/kg	0.033
Aroclor 1248	mg/kg	0.050
Aroclor 1254	mg/kg	0.033
Aroclor 1260	mg/kg	0.033
2,4'-DDD	ng/g	
2,4'-DDE	ng/g	
2,4'-DDT	ng/g	
trans-Nonachlor	ng/g	
Pesticide-GC\ECD (SW846 8080) - Clam Tissue		
Isodrin	mg/kg	0.0017
PCBs GC/ECD - Congeners (SW846 8080) - Clam Tissue		
2,4,5,6-tetrachloro-meta-xylene	ug/kg	
2,4'-dichlorobiphenyl	ug/kg	
2,2',5-trichlorobiphenyl	ug/kg	
2,4,4'-trichlorobiphenyl	ug/kg	
2,2,5,5'-tetrachlorobiphenyl	ug/kg	
2,2',4,5'-tetrachlorobiphenyl	ug/kg	

(a) EA Laboratories has established *Reporting Limits* (RLs) as laboratory quantitation levels. These are the minimum concentrations to be reported for routine laboratory analyses in specified environmental matrices. The RLs are values believed to provide greater than 50% probability of avoiding a false negative.

Updated: May 1995
Supercedes: June 1994

REPORTING LIMITS FOR TISSUE SAMPLES [a]

Parameter	Units	Reporting Limit
2,2',3,5'-tetrachlorobiphenyl	ug/kg	
2,3',4,4'-tetrachlorobiphenyl	ug/kg	
2,2',4,5,5'-pentachlorobiphenyl	ug/kg	
2,2',3,4,5'-pentachlorobiphenyl	ug/kg	
3,3',4,4'-tetrachlorobiphenyl	ug/kg	
2,3',4,4',5-pentachlorobiphenyl	ug/kg	
2,3,3',4,4'-pentachlorobiphenyl	ug/kg	
2,2',4,4',5,5'-hexachlorobiphenyl	ug/kg	
2,2',3,4,4',6,6'-heptachlorobiphenyl	ug/kg	
2,2',3,4,4',5-hexachlorobiphenyl	ug/kg	
3,3',4,4',5-pentachlorobiphenyl	ug/kg	
2,2',3,4,5,5',6-heptachlorobiphenyl	ug/kg	
2,2',3,3',4,4'-hexachlorobiphenyl	ug/kg	
2,2',3,4,4',5',6-heptachlorobiphenyl	ug/kg	
2,3,3',4,4',5-hexachlorobiphenyl	ug/kg	
2,2',3,4,4',5,5'-heptachlorobiphenyl	ug/kg	
3,3',4,4',5,5'-hexachlorobiphenyl	ug/kg	
2,2',3,3',4,4',5-heptachlorobiphenyl	ug/kg	
2,2',3,3',4,4',5,6-octachlorobiphenyl	ug/kg	
2,2',3,3',4,4',5,5',6-nonachlorobiphenyl	ug/kg	
decachlorobiphenyl	ug/kg	
Semivolatile Organics HPLC - PAHs (SW846 8310) - Clam tissue		
Acenaphthene	mg/kg	0.060
Acenaphthylene	mg/kg	0.077
Anthracene	mg/kg	0.0050
Benzo[a]anthracene	mg/kg	0.0050
Benzo[b]fluoranthene	mg/kg	0.0075
Benzo[k]fluoranthene	mg/kg	0.0050
Benzo[a]pyrene	mg/kg	0.0050
Benzo[ghi]perylene	mg/kg	0.0075
Chrysene	mg/kg	0.0050
Dibenzo[a,h]anthracene	mg/kg	0.0075
Fluoranthene	mg/kg	0.0070
Fluorene	mg/kg	0.0070
Indeno[1,2,3-cd]pyrene	mg/kg	0.0050
1-Methyl Naphthalene	mg/kg	0.060
2-Methyl Naphthalene	mg/kg	0.060
Naphthalene	mg/kg	0.060
Phenanthrene	mg/kg	0.0050
Pyrene	mg/kg	0.009
Semivolatile Organics GC - Phenols and Substituted Phenols (SW846 8040) - Clam tissue		
Phenol	mg/kg	0.75
2,4-dimethylphenol	mg/kg	0.75

(a) EA Laboratories has established *Reporting Limits* (RLs) as laboratory quantitation levels. These are the minimum concentrations to be reported for routine laboratory analyses in specified environmental matrices. The RLs are values believed to provide greater than 50% probability of avoiding a false negative.

Updated: May 1995
Supercedes: June 1994

REPORTING LIMITS FOR TISSUE SAMPLES [a]

Parameter	Units	Reporting Limit
2,4,6-Trichlorophenol	mg/kg	0.75
4-Chloro-m-cresol	mg/kg	0.75
2-Chlorophenol	mg/kg	0.75
2,4-Dichlorophenol	mg/kg	0.75
2-Nitrophenol	mg/kg	0.75
4-Nitrophenol	mg/kg	0.75
2,4-Dinitrophenol	mg/kg	0.75
4,6-Dinitro-o-cresol	mg/kg	0.75
Pentachlorophenol	mg/kg	0.75
Volatile organics GC/PID - Aromatic Hydrocarbons (SW846 8020) - Clam tissue		
1,4-Dichlorobenzene	µg/kg	1.0
Inorganic nonmetals - Clam tissue		
Cyanide	mg/kg	
Metals - Cold Vapor (SW846 7471) - Clam tissue		
Mercury	mg/kg	
Metals - Furnace (SW846 7000 Series) - Clam tissue		
Antimony	mg/kg	
Arsenic	mg/kg	
Beryllium	mg/kg	
Cadmium	mg/kg	
Chromium	mg/kg	
Copper	mg/kg	
Lead	mg/kg	
Nickel	mg/kg	
Selenium	mg/kg	
Silver	mg/kg	
Thallium	mg/kg	
Metals - ICP (SW846 6010) - Clam Tissue		
Nickel	mg/kg	
Aluminum	mg/kg	
Chromium	mg/kg	
Copper	mg/kg	
Iron	mg/kg	
Zinc	mg/kg	
Silver	mg/kg	
Metals-ICP (Trace) - Clam Tissue		
Arsenic	mg/kg	
Cadmium	mg/kg	
Lead	mg/kg	

(a) EA Laboratories has established *Reporting Limits* (RLs) as laboratory quantitation levels. These are the minimum concentrations to be reported for routine laboratory analyses in specified environmental matrices. The RLs are values believed to provide greater than 50% probability of avoiding a false negative.

Updated: May 1995
Supercedes: June 1994

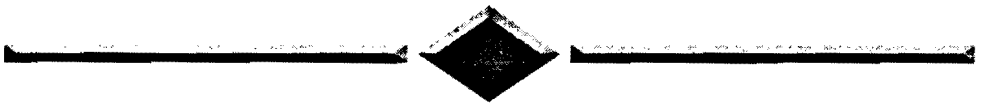
REPORTING LIMITS FOR TISSUE SAMPLES [a]

Parameter	Units	Reporting Limit
Antimony	mg/kg	
Selenium	mg/kg	
Beryllium	mg/kg	

(a) EA Laboratories has established *Reporting Limits* (RLs) as laboratory quantitation levels. These are the minimum concentrations to be reported for routine laboratory analyses in specified environmental matrices. The RLs are values believed to provide greater than 50% probability of avoiding a false negative.

Updated: May 1995
Supercedes: June 1994

Project-Specific MDLs



EA LABORATORIES MDLs for SW 846 Methods in Water

[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]
INORGANICS AND METALS					
Cyanide	SW9012	Colorimetric	mg/L	0.01	NB
Aluminum	SW3010A/6010A	ICP	ug/L	65.0	NB
Antimony	SW3010A/6010A	ICP	ug/L	24.0	500
Arsenic	SW3010A/6010A	ICP	ug/L	29.0	NB
Barium	SW3010A/6010A	ICP	ug/L	8.0	NB
Beryllium	SW3010A/6010A	ICP	ug/L	1.0	NB
Cadmium	SW3010A/6010A	ICP	ug/L	3.0	9
Calcium	SW3010A/6010A	ICP	ug/L	39.0	NB
Chromium	SW3010A/6010A	ICP	ug/L	4.0	NB
Cobalt	SW3010A/6010A	ICP	ug/L	7.0	NB
Copper	SW3010A/6010A	ICP	ug/L	5.0	NB
Iron	SW3010A/6010A	ICP	ug/L	53.0	NB
Lead	SW3010A/6010A	ICP - Trace	ug/L	2.0	8.5
Magnesium	SW3010A/6010A	ICP	ug/L	35.0	NB
Manganese	SW3010A/6010A	ICP	ug/L	2.0	NB
Nickel	SW3010A/6010A	ICP	ug/L	5.0	NB
Potassium	SW3010A/6010A	ICP	ug/L	71.0	NB
Selenium	SW3010A/6010A	ICP	ug/L	44.0	71
Silver	*SW3010A/6010A*	*ICP - Trace*	*ug/L*	*1.0*	*0.92*
Sodium	SW3010A/6010A	ICP	ug/L	81.0	NB
Thallium	SW3010A/6010A	ICP	ug/L	56.0	NB
Vanadium	SW3010A/6010A	ICP	ug/L	3.0	NB
Zinc	SW3010A/6010A	ICP	ug/L	12.0	88
Mercury	*SW7470*	*Cold Vapor*	*ug/L*	*0.2*	*0.025*
ORGANICS					
PCBS					
Aroclor 1016	*SW3520A/8082A*	*GC/ECD*	*ug/L*	*0.05*	*0.03*
Aroclor 1221	*SW3520A/8082A*	*GC/ECD*	*ug/L*	*0.10*	*0.03*
Aroclor 1232	*SW3520A/8082A*	*GC/ECD*	*ug/L*	*0.05*	*0.03*
Aroclor 1242	*SW3520A/8082A*	*GC/ECD*	*ug/L*	*0.05*	*0.03*
Aroclor 1248	*SW3520A/8082A*	*GC/ECD*	*ug/L*	*0.05*	*0.03*
Aroclor 1254	*SW3520A/8082A*	*GC/ECD*	*ug/L*	*0.05*	*0.03*
Aroclor 1260	*SW3520A/8082A*	*GC/ECD*	*ug/L*	*0.05*	*0.03*
(Method modification to 1mL final volume and lower standard.)					
Volatiles					
Acetone	SW5030B/8260B-5mL	GC/MS	ug/L	3.3	NB
Acetonitrile	SW5030B/8260B-5mL	GC/MS	ug/L	30	NB
Acrolein	SW5030B/8260B-5mL	GC/MS	ug/L	18	NB
Acrylonitrile	SW5030B/8260B-5mL	GC/MS	ug/L	12	NB
Allyl Chloride	SW5030B/8260B-5mL	GC/MS	ug/L	0.9	NB
Benzene	SW5030B/8260B-5mL	GC/MS	ug/L	0.5	700
Bromobenzene	SW5030B/8260B-5mL	GC/MS	ug/L	0.4	NB
Bromochloromethane	SW5030B/8260B-5mL	GC/MS	ug/L	0.5	NB

EA LABORATORIES MDLs for SW 846 Methods in Water

Analyte	Method	Description	Units	MDL	Benchmark
Bromodichloromethane	SW5030B/8260B-5mL	GC/MS	ug/L	0.4	NB
Bromofluorobenzene	SW5030B/8260B-5mL	GC/MS	ug/L	2.8	NB
Bromoform	SW5030B/8260B-5mL	GC/MS	ug/L	0.4	NB
Bromomethane	SW5030B/8260B-5mL	GC/MS	ug/L	1.7	NB
2-Butanone	SW5030B/8260B-5mL	GC/MS	ug/L	2.3	NB
n-Butylbenzene	SW5030B/8260B-5mL	GC/MS	ug/L	0.7	NB
sec-Butylbenzene	SW5030B/8260B-5mL	GC/MS	ug/L	0.7	NB
tert-Butylbenzene	SW5030B/8260B-5mL	GC/MS	ug/L	0.6	NB
Carbon Disulfide	SW5030B/8260B-5mL	GC/MS	ug/L	0.8	NB
Carbon tetrachloride	SW5030B/8260B-5mL	GC/MS	ug/L	1.2	NB
Chlorobenzene	SW5030B/8260B-5mL	GC/MS	ug/L	0.4	129
Chlorodibromomethane	SW5030B/8260B-5mL	GC/MS	ug/L	0.4	6400
Chloroethane	SW5030B/8260B-5mL	GC/MS	ug/L	1.8	NB
2-Chloroethylvinyl ether	SW5030B/8260B-5mL	GC/MS	ug/L	2.3	NB
Chloroform	SW5030B/8260B-5mL	GC/MS	ug/L	0.7	NB
1-Chlorohexane	SW5030B/8260B-5mL	GC/MS	ug/L	0.7	NB
Chloromethane	SW5030B/8260B-5mL	GC/MS	ug/L	1.1	NB
Chloroprene	SW5030B/8260B-5mL	GC/MS	ug/L	1.0	NB
2-Chlorotoluene	SW5030B/8260B-5mL	GC/MS	ug/L	0.4	NB
4-Chlorotoluene	SW5030B/8260B-5mL	GC/MS	ug/L	0.6	NB
Dibromochloromethane	SW5030B/8260B-5mL	GC/MS	ug/L	3.5	NB
1,2-Dibromo-3-chloropropane	SW5030B/8260B-5mL	GC/MS	ug/L	0.9	6400
Dibromofluoromethane	SW5030B/8260B-5mL	GC/MS	ug/L	3.5	NB
Dibromomethane	SW5030B/8260B-5mL	GC/MS	ug/L	0.6	6400
1,2-Dibromoethane (EDB)	SW5030B/8260B-5mL	GC/MS	ug/L	0.4	NB
1,2-Dichlorobenzene	SW5030B/8260B-5mL	GC/MS	ug/L	0.4	129
1,3-Dichlorobenzene	SW5030B/8260B-5mL	GC/MS	ug/L	0.5	129
1,4-Dichlorobenzene	SW5030B/8260B-5mL	GC/MS	ug/L	0.4	129
trans-1,4-Dichloro2-Butene	SW5030B/8260B-5mL	GC/MS	ug/L	4.4	NB
Dichlorodifluoromethane	SW5030B/8260B-5mL	GC/MS	ug/L	1.0	6400
1,1-Dichloroethane	SW5030B/8260B-5mL	GC/MS	ug/L	0.8	NB
1,2-Dichloroethane	SW5030B/8260B-5mL	GC/MS	ug/L	0.4	NB
1,1-Dichloroethene	SW5030B/8260B-5mL	GC/MS	ug/L	1.0	NB
cis-1,2-Dichloroethene	SW5030B/8260B-5mL	GC/MS	ug/L	0.7	NB
trans-1,2-Dichloroethene	SW5030B/8260B-5mL	GC/MS	ug/L	0.7	NB
1,2-Dichloroethene (total)	SW5030B/8260B-5mL	GC/MS	ug/L	0.7	NB
1,2-Dichloropropane	SW5030B/8260B-5mL	GC/MS	ug/L	0.5	NB
1,3-Dichloropropane	SW5030B/8260B-5mL	GC/MS	ug/L	0.5	NB
2,2-Dichloropropane	SW5030B/8260B-5mL	GC/MS	ug/L	2.0	NB
1,1-Dichloropropene	SW5030B/8260B-5mL	GC/MS	ug/L	0.6	NB
cis-1,3-Dichloropropene	SW5030B/8260B-5mL	GC/MS	ug/L	0.4	NB
trans-1,3-Dichloropropene	SW5030B/8260B-5mL	GC/MS	ug/L	0.4	NB
Diisopropyl ether	SW5030B/8260B-5mL	GC/MS	ug/L	0.6	NB
Ethyl acetate	SW5030B/8260B-5mL	GC/MS	ug/L	2.6	NB
Ethylbenzene	SW5030B/8260B-5mL	GC/MS	ug/L	0.5	NB
Ethyl ether	SW5030B/8260B-5mL	GC/MS	ug/L	1.3	NB
Ethyl methacrylate	SW5030B/8260B-5mL	GC/MS	ug/L	0.8	NB
Hexachlorobutadiene	SW5030B/8260B-5mL	GC/MS	ug/L	0.7	NB
2-Hexanone	SW5030B/8260B-5mL	GC/MS	ug/L	2.3	NB

EA LABORATORIES MDLs for SW 846 Methods in Water

Analyte	Method	Description	Unit	MDL	Benchmark
Iodomethane	SW5030B/8260B-5mL	GC/MS	ug/L	2.0	NB
Isobutyl Alcohol	SW5030B/8260B-5mL	GC/MS	ug/L	38.0	NB
Isopropylbenzene	SW5030B/8260B-5mL	GC/MS	ug/L	0.6	NB
p-Isopropyltoluene	SW5030B/8260B-5mL	GC/MS	ug/L	0.6	NB
Methacryonitrile	SW5030B/8260B-5mL	GC/MS	ug/L	29.0	NB
Methylene chloride	SW5030B/8260B-5mL	GC/MS	ug/L	0.7	NB
Methyl methacrylate	SW5030B/8260B-5mL	GC/MS	ug/L	0.5	NB
4-Methyl-2-Pentanone	SW5030B/8260B-5mL	GC/MS	ug/L	2.0	NB
Methyl t-butyl ether	SW5030B/8260B-5mL	GC/MS	ug/L	1.9	NB
Naphthalene	SW5030B/8260B-5mL	GC/MS	ug/L	1.1	NB
2-Nitropropane	SW5030B/8260B-5mL	GC/MS	ug/L	3.4	NB
Pentachloroethane	SW5030B/8260B-5mL	GC/MS	ug/L	0.4	281
Propionitrile	SW5030B/8260B-5mL	GC/MS	ug/L	27.0	NB
n-Propylbenzene	SW5030B/8260B-5mL	GC/MS	ug/L	0.5	NB
Styrene	SW5030B/8260B-5mL	GC/MS	ug/L	0.4	NB
1,1,1,2-Tetrachloroethane	SW5030B/8260B-5mL	GC/MS	ug/L	0.4	NB
1,1,2,2-Tetrachloroethane	SW5030B/8260B-5mL	GC/MS	ug/L	0.9	NB
Tetrachloroethene	SW5030B/8260B-5mL	GC/MS	ug/L	0.6	NB
Tetrahydrofuran	SW5030B/8260B-5mL	GC/MS	ug/L	7.6	NB
Toluene	SW5030B/8260B-5mL	GC/MS	ug/L	0.4	NB
1,2,3-Trichlorobenzene	SW5030B/8260B-5mL	GC/MS	ug/L	0.7	NB
1,2,4-Trichlorobenzene	SW5030B/8260B-5mL	GC/MS	ug/L	0.8	NB
1,1,1-Trichloroethane	SW5030B/8260B-5mL	GC/MS	ug/L	1.6	NB
1,1,2-Trichloroethane	SW5030B/8260B-5mL	GC/MS	ug/L	0.5	NB
Trichloroethene	SW5030B/8260B-5mL	GC/MS	ug/L	0.5	NB
Trichlorofluoromethane	SW5030B/8260B-5mL	GC/MS	ug/L	1.6	NB
1,2,3-Trichloropropane	SW5030B/8260B-5mL	GC/MS	ug/L	0.5	NB
1,1,2-Trichlorotrifluoroethane	SW5030B/8260B-5mL	GC/MS	ug/L	1.0	NB
1,2,3-Trimethylbenzene	SW5030B/8260B-5mL	GC/MS	ug/L	0.4	NB
1,2,4-Trimethylbenzene	SW5030B/8260B-5mL	GC/MS	ug/L	0.4	NB
1,3,5-Trimethylbenzene	SW5030B/8260B-5mL	GC/MS	ug/L	0.4	NB
Vinyl acetate	SW5030B/8260B-5mL	GC/MS	ug/L	1.4	NB
Vinyl chloride	SW5030B/8260B-5mL	GC/MS	ug/L	0.9	NB
m&p-Xylenes	SW5030B/8260B-5mL	GC/MS	ug/L	0.8	NB
o-Xylene	SW5030B/8260B-5mL	GC/MS	ug/L	0.4	NB
Xylenes	SW5030B/8260B-5mL	GC/MS	ug/L	1.3	NB
Polynuclear Aromatic Hydrocarbons (PAHs)					
Acenaphthene	SW3520A/8310	HPLC	ug/L	0.08	710
Acenaphthylene	SW3520A/8310	HPLC	ug/L	0.53	NB
Anthracene	SW3520A/8310	HPLC	ug/L	0.02	NB
Benzo[a]anthracene	SW3520A/8310	HPLC	ug/L	0.02	NB
Benzo[b]fluoranthene	SW3520A/8310	HPLC	ug/L	0.03	NB
Benzo[k]fluoranthene	SW3520A/8310	HPLC	ug/L	0.008	NB
Benzo[a]pyrene	SW3520A/8310	HPLC	ug/L	0.021	NB
Benzo[ghi]perylene	SW3520A/8310	HPLC	ug/L	0.028	NB
Chrysene	SW3520A/8310	HPLC	ug/L	0.014	NB
Dibenzo[a,h]anthracene	SW3520A/8310	HPLC	ug/L	0.022	NB
Fluoranthene	SW3520A/8310	HPLC	ug/L	0.047	16

EA LABORATORIES MDLs for SW 846 Methods in Water

Analyte	Method	Description	Units	MDL	Benchmark
Fluorene	SW3520A/8310	HPLC	ug/L	0.041	NB
Indeno[1,2,3-cd]pyrene	SW3520A/8310	HPLC	ug/L	0.042	NB
1-Methylnaphthalene	SW3520A/8310	HPLC	ug/L	0.10	NB
2-Methylnaphthalene	SW3520A/8310	HPLC	ug/L	0.13	NB
Naphthalene	SW3520A/8310	HPLC	ug/L	0.15	NB
Phenanthrene	SW3520A/8310	HPLC	ug/L	0.007	4.6
Pyrene	SW3520A/8310	HPLC	ug/L	0.016	NB

[1] The benchmark used for determining the appropriate analytical detection limits was based on the NOAA Screening Guidelines for Inorganics and Organics. The values selected are reflective of the US EPA Ambient Water Quality Criteria for marine waters and chronic exposures. It is our opinion, based on our understanding of the project, that drinking water criteria are not appropriate because all of the samples will be collected from a marine/estuarine system; thus, the sample stations are not drinking water resources. Ambient Water Quality Criteria are the appropriate values for situations such as this program.

Parameters indicated with italics represent those analytes/compounds whose MDL is higher than the Benchmark.

EA LABORATORIES MDLs for SW846 Methods in Solids

INORGANICS AND METALS					
Cyanide	SW9012	Colorimetric	mg/kg	0.19	NB
Total Organic Carbon	SW9060	Oxidation IR	mg/kg	4880	NB
Hexavalent Chromium	SW7196	Colorimetric	mg/kg	0.05	NB
Aluminum	SW3010A/6010A	ICP	mg/kg	6.5	NB
Antimony	SW3010A/6010A	ICP	mg/kg	2.4	NB
Arsenic	SW3050A/6010A	ICP	mg/kg	2.9	8.2
Barium	SW3050A/6010A	ICP	mg/kg	0.80	NB
Beryllium	SW3050A/6010A	ICP	mg/kg	0.10	NB
Cadmium	SW3050A/6010A	ICP	mg/kg	0.30	1.2
Calcium	SW3050A/6010A	ICP	mg/kg	3.9	NB
Chromium	SW3050A/6010A	ICP	mg/kg	0.40	81
Cobalt	SW3050A/6010A	ICP	mg/kg	0.70	NB
Copper	SW3050A/6010A	ICP	mg/kg	0.50	34
Iron	SW3050A/6010A	ICP	mg/kg	5.3	NB
Lead	SW3050A/6010A	ICP	mg/kg	5.1	46.7
Magnesium	SW3050A/6010A	ICP	mg/kg	3.5	NB
Manganese	SW3050A/6010A	ICP	mg/kg	0.20	NB
Nickel	SW3050A/6010A	ICP	mg/kg	0.50	20.9
Potassium	SW3050A/6010A	ICP	mg/kg	7.1	NB
Selenium	SW3050A/6010A	ICP	mg/kg	4.4	NB
Silver	SW3050A/6010A	ICP	mg/kg	0.40	1
Sodium	SW3050A/6010A	ICP	mg/kg	8.1	NB
Thallium	SW3050A/6010A	ICP	mg/kg	5.6	NB
Vanadium	SW3050A/6010A	ICP	mg/kg	0.30	NB
Zinc	SW3050A/6010A	ICP	mg/kg	1.2	150
Mercury	SW7471	Cold Vapor	mg/kg	0.10	0.15
ORGANICS					
PCBs					
Aroclor 1016	SW3540A8082	GC/ECD	ug/kg	9.3	22.7
Aroclor 1221	SW3540A8082	GC/ECD	ug/kg	7.4	22.7
Aroclor 1232	SW3540A8082	GC/ECD	ug/kg	10	22.7
Aroclor 1242	SW3540A8082	GC/ECD	ug/kg	4.7	22.7
Aroclor 1248	SW3540A8082	GC/ECD	ug/kg	8.4	22.7
Aroclor 1254	SW3540A8082	GC/ECD	ug/kg	3	22.7
Aroclor 1260	SW3540A8082	GC/ECD	ug/kg	8	22.7
Aroclor 5432	SW3540A8082	GC/ECD	ug/kg	18	22.7
Aroclor 5460	SW3540A8082	GC/ECD	ug/kg	28	22.7
Volatile Organics					
Acetone	SW5030A/8260B	GC/MS	ug/kg	3	NB
Acetonitrile	SW5030A/8260B	GC/MS	ug/kg	18	NB
Acrolein	SW5030A/8260B	GC/MS	ug/kg	12	NB
Acrylonitrile	SW5030A/8260B	GC/MS	ug/kg	14	NB
Allyl Chloride	SW5030A/8260B	GC/MS	ug/kg	3	NB
Benzene	SW5030A/8260B	GC/MS	ug/kg	1	NB
Bromobenzene	SW5030A/8260B	GC/MS	ug/kg	1	NB
Bromochloromethane	SW5030A/8260B	GC/MS	ug/kg	0.80	NB
Bromodichloromethane	SW5030A/8260B	GC/MS	ug/kg	0.90	NB
Bromofluorobenzene	SW5030A/8260B	GC/MS	ug/kg	0.80	NB
Bromoform	SW5030A/8260B	GC/MS	ug/kg	1	NB
Bromomethane	SW5030A/8260B	GC/MS	ug/kg	3	NB

EA LABORATORIES MDLs for SW846 Methods in Solids

2-Butanone	SW5030A/8260B	GC/MS	ug/kg	4	NB
n-Butylbenzene	SW5030A/8260B	GC/MS	ug/kg	1	NB
sec-Butylbenzene	SW5030A/8260B	GC/MS	ug/kg	1	NB
tert-Butylbenzene	SW5030A/8260B	GC/MS	ug/kg	0.90	NB
Carbon Disulfide	SW5030A/8260B	GC/MS	ug/kg	2	NB
Carbon tetrachloride	SW5030A/8260B	GC/MS	ug/kg	1	NB
Chlorobenzene	SW5030A/8260B	GC/MS	ug/kg	0.90	NB
Chlorodibromomethane	SW5030A/8260B	GC/MS	ug/kg		NB
Chloroethane	SW5030A/8260B	GC/MS	ug/kg	1	NB
2-Chloroethylvinyl ether	SW5030A/8260B	GC/MS	ug/kg	4	NB
Chloroform	SW5030A/8260B	GC/MS	ug/kg	1	NB
1-Chlorohexane	SW5030A/8260B	GC/MS	ug/kg	2	NB
Chloromethane	SW5030A/8260B	GC/MS	ug/kg	2	NB
Chloroprene	SW5030A/8260B	GC/MS	ug/kg	1	NB
2-Chlorotoluene	SW5030A/8260B	GC/MS	ug/kg	0.90	NB
4-Chlorotoluene	SW5030A/8260B	GC/MS	ug/kg	2	NB
Dibromochloromethane	SW5030A/8260B	GC/MS	ug/kg	0.80	NB
1,2-Dibromo-3-chloropropane	SW5030A/8260B	GC/MS	ug/kg	2	NB
Dibromofluoromethane	SW5030A/8260B	GC/MS	ug/kg	1	NB
Dibromomethane	SW5030A/8260B	GC/MS	ug/kg	1	NB
1,2-Dibromoethane (EDB)	SW5030A/8260B	GC/MS	ug/kg	0.90	NB
1,2-Dichlorobenzene	SW5030A/8260B	GC/MS	ug/kg	0.90	NB
1,3-Dichlorobenzene	SW5030A/8260B	GC/MS	ug/kg	1	NB
1,4-Dichlorobenzene	SW5030A/8260B	GC/MS	ug/kg	1	NB
trans-1,4-Dichloro2-Butene	SW5030A/8260B	GC/MS	ug/kg	4	NB
Dichlorodifluoromethane	SW5030A/8260B	GC/MS	ug/kg	2	NB
1,1-Dichloroethane	SW5030A/8260B	GC/MS	ug/kg	1	NB
1,2-Dichloroethane	SW5030A/8260B	GC/MS	ug/kg	1	NB
1,1-Dichloroethene	SW5030A/8260B	GC/MS	ug/kg	2	NB
cis-1,2-Dichloroethene	SW5030A/8260B	GC/MS	ug/kg	1	NB
trans-1,2-Dichloroethene	SW5030A/8260B	GC/MS	ug/kg	2	NB
1,2-Dichloroethene (total)	SW5030A/8260B	GC/MS	ug/kg	2	NB
1,2-Dichloropropane	SW5030A/8260B	GC/MS	ug/kg	4	NB
1,3-Dichloropropane	SW5030A/8260B	GC/MS	ug/kg	1	NB
2,2-Dichloropropane	SW5030A/8260B	GC/MS	ug/kg	2	NB
1,1-Dichloropropene	SW5030A/8260B	GC/MS	ug/kg	1	NB
cis-1,3-Dichloropropene	SW5030A/8260B	GC/MS	ug/kg	0.80	NB
trans-1,3-Dichloropropene	SW5030A/8260B	GC/MS	ug/kg	0.80	NB
Diisopropyl ether	SW5030A/8260B	GC/MS	ug/kg	2	NB
Ethyl acetate	SW5030A/8260B	GC/MS	ug/kg	2	NB
Ethylbenzene	SW5030A/8260B	GC/MS	ug/kg	2	NB
Ethyl ether	SW5030A/8260B	GC/MS	ug/kg	2	NB
Ethyl methacrylate	SW5030A/8260B	GC/MS	ug/kg	2	NB
Hexachlorobutadiene	SW5030A/8260B	GC/MS	ug/kg	1	NB
2-Hexanone	SW5030A/8260B	GC/MS	ug/kg	4	NB
Iodomethane	SW5030A/8260B	GC/MS	ug/kg	0.80	NB
Isobutyl Alcohol	SW5030A/8260B	GC/MS	ug/kg	40	NB
Isopropylbenzene	SW5030A/8260B	GC/MS	ug/kg	1	NB
p-Isopropyltoluene	SW5030A/8260B	GC/MS	ug/kg	0.90	NB
Methacryonitrile	SW5030A/8260B	GC/MS	ug/kg	3	NB
Methylene chloride	SW5030A/8260B	GC/MS	ug/kg	1	NB
Methyl methacrylate	SW5030A/8260B	GC/MS	ug/kg	2	NB
4-Methyl-2-Pentanone	SW5030A/8260B	GC/MS	ug/kg	5	NB
Methyl t-butyl ether	SW5030A/8260B	GC/MS	ug/kg	2	NB
Naphthalene	SW5030A/8260B	GC/MS	ug/kg	2	NB
2-Nitropropane	SW5030A/8260B	GC/MS	ug/kg	3	NB

EA LABORATORIES MDLs for SW846 Methods in Solids

Analyte	Method	Description	Units	MDL	Benchmark [1]
Pentachloroethane	SW5030A/8260B	GC/MS	ug/kg	2	NB
Propionitrile	SW5030A/8260B	GC/MS	ug/kg	20	NB
n-Propylbenzene	SW5030A/8260B	GC/MS	ug/kg	1	NB
Styrene	SW5030A/8260B	GC/MS	ug/kg	0.80	NB
1,1,1,2-Tetrachloroethane	SW5030A/8260B	GC/MS	ug/kg	1	NB
1,1,2,2-Tetrachloroethane	SW5030A/8260B	GC/MS	ug/kg	1	NB
Tetrachloroethene	SW5030A/8260B	GC/MS	ug/kg	2	NB
Tetrahydrofuran	SW5030A/8260B	GC/MS	ug/kg	4	NB
Toluene	SW5030A/8260B	GC/MS	ug/kg	1	NB
1,2,3-Trichlorobenzene	SW5030A/8260B	GC/MS	ug/kg	1	NB
1,2,4-Trichlorobenzene	SW5030A/8260B	GC/MS	ug/kg	1	NB
1,1,1-Trichloroethane	SW5030A/8260B	GC/MS	ug/kg	1	NB
1,1,2-Trichloroethane	SW5030A/8260B	GC/MS	ug/kg	1	NB
Trichloroethene	SW5030A/8260B	GC/MS	ug/kg	0.90	NB
Trichlorofluoromethane	SW5030A/8260B	GC/MS	ug/kg	2	NB
1,2,3-Trichloropropane	SW5030A/8260B	GC/MS	ug/kg	1	NB
1,1,2-Trichlorotrifluoroethane	SW5030A/8260B	GC/MS	ug/kg	3	NB
1,2,3-Trimethylbenzene	SW5030A/8260B	GC/MS	ug/kg	2	NB
1,2,4-Trimethylbenzene	SW5030A/8260B	GC/MS	ug/kg	1	NB
1,3,5-Trimethylbenzene	SW5030A/8260B	GC/MS	ug/kg	1	NB
Vinyl acetate	SW5030A/8260B	GC/MS	ug/kg	1	NB
Vinyl chloride	SW5030A/8260B	GC/MS	ug/kg	2	NB
m&p-Xylenes	SW5030A/8260B	GC/MS	ug/kg	2	NB
o-Xylene	SW5030A/8260B	GC/MS	ug/kg	1	NB
Xylenes	SW5030A/8260B	GC/MS	ug/kg	2	NB
Polynuclear Aromatic Hydrocarbons (PAHs)					
Acenaphthene	SW3540A/8310	HPLC	ug/kg	11	16
Acenaphthylene	SW3540A/8310	HPLC	ug/kg	51	44
Anthracene	SW3540A/8310	HPLC	ug/kg	0.68	85.3
Benzo[a]anthracene	SW3540A/8310	HPLC	ug/kg	0.52	261
Benzo[b]fluoranthene	SW3540A/8310	HPLC	ug/kg	1.1	NB
Benzo[k]fluoranthene	SW3540A/8310	HPLC	ug/kg	0.44	NB
Benzo[a]pyrene	SW3540A/8310	HPLC	ug/kg	1.8	430
Benzo[ghi]perylene	SW3540A/8310	HPLC	ug/kg	1.3	NB
Chrysene	SW3540A/8310	HPLC	ug/kg	0.52	384
Dibenzo[a,h]anthracene	SW3540A/8310	HPLC	ug/kg	1.4	69.4
Fluoranthene	SW3540A/8310	HPLC	ug/kg	0.84	600
Fluorene	SW3540A/8310	HPLC	ug/kg	2.0	19
Indeno[1,2,3-cd]pyrene	SW3540A/8310	HPLC	ug/kg	0.93	NB
1-Methylnaphthalene	SW3540A/8310	HPLC	ug/kg	10	NB
2-Methylnaphthalene	SW3540A/8310	HPLC	ug/kg	13	70
Naphthalene	SW3540A/8310	HPLC	ug/kg	7.4	160
Phenanthrene	SW3540A/8310	HPLC	ug/kg	0.65	240
Pyrene	SW3540A/8310	HPLC	ug/kg	0.48	665

[1] Benchmark based on EPA Region I ERL Guidelines.

EA LABORATORIES MDLs for SW846 Methods in Tissue

INORGANICS AND METALS					
Aluminum	SW3010A/6010A	ICP	mg/kg	11	NB
Antimony	SW3010A/6010A	ICP - Trace	mg/kg	0.9	NB
Arsenic	SW3050A/6010A	ICP - Trace	mg/kg	0.8	NB
Barium	SW3050A/6010A	ICP	mg/kg	12	NB
Beryllium	SW3050A/6010A	ICP	mg/kg	0.3	NB
Cadmium	SW3050A/6010A	ICP - Trace	mg/kg	0.04	NB
Calcium	SW3050A/6010A	ICP	mg/kg	191	NB
Chromium	SW3050A/6010A	ICP	mg/kg	1.4	NB
Cobalt	SW3050A/6010A	ICP	mg/kg	3.1	NB
Copper	SW3050A/6010A	ICP	mg/kg	1.5	NB
Iron	SW3050A/6010A	ICP	mg/kg	16	NB
Lead	SW3050A/6010A	ICP - Trace	mg/kg	0.23	NB
Magnesium	SW3050A/6010A	ICP	mg/kg	69	NB
Manganese	SW3050A/6010A	ICP	mg/kg	2.9	NB
Nickel	SW3050A/6010A	ICP	mg/kg	3.0	NB
Potassium	SW3050A/6010A	ICP	mg/kg	579	NB
Selenium	SW3050A/7740	GFAA	mg/kg	0.9	NB
Silver	SW3050A/6010A	ICP - Trace	mg/kg	0.27	NB
Sodium	SW3050A/6010A	ICP	mg/kg	1058	NB
Thallium	SW3050A/7841	GFAA	mg/kg	0.3	NB
Vanadium	SW3050A/6010A	ICP	mg/kg	2.8	NB
Zinc	SW3050A/6010A	ICP	mg/kg	9.1	NB
Mercury	SW7471	Cold Vapor	mg/kg	0.14	NB
ORGANICS					
PCBs					
Aroclor 1016	SW3540A/3640A/8082	GC/ECD	ug/kg	12.0	NB
Aroclor 1221	SW3540A/3640A/8082	GC/ECD	ug/kg	13.0	NB
Aroclor 1232	SW3540A/3640A/8082	GC/ECD	ug/kg	17	NB
Aroclor 1242	SW3540A/3640A/8082	GC/ECD	ug/kg	23.0	NB
Aroclor 1248	SW3540A/3640A/8082	GC/ECD	ug/kg	12.0	NB
Aroclor 1254	SW3540A/3640A/8082	GC/ECD	ug/kg	14	NB
Aroclor 1260	SW3540A/3640A/8082	GC/ECD	ug/kg	14	NB

Laboratory Standard RLs

EA LABORATORIES

REPORTING LIMITS FOR SOIL SAMPLES [a]

Parameter	Units	Reporting Limit
Pesticides and PCBs GC/ECD - organochlorine compounds (SW846 3540/8082A)		
Aroclor 1016	ug/kg	33
Aroclor 1221	ug/kg	67
Aroclor 1232	ug/kg	33
Aroclor 1242	ug/kg	33
Aroclor 1248	ug/kg	33
Aroclor 1254	ug/kg	33
Aroclor 1260	ug/kg	33
Semivolatile organics HPLC - PAHs (SW846 3540A/8310)		
Acenaphthene	ug/kg	40
Acenaphthylene	ug/kg	70
Anthracene	ug/kg	5.0
Benzo[a]anthracene	ug/kg	2.0
Benzo[b]fluoranthene	ug/kg	2.0
Benzo[k]fluoranthene	ug/kg	2.0
Benzo[a]pyrene	ug/kg	2.0
Benzo[ghi]perylene	ug/kg	2.0
Chrysene	ug/kg	5.0
Dibenzo[a,h]anthracene	ug/kg	2.0
Fluoranthene	ug/kg	7.0
Fluorene	ug/kg	7.0
Indeno[1,2,3-cd]pyrene	ug/kg	2.0
Naphthalene	ug/kg	40
Phenanthrene	ug/kg	5.0
Pyrene	ug/kg	9.0
Volatile organics GC/MS - (SW846 5030A/8260B)		
Acetone	ug/kg	10
Benzene	ug/kg	5
Bromodichloromethane	ug/kg	5
Bromobenzene	ug/kg	5
Bromochloromethane	ug/kg	5
Bromoform	ug/kg	5
Bromomethane	ug/kg	5
n-Butylbenzene	ug/kg	5
sec-Butylbenzene	ug/kg	5
tert-Butylbenzene	ug/kg	5
2-Butanone	ug/kg	10
Carbon disulfide	ug/kg	5
Carbon tetrachloride	ug/kg	5
Chlorobenzene	ug/kg	5

(a) EA Laboratories has established *Reporting Limits* (RLs) as laboratory quantitation levels. These are the minimum concentrations reported within the method precision and accuracy, and are indicative of routine laboratory analyses in clean environmental matrices.

Updated: 09 SEP 97/nks
Supercedes: 13 JAN 97/amt

EA LABORATORIES

REPORTING LIMITS FOR SOIL SAMPLES [a]

Parameter	Units	Reporting Limit
Chloroethane	ug/kg	5
2-Chloroethylvinyl ether	ug/kg	10
Chloroform	ug/kg	5
Chloromethane	ug/kg	5
2-Chlorotoluene	ug/kg	5
4-Chlorotoluene	ug/kg	5
Dibromochloromethane	ug/kg	5
1,2-Dibromo-3-chloropropane	ug/kg	5
1,2-Dibromoethane	ug/kg	5
Dibromomethane	ug/kg	5
1,2-Dichlorobenzene	ug/kg	5
1,3-Dichlorobenzene	ug/kg	5
1,4-Dichlorobenzene	ug/kg	5
1 Dichlorodifluoromethane	ug/kg	5
1,1-Dichloroethane	ug/kg	5
1,2-Dichloroethane	ug/kg	5
1,1-Dichloroethene	ug/kg	5
cis-1,2-Dichloroethene	ug/kg	5
trans-1,2-Dichloroethene	ug/kg	5
1,2-Dichloropropane	ug/kg	5
1,3-Dichloropropane	ug/kg	5
2,2-Dichloropropane	ug/kg	5
1,1-Dichloropropene	ug/kg	5
cis-1,3-Dichloropropene	ug/kg	5
trans-1,3-Dichloropropene	ug/kg	5
Diisopropyl ether	ug/kg	5
Ethylbenzene	ug/kg	5
Hexachlorobutadiene	ug/kg	5
2-Hexanone	ug/kg	10
Isopropylbenzene	ug/kg	5
4-Isopropyltoluene	ug/kg	5
Methylene chloride	ug/kg	5
Methyl tert-butyl ether (MTBE)	ug/kg	5
4-Methyl-2-pentanone	ug/kg	10
Naphthalene	ug/kg	5
n-Propylbenzene	ug/kg	5
Styrene	ug/kg	5
1,1,1,2-Tetrachloroethane	ug/kg	5
1,1,2,2-Tetrachloroethane	ug/kg	5
Tetrachloroethene	ug/kg	5
Toluene	ug/kg	5
1,2,3-Trichlorobenzene	ug/kg	5

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Updated: 09 SEP 97/nks
Supercedes: 13 JAN 97/amt

Parameter	Units	Reporting Limit
1,2,4-Trichlorobenzene	ug/kg	5
1,1,1-Trichloroethane	ug/kg	5
1,1,2-Trichloroethane	ug/kg	5
Trichloroethene	ug/kg	5
Trichlorofluoromethane	ug/kg	5
1,2,3-Trichloropropane	ug/kg	5
1,2,4-Trimethylbenzene	ug/kg	5
1,3,5-Trimethylbenzene	ug/kg	5
Vinyl acetate	ug/kg	10
Vinyl chloride	ug/kg	5
m-Xylene	ug/kg	5
m&p-Xylene	ug/kg	5
o&p-Xylene	ug/kg	5
o-Xylene	ug/kg	5
Inorganic nonmetals		
Cyanide	mg/kg	0.2
TOC (SW846 9060)	mg/kg	6000
Metals - Cold Vapor		
Mercury	mg/kg	0.10
Metals - ICP(SW-846 3050A/6010A)		
Aluminum	mg/kg	20.0
Antimony	mg/kg	6.0
Arsenic	mg/kg	10.0
Barium	mg/kg	20.0
Beryllium	mg/kg	0.50
Cadmium	mg/kg	0.50
Calcium	mg/kg	100
Chromium	mg/kg	1.0
Cobalt	mg/kg	5.0
Copper	mg/kg	1.0
Iron	mg/kg	10.0
Lead	mg/kg	10.0
Magnesium	mg/kg	100.0
Manganese	mg/kg	1.5
Nickel	mg/kg	4.0
Potassium	mg/kg	100
Selenium	mg/kg	10.0
Silver	mg/kg	1.0
Sodium	mg/kg	100

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Updated: 09 SEP 97/nks
Supercedes: 13 JAN 97/amt

REPORTING LIMITS FOR SOIL SAMPLES [a]

Parameter	Units	Reporting Limit
Thallium	mg/kg	10.0
Vanadium	mg/kg	5.0
Zinc	mg/kg	2.0

(a) EA Laboratories has established *Reporting Limits* (RLs) as laboratory quantitation levels. These are the minimum concentrations reported within the method precision and accuracy, and are indicative of routine laboratory analyses in clean environmental matrices.

Updated: 09 SEP 97/nks
Supercedes: 13 JAN 97/amt

EA LABORATORIES

REPORTING LIMITS FOR WATER SAMPLES [a]

Parameter	Units	Reporting Limit
Pesticides and PCBs GC/ECD - organochlorine compounds (SW846 3520A/8082)		
Aroclor 1016	ug/L	1.0
Aroclor 1221	ug/L	2.0
Aroclor 1232	ug/L	1.0
Aroclor 1242	ug/L	1.0
Aroclor 1248	ug/L	1.0
Aroclor 1254	ug/L	1.0
Aroclor 1260	ug/L	1.0
Semivolatile organics HPLC - PAHs (SW846 3520A/8310)		
Acenaphthene	ug/L	1.0
Acenaphthylene	ug/L	2.0
Anthracene	ug/L	0.20
Benzo[a]anthracene	ug/L	0.10
Benzo[b]fluoranthene	ug/L	0.15
Benzo[k]fluoranthene	ug/L	0.10
Benzo[a]pyrene	ug/L	0.10
Benzo[ghi]perylene	ug/L	0.20
Chrysene	ug/L	0.10
Dibenzo[a,h]anthracene	ug/L	0.20
Fluoranthene	ug/L	0.20
Fluorene	ug/L	0.20
Indeno[1,2,3-cd]pyrene	ug/L	0.10
Naphthalene	ug/L	1.0
Phenanthrene	ug/L	0.20
Pyrene	ug/L	0.20
Volatile organics GC/MS - 5 mL purge (SW846 5030A/8260B) - including Appendix IX compounds		
Acetone	ug/L	10
Acetonitrile	ug/L	100
Acrolein	ug/L	50
Acrylonitrile	ug/L	50
Allyl chloride	ug/L	5
Benzene	ug/L	5
Bromodichloromethane	ug/L	5
Bromobenzene	ug/L	5
Bromochloromethane	ug/L	5
Bromoform	ug/L	5
Bromomethane	ug/L	5
n-Butylbenzene	ug/L	5
sec-Butylbenzene	ug/L	5
tert-Butylbenzene	ug/L	5
2-Butanone	ug/L	10
Carbon disulfide	ug/L	5
Carbon tetrachloride	ug/L	5
Chlorobenzene	ug/L	5
Chloroethane	ug/L	5
2-Chloroethylvinyl ether	ug/L	10

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Updated: 09SEP97/nks
Supercedes:April 1, 1996/HJG

EA LABORATORIES

REPORTING LIMITS FOR WATER SAMPLES [a]

Parameter	Units	Reporting Limit
Chloroform	ug/L	5
Chloromethane	ug/L	5
Chloroprene	ug/L	10
2-Chlorotoluene	ug/L	5
4-Chlorotoluene	ug/L	5
Dibromochloromethane	ug/L	5
1,2-Dibromo-3-chloropropane	ug/L	5
1,2-Dibromoethane	ug/L	5
Dibromomethane	ug/L	5
1,2-Dichlorobenzene	ug/L	5
1,3-Dichlorobenzene	ug/L	5
1,4-Dichlorobenzene	ug/L	5
trans-1,4-dichloro-2-butene	ug/L	100
Dichlorodifluoromethane	ug/L	5
1,1-Dichloroethane	ug/L	5
1,2-Dichloroethane	ug/L	5
1,1-Dichloroethene	ug/L	5
cis-1,2-Dichloroethene	ug/L	5
trans-1,2-Dichloroethene	ug/L	5
1,2-Dichloropropane	ug/L	5
1,3-Dichloropropane	ug/L	5
2,2-Dichloropropane	ug/L	5
1,1-Dichloropropene	ug/L	5
cis-1,3-Dichloropropene	ug/L	5
trans-1,3-Dichloropropene	ug/L	5
Diisopropyl ether	ug/L	5
Ethylbenzene	ug/L	5
Ethyl methacrylate	ug/L	5
Hexachlorobutadiene	ug/L	5
2-Hexanone	ug/L	10
Isobutyl alcohol	ug/L	100
Isopropylbenzene	ug/L	5
4-Isopropyltoluene	ug/L	5
Methacrylonitrile	ug/L	100
Methylene chloride	ug/L	5
Methyl iodide	ug/L	5
Methyl methylacrylate	ug/L	5
Methyl tert-butyl ether (MTBE)	ug/L	5
4-Methyl-2-pentanone	ug/L	10
Naphthalene	ug/L	5
Pentachloroethane	ug/L	10
Propionitrile	ug/L	100
n-Propylbenzene	ug/L	5
Styrene	ug/L	5
1,1,1,2-Tetrachloroethane	ug/L	5
1,1,2,2-Tetrachloroethane	ug/L	5
Tetrachloroethene	ug/L	5

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Updated: 09SEP97/nks
Supercedes:April 1, 1996/HJG

EA LABORATORIES

REPORTING LIMITS FOR WATER SAMPLES [a]

Parameter	Units	Reporting Limit
Toluene	ug/L	5
1,2,3-Trichlorobenzene	ug/L	5
1,2,4-Trichlorobenzene	ug/L	5
1,1,1-Trichloroethane	ug/L	5
1,1,2-Trichloroethane	ug/L	5
Trichloroethene	ug/L	5
Trichlorofluoromethane	ug/L	5
1,2,3-Trichloropropane	ug/L	5
1,2,4-Trimethylbenzene	ug/L	5
1,3,5-Trimethylbenzene	ug/L	5
Vinyl acetate	ug/L	10
Vinyl chloride	ug/L	5
m-Xylene	ug/L	5
m&p-Xylene	ug/L	5
o&p-Xylene	ug/L	5
o-Xylene	ug/L	5
Inorganic Nonmetals/General Organics		
Cyanide (EPA 335.2)	mg/L	0.01
Metals - Cold Vapor (SW846 7470)		
Mercury	ug/L	0.20
Metals - ICP (SW846 3010A/6010A)		
Aluminum	ug/L	200
Antimony	ug/L	60.0
Arsenic	ug/L	100
Barium	ug/L	200
Beryllium	ug/L	5.0
Cadmium	ug/L	5.0
Calcium	ug/L	1000
Chromium	ug/L	10.0
Cobalt	ug/L	50.0
Copper	ug/L	10.0
Iron	ug/L	100
Magnesium	ug/L	1000
Manganese	ug/L	15.0
Nickel	ug/L	40.0
Phosphorus	ug/L	100
Potassium	ug/L	1000
Selenium	ug/L	100
Sodium	ug/L	1000
Strontium	ug/L	100
Thallium	ug/L	100
Vanadium	ug/L	50.0
Zinc	ug/L	20.0

(a) EA Laboratories has established *Reporting Limits* (RLs) as laboratory quantitation levels. These are the minimum concentrations reported within the method precision and accuracy, and are indicative of routine laboratory analyses in clean environmental matrices.

Updated: 09SEP97/nks
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EA LABORATORIES

REPORTING LIMITS FOR WATER SAMPLES [a]

Parameter	Units	Reporting Limit
Metals - TRACE ICP (SW846 3010A/6010A)		
Lead	ug/L	3.0

(a) EA Laboratories has established *Reporting Limits* (RLs) as laboratory quantitation levels. These are the minimum concentrations reported within the method precision and accuracy, and are indicative of routine laboratory analyses in clean environmental matrices.

Updated: 09SEP97/nks
Supercedes:April 1, 1996/HJG

Laboratory Standard MDLs

EA LABORATORIES

METHOD DETECTION LIMITS (MDLS) FOR WATER SAMPLES

Parameter	Units	MDL (a)	Date
Pesticides and PCBs GC/ECD - organochlorine compounds (SW846 3520A/8082)			
Aroclor 1016	ug/L	0.62	DEC 97
Aroclor 1221	ug/L	0.61	DEC 97
Aroclor 1232	ug/L	0.76	DEC 97
Aroclor 1242	ug/L	0.54	DEC 97
Aroclor 1248	ug/L	0.54	DEC 97
Aroclor 1254	ug/L	0.68	DEC 97
Aroclor 1260	ug/L	0.14	DEC 97
Volatile Organics GC/MS -5 mL purge - Capillary Column (SW846 5030A/8260B)			
Acetone	ug/L	3.3	18 FEB 98
Acetonitrile	ug/L	30	18 FEB 98
Acrolein	ug/L	18	16 JAN 97
Allyl Chloride	ug/L	0.9	18 FEB 98
Acrylonitrile	ug/L	12	18 FEB 98
Benzene	ug/L	0.5	18 FEB 98
Bromobenzene	ug/L	0.4	18 FEB 98
Bromochloromethane	ug/L	0.5	18 FEB 98
Bromodichloromethane	ug/L	0.4	18 FEB 98
Bromofluorobenzene	ug/L	2.8	18 FEB 98
Bromoform	ug/L	0.4	18 FEB 98
Bromomethane	ug/L	1.7	18 FEB 98
2-Butanone	ug/L	2.3	18 FEB 98
sec-Butylbenzene	ug/L	0.7	18 FEB 98
n-Butylbenzene	ug/L	0.7	18 FEB 98
tert-Butylbenzene	ug/L	0.6	18 FEB 98
Carbon Disulfide	ug/L	0.8	18 FEB 98
Carbon tetrachloride	ug/L	1.2	18 FEB 98
Chlorobenzene	ug/L	0.4	18 FEB 98
Chlorodibromomethane	ug/L	0.4	18 FEB 98
Chloroethane	ug/L	1.8	18 FEB 98
2-Chloroethylvinyl ether	ug/L	2.3	18 FEB 98
Chloroform	ug/L	0.7	18 FEB 98
1-Chlorohexane	ug/L	0.7	18 FEB 98
Chloromethane	ug/L	1.1	18 FEB 98
Chloroprene	ug/L	1.0	11 DEC 94
2-Chlorotoluene	ug/L	0.4	18 FEB 98
4-Chlorotoluene	ug/L	0.6	18 FEB 98
Dibromochloromethane	ug/L	3.5	18 FEB 98
1,2-Dibromo-3-chloropropane	ug/L	0.9	18 FEB 98
1,2-Dibromoethane (EDB)	ug/L	0.4	18 FEB 98
Dibromofluoromethane	ug/L	3.5	18 FEB 98
Dibromomethane	ug/L	0.6	18 FEB 98
1,2-Dichlorobenzene	ug/L	0.4	18 FEB 98
1,3-Dichlorobenzene	ug/L	0.5	18 FEB 98
1,4-Dichlorobenzene	ug/L	0.4	18 FEB 98

(a) Determined according to 40 CFR 136 Appendix B.

Updated: 5 JUNE/mjb
Supercedes: 31MAR98/mjb

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METHOD DETECTION LIMITS (MDLS) FOR WATER SAMPLES

Parameter	Units	MDL (a)	Date
trans 1,4-dichloro2-Butene	ug/L	4.4	18 FEB 98
Dichlorodifluoromethane	ug/L	1.0	18 FEB 98
1,1-Dichloroethane	ug/L	0.8	18 FEB 98
1,2-Dichloroethane	ug/L	0.4	18 FEB 98
1,2-Dichloroehtane-d_4	ug/L	2.3	18 FEB 98
1,1-Dichloroethene	ug/L	1.0	18 FEB 98
cis-1,2-Dichloroethene	ug/L	0.7	18 FEB 98
trans-1,2-Dichloroethene	ug/L	0.7	18 FEB 98
1,2-Dichloropropane	ug/L	0.5	18 FEB 98
1,3-Dichloropropane	ug/L	0.5	18 FEB 98
2,2-Dichloropropane	ug/L	2.0	18 FEB 98
1,1-Dichloropropene	ug/L	0.6	18 FEB 98
cis-1,3-Dichloropropene	ug/L	0.4	18 FEB 98
trans-1,3-Dichloropropene	ug/L	0.4	18 FEB 98
Diisopropyl ether	ug/L	0.6	18 FEB 98
Ethylbenzene	ug/L	0.5	18 FEB 98
Ethyl Acetate	ug/L	2.6	18 FEB 98
Ethyl Ether	ug/L	1.3	18 FEB 98
Ethyl Methacrylate	ug/L	0.8	18 FEB 98
Hexachlorobutadiene	ug/L	0.7	18 FEB 98
2-Hexanone	ug/L	2.3	18 FEB 98
Iodomethane	ug/L	2	16 JAN 97
Isobutyl Alcohol	ug/L	38	18 FEB 98
Isopropylbenzene	ug/L	0.6	18 FEB 98
p-Isopropyltoluene	ug/L	0.6	18 FEB 98
Methacrylonitrile	ug/L	29	18 FEB 98
Methylene chloride	ug/L	0.7	18 FEB 98
Methyl Methacrylate	ug/L	0.5	18 FEB 98
4-Methyl-2-Pentanone	ug/L	2.0	18 FEB 98
Methyl t-butyl ether	ug/L	1.9	18 FEB 98
Naphthalene	ug/L	1.1	18 FEB 98
2-Nitropropane	ug/L	3.4	18 FEB 98
Pentachloroethane	ug/L	0.4	18 FEB 98
Propionitrile	ug/L	27	18 FEB 98
n-Propylbenzene	ug/L	0.5	18 FEB 98
Styrene	ug/L	0.4	18 FEB 98
1,1,1,2-Tetrachloroethane	ug/L	0.4	18 FEB 98
1,1,2,2-Tetrachloroethane	ug/L	0.9	18 FEB 98
Tetrachloroethene	ug/L	0.6	18 FEB 98
Tetrahydrofuran	ug/L	7.6	18 FEB 98
Toluene-d_8	ug/L	2.5	18 FEB 98
Toluene	ug/L	0.4	18 FEB 98
1,2,3-Trichlorobenzene	ug/L	0.7	18 FEB 98
1,2,4-Trichlorobenzene	ug/L	0.8	18 FEB 98
1,1,1-Trichloroethane	ug/L	1.6	18 FEB 98
1,1,2-Trichloroethane	ug/L	0.5	18 FEB 98

(a) Determined according to 40 CFR 136 Appendix B.

Updated: 5 JUNE/mjb
Supercedes: 31MAR98/mjb

EA LABORATORIES

METHOD DETECTION LIMITS (MDLS) FOR WATER SAMPLES

Parameter	Units	MDL (a)	Date
Trichloroethene	ug/L	0.5	18 FEB 98
Trichlorofluoromethane	ug/L	1.6	18 FEB 98
1,2,3-Trichloropropane	ug/L	0.5	18 FEB 98
1,1,2-Trichlorotrifluoroethane	ug/L	1.0	18 FEB 98
1,2,3-Trimethylbenzene	ug/L	0.4	18 FEB 98
1,2,4-Trimethylbenzene	ug/L	0.4	18 FEB 98
1,3,5-Trimethylbenzene	ug/L	0.4	18 FEB 98
Vinyl chloride	ug/L	0.9	18 FEB 98
Vinyl Acetate	ug/L	1.4	18 FEB 98
m&p-Xylenes	ug/L	0.8	18 FEB 98
o-Xylene	ug/L	0.4	18 FEB 98
Xylenes	ug/L	1.3	18 FEB 98
Semivolatile organics HPLC - PAHs (SW846 3520A/8310)			
Acenaphthene	ug/L	0.08	30 JUL 97
Acenaphthylene	ug/L	0.53	30 JUL 97
Anthracene	ug/L	0.024	30 JUL 97
Benzo[a]anthracene	ug/L	0.017	30 JUL 97
Benzo[b]fluoranthene	ug/L	0.027	30 JUL 97
Benzo[k]fluoranthene	ug/L	0.008	30 JUL 97
Benzo[a]pyrene	ug/L	0.021	30 JUL 97
Benzo[ghi]perylene	ug/L	0.028	30 JUL 97
Chrysene	ug/L	0.014	30 JUL 97
Dibenzo[a,h]anthracene	ug/L	0.022	30 JUL 97
Fluoranthene	ug/L	0.047	30 JUL 97
Fluorene	ug/L	0.041	30 JUL 97
Indeno[1,2,3-cd]pyrene	ug/L	0.042	30 JUL 97
1-Methylnaphthalene	ug/L	0.10	30 JUL 97
2-Methylnaphthalene	ug/L	0.13	30 JUL 97
Naphthalene	ug/L	0.15	30 JUL 97
Phenanthrene	ug/L	0.007	30 JUL 97
Pyrene	ug/L	0.016	30 JUL 97
Inorganic nonmetals/general organics			
Cyanide (EAL-M-9012)	mg/L	0.007	25 JUL 97
Metals - Cold Vapor (SW846 7470)			
Mercury	ug/L	0.10	24 APR 97
Metals - ICP (SW846 3010A/6010A)			
Aluminum	ug/L	65	11 JUL 97
Antimony	ug/L	24	11 JUL 97
Arsenic	ug/L	29	11 JUL 97
Barium	ug/L	8.0	11 JUL 97
Beryllium	ug/L	1.0	11 JUL 97
Cadmium	ug/L	3.0	11 JUL 97

(a) Determined according to 40 CFR 136 Appendix B.

Updated: 5 JUNE/mjb
Supercedes: 31MAR98/mjb

EA LABORATORIES

METHOD DETECTION LIMITS (MDLS) FOR WATER SAMPLES

Parameter	Units	MDL (a)	Date
Calcium	ug/L	39	11 JUL 97
Chromium	ug/L	4.0	11 JUL 97
Cobalt	ug/L	7.0	11 JUL 97
Copper	ug/L	5.0	11 JUL 97
Iron	ug/L	53	11 JUL 97
Magnesium	ug/L	35	11 JUL 97
Manganese	ug/L	2.0	11 JUL 97
Nickel	ug/L	5.0	11 JUL 97
Potassium	ug/L	71	11 JUL 97
Selenium	ug/L	44	11 JUL 97
Silver	ug/L	4.0	11 JUL 97
Sodium	ug/L	81	11 JUL 97
Thallium	ug/L	56	11 JUL 97
Vanadium	ug/L	3.0	11 JUL 97
Zinc	ug/L	12.0	11 JUL 97
Metals-Trace ICP (SW846 3010A/6010A)			
Lead	ug/L	1.0	11 JUL 97
Silver	ug/L	1.0	11 JUL 97

(a) Determined according to 40 CFR 136 Appendix B.

Updated: 5 JUNE/mjb
Supercedes: 31MAR98/mjb

EA LABORATORIES

METHOD DETECTION LIMITS (MDLS) FOR SOIL SAMPLES [a]

Parameter	Units	MDL	Date
Pesticides and PCBs GC/ECD - organochlorine compounds (SW846 3450A//8082)			
Aroclor 1016	ug/kg	9.3	DEC 97
Aroclor 1221	ug/kg	7.4	DEC 97
Aroclor 1232	ug/kg	10	DEC 97
Aroclor 1242	ug/kg	4.7	DEC 97
Aroclor 1248	ug/kg	8.4	DEC 97
Aroclor 1254	ug/kg	3.0	DEC 97
Aroclor 1260	ug/kg	8.0	DEC 97
Semivolatile organics HPLC - PAHs (SW846 3540A/8310)-30 grams - Soil			
Acenaphthene	ug/kg	11	30 JUL 97
Acenaphthylene	ug/kg	51	30 JUL 97
Anthracene	ug/kg	0.68	30 JUL 97
Benzo[a]anthracene	ug/kg	0.52	30 JUL 97
Benzo[b]fluoranthene	ug/kg	1.1	30 JUL 97
Benzo[k]fluoranthene	ug/kg	0.44	30 JUL 97
Benzo[a]pyrene	ug/kg	1.8	30 JUL 97
Benzo[ghi]perylene	ug/kg	1.3	30 JUL 97
Chrysene	ug/kg	0.52	30 JUL 97
Dibenzo[a,h]anthracene	ug/kg	1.4	30 JUL 97
Fluoranthene	ug/kg	0.84	30 JUL 97
Fluorene	ug/kg	2.0	30 JUL 97
Indeno[1,2,3-cd]pyrene	ug/kg	0.93	30 JUL 97
Naphthalene	ug/kg	7.4	30 JUL 97
Phenanthrene	ug/kg	0.65	30 JUL 97
Pyrene	ug/kg	0.48	30 JUL 97
1-Methylnaphthalene	ug/kg	10.0	30 JUL 97
2-Methylnaphthalene	ug/kg	13	30 JUL 97
Volatile Organics GC/MS -Capillary Column (SW846 5030A/8260B)			
Acetone	ug/kg	3	27 FEB 97
Acetonitrile	ug/kg	18	27 FEB 97
Acrolein	ug/kg	12	27 FEB 97
Allyl Chloride	ug/kg	3	27 FEB 97
Acrylonitrile	ug/kg	14	27 FEB 97
Benzene	ug/kg	1	27 FEB 97
Bromobenzene	ug/kg	1	27 FEB 97
Bromochloromethane	ug/kg	0.8	27 FEB 97
Bromodichloromethane	ug/kg	0.9	27 FEB 97
Bromofluorobenzene	ug/kg	0.8	27 FEB 97
Bromoform	ug/kg	1	27 FEB 97
Bromomethane	ug/kg	3	27 FEB 97
2-Butanone	ug/kg	4	27 FEB 97
sec-Butylbenzene	ug/kg	1	27 FEB 97

(a) Determined according to the procedure specified in 40 CFR 136, Appendix B.
(c) Quantitated using DB-5 column due to coelution with interferent on DB-35.
(d) Quantitated using DB-5 column due to coelution on DB-35.

EA LABORATORIES

METHOD DETECTION LIMITS (MDLS) FOR SOIL SAMPLES [a]

Parameter	Units	MDL	Date
n-Butylbenzene	ug/kg	1	27 FEB 97
tert-Butylbenzene	ug/kg	0.9	27 FEB 97
Carbon Disulfide	ug/kg	2	27 FEB 97
Carbon tetrachloride	ug/kg	1	27 FEB 97
Chlorobenzene	ug/kg	0.9	27 FEB 97
Chloroethane	ug/kg	1	27 FEB 97
2-Chloroethylvinyl ether	ug/kg	4	27 FEB 97
Chloroform	ug/kg	1	27 FEB 97
1-Chlorohexane	ug/kg	2	27 FEB 97
Chloromethane	ug/kg	2	27 FEB 97
Chloroprene	ug/kg	1	09 JAN 95
2-Chlorotoluene	ug/kg	0.9	27 FEB 97
4-Chlorotoluene	ug/kg	2	27 FEB 97
Dibromochloromethane	ug/kg	0.8	27 FEB 97
1,2-Dibromo-3-chloropropane (DBCP)	ug/kg	2	27 FEB 97
1,2-Dibromoethane (EDB)	ug/kg	0.9	27 FEB 97
Dibromofluoromethane	ug/kg	1	27 FEB 97
Dibromomethane	ug/kg	1	27 FEB 97
1,2-Dichlorobenzene	ug/kg	0.9	27 FEB 97
1,3-Dichlorobenzene	ug/kg	1	27 FEB 97
1,4-Dichlorobenzene	ug/kg	1	27 FEB 97
trans 1,4-dichloro2-Butene	ug/kg	4	27 FEB 97
Dichlorodifluoromethane	ug/kg	2	27 FEB 97
1,1-Dichloroethane	ug/kg	1	27 FEB 97
1,2-Dichloroethane	ug/kg	1	27 FEB 97
1,2-Dichloroethane-d4	ug/kg	0.8	27 FEB 97
1,1-Dichloroethene	ug/kg	2	27 FEB 97
cis-1,2-Dichloroethene	ug/kg	1	27 FEB 97
trans-1,2-Dichloroethene	ug/kg	2	27 FEB 97
1,2-Dichloroethene, total	ug/kg	2	27 FEB 97
1,2-Dichloropropane	ug/kg	4	27 FEB 97
1,3-Dichloropropane	ug/kg	1	27 FEB 97
2,2-Dichloropropane	ug/kg	2	27 FEB 97
1,1-Dichloropropene	ug/kg	1	27 FEB 97
cis-1,3-Dichloropropene	ug/kg	0.8	27 FEB 97
trans-1,3-Dichloropropene	ug/kg	0.8	27 FEB 97
Diisopropyl ether	ug/kg	2	27 FEB 97
Ethylbenzene	ug/kg	2	27 FEB 97
Ethyl Acetate	ug/kg	2	27 FEB 97
Ethyl Ether	ug/kg	2	27 FEB 97
Ethyl Methacrylate	ug/kg	2	27 FEB 97
Hexachlorobutadiene	ug/kg	1	27 FEB 97
2-Hexanone	ug/kg	4	27 FEB 97
Iodomethane	ug/kg	0.8	27 FEB 97

(a) Determined according to the procedure specified in 40 CFR 136, Appendix B.
(c) Quantitated using DB-5 column due to coelution with interferent on DB-35.
(d) Quantitated using DB-5 column due to coelution on DB-35.

EA LABORATORIES

METHOD DETECTION LIMITS (MDLS) FOR SOIL SAMPLES [a]

Parameter	Units	MDL	Date
Isobutyl Alchol	ug/kg	40	27 FEB 97
Isopropylbenzene	ug/kg	1	27 FEB 97
p-Isopropyltoluene	ug/kg	0.9	27 FEB 97
Methacrylonitrile	ug/kg	3	27 FEB 97
Methylene chloride	ug/kg	1	27 FEB 97
Methyl Methacrylate	ug/kg	2	27 FEB 97
4-Methyl-2-Pentanone	ug/kg	5	27 FEB 97
Methyl t-butyl ether	ug/kg	2	27 FEB 97
Naphthalene	ug/kg	2	27 FEB 97
2-Nitropropane	ug/kg	3	27 FEB 97
Pentachloroethane	ug/kg	2	27 FEB 97
Propionitrile	ug/kg	20	27 FEB 97
n-Propylbenzene	ug/kg	1	27 FEB 97
Styrene	ug/kg	0.8	27 FEB 97
1,1,1,2-Tetrachloroethane	ug/kg	1	27 FEB 97
1,1,2,2-Tetrachloroethane	ug/kg	1	27 FEB 97
Tetrachloroethene	ug/kg	2	27 FEB 97
Tetrahydrofuran	ug/kg	4	27 FEB 97
Toluene-d_8	ug/kg	1	27 FEB 97
Toluene	ug/kg	1	27 FEB 97
1,2,3-Trichlorobenzene	ug/kg	1	27 FEB 97
1,2,4-Trichlorobenzene	ug/kg	1	27 FEB 97
1,1,1-Trichloroethane	ug/kg	1	27 FEB 97
1,1,2-Trichloroethane	ug/kg	1	27 FEB 97
Trichloroethene	ug/kg	0.9	27 FEB 97
Trichlorofluoromethane	ug/kg	2	27 FEB 97
1,2,3-Trichloropropane	ug/kg	1	27 FEB 97
1,1,2-Trichlorotrifluoroethane	ug/kg	3	27 FEB 97
1,2,3-Trimethylbenzene	ug/kg	2	27 FEB 97
1,2,4-Trimethylbenzene	ug/kg	1	27 FEB 97
1,3,5-Trimethylbenzene	ug/kg	1	27 FEB 97
Vinyl chloride	ug/kg	2	27 FEB 97
Vinyl Acetate	ug/kg	1	27 FEB 97
m&p-Xylenes	ug/kg	2	27 FEB 97
o-Xylene	ug/kg	1	27 FEB 97
Xylenes (total)	ug/kg	2	27 FEB 97
Inorganic nonmetals			
Chromium, hexavalent (SW846 7196A)	mg/kg	0.05	
Cyanide (SW846 9012)	mg/kg	0.19	03 MAR 98
TOC (SW846 9060)	mg/kg	4880	16 JUL 96
Metals - Cold Vapor (SW846 7471)			
Mercury	mg/kg	0.10	16 MAY 96

(a) Determined according to the procedure specified in 40 CFR 136, Appendix B.
(c) Quantitated using DB-5 column due to coelution with interferent on DB-35.
(d) Quantitated using DB-5 column due to coelution on DB-35.

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Updated: 03FEB98/ams
Supercedes: 09SEP97/ams

EA LABORATORIES

METHOD DETECTION LIMITS (MDLS) FOR SOIL SAMPLES [a]

Parameter	Units	MDL	Date
Metals - ICP (SW846 3050A/6010A)			
Aluminum	mg/kg	6.5	11 JUL 97
Antimony	mg/kg	2.4	11 JUL 97
Arsenic	mg/kg	2.9	11 JUL 97
Barium	mg/kg	0.8	11 JUL 97
Beryllium	mg/kg	0.1	11 JUL 97
Cadmium	mg/kg	0.3	11 JUL 97
Calcium	mg/kg	3.9	11 JUL 97
Chromium	mg/kg	0.4	11 JUL 97
Cobalt	mg/kg	0.7	11 JUL 97
Copper	mg/kg	0.5	11 JUL 97
Iron	mg/kg	5.3	11 JUL 97
Lead	mg/kg	5.1	11 JUL 97
Magnesium	mg/kg	3.5	11 JUL 97
Manganese	mg/kg	0.2	11 JUL 97
Nickel	mg/kg	0.5	11 JUL 97
Potassium	mg/kg	7.1	11 JUL 97
Selenium	mg/kg	4.4	11 JUL 97
Silver	mg/kg	0.4	11 JUL 97
Sodium	mg/kg	8.1	11 JUL 97
Thallium	mg/kg	5.6	11 JUL 97
Vanadium	mg/kg	0.3	11 JUL 97
Zinc	mg/kg	1.2	11 JUL 97
Metals - Trace ICP (SW846 3050A/6010A)			
Lead	mg/kg	0.1	11 JUL 97

(a) Determined according to the procedure specified in 40 CFR 136, Appendix B.
(c) Quantitated using DB-5 column due to coelution with interferent on DB-35.
(d) Quantitated using DB-5 column due to coelution on DB-35.

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Updated: 03FEB98/ams
Supercedes: 09SEP97/ams

EA LABORATORIES

METHOD DETECTION LIMITS (MDLS) FOR TISSUE SAMPLES [a]

Parameter	Units	MDL [b]	Date
Pesticides and PCBs - GC/ECD organochlorine compounds (SW846 3540/8081/8082) - Clam tissue			
Aroclor 1016	ug/kg	12	10 FEB 98
Aroclor 1221	ug/kg	13	10 FEB 98
Aroclor 1232	ug/kg	17	10 FEB 98
Aroclor 1242	ug/kg	23	24 JUN 98
Aroclor 1248	ug/kg	12	10 FEB 98
Aroclor 1254	ug/kg	14	10 FEB 98
Aroclor 1260	ug/kg	14	10 FEB 98
Metals - Cold Vapor (SW846 7471) - Clam tissue			
Mercury	mg/kg	0.14	05 FEB 98
Metals - Furnace (SW846 3050/7000 Series) - Clam tissue			
Selenium	mg/kg	0.9	05 FEB 98
Thallium	mg/kg	0.3	05 FEB 98
Metals - ICP (SW846 3050/6010) - Clam Tissue			
Aluminum	mg/kg	11	05 FEB 98
Barium	mg/kg	12	05 FEB 98
Beryllium	mg/kg	0.3	05 FEB 98
Calcium	mg/kg	191	05 FEB 98
Chromium	mg/kg	1.4	05 FEB 98
Cobalt	mg/kg	3.1	05 FEB 98
Copper	mg/kg	1.5	05 FEB 98
Iron	mg/kg	16	05 FEB 98
Magnesium	mg/kg	69	05 FEB 98
Manganese	mg/kg	2.9	05 FEB 98
Nickel	mg/kg	3.0	05 FEB 98
Potassium	mg/kg	579	05 FEB 98
Sodium	mg/kg	1058	05 FEB 98
Vanadium	mg/kg	2.8	05 FEB 98
Zinc	mg/kg	9.1	05 FEB 98
Metals-ICP(Trace) - Clam Tissue			
Antimony	mg/kg	0.9	05 FEB 98
Arsenic	mg/kg	0.8	05 FEB 98
Cadmium	mg/kg	0.04	05 FEB 98
Lead	mg/kg	0.23	17 MAR 98
Silver	mg/kg	0.27	05 FEB 98

(a) Determined by according 40 CFR 136 Appendix B.
(b) MDLs are given on a wet weight basis.
(c) Coplanar PCB congener.

Updated: 12 JUN 98/AMS
Supercedes: 30 APR 97/ams

Standard Operating Procedures

Volatiles Prep
SW 5030

EA Engineering, Science, and Technology, Inc.

EA Laboratories

Method

Number: 5030 **Rev. No.:** 0

Title: Sample Preparation of Purge and Trap Volatiles

Prepared By: D.C. Miser, Volatile Group Leader — 24 August 1995

Revised By: Shelly Blough for Dennis Miser — 10/11/96
D.C.Miser, Volatile Group Leader — Date

Approved By: M.M. Uhlfelder — 10/11/96
M.M. Uhlfelder, Quality Services Manager — Date

Approved By: — 10/10/96
P.A. Christopher, Operations Manager — Date

WHEN STAMPED IN RED, THIS IS A CONTROLLED COPY. IF STAMP IS NOT RED, VERIFY THAT YOU ARE USING THE CURRENT REVISION BEFORE PROCEEDING WITH WORK UNDER THIS PROCEDURE

EA Engineering, Science, and Technology, Inc.

EA Laboratories

Method

Procedure No.: 5030 **Revision No.:** 0

Revisions		
Rev. No.	**Date of Release**	**Description**
0		Initial distribution. Naval Audit Findings requirements.

EA Engineering, Science, and Technology, Inc.

EA Laboratories

Method

Procedure No.: 5030 **Revision No.:** 0

Controlled Distribution

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Walt Miller	2
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Jeffrey Black	10
Carl Simmons	12
Phyllis Christopher	13
Sharon Albaugh	17
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Cathy Atkinson	19

1.0 SCOPE AND APPLICATION

1.1 This method describes sample preparation and extraction for the analysis of volatile organics by a purge-and-trap procedure. The gas chromatographic determinative steps are found in Methods 8010, 8015, 8020, 8021 and 8030. Although applicable to Methods 8240 and 8260, the purge-and-trap procedure is already incorporated into Methods 8240 and 8260.
1.2 Method 5030 can be used for most volatile organic compounds that have boiling points below 200°C and are insoluble or slightly soluble in water. Volatile water-soluble compounds can be included in this analytical technique; however, quantitation limits (by GC or GC/MS) are approximately ten times higher because of poor purging efficiency.
1.3 Water samples can be analyzed directly for volatile organic compounds by purge-and-trap extraction and gas chromatography. Higher concentrations of these analytes in water can be determined by direct injection of the sample into the chromatographic system.
1.4 This method also describes the preparation of water-miscible liquids, solids, wastes, and soils/sediments for analysis by the purge-and-trap procedure.
1.6 Any changes in the analytical procedures must be approved by the Operations Manager and the Quality Services Manager before samples can be analyzed.

2.0 SUMMARY OF METHOD

2.1 The purge-and-trap process: An inert gas is bubbled through the solution at ambient temperature, and the volatile components are efficiently transferred from the aqueous phase to the vapor phase. The vapor is swept through a sorbent column where the volatile components are adsorbed. After purging is completed, the sorbent column is heated and backflushed with inert gas to desorb the components onto a gas chromatographic column.
2.2 If the sample introduction technique in Section 2.1 is not applicable, a portion of the sample is dispersed in methanol to dissolve the volatile organic constituents. A portion of the methanol solution is combined with water in a specially designed purging chamber. It is then analyzed by purge-and-trap GC following the normal water method.

3.0 DEFINITIONS

Method and project specific quality control components requirements will be defined in each determinative method.

4.0 INTERFERENCES

4.1 Impurities in the purge gas, and from organic compounds out-gassing from the plumbing ahead of the trap, account for the majority of contamination problems. The analytical system must be demonstrated to be free from contamination under the conditions of the analysis by running laboratory reagent blanks. The use of non-TFE plastic coating, non-TFE thread sealants, or flow controllers with rubber components in the purging device should be avoided.
4.2 Samples can be contaminated by diffusion of volatile organics (particularly methylene chloride and fluorocarbons) through the septum seal of the sample vial during shipment and storage. A trip blank prepared from organic-free reagent water and carried through sampling and handling protocols serves as a check on such contamination.
4.3 Contamination by carryover can occur whenever high-concentration and low-concentration samples are analyzed sequentially. Whenever an unusually concentrated sample is analyzed, it should be followed by an analysis of organic-free reagent water to check for cross-contamination. The trap and other parts of the system are subject to contamination. Therefore, frequent bake-out and purging of the entire system may be required.

5.0 APPARATUS AND MATERIALS

5.1 Syringe - 5 ml, gas-tight with shutoff valve.
5.2 Purge-and-trap device: The purge-and-trap device consists of three separate pieces of equipment: the sample purger (TEKMAR, LCS 2000), the trap, and the desorber (model ALS 2016).

5.2.1 The recommended purging chamber is designed to accept 5 ml samples with a water column at least 3 cm deep. The gaseous headspace between the water column and the trap must have a total volume of less than 15 ml. The purge gas must pass through the water column as finely divided bubbles with a diameter of less than 3 mm at the origin. The purge gas must be introduced no more than 5 mm from the base of the water column.
5.2.3 The trap must be at least 25 cm long and have an inside diameter of at least 0.105 in. Vocarb 3000 used μL carbo pack B Carboxen 1000 & 1001. Trap condition at 270 C prior to analysis.
5.2.4 The desorber should be capable of rapidly heating the trap to 250°C for desorption. The polymer section of the trap should not be heated higher than 250°C, and the remaining sections should not exceed 260°C during bake-out mode.

5.3 Heater jackets used to heat soil/solid samples.

6.0 REAGENTS

6.1 Organic-free reagent water - All references to water in this method refer to organic-free reagent water.
6.2 Methanol, CH_3OH - Pesticide quality or equivalent. Store away from other solvents.

7.0 SAMPLE PRESERVATION, AND HANDLING

Samples should be stored in capped bottles, with minimum headspace, at 4°C or less and the pH measured and recorded after the aliquot has been removed for analysis.

8.0 PROCEDURE

8.1 Initial calibration: Prior to using this introduction technique for any GC method, the system must be calibrated. General calibration procedures are discussed in the determinative methods.

8.1.1 The samples and required quality control samples are prepared as described in the determinative methods.

8.2 On-going calibration: Refer to the determinative method for details on continuing calibration.
8.3 Water samples:

8.3.1 Screening of the sample prior to purge-and-trap analysis will provide guidance on whether sample dilution is necessary and will prevent contamination of the purge-and-trap system. With FID detector and headspace analyzer.
8.3.2 All samples and standard solutions must be allowed to warm to ambient temperature before analysis.
8.3.3 Daily GC calibration criteria as specified in the determinative method must be met before analyzing samples.
8.3.4 Adjust the purge gas flow rate to 40 mL/min.
8.3.5 Remove the plunger from a 5 mL syringe and attach a closed syringe valve. Open the sample or standard bottle, which has been allowed to come to ambient temperature, and carefully pour the sample into the syringe barrel to just short of overflowing. Replace the syringe plunger and compress the sample. Open the syringe valve and vent any residual air while adjusting the sample volume to 5.0 mL. This process of taking an aliquot destroys the validity of the liquid sample for future analysis; therefore, if there is only one VOA vial, the analyst should fill a second sample bottle at this time to protect against possible loss of sample integrity. This second sample is maintained only until such time when the analyst has determined that the first sample has been analyzed properly. Filling one 20 mL bottle would allow the use of only one syringe.
8.3.6 The following procedure is appropriate for diluting purgeable samples. All steps must be performed without delays until the diluted sample is in a gas-tight syringe.

8.3.6.1 Dilutions may be made in volumetric flasks (10 mL to 100 mL). Select the volumetric flask that will allow for the necessary dilution. Intermediate dilutions may be necessary for extremely large dilutions.
8.3.6.2 Calculate the approximate volume of organic-free reagent water to be added to the volumetric flask selected

and add slightly less than this quantity of organic-free reagent water to the flask.
8.3.6.3 Inject the proper aliquot of samples from the syringe prepared in Section 8.3.5 into the flask. Aliquots of less than 1 mL are not recommended. Dilute the sample to the mark with organic-free reagent water. Cap the flask, invert, and shake three times. Repeat the above procedure for additional dilutions.
8.3.6.4 Fill a 5 mL syringe with the diluted sample as in Section 8.3.5.

8.3.7 Add 1.25 μL of surrogate spiking spiking solution (found in each determinative method, Section 5.0). Matrix spiking solutions, if indicated, for 8015 gas add 1.25 μL to the sample at this time.
8.3.8 Attach the syringe-syringe valve assembly to the syringe valve on the purging device. Open the syringe valves and inject the sample into the purging chamber.
8.3.9 Close both valves and purge the sample for the time and at the temperature specified in Table 1.
8.3.10 At the conclusion of the purge time, attach the trap to the chromatograph, adjust the device to the desorb mode, and begin the gas chromatographic temperature program and GC data acquisition. Concurrently, introduce the trapped materials to the gas chromatographic column by rapidly heating the trap to 260°C while backflushing the trap with inert gas between 20 and 60 mL/min for the time specified in Table 1.
8.3.11 While the trap is being desorbed into the gas chromatograph, empty the purging chamber. Wash the chamber with a minimum of two 5 mL flushes of organic-free reagent water to avoid carryover of pollutant compounds into subsequent analyses.
8.3.12 After desorbing the sample, recondition the trap by returning the purge-and-trap device to the purge mode. Wait 15 sec; then close the syringe valve on the purging device to begin gas flow through the trap. The trap temperature should be maintained at 260°C for Methods 8010, 8020, 8021, 8240, 8260, and Methods 8015 and 8030. Trap temperatures up to 270°C may be employed. However, the higher temperatures will shorten the useful life of the trap. After approximately 7 min, turn off the trap heater and open the syringe valve to stop the gas flow through the trap. When cool, the trap is ready for the next sample.
8.3.13 If the initial analysis of a sample or a dilution of the sample has a concentration of analytes that exceeds the initial calibration range, the sample must be reanalyzed at a higher dilution. When a sample is analyzed that has saturated response from a compound, this analysis must be followed by a blank organic-free reagent water analysis. If the blank analysis is not free of interferences, the system must be decontaminated. Sample analysis may not resume until a blank can be analyzed that is free of interferences.
8.3.14 All dilutions should keep the response of the major constituents (previously saturated peaks) in the upper half of the linear range of the curve. Proceed to the specific determinative method for details on calculating analyze response. Unless all determinative method compounds are detected or an undiluted analysis confirms level of compounds found in secondary dilution.

8.4 Sediment/soil and waste samples:

Samples requiring GC/MS analysis are screened if suspect, display background or sample emits a petroleum odor. It is highly recommended that all samples of this type be screened by using GC/FID prior to the purge-and-trap GC analysis. These samples may contain percent quantities of purgeable organics that will contaminate the purge-and-trap system, and require extensive cleanup and instrument downtime. Use the screening data to determine whether to use the low-concentration method (0.005-1 mg/Kg) or the high concentration method (>1 mg/Kg).

8.4.1 Low-concentration method: This is designed for samples containing individual purgeable compounds of <1 mg/Kg. It is limited to sediment/soil samples and waste that is of a similar consistency (granular and porous). The low-concentration method is based on purging a heated sediment/soil sample mixed with organic-free reagent water containing the surrogate and, if applicable, internal and matrix spiking standards. Analyze all reagent blanks and standards under the same conditions as the samples.

8.4.2 Use a 5 g sample if the expected concentration is <0.1 mg/Kg or a 1 g sample for expected concentrations between 0.1 and 1 mg/Kg.
8.4.3 The GC system should be set up as described in the specific determinative method. This should be done prior

to the preparation of the sample to avoid loss of volatile from standards and samples. A heated purge calibration curve must be prepared and used for the quantitation of all samples analyzed with the low-concentration method. Follow the initial and daily calibration instructions, except for the addition of a 40°C purge temperature for Methods 8010, 8020, and 8021.

8.4.4 Remove the plunger from a 5 mL Luerlock type syringe equipped with a syringe valve and fill until overflowing with organic-free reagent water. Replace the plunger and compress the reagent water to vent trapped air. Adjust the volume to 5.0 mL. Add 10 μL each of surrogate spiking solution to the syringe through the valve. Matrix spiking solutions, if indicated, should be added (10 μL) to the sample at this time.

8.4.5 The sample (for volatile organics) consists of the entire contents of the sample container. Do not discard any supernatant liquids. Mix the contents of the sample container with a narrow metal spatula. Weigh the amount determined in Section 8.4.2 into a tared purge device. Note and record the actual weight to the nearest 0.1 g.

8.4.6 Add the spiked organic-free reagent water to the purge device, which contains the weighed amount of sample, and connect the device to the purge-and-trap system.

NOTE: Prior to the attachment of the purge device, Sections 8.4.5 and 8.4.6 must be performed rapidly and without interruption to avoid loss of volatile organics. These steps must be performed in a laboratory free of solvent fumes.

8.4.7 Heat the sample to 40°C ± 1°C (GC Methods 8010, 8020, 8021; GC/MS Methods 8240, 8260, and CLP) or to 85°C± 2°C (GC Methods 8015 and 8030) and purge the sample for the time shown in Table 1.

8.4.8 Proceed with the analysis as outlined in Sections 8.3.11–8.3.14. Use 5 mL of the same organic-free reagent water as in the reagent blank. If saturated peaks occurred or would occur if a 1 g sample were analyzed, the high-concentration method must be followed.

8.4.9 For matrix spike analysis of low-concentration sediment/soils, add 10 μL of the matrix spike solution to 5 mL of organic-free reagent water. The concentration for a 5 g sample would be equivalent to 50 μg/kg of each matrix spike standard.

8.5 High-concentration method: The method is based on extracting the sediment/soil with methanol. A waste sample is either extracted or diluted, depending on its solubility in methanol.

8.5.1 The sample (for volatile organics) consists of the entire contents of the sample container. Do not discard any supernatant liquids. Mix the contents of the sample container with a narrow metal spatula. For sediment/soil and waste that are insoluble in methanol, weigh 4 g (wet weight) of sample into a tared 20 mL vial. Use a top-loading balance. Note and record the actual weight to 0.1 gram and determine the percent dry weight of the sample using the procedure in Section 8.4.2. For waste that is soluble in methanol, weigh 1 g (wet weight) into a tared scintillation vial or culture tube or a 10 mL volumetric flask. (If a vial or tube is used, it must be calibrated prior to use. Pipet 10.0 mL of methanol into tile vial and mark the bottom of the meniscus. Discard this solvent.)

8.5.2 For sediment/soil or solid waste, quickly add 9.0 mL of appropriate solvent; then add 1.0 mL of the surrogate spiking solution to the vial. For a solvent miscible sample, dilute the sample to 10 mL with the appropriate solvent after adding 1.0 mL of the surrogate spiking solution. Cap and shake for 2 min.

NOTE: Sections 8.5.1 and 8.5.2 must be performed rapidly and without interruption to avoid loss of volatile organics. These steps must be performed in a laboratory free from solvent fumes.]

8.5.3 Pipet approximately 1 mL of the extract into a GC vial for storage, using a disposable pipet. The remainder may be discarded. Transfer approximately 1 mL of reagent methanol to a separate GC vial for use as the method blank for each set of samples. These extracts may be stored at 4°C in the dark, prior to analysis.

8.5.4 The GC system should be set up as in Section 8.0 of the specific determinative method. This should be done prior to the addition of the methanol extract to organic-free reagent water.

8.5.5 Table 2 can be used to determine the volume of methanol extract to add to the 5 mL of organic-free reagent water for analysis. If a screening procedure was followed, use the estimated concentration to determine the appropriate volume. Otherwise, estimate the concentration range of the sample from the low-concentration analysis to determine

the appropriate volume. If the sample was submitted as a high-concentration sample, start with 100 μL. All dilutions must keep the response of the major constituents (previously saturated peaks) in the upper half of the linear range of the curve.

8.5.6 Remove the plunger from a 5.0 mL Luerlock type syringe equipped with a syringe valve and fill until overflowing with organic-free reagent water. Replace the plunger and compress the water to vent trapped air. Adjust the volume to 4.9 mL. Pull the plunger back to 5.0 mL to allow volume for the addition of the sample extract and of standards. Add 10 μL of internal standard solution. Also add the volume of methanol extract determined in Section 8.5.5 and a volume of methanol solvent to total 100 μL (excluding methanol in standards).

8.5.7 Attach the syringe-syringe valve assembly to the syringe valve on the purging device. Open the syringe valve and inject the water/methanol sample into the purging chamber.

8.5.8 Proceed with the analysis as outlined in the specific determinative method. Analyze all reagent blanks on the same instrument as that used for the samples. The standards and blanks should also contain 100 μL of methanol to simulate the sample conditions.

8.5.9 For a matrix spike in the high-concentration sediment/soil samples, add 8.0 mL of methanol, 1.0 mL of surrogate spike solution and 1.0 mL of matrix spike solution. Add a 100 μL aliquot of this extract to 5 mL of water for purging (as per Section 8.5.6).

8.6 Sample analysis:

The samples prepared by this method may be analyzed by Methods 8010, 8015, 8020, 8021, 8030, 8240, and 8260. Refer to these methods for appropriate analysis conditions.

9.0 QUALITY CONTROL

9.1 Method blanks, LCS, and MS/MSD samples are be subjected to exactly the same procedures as those used upon actual samples.

9.2 Initial Demonstration of Precision and Accuracy: Prior to the extraction of samples, each analyst is required to demonstrate the ability to generate data of acceptable bias and precision by meeting the acceptance criteria specified in the determinative method for the method blank and LCS in three extraction batches. The documentation of this demonstration is included in the analyst's Training Record.

10.0 REFERENCES

10.1 U.S. EPA 40 CFR Part 136, "Guidelines Establishing Test Procedures for the Analysis of Pollutants Under the Clean Water Act; Final Rule and Interim Final Rule and Proposed Rule, " October 26, 1984.

10.2 U.S. EPA, "Test Methods for Evaluating Solid Waste," SW-846, 1995, third edition, Office of Solid Waste and Emergency Response, Washington, D.C.

TABLE 1. PURGE-AND-TRAP OPERATING PARAMETERS

	ANALYSIS METHOD					
	8010	***8240***	***8240 25 mL Purge***	***8020/ 8021***	***8260***	***8260 25 mL Purge***
Purge gas	Helium	Helium	Helium	Helium	Helium	Helium
Purge gas flow rate (mL/min)	40	40	40	40	40	40
Purge time (min)	11.0 ± 0.1	11.0 ± 0.1	11.0 ± 0.1	11.0 ± 0.1	11.0 ± 0.1	11.0 ± 0.1
Purge temperature (°C)	35 ± 2	35 ± 2	35 ± 2	35 ± 2	35 ± 2	35 ± 2
Desorb temperature (°C)	250	250	250	250	250	250
Backflush inert gas flow (mL/min)	20-60	20-60	20-60	20-60	20-60	20-60
Desorb time (min)	4	4	4	4	4	4

TABLE 2. QUANTITY OF METHANOL EXTRACT REQUIRED FOR ANALYSIS OF HIGH-CONCENTRATION SOILS/SEDIMENTS

Approximate Concentration Range	Volume of Methanol Extract (a)
500-10,000 µg/Kg	100 µL
1,000 – 20,000 µg/Kg	50 µL
5,000 – 100,000 µg/Kg	10 µL
25,000 – 500,000 µg/Kg	100 µL of 1 / 50 dilution (b)

Calculate the appropriate dilution factor for concentrations exceeding this table.

(a) The volume of methanol added to 5 mL of water being purged should be kept constant. Therefore, add to the 5 mL syringe whatever volume of methanol is necessary to maintain a volume of 100µL added to the syringe.
(b) Dilute an aliquot if the methanol extract and then take 100 µL for analysis.

Volatiles
SW 8260B

EA Engineering, Science, and Technology, Inc.

EA Laboratories

ANALYTICAL METHOD

Number: EAL-M-8260B **Rev. No.:** 2

Title: VOLATILE ORGANIC COMPOUNDS BY GC/MS: CAPILLARY COLUMN TECHNIQUE

Approved By: Craig Schenning, Section Chief — Date: 10/7/98

Approved By: M. M. Uhlfelder, Quality Services Manager — Date: 10/7/98

Approved By: A. R. Karimi, Laboratory Director — Date: 10/7/98

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EA Engineering, Science, and Technology, Inc.

EA Laboratories

ANALYTICAL METHOD

Number: EAL-M-8260B **Rev. No.:** 2

Revisions		
Rev. No.	**Date of Release**	**Description**
0	N/A	Initial distribution. Naval Audit Finding requirements. Revised prior to release
1	12/27/97	Revised per Naval Auditor response CTO#004.
2		Revised per annual review

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ANALYTICAL METHOD

Number: EAL- 8260B **Rev. No.:** 2

Controlled Distribution

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John Pence	12
Craig Schenning	17
Reza Karimi	18
Mohammed Haq	19

1.0 SCOPE AND APPLICATION

1.1 This method is used to determine the concentration of volatile organic compounds in a variety of solid waste matrices. This method is applicable to nearly all types of samples, regardless of water content, including ground water, aqueous sludges, caustic liquors, acid liquors, waste solvents, oily wastes, mousses, tars, fibrous wastes, polymeric emulsions, filter cakes, spent carbons, spent catalysts, soils, and sediments.

1.2 Table 1 lists the compounds that are routinely determined by this method. Other compounds which can be determined are in the reference method. The laboratory Reporting Limits are listed in Table 2. Modifications to the analyte list or procedural changes to reach lower Reporting Limits are allowed if required by client, project or program. Any changes in the analytical procedures must be approved by the Section Chief, QC Chemist, and the Quality Services Manager before samples can be analyzed.

1.3 This method is based upon a purge-and-trap, gas chromatographic/mass spectrometric (GC/MS) procedure. It is restricted to use by, or under the supervision of, analysts experienced in the use of purge-and-trap systems and gas chromatograph/mass spectrometers, and skilled in the interpretation of mass spectra and their use as a quantitative tool.

Table 1. Method Analytes and Characteristic Masses (M/Z)

Analyte	CAS#	Primary Characteristic Ion	Secondary Characteristic Ion(s)
Acetone	67-64-1	58	43
Acetonitrile	75-05-8	41	40, 39
Acrolein	107-02-8	56	55, 58
Acrylonitrile	107-13-1	53	52, 51
Allyl chloride	107-05-1	76	41, 39, 78
Benzene	71-43-2	78	--
Bromobenzene	108-86-1	156	77, 158
Bromochloromethane	74-97-5	128	49, 130
Bromodichloromethane	75-27-4	83	85, 127
Bromoform	75-25-2	173	175, 254
Bromomethane	74-83-9	94	96
2-Butanone (MEK)	78-36-3	56	41
n-Butylbenzene	104-51-8	91	92, 134
sec-Butylbenzene	135-98-8	105	134
tert-Butylbenzene	98-06-6	119	91, 134
Carbon disulfide	75-15-0	76	78
Carbon tetrachloride	56-23-5	117	119
Chlorobenzene	108-90-7	112	77, 114
Chloroethane	124-48-1	64	66
2-Chloroethyl vinyl ether	110-75-8	63	65, 106
Chloroform	75-00-3	83	85
Chloromethane	67-66-3	50	52
2-Chlorotoluene	74-87-3	91	126
4-Chlorotoluene	95-49-8	91	126
1,2-Dibromo-3-chloropropane	106-43-4	75	155, 157
Dibromochloromethane	96-12-8	129	127
1,2-Dibromoethane	106-93-4	107	109, 188
Dibromomethane	74-95-3	93	95, 174
1,2-Dichlorobenzene	95-50-1	146	111, 148
1,3-Dichlorobenzene	541-73-1	146	111, 148
1,4-Dichlorobenzene	106-46-7	146	111, 148

Table 1. Method Analytes and Characteristic Masses (M/Z)

Analyte	CAS#	Primary Characteristic Ion	Secondary Characteristic Ion(s)
Dichlorodifluoromethane	75-71-8	85	87
1,1-Dichloroethane	75-34-3	63	65, 83
1,2-Dichloroethane	107-06-2	62	98
1,1-Dichloroethene	75-35-4	96	61, 63
cis-1,2-Dichloroethene	156-59-2	96	61, 98
trans-1,2-Dichloroethene	156-60-5	96	61, 98
1,2-Dichloropropane	78-87-5	63	112
1,3-Dichloropropane	142-28-9	76	78
2,2-Dichloropropane	594-20-7	77	97
1,1-Dichloropropene	563-58-6	75	110, 77
cis-1,2-Dichloropropene	10061-01-5	75	77, 39
trans-1,2-Dichloropropene	10061-02-6	75	77, 39
Ethyl acetate	141-78-6	88	43, 45, 61
Ethyl methacrylate	97-63-2	69	41, 99, 86, 114
Ethylbenzene	100-41-4	91	106
Hexachlorobutadiene	87-68-3	225	223, 227
2-Hexanone	591-78-6	43	58, 57, 100
Iodomethane	74-88-4	142	127, 141
Isopropylbenzene	98-82-8	105	120
p-Isopropyltoluene	99-87-6	119	134, 91
Methacrylonitrile	126-98-7	41	67, 39, 52, 66
4-Methyl-2-pentanone (MIBK)	108-10-1	100	43, 58, 85
Methylene chloride	75-09-2	84	86, 49
Naphthalene	91-20-3	128	--
Pentachloroethane	76-01-7	167	130, 132, 165, 169
n-Propylbenzene	103-65-1	91	120
Styrene	100-42-5	104	78
1,1,1,2-Tetrachloroethane	630-20-6	131	133, 119
1,1,2,2-Tetrachloroethane	79-34-5	83	131, 85
Tetrachloroethene	127-18-4	166	168, 129
Toluene	108-88-3	92	91
1,2,3-Trichlorobenzene	87-61-6	180	182, 145
1,2,4-Trichlorobenzene	120-82-1	180	182, 145
1,1,1-Trichloroethane	71-55-6	97	99, 61
1,1,2-Trichloroethane	79-00-5	83	97, 85
Trichloroethene	79-01-6	95	130, 132
Trichlorofluoromethane	75-69-4	101	103
1,2,3-Trichloropropane	96-18-4	75	77
1,2,4-Trimethylbenzene	95-63-6	105	120
1,3,5-Trimethylbenzene	108-67-8	105	120
Vinyl acetate	108-05-4	43	86
Vinyl chloride	75-01-4	62	64
o-Xylene	95-47-6	106	91
m-Xylene	108-38-3	106	91
p-Xylene	106-42-3	106	91
INTERNAL STANDARDS/SURROGATES:			
4-Bromofluorobenzene	460-00-4	95	174, 176
Dibromofluoromethane		113	
Toluene-d(8)	2037-26-5	98	

Table 1. Method Analytes and Characteristic Masses (M/Z)

Analyte	CAS#	Primary Characteristic Ion	Secondary Characteristic Ion(s)
Pentafluorobenzene	363-72-4	168	
1,4-Difluorobenzene	540-36-3	114	
Chlorobenzene-d(5)		117	
1,4-Dichlorobenzene-d(4)		152	115, 150

Table 2. Reporting Limits for Volatile Analytes (a)

	(ug/L)	(ug/kg) (b)
Water (5 mL Purge)	5 (Ketones - 25)	--
Water (25 mL Purge)	1 (Ketones - 5)	--
Soil/Sediment	--	5

(a) Reporting Limit (RL) - The lowest concentration that can be reliably achieved within specified limits of precision and accuracy during routine laboratory operating conditions. The RL is derived from the MDL and the sensitivity of the analytical technique. Sample RL are highly matrix-dependent and may not always be achievable.
(b) RL listed for soil/sediment are based on wet weight. Normally data are reported on a dry weight basis; therefore, RL will be higher, based on the percent dry weight in each sample.

2.0 SUMMARY OF METHOD

2.1 Volatile compounds are introduced into the gas chromatograph by the purge-and-trap method. Purged sample components are trapped in a tube containing suitable sorbent materials. When purging is complete, the sorbent tube is heated and back flushed with helium to desorb trapped sample components. The analytes are desorbed directly to a large bore capillary for analysis. The column is temperature programmed to separate the analytes which are then detected with a mass spectrometer (MS) interfaced to the gas chromatograph.

2.2 If the above sample introduction techniques are not applicable, a portion of the sample is dispersed in solvent to dissolve the volatile organic constituents. A portion of the solution is combined with organic-free reagent water in the purge chamber. It is then analyzed by purge-and-trap GC/MS following the normal water method.

2.3 Qualitative identifications are confirmed by analyzing standards under the same conditions used for samples and comparing resultant mass spectra and GC retention times. Each identified component is quantitated by relating the MS response for an appropriate selected ion produced by that compound to the MS response for another ion produced by an internal standard.

3.0 DEFINITIONS

3.1 **Organic-free reagent water** refers to water in which no target analyte is observed at the Reporting Limit of the compounds of interest. EA Laboratories uses a Culligan reverse osmosis (R/O) water purification system to generate organic-free deionized water.

3.2 **Initial Calibration Verification (ICV)** is a second source calibration standard used to verify the initial calibration and evaluate method performance. It contains all the analytes listed in Table 1. The stock used to prepare the ICV must be from a source that is different from the stocks used to prepare the calibration standards.

3.3 **Continuing Calibration Verification(CCV)** is a mid-level calibration standard used to verify the initial calibration throughout the analytical sequence.

3.4 **Method Blank** is a reagent water or standard solid matrix spiked with all surrogates of interest and taken through the entire analytical procedure.

3.5 **Surrogate** is a non-target compound spiked into all samples and QC samples and taken through the entire analytical procedure to determine purging efficiency, and any possible matrix bias.

3.6 **Laboratory Control Sample (LCS)** is an aliquot of reagent water or a standard solid matrix, e.g. Na_2SO_4 or sand, spiked with a representative subset of the analytes of interest and taken through the entire analytical procedure. It is used to monitor the analytical process and recoveries of target analytes are compared to laboratory or project specified control limits for precision and accuracy.

3.7 **Matrix Spike/Matrix Spike Duplicate (MS/MSD)** are two sample duplicates spiked with a representative subset of the analytes of interest and taken through the entire analytical procedure. Results are used to evaluate measurement bias due to the sample matrix. Recoveries of target analytes are compared to LCS control limits.

3.8 **Reference** the terminology used to identify the native sample used for matrix spiking purposes.

4.0 SAFETY AND ALCHEMICAL HYGIENE

4.1 The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; however, each chemical compound should be treated as a potential health hazard, and exposure to these chemicals must be reduced to the lowest possible level by whatever means available. The laboratory maintains a reference file of material safety data sheets for the chemicals specified in this method. Additional information on general laboratory safety is available from the Laboratory Safety Officer.

4.2 Good laboratory technique dictates the use of appropriate dermal protection. A laboratory coat, eye protection, and gloves are the minimum requirements.

4.3 All wastes must be disposed of following the procedure outlined in EAL-SOP-018.

5.0 SAMPLE HANDLING AND PRESERVATION

5.1 Samples are stored in the laboratory at 4°C ± 2°C .

5.2 Prior to analysis the analyst should check the sample for air bubbles, and should notify the laboratory Project Manager (LPM) immediately if present. Analysis will proceed if holding times will be affected.

5.3 Two vials are sent to the laboratory for 5 mL purge analysis; three vials are needed for 25 mL purge. The pH is determined for all samples received. Notify the LPM if the pH > 2.

Table 3. Recommended Sample Containers, Preservation Techniques, and Holding Times

Parameter	Container	Preservative	Holding Time
Concentrated Waste Samples	8-oz. widemouth glass with Teflon liner	None	14 days
Liquid Samples: No Residual Chlorine Present	2 40-mL vials with Teflon lined septum caps	4 drops conc. HCl, Cool to 4°± 2°C	14 days
Liquid Samples: Residual Chlorine Present	2 40-mL vials with Teflon lined septum caps	Collect sample in a 4 oz. soil VOA container which has been pre-preserved with 4 drops of 10% sodium thiosulfate. Gently mix sample and transfer to a 40-mL VOA vial that has been pre-preserved with 4 drops conc. HCl, Cool to 4°C ± 2°C	14 days
Soil/Sediments and Sludges	4 oz (120-mL) widemouth glass with Teflon liner	Cool to 4°± 2°C	14 days

6.0 INTERFERENCES

6.1 Major contaminant sources are volatile materials in the laboratory and impurities in the inert purging gas and in the sorbent trap. The use of non-polytetrafluoroethylene (PTFE) thread sealants, plastic tubing, or flow controllers with rubber components should be avoided since such materials out-gas organic compounds which will be concentrated in the trap during the purge operation. Analyses of calibration and reagent blanks provide information about the presence of contaminants. When potential interfering peaks are noted in blanks, the analyst should change the purge gas source and regenerate the molecular sieve purge gas filter. Subtracting blank values from sample results is not permitted. If reporting values not corrected for blanks result in what the laboratory feels to be a false positive for that sample, an detailed explanation with accompany the uncorrected data.

6.2 Interfering contamination may occur when a sample containing low concentrations of volatile organic compounds is analyzed immediately after a sample containing high concentrations of volatile organic compounds. After each sample batch is complete, the auto-sampler is purged to remove residual water. To assure that any contamination is eliminated the auto-sampler position that contained samples that may have been saturated is purged with 1:1 methanol/water and instrument is programed to bake for 9 minutes and 30 seconds. Prior to and after analyses the glassware used is rinsed and baked in an oven at 250°C.

6.3 Special precautions must be taken to analyze for methylene chloride. The analytical and sample storage area should be isolated from all atmospheric sources of methylene chloride. Otherwise random background levels will result. Since methylene chloride will permeate through PTFE tubing, all gas chromatography carrier gas lines and purge gas plumbing should be constructed from stainless steel or copper tubing. Laboratory clothing worn by the analyst should be clean since clothing previously exposed to methylene chloride fumes during liquid/liquid extraction procedures can contribute to sample contamination.

6.4 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 trip blank prepared from organic-free reagent water and carried through the sampling and handling protocol can serve as a check on such contamination.

7.0 APPARATUS/INSTRUMENTATION

7.1 Microsyringes - 5, 10, 25, 100, 250, 500, and 1,000-uL.

7.2 Syringes - 5, 10, or 25-mL, gas-tight.

7.3 Balance - Analytical , 0.0001 g, and top-loading, 0.1 g.

7.4 Glass scintillation vials, 1 and 2-mL, with Teflon lined screw-caps

7.5 Disposable Pasteur pipets.

7.6 Volumetric flasks, Class A - 10 mL, 40 mL, 100 mL, 200 mL, 250 mL, and 1000 mL with ground-glass stoppers.

7.7 Spatula - Stainless steel.

7.8 Purge-and-Trap device - The purge-and-trap device, a TEKMAR 2000 or equivalent, consists of three separate pieces of equipment: the sample purger, the trap, and the desorber

7.8.1 Sample Purger: The purging chamber is designed to accept 5 mL (and 25 mL if the lowest detection limit is required) samples.

7.8.2 The traps are purchased from Supelco K (VOCARB 3000) with Carbopack B/Carboxen 1000 & 1001.

7.8.3 The desorber should be capable of rapidly heating the trap to 250°C for desorption. The trap bake-out temperature should not exceed 260°C

7.9 Heater - The heater used is capable of maintaining the purging chamber to within 1°C over the

temperature range of ambient to 100°C.

7.10 Gas chromatograph

7.10.1 HP 5890 GCs are equipped complete with a temperature-programmable gas chromatograph suitable for splitless injection and all required accessories, including syringes, analytical columns, and gases.

7.10.2 Gas chromatographic columns: Rtx 502.2, 105 m x 0.53 mm ID, 3.0 µm film thickness (or equivalent).

Injector temperature	250°C
Transfer line temperature	250-300°C
Carrier gas (He) flow rate	40 mL/min
Initial temperature	35°C, hold for 5 minutes
Temperature program:	5°C/min to 85°C, then 8°C/min to 230°C.
Final temperature	230°C, hold until all expected compounds have eluted.

7.11 Mass spectrometer - Capable of scanning from 35 to 300 amu every 2 seconds or less, using 70 volts (nominal) electron energy in the electron impact ionization mode.

7.12 GC/MS interface - The GC is interfaced to the MS with an all glass enrichment device and an all glass transfer line that gives acceptable calibration points at 50 ng or less per injection for each of the analytes and achieves all acceptable performance criteria (see Table 8) .

7.13 Data system - HP Enviroquant data system allows the continuous acquisition and storage on machine-readable media of all mass spectra obtained throughout the duration of the chromatographic program must be interfaced to the mass spectrometer. The computer must have software that allows searching any GC/MS data file for ions of a specified mass and plotting such ion abundances versus time or scan number. This type of plot is defined as an Extracted Ion Current Profile (EICP). Software must also be available that allows integrating the abundances in any EICP between specified time or scan-number limits. The most recent version of the EPA/NIST Mass Spectral Library is used.

8.0 STANDARDS AND REAGENTS

8.1 Reagents

8.1.1 The Reagent log book will be filled out completely following EA-SOP-299.

8.1.2 Organic Free Water, laboratory reagent water purged with helium.

8.1.3 Methanol, CH_3OH - Purge and Trap Grade or equivalent, demonstrated to be free of analytes. Store apart from other solvents.

8.2 Standards

8.2.1 Stock Calibration Standards

8.2.1.1 Stock solution for all method analytes are purchased from Supelco and Accustandard as certified solution kits at a concentrations of 200 mg/L.

8.2.1.2 Gas standards should be replaced after 1 week or as recommended by manufacturer's recommendation.

8.2.1.3 Other standards should be replaced after 6 months.

8.2.1.4 All standards should be stored at -10°C - -20°C in amber bottles with Teflon lined-screw caps.

8.2.2 Intermediate Calibration Standards

8.2.2.1 Prepared by diluting (Tables 4a and 4b).
8.2.2.2 Intermediate standards are stored with no headspace at -10°C - -20°C, and should be replaced after 1 week.

TABLE 4a - 5 mL Purge

	4 PPB	200 PPB	100 PPB	50 PPB	20 PPB	10 PPB
502+	2 uL	5 uL	2.5 uL	1.25 uL	5 uL	2.5 uL
8260VOC1	2 uL	5 uL	2.5 uL	1.25 uL	5 uL	2.5 uL
8260VOC2	2 uL	5 uL	2.5 uL	1.25 uL	5 uL	2.5 uL
8260VOC3	2 uL	5 uL	2.5 uL	1.25 uL	5 uL	2.5 uL
8260SS	2 uL	5 uL	2.5 uL	1.25 uL	5 uL	2.5 uL
KETONES	1.6 uL	4 uL	2 uL	1 uL	4 uL	2 uL
2-CLEVE	8 uL	20 uL	10 uL	5 uL	20 uL	10 uL
ACROLEIN	2 uL	5 uL	2.5 uL	1.25 uL	5 uL	2.5 uL
FINAL VOLUME	100 mL	5 mL	5 mL	5 mL	50 mL	50 mL

TABLE 4b - 25 mL purge

	0.8 PPB	40 PPB	20 PPB	10 PPB	5 PPB	2 PPB
502+	0.8 uL	5 uL	2.5 uL	1.25 uL	2.5 uL	1 uL
8260VOC1	0.8 uL	5 uL	2.5 uL	1.25 uL	2.5 uL	1 uL
8260VOC2	0.8 uL	5 uL	2.5 uL	1.25 uL	2.5 uL	1 uL
8260VOC3	0.8 uL	5 uL	2.5 uL	1.25 uL	2.5 uL	1 uL
8260SS	0.8 uL	5 uL	2.5 uL	1.25 uL	2.5 uL	1 uL
KETONES	1.6 uL	10 uL	5 uL	2.5 uL	5 uL	2 uL
2-CLEVE	3.2 uL	20 uL	10 uL	5 uL	10 uL	4 uL
ACROLEIN	1.6 uL	10 uL	5 uL	2.5 uL	5 uL	2 uL
FINAL VOLUME	200 mL	25 mL	25 mL	25 mL	100 mL	100 mL

8.2.3 Working Calibration Standards

8.2.3.1 Calibration standards are prepared at a minimum of five concentrations as directed in Table 5.

8.2.3.2 To prepare a calibration standard, add an appropriate volume of a secondary standard solution to an aliquot of DI water in a volumetric flask. Use a microsyringe and rapidly inject the alcoholic standard into the expanded area of the filled volumetric flask. Remove the needle as quickly as possible after injection.

8.2.3.3 Mix by inverting the flask three times only. Discard the contents contained in the neck of the flask.

Table 5. Calibrations Standard Preparation

200 ppm Stock Standard (uL)	200 ppm Surrogate Standard (uL)	Final Volume (mL)	Standard Concentration (ng/mL)
5	5	5	200
2.5	2.5	5	100
1.25	1.25	5	50
0.5	0.5	5	20
0.5	0.5	10	10
2	2	100	4

8.2.4 Surrogate standards - The surrogates recommended are toluene-d8, 4-bromofluorobenzene, dibromofluoromethane, and 1,2-dichloroethane-d4.

8.2.4.1 A stock surrogate solution in methanol is purchased as a vendor certified solution at a concentration of 25 µg/mL.

8.2.4.2 An intermediate surrogate standard spiking solution is prepared from the stock at a concentration of 50-250 ug/10 mL in methanol.

8.2.4.3 Each sample undergoing GC/MS analysis is be spiked with 10 uL of the surrogate spiking solution prior to analysis.

8.2.5 Internal standards

8.2.5.1 Internal standards are chlorobenzene-d5, 1,4-difluorobenzene, 1,4-dichlorobenzene-d4, and pentafluorobenzene.

8.2.5.2 A stock internal standard solution in methanol is purchased as a vendor certified solution.

8.2.5.3 Prepare the internal standard spiking solution from the stock at a concentration of 250 ug/10 mL in methanol.

8.2.5.4 10 uL of the internal standard solution to 5.0 mL of sample or calibration standard results in 50 ug/L.

8.2.6 Initial Calibration Verification (ICV)

8.2.6.1 A second source calibration standard is purchased as a vendor certified solution.

8.2.6.2 10 uL of the internal standard solution to 5.0 mL of sample or calibration standard results in 50 ug/L.

8.2.7 Tuning Standard

8.2.7.1 4-Bromofluorobenzene (BFB) neat solution is purchased from the vendor.
8.2.7.2 BFB Intermediate Solution: Mix 0.1 g neat BFB to 10 mL methanol.
8.2.7.3 A standard solution containing 50 ng/uL of BFB in methanol is prepared by diluting 125 μL of the intermediate solution to 25 mL.

8.2.8 LCS and Matrix Spiking Standards

8.2.8.1 Prepared from separate solutions as the calibration standards.
8.2.8.2 Matrix spiking standards should be prepared from volatile organic compounds which will be representative of the compounds being investigated. At a minimum, the matrix spike should include 1,1-dichloroethene, trichloroethene, chlorobenzene, toluene, and benzene. The analyst must review the project summary for information on spiking compounds. It is desirable to perform a matrix spike using compounds found in samples. Some projects may require spiking specific compounds of interest, especially if they are polar and would not be represented by the above listed compounds. The standard should be prepared in methanol, with each compound present at a concentration of 250 ug/10.0 mL.

8.2.9 Great care must be taken to maintain the integrity of all standard solutions. It is recommended all standards in methanol be stored at -10°C to -20°C in amber bottles with Teflon lined screw-caps.

9.0 PROCEDURES

9.1 Instrument Set Up

9.1.1 Instrument Tuning

9.1.1.1 For each 12 hour shift, prior to initial calibration of the instrument, daily calibration of the instrument, and analysis of samples, a GC/MS tuning standard must be analyzed.
9.1.1.2 Each GC/MS system must be hardware-tuned to meet the criteria in Table 5 for a 50 ng injection of 4-bromofluorobenzene (1 uL injection of the BFB standard). Analyses must not begin until these criteria are met.

Table 6. BFB Mass - Intensity Specifications (4 -Bromofluorobenzene)

Mass	Intensity Required (relative abundance)
50	15 to 40% of mass 95
75	30 to 60% of mass 95
95	base peak, 100% relative abundance
96	5 to 9% of mass 95
173	less than 2% of mass 174
174	greater than 50% of mass 95
175	5 to 9% of mass 174
176	greater than 95% but less than 101% of mass 174
177	5 to 9% of mass 176

9.1.1.3 Three scans (the peak apex can and the scans immediately preceding and following the apex) are acquired and averaged. Background subtraction is required, and must be accomplished using a single scan acquired no more than 20 scans prior to the elution of BFB.

NOTE: All subsequent standard, sample, MS/MSDs and blanks associated with a BFB analysis must use the identical mass spectrometer instrument conditions.

9.1.2 Purge and Trap

9.1.2.1 Assemble a purge-and-trap device. For new traps, condition the trap at 260°C in the purge mode with an inert gas flow of at least 20 mL/min for 30 minutes. Prior to analysis, bake out the trap at 260°C for 9 minutes at an oven temperature of 150°C.

9.1.2.2 Sample desorption - After the 11 minute purge, adjust the purge and trap system to the desorb mode and initiate the temperature program sequence of the gas chromatograph and start data acquisition. Introduce the trapped materials to the GC column by rapidly heating the trap to 250°C while back flushing the trap with an inert gas at 10 mL/min for 4 minutes. If the non-cryogenic cooling technique is followed, the trap must be preheated to 245°C just prior to trap desorption at 250°C. While the purged analytes are being introduced into the gas chromatograph.

9.1.2.3 Hold the column temperature at 10°C for 5 minutes, then program at 6°C/min to 160°C and hold until all analytes elute. After desorbing the sample for 4 minutes, bake trap for 9.5 minutes at 260°C using "bake gas by-pass" mode for 120 seconds.

9.2 Initial Calibration

9.2.1 Transfer 5.0 mL (or 25 mL if lower detection limits are required) of each calibration standard (Table 5) to a gas tight syringe along with 10 uL of internal standard solution. Open the flask containing the standard solution and carefully pour the standard into a designated 5 mL syringe barrel to just short of overflowing. Replace the syringe plunger and compress the standard solution. Vent any residual air while adjusting the standard volume to 5.0 mL. Then transfer the contents to a purging device. Analyze the calibration standards with the volume of sample that will be analyzed (5 mL or 25 mL).

9.2.2 Carry out the purge-and-trap analysis procedure for each standard.

9.2.2.1 Traditionally, the purge-and-trap of aqueous samples is performed at ambient temperature, while purging of soil/solid samples is performed at 40°C, to improve purging efficiency.

9.2.2.2 Aqueous and soil/solid samples may also be purged at temperatures above those being recommended as long as all calibration standards, samples, and QC samples are purges at the same temperature, appropriate trapping material is used to handle the excess water, and the laboratory demonstrates acceptable method performance for the project. Purging of aqueous samples at elevated temperatures (e.g., 40°C) may improve the purging performance of many of the water soluble compounds which have poor purging efficiencies at ambient temperatures.

9.2.3 Tabulate the area response of the characteristic ions (see Table 1) against concentration for each compound and each internal standard. Calculate relative response factors (RRFs) for each compound relative to one of the internal standards. The internal standard selected for the calculation of the RRF for a compound should be the internal standard that has a retention time closest to the compound being measured. Calculate relative response factors (RRFs) for each compound as follows:

$$RRF = \frac{A_X C_{IS}}{A_{IS} C_X}$$

where:

RRF	=	Relative response factor
A_X	=	Area of the characteristic ion for the compound being measured
A_{IS}	=	Area of the characteristic ion for the specific internal standard
C_X	=	Concentration of the compound being measured (ng on column)
C_{IS}	=	Concentration of the specific internal standard (ng on column)

9.2.4 The percent relative standard deviation (%RSD) is calculated for each compound:

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

where:

%RSD	=	Percent Relative Standard Deviation
SD	=	Standard Deviation
RRF_A	=	Average Relative response factor

9.2.5 System performance check compounds (SPCC)

9.2.5.1 A system performance check must be made before the calibration curve is used. Five System Performance Check Compounds (SPCCs) are checked for a minimum average relative response factor.

Compound	Minimum RRF
Chloromethane	0.10
1,1,-Dichloroethane	0.10
Bromoform	0.10
Chlorobenzene	0.30
1,1,2,2,-Tetrachloroethane	0.30 (0.1 for 25 mL purge)

9.2.5.2 These compounds are used to check compound instability and to check for degradation caused by contaminated lines or active sites in the system.

9.2.5.2.1 chloromethane is the most likely compound to be lost if the purge flow is too fast.

9.2.5.2.2 Bromoform is one of the compounds most likely to be purged very poorly if the purge flow is too slow. Cold spots and/or active sites in the transfer lines may adversely affect response. Response of the quantitation ion (m/z 173) is directly affected by the tuning of BFB at ions m/z 174/176. Increasing the m/z 174/176 ration relative to m/z 95 may improve bromoform response.

9.2.5.2.3 Tetrachloroethane and 1,1-Dichloroethane are degraded by contaminated transfer lines in purge-and-trap systems and/or active sites.

9.2.5.3 If the minimum response factors are not met, the system must be evaluated, and

corrective action must be taken before sample analysis begins. Possible problems include standard mixture degradation, injection port inlet contamination, contamination at the from end of the analytical column, and active sites in the column or chromatographic system.

9.2.6 Calibration Check Compounds (CCC)

9.2.6.1 The purpose of the CCCs are to evaluate the calibration from the standpoint of the integrity of the system. High variability for these compounds may be indicative of system leaks or reactive sites on the column. Meeting the CCC criteria is not a substitute for successful calibration of the target analytes.

9.2.6.2 The RSD for each individual Calibration Check Compound (CCC) should be ≤15%. The CCCs are:

1,1-Dichloroethene	Toluene
Chloroform	Ethylbenzene
1,2-Dichloropropane	Vinyl chloride

9.2.6.3 If a % RSD of greater than 30% is measured for any CCC, then corrective action to eliminate a system leak and/or column reactive sites is necessary before reattempting calibration. The relative retention times of each compound in each calibration standard should agree within 0.06 relative retention time units.

9.2.7 The relative retention times (RRT) of each compound in each calibration run should agree to ±0.06 relative retention time units.

9.3 If the percent relative standard deviation (%RSD) of the response factor for any target analyte is ≤ 15% over the working range, linearity through the origin can be assumed, and the average response factor can be used in place of a calibration curve.

9.3.1 If the %RSD criteria for initial calibration is >15%, the laboratory has the following options:

9.3.1.1 Review the results (area counts, response factors) for those analytes which failed the %RSD acceptance criteria to determine if the problem is with just one of the standards. Should this be the case, the analyst has the option to reanalyze and replace the standard in question.

9.3.1.2 The calibration range may be narrowed by replacing one or more of the standards with standards of different concentrations.

9.3.1.2.1 If the high standard is dropped, more dilutions may be required.

9.3.1.2.2 If the low standards are dropped, the analyst must verify that changing the low standard concentration would not effect any client DQO's (that the new quantitation level is at least as low as any required regulatory limits or action levels).

9.3.1.3 The laboratory may use a linear regression analysis to establish the curve and use for quantitation.

9.3.1.3.1 Make certain that the instrument response is treated as the dependent variable (y) and the concentration as the independent variable (x). The regression will produce the slope and intercept terms for a linear equation in the form:

$$y = mx + b$$

where:

y = Instrument response (ratio of standard response to IS response) [Ax/Ais]

m = Slope of the line (also called the coefficient of x)(ratio of standard concentration to IS concentration) [Cis/Cx]

x = Concentration of the calibration standard

b = intercept

9.3.1.3.2 The line must not be forced through the origin. (Do not include the origin [0,0] as the sixth point!)

9.3.1.3.2.1 The regression calculation will generate a correlation coefficient (r) that is a measure of the "goodness of fit" of the regression line to the data. A value of 1.00 indicates a perfect fit. The correlation coefficient must be $r \geq 0.990$ ($r^2 \geq 0.980$).

9.3.1.3.2.2 The calculated intercept value must also be evaluated before reporting sample results. A positive value for the intercept indicates that there is some threshold instrument response which is the limiting factor in establishing linearity. A negative intercept value can be transformed into an x-intercept value that represents a threshold concentration which is the limitation. If the intercept is positive, then, as a general rule, results where the instrument response is less than three times (3x) the intercept value may be unreliable. This will afford some protection against false positive results. If the intercept is negative, results below the concentration of the lowest concentration calibration standard may be unreliable. These adjustments to the quantitation limits will apply to all samples analyzed using the regression line. In calculating sample concentrations, the regression equation is rearranged to solve for the concentration (x), as shown below.

$$x = \frac{y - b}{m}$$

9.3.1.3.2.3 An acceptable approach is to raise the reporting limit to above the y-intercept (or at least meet the y-intercept) if it would not change required client reporting limits. **The analyst must first check the project summary to determine if raising the RL exceeds action levels. This must be approved by the QC Chemist, QSM, and the LPM prior to reporting of the data package.**

9.4 Initial Calibration Verification:

9.4.1 The initial calibration curve or calibration factor must be verified immediately after the calibration is performed with a mid-level Initial Calibration Verification(ICV) standard (50 µg/mL).

9.4.1.1 The percent difference from the initial calibration must be within ± 20% for CCC compounds. If the ICV standard fails the acceptance criteria the standard should be reanalyzed immediately. If the standard fails again, the analysis is stopped and a new initial calibration is performed.

$$\%\ Difference = \frac{\overline{RRF_A} - RF}{\overline{RRF}} \times 100$$

where:

RF = Calibration factor from the analysis of the verification standard

RRF_A = Mean calibration factor from the initial calibration

Note: concentrations are used when linear regression is the quantitation technique

9.4.2 Retention Times

9.4.2.1 Target Analytes - The relative retention times of the target analyte in each calibration standard should agree within 0.06 relative retention time units. Late-eluting compounds usually have much better agreement.

9.4.2.2 Internal standard - The retention times of the internal standards in the calibration verification standard must be evaluated immediately after or during data acquisition. If the retention time for any internal standard changes by more than 30 seconds from the that in the mid-point standard level of the most recent initial calibration sequence, then the chromatographic system must be inspected for malfunctions and corrections must be made, as required. When corrections are made, reanalysis of samples analyzed while the system was malfunctioning is required.

9.4.3 Continuing Calibration Verification (CCV)

9.4.3.1 Prior to the analysis of samples, inject or purge 50 ng of the 4-bromofluorobenzene standard. The resultant mass spectra for the BFB must meet all of the criteria given in Table 5 before sample analysis begins. These criteria must be demonstrated for each 12-hour tune period.

9.4.3.2 The calibration curve for each compound of interest must be verified initially, and once every 12 hours of analysis time. This is accomplished by analyzing a mid-level calibration standard and evaluating it against the following SPCC and CCC criteria.

9.4.3.2.1 System Performance Check Compounds (SPCCs) - A system performance check must be made each 12 hours. If the SPCC criteria are met, a comparison of relative response factors is made for all compounds. This is the same check that is applied during the initial calibration. If the minimum relative response factors are not met, the system must be evaluated, and corrective action must be taken before

sample analysis begins.

9.4.3.2.2 Calibration Check Compounds (CCCs) - After the system performance check is met, CCCs are used to check the validity of the initial calibration. Calculate the percent difference using the following equation:

$$\%D = \frac{RRF_i - RRF_c}{RRF_i} \times 100$$

where:

% D	=	Percent Deviation (SW-846, 8270 percent difference)
RRF_i	=	Average relative response factor from initial calibration.
RRF_c	=	Relative response factor from current verification check standard.

9.4.3.2.3 If the percent deviation for each CCC is≤ 20%, the initial calibration is assumed to be valid. If the criterion is not met (>20%D), for any one CCC, remake and reanalyze the CCV.

9.4.3.2.4 If the CCV still fails to meet acceptance criteria, the instrument must be recalibrated prior to sample analysis.

9.4.4 Internal Standard

9.4.4.1 The internal standard responses and retention times in the check calibration standard must be evaluated immediately after or during data acquisition. If the retention time for any internal standard changes by more than 30 seconds from the last calibration check (12 hours), the chromatographic system must be inspected for malfunctions and corrections must be made, as required.

9.4.4.2 If the EICP area for any of the internal standards changes by a factor of two (-50% to +100%) from the midpoint of the curve, the mass spectrometer must be inspected for malfunctions and corrections must be made, as appropriate. When corrections are made, samples analyzed during the period of malfunction must be reanalyzed.

9.5 Sample Analysis

9.5.1 Screening of the sample, using a headspace analysis by gas chromatography with a FID, prior to purge-and-trap analysis will provide guidance on whether sample dilution is necessary and will prevent contamination of the purge-and-trap system.

9.5.2 All samples and standard solutions must be allowed to warm to ambient temperature before analysis.

9.5.3 Analysis of Water Samples

9.5.3.1 If lower detection limits are required, use a 25 mL syringe. Open the sample or standard bottle and carefully pour the sample into the syringe barrel to just short of overflowing. Replace the syringe plunger and compress the sample. Vent any residual air while adjusting the sample volume to 5.0 mL.

9.5.3.2 The following procedure is appropriate for diluting purgeable samples. All steps must be performed without delays until the diluted sample is in a gas-tight syringe. Dilutions may be made in volumetric flasks (10 to 100 mL). Select the

volumetric flask that will allow for the necessary dilution. Intermediate dilutions may be necessary for extremely large dilutions. Calculate the approximate volume of DI water to be added to the volumetric flask selected and add slightly less than this quantity of DI water to the flask. Inject the proper aliquot of sample from the syringe into the flask. Aliquots of less than 1 mL are not recommended. Dilute the sample to the mark with DI water. Cap the flask, invert, and shake three times. Repeat above procedure for additional dilutions. Fill a 5 mL syringe with the diluted sample.

9.5.3.3 Add 10.0 uL of the surrogate spiking solution and 10.0 uL of the internal standard spiking solution to each sample. The surrogate and internal standards may be mixed and added as a single spiking solution. The addition of 10 uL of the surrogate spiking solution to 5 mL of aqueous sample will yield a concentration of 50 ug/L of each surrogate standard. The addition of 10 uL of the surrogate spiking solution to 5 g of a non-aqueous sample will yield a concentration of 50 ug/kg of each standard.

9.5.3.4 Add 10 uL of the matrix spike solution (Section 8.8) to a 5-mL aliquot of the sample chosen for spiking. Disregarding any dilutions, this is equivalent to a concentration of 50 ug/L of each matrix spike standard. Follow the same procedure in preparing the laboratory control sample (LCS) and method blank (MB), except the spike is added to a clean matrix.

9.5.3.5 Inject the sample into the purging chamber. Purge the sample for 11.0 +/- 0.1 minutes at ambient temperature. Trap temperature is set to 35°C.

9.5.4 Analysis of Sediment/Soil and Waste Samples

9.5.4.1 Use the screening data to determine whether to use the low-concentration method (0.005-1 mg/Kg) or the high-concentration method (> 1 mg/Kg).

9.5.4.2 Low-concentration method - This is designed for samples containing individual purgeable compounds of <1 mg/Kg. The low-concentration method is based on purging a heated sediment/soil sample mixed with DI water containing the surrogate and internal standards. Analyze all blanks and standards under the same conditions as the samples. Use a 5 g sample if the expected concentration is < 0.1 mg/Kg or a 1 g sample for expected concentrations between 0.1 and 1 mg/Kg. A heated purge calibration curve must be prepared and used for the quantitation of all samples analyzed with the low-concentration method. Follow the initial and daily calibration instructions, except for the addition of a 40°C purge temperature.

9.5.4.3 Remove the plunger from a 5 mL Luerlock type syringe equipped with a syringe valve and fill until overflowing with DI water. Replace the plunger and compress the water to vent trapped air. Adjust the volume to 5.0 mL. Add 10 uL each of surrogate spiking solution and internal standard solution to the syringe through the valve. The addition of 10 uL of the surrogate spiking solution to 5 g of sediment/soil is equivalent to 50 ug/Kg of each surrogate standard. The sample (for volatile organics) consists of the entire contents of the sample container. Do not discard any supernatant liquids. Mix the contents of the sample container with a narrow metal spatula. Weigh the sample into a tared purge device.

9.5.4.4 Add the spiked DI water to the purging device, which contains the weighed amount of sample, and connect the device to the purge-and-trap system. Heat the sample to 40° ± 1°C and purge the sample for 11.0 ± 0.1 minutes. The initial trap temperature is set at 35°C. For matrix spike analysis of low-concentration sediment/soils, add 10 uL of the matrix spike solution to the 5 mL of DI water. The concentration for a 5 g sample would be equivalent to 50 ug/Kg of each

matrix spike standard.

9.5.4.5 High-concentration method - The method is based on extracting the sediment/soil with methanol. An aliquot of the extract is added to DI water containing surrogate and internal standards. This is purged at ambient temperature. All samples with an expected concentration of > 1.0 mg/Kg should be analyzed by this method. The sample (for volatile organics) consists of the entire contents of the sample container. Do not discard any supernatant liquids. Mix the contents of the sample container with a narrow metal spatula. For sediment/soil and solid wastes that are insoluble in methanol weigh 4 g (wet weight) of sample into a tared 20 mL vial. Use a top-loading balance. Note and record the actual weight. Quickly add 10.0 mL of methanol. Cap and shake for 2 minutes.

9.5.4.6 Pipet approximately 1 mL of the extract to a 2-mL screw cap vial for storage, using a disposable pipet. The remainder may be disposed. Transfer approximately 1 mL of appropriate solvent to a separate GC vial for use as the method blank for each set of samples. These extracts may be stored at 4° ± 2°C in the dark, prior to analysis.

9.5.4.7 Setup up the GC/MS system prior to the addition of the solvent extract to DI water. Add 100 uL of the solvent extract to the 5 mL of DI water for analysis. Remove the plunger from a 5.0 mL Luerlock type syringe and fill until overflowing with water. Replace the plunger and compress the water to vent trapped air. Adjust the volume to 4.9 mL. Pull the plunger back to 5.0 mL to allow volume for the addition of the sample extract and of standards.

9.5.4.8 If a screening procedure was followed, use the estimated concentration to determine the appropriate volume of solvent extract. Otherwise, estimate the concentration range of the sample from the low concentration analysis to determine the appropriate volume. If the sample was submitted as a high-concentration sample, start with 100 uL. All dilutions must keep the response of the major constituents (previously saturated peaks) in the upper half of the linear range of the curve. Add 10 uL of internal standard/surrogate solution. Also add the volume of solvent extract and a volume of extraction or dissolution solvent to total 100 uL (excluding solvent in standards).

9.5.4.9 Attach the syringe-syringe valve assembly to the syringe valve on the purging device. Open the syringe valve and inject the water/solvent sample into the purging chamber. Proceed with the analysis. Analyze all blanks on the same instrument as that used for the samples. The standards and blanks should also contain 100 uL of the dilution solvent to simulate the sample conditions.

9.5.4.10 For a matrix spike in the high-concentration sediment/soil samples, add 9.0 mL of methanol and 1.0 mL of matrix spike solution. This results in a 6,200 ug/Kg concentration of each matrix spike standard when added to a 4 g sample. Add a 100 uL aliquot of this extract to 5 mL of DI water for purging.

9.6 Laboratory Control Samples - are prepared by spiking a deionized water blank sample with a secondary independent source vendor certified gas standard solution (containing Chloromethane, Vinyl Chloride, Bromomethane and Chloroethane) and matrix spike standard solution. The gas standard is added at 1.25 uL into 25 mL of the blank and 10 uL of matrix spike standard containing the reminding target compounds to achieve 50 ppb concentration.

9.7 If the initial analysis of the sample or dilution of the sample has a concentration of analytes that exceeds the initial calibration range, the sample must be reanalyzed at a higher dilution. All dilutions should keep the response of the major constituents in the upper half of the linear range of the curve.

10.0 CALCULATIONS

10.1 Qualitative

10.1.1 The qualitative identification of compounds determined by this method is based on retention time, and on comparison of the sample mass spectrum, after background correction, with characteristic ions in a reference mass spectrum. The reference mass spectrum must be generated by the laboratory using the conditions of this method. The characteristic ions from the reference mass spectrum are defined to be the three ions of greatest relative intensity, or any ions over 30% relative intensity if less than three such ions occur in the reference spectrum. Compounds are identified as present when the following criteria are met.

10.1.1.1 The intensities of the characteristic ions of a compound maximize in the same scan or within one scan of each other. Selection of a peak by a data system target compound search routine where the search is based on the presence of a target chromatographic peak containing ions specific for the target compound at a compound-specific retention time will be accepted as meeting this criterion.

10.1.1.2 The relative retention time (RRT) of the sample component is within ± 0.06 RRT units of the RRT of the standard component.

10.1.1.3 The relative intensities of the characteristic ions agree within 30% of the relative intensities of these ions in the reference spectrum.

EXAMPLE: For an ion with an abundance of 50% in the reference spectrum, the corresponding abundance in a sample spectrum can range between 20% and 80%.

10.1.1.4 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 two isomer peaks is less than 25% of the sum of the two peak heights. Otherwise, structural isomers are identified as isomeric pairs.

10.1.1.5 Identification is hampered when sample components are not resolved chromatographically and produce mass spectra containing ions contributed by more than one analyte. When gas chromatographic peaks obviously represent more than one sample component (i.e., a broadened peak with shoulder(s) or a valley between two or more maxima), appropriate selection of analyte spectra and background spectra is important.

10.1.1.6 Examination of extracted ion current profiles of appropriate ions can aid in the selection of spectra, and in qualitative identification of compounds. When analytes coelute (i.e., only one chromatographic peak is apparent), the identification criteria may be met, but each analyte spectrum will contain extraneous ions contributed by the coeluting compound.

10.1.2 For samples containing components not associated with the calibration standards, a library search may be made for the purpose of tentative identification. The analyst must review the Project Summary or contract the LPM if there are any questions. Data system library search routines should not use normalization routines that would misrepresent the library or unknown spectra when compared to each other. Use the following guidelines for making tentative identifications:

10.1.2.1 Relative intensities of major ions in the reference spectrum (ions greater than 10% of the most abundant ion) should be present in the sample spectrum.

10.1.2.2 The relative intensities of the major ions should agree within +/- 20%. For example, an ion with an abundance of 50% in the standard spectrum, the corresponding sample ion abundance must be between 30 and 70%.

10.1.2.3 Molecular ions present in the reference spectrum should be present in the sample spectrum.

10.1.2.4 Ions present in the sample spectrum but not in the reference spectrum should be reviewed for possible background contamination or presence of coeluting compounds.

10.1.2.5 Ions present in the reference spectrum but not in the sample spectrum should be reviewed for possible subtraction from the sample spectrum because of background contamination or coeluting peaks. Data system library reduction programs can sometimes create these discrepancies.

10.2 Quantitative Analysis

10.2.1 When a compound has been identified, the quantitation of that compound will be based on the integrated abundance from the EICP of the primary characteristic ion. Quantitation will take place using the internal standard technique. The internal standard used shall be the one nearest the retention time of the given analyte.

10.2.2 Calculate the concentration of each identified analyte in the sample as follows:

10.2.2.1 Water and Water-Miscible Wastes:

$$Conc_{sample} = \frac{A_{sample} \times C_{is}}{A_{is} \times \overline{RRF}} \times DF$$

where

$Conc_{sample}$	=	Sample concentration in ug/L
A_{sample}	=	Area of characteristic ion for compound being measured in the sample.
C_{is}	=	Concentration of internal standard injected (ng/uL).
A_{is}	=	Area of characteristic ion for the internal standard.
RRF	=	Average relative response factor of compound being measured from initial calibration.
DF	=	Dilution factor

10.2.2.2 Sediment/Soil, Sludge, and Waste

$$Conc_{sample} = \frac{A_{sample} \times C_{is}}{A_{is} \times \overline{RRF}} \times DWF \times DF$$

where:

$Conc_{sample}$	=	Sample concentration in ug/kg
A_{sample}	=	Area of characteristic ion for compound being measured.
C_{is}	=	Concentration of internal standard injected (ug/kg).
A_{is}	=	Area of characteristic ion for the internal standard.
RRF	=	Average relative response factor of compound being

measured from initial calibration.

DWF = Dry Weight Factor

DF = Dilution factor

10.2.2.2.1 Sediment/soil samples are generally reported on a dry weight basis, while sludges and wastes are reported on a wet weight basis.

10.2.2.2.2 The dry weight factor (DWF) of the sample is calculated using the following equation.

$$DWF = \frac{100}{100 - \%Moisture}$$

10.2.3 Where applicable, an estimate of concentration for noncalibrated components in the sample should be made. The formulae given above should be used with the following modifications: The areas A_{sample} and A_{is} should be from the total ion chromatograms, and the RRF for the compound should be assumed to be 1. The concentration obtained should be reported indicating (1) that the value is an estimate and (2) which internal standard was used to determine concentration. Use the nearest internal standard free of interferences.

11.0 QUALITY CONTROL

11.1 Quality Control Acceptance Criteria for this method, including the frequency and corrective actions are shown in Table 6.

11.2 Initial Calibration

11.2.1 In order to produce acceptable sample results, the response of the instrument must be within the working range established by the initial calibration.

11.2.2 The extrapolation of the calibration to concentrations above or below those of the actual calibration standards is not permitted.

11.3 The standard curve is verified using a second source standard (ICV).

11.4 The standard curve is verified every 12 hour analytical shift using a continuing calibration verification standard (CCV).

11.5 Method Blank

11.5.1 A method blank is analyzed once per analytical batch of 20 or fewer samples to determine whether or not the analysis has introduced any contamination to the samples.

11.5.2 Results from the method blank are not subtracted from the samples.

11.5.3 When method or solvent blanks are not used after the analysis of high level samples, the results for at least two of the following samples are carefully reviewed to determine if there was any contamination. If the analytes are not present in the samples following the highly concentrated sample, that data is usable. Otherwise, the sample are reanalyzed.

11.6 Laboratory Control Sample: Analyzed once per analytical batch of 20 or fewer samples.

11.6.1 LCS Recovery

$$\%\ LCS\ Recovery\ (\%R) = \frac{Found}{True} \times 100$$

11.7 Matrix Spike/Matrix Spike Duplicate: Analyze one MS/MSD pair once per analytical batch of every 20 or fewer samples.

11.7.1 Report the results of the both the %R in the MS and MSD samples and the %RPD between the MS and MSD. Note results in the case narrative.

11.7.1.1 Spike Recovery

$$\%\ Spike\ Recovery\ (\%R) = \frac{SSR - SR}{SA} \times 100$$

where:

SSR = Spiked Sample Result
SR = Sample Result
SA = Spike Added

11.7.1.2 %RPD

$$\%\ RPD = \frac{|MSR - MSRD|}{(MSR + MSRD)/2} \times 100$$

where:

MSR = Matrix spike recovery
MSRD = Matrix spike duplicate recovery

(The vertical bars in the formula above indicate the absolute value of the difference, therefore, RPD is always expressed as a positive value.)

12.0 REFERENCES

12.1 United States Environmental Protection Agency. 1997. Test Methods for Evaluating Solid Waste. Physical/Chemical Methods. EPA SW-846, 3rd Edition, Method 8260B. U.S. EPA, Washington, D.C.

12.2 American Public Health Association (APHA), American Water Works Association, and Water Environmental Federation. 1992. Standard Methods For The Examination of Water and Wastewater (18th edition). APHA, Washington, D.C.

12.3 United States Environmental Protection Agency. 1995. Contract Lab Program , Statement of Work OLM03.2. U.S. EPA, Washington, D.C.

TABLE 8. SUMMARY OF LABORATORY QUALITY CONTROL REQUIREMENTS AND CORRECTIVE ACTION PROCEDURES FOR SW-846 METHOD 8260

QC Check	Frequency	Acceptance Criteria	Laboratory Corrective Action
Holding time	See Table 3	Analysis is completed within holding time.	Notify LPM, determine if laboratory is to proceed or if client will resample.
Tuning	Every 12 hours	Within limits of method	Adjust instrument parameters.
Calibration curve	Established initially at 5 concentration levels, verified daily at mid level	1. Initial calibration %RSD for all CCCs is less than 30 percent. 2. %RSD for all other analytes must by ≤15%. 3. RRF for SPCCs is >0.05. If %RSD fails, there are several options: 1. Reanalyze any outlier standards, 2. Average of all analytes ≤15%, ICAL valid, Linear regression with r≤0.99, ICAL valid.	1. Recalibrate instrument. 2. Reanalyze samples since last criteria met. 3. Document actions taken.
Initial Calibration Verification	After each initial calibration using second source standard	Recoveries for target analytes must be ±20%	1. Verify ICV preparation and reanalyze the ICV standard once to verify. 2. If still fails, recalibrate the instrument.
Continuing Calibration Verification	Verified every 12 hour analytical shift at mid-level	1. %D for each CCC must be ≤20%, then the ICAL is assumed to be valid; if the criteria is not met (>20%D) for any one CCC, then corrective action must be taken. 2. %D for all target analytes, on average, ±20%. 3. RF for SPCC is >0.05.	1. If acceptance criteria is not met, reanalyze the CCV.. 2. If still fails, recalibrate the instrument.
Method Blank	1 per analytical batch	Analyte concentration ≤RL, except that the common laboratory contaminates CH_2C1_2 and CCl_4 are ≤ 5X the MDL.	1. Determine source of contamination, i.e. instrument, blank water, reagents. 2. Take appropriate corrective action and document. 3. Reanalyze or prepare analytical batch. 4. If samples cannot be reanalyzed or reprepared, qualify data. 5. Document actions taken.

TABLE 8. SUMMARY OF LABORATORY QUALITY CONTROL REQUIREMENTS AND CORRECTIVE ACTION PROCEDURES FOR SW-846 METHOD 8260

QC Check	Frequency	Acceptance Criteria	Laboratory Corrective Action
LCS	1 per analytical batch	All recoveries must be within laboratory control limits.	1. Examine instrument parameters, sensitivity and linearity. Correct problems and document. 2. Review standard and LCS preparation. Correct any problems and document. 3. Evaluate against project specific DQOs and report data if there is not impact on data usability. 4. If data is not usable, reprepare/reanalyze the method blank, LCS and all field samples in the batch. 5. If repreparation of samples is not possible, qualify data. 6. Document all actions taken in a Nonconformance Record and in the report narrative.
Internal Standard Responses and Retention Times	Internal standards are added to all calibration standards, LCS, samples and blanks	1. Retention time for any internal standard must be within 30 seconds of the mid-point standard level of the most recent initial calibration sequence. For samples analyzed with an initial calibration, the retention times are compared to the 10 µg/L standard (waters), 50 µg/L standard (soils). 2. The area counts for all internal standards in the CCV must be within a factor of two (-50% to +100%) of the initial calibration.	1. Inspect the mass spectrometric system for malfunction and correct. 2. Reanalyze affected samples. If the areas meet criteria, report data from the compliant analysis. 3. If reanalysis of the sample does not solve the problem, submit data from both runs, and document all inspection and corrective actions taken in the analytical narrative.
Surrogate spike	All field and QC samples	Surrogate recoveries must be within laboratory limits.	1. Examine all QC, including calibration and quantitation. 2. If surrogates in LCS and/or MB are out-of-control reprepare/reanalyze the entire batch. 3. If samples cannot be reprepared, qualify data. 5. If surrogate spike in LCS and MB are acceptable but out-of-control for samples, examine preparation of samples. If no errors or problems are discovered for samples preparation, a matrix effect is assumed. 6. If errors are discovered in preparation of samples, reprepare/reanalyze the entire batch. 7. Document actions taken in a Nonconformance Record, and in the analytical report.

Soxhlet Extraction
SW 3540C

EA Engineering, Science, and Technology, Inc.

EA Laboratories

Method

Number: 3540C **Rev. No.:** 1

Title: Soxhlet Extraction

Approved By: G.M. Gregory, Semivolatiles Section Chief — 2/23/98 Date

Approved By: W.E. Miller, Semivolatiles QC Chemist — 2/23/98 Date

Approved By: M.M. Uhlfelder, Quality Services Manager — 2/26/98 Date

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1.0 SCOPE AND APPLICATION

1.1 Method 3540 is a procedure for extracting nonvolatile and semivolatile organic compounds from solids such as soils, sludges, tissues, and wastes. The Soxhlet extraction process ensures intimate contact of the sample matrix with the extraction solvent.
1.2 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 OF METHOD

2.1 The solid sample is mixed with anhydrous sodium sulfate and extracted using an appropriate solvent in a Soxhlet extractor.
2.2 The extract is then dried, concentrated, and, as necessary, exchanged into a solvent compatible with the cleanup or determinative step being employed.
2.3 Samples of interest are organized into analytical batches by the Section Chief and Group Leader based on specific project summaries. At a minimum, each batch will contain the required quality control samples consisting of one method blank, one LCS, and one MS/MSD for every 20 samples.
2.4 Sample amounts and spiking concentration and volumes are prepared by the Section Chief and Group Leader based on specific project summaries.

3.0 DEFINITIONS

3.1 **Organic-free reagent water** refers to water in which no target analyte is observed at the Reporting Limit of the compounds of interest. EA Laboratories uses a Culligan reverse osmosis (R/O) water purification system to generate organic-free deionized water.
3.2 **Laboratory Control Sample (LCS)** is a standard used to evaluate method performance and contains some or all of the target analytes specified in the determinative method. The stock used to prepare the LCS must be from a source that is different from the stocks used to prepare the calibration standards and taken through the entire analytical procedure.
3.3 **Matrix Spike/Matrix Spike Duplicate (MS/MSD)** are two sample duplicates spiked with the same standard used to prepare the LCS and taken through the entire analytical procedure.
3.4 **Method Blank** is a reagent water or standard soil spiked with all surrogates of interest and taken through the entire analytical procedure.
3.5 **Surrogate** is a non-target compound spiked into all samples and QC samples and taken through the entire analytical procedure to determine matrix bias and overall system performance.

4.0 SAMPLE HANDLING, PRESERVATION, AND HOLDING TIME

Sample container, preservation and holding time requirements are given in Table 1. While sample extracts are in the custody of the laboratory, they are stored in the semivolatiles laboratory at 4°C ± 2°C prior to and during analysis. After analysis is completed, samples are stored by the Sample Management Office in laboratory walk-ins until disposal.

TABLE 1. RECOMMENDED SAMPLE CONTAINERS, PRESERVATION TECHNIQUES, AND HOLDING TIMES

Parameter	Container	Preservative	Holding Time
Soil/Sediments and Sludges	8 oz widemouth glass with Teflon liner	Cool to 4° ± 2°C	Samples must be extracted within 14 days and extracts analyzed within 40 days following extraction.
Tissues	8 oz glass jar or wrapped in aluminum foil	Frozen	Samples must be extracted within 1 year of collection and extracts analyzed within 40 days following extraction.

5.0 INTERFERENCES

5.1 Solvents, reagent, glassware, and other sample processing hardware may yield artifacts and/or interferences to sample analysis. All these materials must be demonstrated to be free from interferences under the conditions of the analysis by analyzing method blanks.
5.2 Interferences coextracted from the samples will vary considerably from source to source. If analysis of an extracted sample is prevented due to interferences, further cleanup of the sample extract may be necessary.
5.3 Phthalate esters contaminate many types of products commonly found in the laboratory. Plastics, in particular, must be avoided because phthalates are commonly used as plasticizers and are easily extracted from plastic materials. Serious phthalate contamination may result at any time if consistent quality control is not practiced.
5.4 Glassware contamination resulting in analyte degradation: Soap residue on glassware may cause degradation of certain analytes. Specifically, aldrin, heptachlor, and most organophosphorous pesticides will degrade in this situation. This problem is especially pronounced with glassware that may be difficult to rinse (e.g., 500-mL K-D flask). These items should be hand-rinsed very carefully to avoid this problem.

6.0 APPARATUS AND MATERIALS

6.1 Soxhlet extractor - 40 mm ID, with 500-mL round bottom flask.
6.2 Drying column - 20 mm ID Pyrex® column with sodium sulfate and Pyrex® glass wool at bottom (both purified y by heating at 400°C for 4 hours).
6.3 Kuderna-Danish (K-D) apparatus

6.3.1 Concentrator tube - 10-mL, graduate (Kontes K-570050-1025 or equivalent).
6.3.2 Evaporation flask - 500-mL (Kontes 570001-500 or equivalent). Attach to concentrator tube with clamps or equivalent.
6.3.3 Snyder column - Three-ball macro (Kontes K-503000-0121 or equivalent).

6.4 Boiling chips - Solvent extracted, approximately 10/40 mesh (silicon carbide or equivalent).
6.5 Water bath - Heated, with concentric ring cover, capable of temperature control (±5°C). Baths are used in the hood.
6.6 Vials - Glass, 2-mL capacity, with polytetrafluoroethylene (PTFE)-lined screw or crimp top.
6.7 Hot plate - thermostat controlled
6.8 Disposable glass Pasteur pipets and bulb.
6.9 Apparatus for grinding/mixing the sample with anhydrous sodium sulfate.
6.10 Analytical balance - capable of weighing to 0.01 g.

7.0 SAFETY

The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; however, each chemical compound should be treated as a potential health hazard, and exposure to these chemicals must be reduced to the lowest possible level by whatever means available. The laboratory maintains a reference file of Material Safety Data Sheets (MSDS) for the chemicals specified in this method. Additional information on laboratory safety is available in the Laboratory Safety Plan and from the Laboratory Safety Officer (LSO).

8.0 REAGENTS

8.1 Reagent grade inorganic chemicals shall be used in all tests. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.
8.2 Organic-free reagent water. All references to water in this method refer to organic-free reagent water, defined as water in which no target analyte is observed at the Reporting Limit of the compounds of interest. EA Laboratories uses a Culligan reverse osmosis (R/O) water purification system to generate organic-free deionized water which meets or exceeds the specifications of ASTM Type II water.
8.3 Sodium sulfate (granular, anhydrous), Na_2SO_4. Purify by heating at 400°C for 4 hours in a shallow tray.
8.4 Extraction solvents - All solvents must be pesticide quality or equivalent. Samples shall be extracted using one of the following solvent systems:

8.4.1 Methylene chloride, CH_2Cl_2.

8.5 Exchange solvents - All Solvents must be pesticide quality or equivalent.

8.5.1 Hexane, C_6H_{14}.
8.5.2 Acetonitrile, CH_3CN.

8.6 Stock Solutions: Refer to EAL-SOP-331; *Organic LCS, MS, MSD, and Surrogate Stock Solution Preparation.*

9.0 PROCEDURE

9.1 Set Up

9.1.1 Rinse beakers, flasks and Soxhlet extractors with CH_2Cl_2. Check glassware for cracks.
9.1.2 Pour approximately 300 mL of extraction solvent into a 500-mL Erlenmeyer flasks and add boiling chips.
9.1.3 Prepare Soxhlet extractor by plugging the bottom of the Soxhlet with glass wool. The glass wool should cover the arm of the extractor to prevent sample particles from clogging the arm and decreasing efficiency of the extraction.
9.1.4 Using the extraction sheet as a reference, prepare labels and attach to the extractors.
9.1.5 Calibrate the scale(s) which will be used to weigh the sample and sodium sulfate (EAL-SOP-015). Use weights that approximate the amount of sample and sodium sulfate that will be added.

9.2 Sample Handling

9.2.1 Retrieve samples according to EAL-SOP-039; *Internal Custody Transfer of Samples.*
9.2.2 Sediment/soil samples - Decant and discard any water layer on a sediment sample. Mix sample thoroughly,

especially composited samples. Discard any objects such as sticks, leaves, and rocks.
9.2.3 Waste samples - Samples consisting of multiphases must be prepared by phase separation. This extraction procedure is for solids only.
9.2.4 Tissue samples are homogenized prior to extraction. See EAL-SOP-289.

9.3 Using a top loader balance or equivalent, weigh appropriate amount of sample into a beaker and add sufficient amount of anhydrous sodium sulfate to dry sample.
9.4 Mix sample and sodium sulfate with a spatula until a loose pouring consistency is achieved. Pour into the Soxhlet extractor, and rinse beaker into extractor. Rinse the spatula with CH_2Cl_2 after each sample to prevent potential contamination.
9.5 Once all samples have been weighed and transferred into the Soxhlet, select the surrogate and matrix spike solutions as indicated on the extraction sheet. Check expiration dates and note the identification number of the solutions on the extraction sheet. Have the appropriate QC Chemist review the extraction sheet to ensure that the solutions and spiking levels are correct.
9.6 Before spiking, check that the correct solution is being used (i.e. surrogate for surrogate spike, matrix for matrix spike).

Note: Another person shall assist with the spiking. One person pipets appropriate volume of solution into the Soxhlet. The other person keeps track of which samples have been spiked.

9.6.1 Using a disposable pipet, add the surrogate standard spiking solution to each sample, method blank, and LCS. Avoid air bubbles in the pipet as this could affect recovery.
9.6.2 For the LCS and sample in each analytical batch selected for spiking, add matrix spiking standard.

Note: The amount of surrogate spiking solution and matrix spiking solution to be added are determined by the Section Chief and/or the Group Leader and will be indicated on the extraction sheet.

9.7 Sample Extraction

9.7.1 Attach the Soxhlet extractor and Erlenmeyer flask to the condenser. The flask should rest even on the hot plate.
9.7.2 Transfer the label from the Soxhlet to the flask.
9.7.3 Once all samples have been connected to the condenser, check that hoses connected to condensers are not kinked. Check that water is running. Turn on hot plates and extract samples for 16-24 hours. Verify the extraction rate once started.
9.7.4 Give the extraction sheet to the Group Leader or Section Chief to log information into the sample status database.
9.7.5 Turn off the hotplate. Allow the extract to cool. Remove Soxhlet and flask. It may be necessary to gently tap the connection points with a smaller wooden hammer if any of the glass joints are adhered to one another.

9.8 Drying the Sample

9.8.1 Assemble a Kuderna-Danish (K-D) apparatus by attaching a 10-mL concentrator tube to a 500-mL evaporation flask. Rinse with CH_2Cl_2 and add boiling chips.
9.8.2 Using large tweezers, insert a piece of pre-baked glass wool into the drying column. Scoop anhydrous sodium sulfate with a beaker and pour into the column. Fill the column approximately 2/3 full. Rinse with $MeCl_2$.
9.8.3 Inspect the extracted sample for water before pouring into the K-D apparatus. If water is present, add anhydrous sodium sulfate directly into the Erlenmeyer flask and swirl. Pass the extract through the drying column. Rinse the flask

twice with CH_2Cl_2 using the appropriate extraction solvent and pass each rinse through the column. Rinse the column to complete the quantitative transfer.

Note: Excessive water in extract must be pipeted out before passing through the drying column.

9.9 Transfer the label from the flask to the K-D apparatus.
9.10 Concentrating the Sample

9.10.1 Pre-wet a Snyder column by adding about 1 mL of methylene chloride to the top of the column. Insert the Snyder column into the K-D apparatus. Place on a hot water bath (15 to 20°C above the boiling point of the solvent) so that the concentrator tube is partially immersed in the hot water and the entire lower rounded surface of the flask is bathed with hot vapor. Adjust the vertical position of the apparatus and the water temperature, as required, to complete the concentration. At the proper rate of distillation the balls of the column will actively chatter, but the chambers will not flood. When the apparent volume of liquid reaches 10 -15 mL, remove the K-D apparatus from the water bath and allow it to drain and cool.

Note: Before placing apparatus on hot water bath, check temperature of water bath and ensure that boiling chips have been added to the K-D apparatus.

9.10.2 If a solvent exchange is required, remove the K-D apparatus from the water bath, pour approximately 50 mL of the exchange solvent into the top the Snyder column, and return K-D apparatus to the water bath. Concentrate the extract as described above (Section 9.10.1), raising the temperature of the water bath, if necessary, to maintain proper distillation. When the apparent volume again reaches 10-15 mL, remove the K-D apparatus from the water bath and allow it to drain and cool for several minutes. If GPC cleanup is required, the extract must remain in $MeCl_2$. Solvent exchange will occur after this cleanup.

Note: It is important that tubes do not become dry as this may severely affect recoveries.

9.10.3 Remove the Snyder column and rinse the flask and its lower joints into the concentrator tube with 1-2 mL of methylene chloride or exchange solvent. Transfer the label from the K-D flask to the concentrator tube.

9.11 Nitrogen blowdown technique

9.11.1 Place the concentrator tube in a warm water bath (approximately 35°C) and evaporate the solvent to the required level using a gentle stream of clean, dry nitrogen. (Proper flow should just ripple the surface of the extract.)
9.11.2 Rinse the internal wall of the tube several times with the appropriate solvent during the operation. During evaporation, the solvent level in the tube must be positioned to prevent water from condensing into sample (i.e., the solvent level should be below the level of the water bath). The extract should **NOT** be allowed to become dry.
9.11.3 Transfer the extract from the concentrator tube to an appropriate volumetric flask once it has reached slightly below final volume. Rinse the concentrator tube with appropriate exchange solvent. Add the rinse to the volumetric flask and bring to final volume (or other appropriate volume).

9.12 See the appropriate Method SOPs for the clean-up methods that are required.
9.13 Transfer the extract from the volumetric flask to a labeled PTFE-lined screw or crimp top vial. Amber-colored vials should be used to store extracts for PAH or BNA analysis.
9.14 When finished, complete the extraction sheet and have it reviewed and signed by the Group Leader or Section

Chief before transferring samples and internal chain of custody to the instrumentation laboratory.
9.15 Relinquish custody of the extracted samples to the instrumentation laboratory for analysis by signing and dating the extraction sheet after the instrumentation analyst has inspected the sample extracts. The receiving instrumentation analyst will then sign and date the extraction sheet. Make a copy of the sheet to accompany the sample extracts in the instrumentation laboratory. Keep the original extraction sheet on file in the Organic Extraction Laboratory.
9.16 Table 2 below lists the appropriate weights, volumes, and solvents for the determinative methods.

Table 2. Determinative Method Weights, Volumes and Solvents

Method	Inital Sample Weight	Fianl Extract Volume	Initial Extraction Solvent	Solvent Exchange ?	Final Extract Solvent
8015B	20 grams	5 mL	$MeCl_2$	N	$MeCl_2$
8080	30 grams	10 mL	$MeCl_2$	Y	Hexane
8081	30 grams	10 mL	$MeCl_2$	Y	Hexane
8082	30 grams	10 mL	$MeCl_2$	Y	Hexane
8100	30 grams	1 mL	$MeCl_2$	N	$MeCl_2$
8140	30 grams	1 mL	$MeCl_2$	Y	Hexane
8141	30 grams	1 mL	$MeCl_2$	Y	Hexane
8270	30 grams	1 mL	$MeCl_2$	N	$MeCl_2$
8310	30 grams	1 mL	$MeCl_2$	Y	Acetonitrile

10.0 QUALITY CONTROL

10.1 Method blanks, LCS, and MS/MSD samples are be subjected to exactly the same procedures as those used upon actual samples.
10.2 Initial Demonstration of Precision and Accuracy: Prior to the extraction of samples, each analyst is required to demonstrate the ability to generate data of acceptable bias and precision by meeting the acceptance criteria specified in EAL-SOP 293. The documentation of this demonstration is included in the analyst's Training Record.

11.0 REFERENCES

11.1 U.S. EPA 40 CFR Part 136, "Guidelines Establishing Test Procedures for the Analysis of Pollutants Under the Clean Water Act; Final Rule and Interim Final Rule and Proposed Rule," October 26, 1984.
11.2 U.S. EPA, "Test Methods for Evaluating Solid Waste," SW-846, 1997, third edition including Update III, Office of Solid Waste and Emergency Response, Washington, D.C.

Continuous L-L Extraction
SW 3520

EA Engineering, Science, and Technology, Inc.

EA Laboratories

Method

Number: 3520 **Rev. No.:** 0

Title: Continuous Liquid-Liquid Extraction

Prepared By: M.J. Anzalone, Extraction Group Leader — 29 August 1994

Revised By: J.G. O'Donnell, Organic Extraction Supervisor — 8/2/96 Date

Approved By: M.M. Uhlfelder, Quality Services Manager — 1 August 1996 Date

Approved By: P.A. Christopher, Operations Manager — 2 Aug 1996 Date

EA Engineering, Science, and Technology, Inc.

EA Laboratories

Method

Procedure No.: 3520 **Revision No.:** 0

Revisions		
Rev. No.	**Date of Release**	**Description**
0		Initial distribution.

EA Engineering, Science, and Technology, Inc.

EA Laboratories

Method

Procedure No.: 3520 **Revision No.:** 0

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Sharon Albaugh	17
Reza Karimi	18
Cathy Atkinson	19

12/4/96

1.0 SCOPE AND APPLICATION

1.1 This method describes a procedure 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 This method is applicable to the isolation and concentration of water-insoluble and slightly soluble organics in preparation for a variety of chromatographic procedures.

2.0 SUMMARY OF METHOD

A measured volume of sample, usually 1 liter, is placed into a continuous liquid-liquid extractor, adjusted, if necessary, to a specific pH (see Table 2), and extracted with organic solvent for 18-24 hours. The extract is dried, concentrated (if necessary), and, as necessary, exchanged into a solvent compatible with the cleanup or determinative method being employed.

3.0 DEFINITIONS

3.1 **Organic-free reagent water** refers to water in which no target analyte is observed at the Reporting Limit of the compounds of interest. EA Laboratories uses a Culligan reverse osmosis (R/O) water purification system to generate organic-free deionized water.
3.2 **Laboratory Control Sample (LCS)** is a standard used to evaluate method performance and contains all the analtyes specified in the determinative method. The stock used to prepare the LCS must be from a source that is different from the stocks used to prepare the calibration standards and taken through the entire analytical procedure.
3.3 **Matrix Spike/Matrix Spike Duplicate (MS/MSD)** are two sample duplicates spiked with the same standard used to prepare the LCS and taken through the entire analytical procedure.
3.4 **Method Blank** is a reagent water or standard soil spiked with all surrogates of interest and taken through the entire analytical procedure.
3.5 **Surrogate** is a non-target compound spiked into all samples and QC samples and taken through the entire analytical procedure to determine matrix bias and overall system performance.

4.0 SAMPLE HANDLING, PRESERVATION, AND HOLDING TIME

Sample container, preservation and holding time requirements are given in Table 1. While sample extracts are in the custody of the laboratory, they are stored in the semivolatiles laboratory at 4°C ± 2°C prior to and during analysis. After analysis is completed, samples are stored by the Sample Management Office in laboratory walk-ins until disposal.

Table 1. Recommended Sample Containers, Preservation Techniques, and Holding Times

Parameter	Container	Preservative	Holding Time
Liquid Samples:			
No Residual Chlorine Present	1-gal. or 0.5 gal. or 2 x 1L amber glass with Teflon liner	Cool, 4°C	Samples must be extracted within 7 days and extracts analyzed within 40 days following extraction.
Residual Chlorine Present	1-gal. or 0.5 gal. or 2 x 1L amber glass with Teflon liner	Add 3 mL of 10% sodium thiosulfate per gallon. Cool to 4°C	Samples must be extracted within 7 days and extracts analyzed within 40 days following extraction.

5.0 INTERFERENCES

5.1 Solvents, reagent, glassware, and other sample processing hardware may yield artifacts and/or interferences to sample analysis. All these materials must be demonstrated to be free from interferences under the conditions of the analysis by analyzing method blanks. Specific selection of reagents and purification of solvents by distillation in all-glass systems may be required. Refer to the specific determinative method for specific guidance on quality control procedures.

5.2 Interferences coextracted from the samples will vary considerably from source to source. If analysis of an extracted sample is prevented due to interferences, further cleanup of the sample extract may be necessary. Refer to Method 3600 for guidance on cleanup procedures.
5.3 Phthalate esters contaminate many types of products commonly found in the laboratory. Plastics, in particular, must be avoided because phthalates are commonly used as plasticizers and are easily extracted from plastic materials. Serious phthalate contamination may result at any time if consistent quality control is not practiced.
5.4 Glassware contamination resulting in analyte degradation: Soap residue on glassware may cause degradation of certain analytes. Specifically, aldrin, heptachlor, and most organophosphorous pesticides will degrade in this situation. This problem is especially pronounced with glassware that may be difficult to rinse (e.g., 500-mL K-D flask). These items should be hand-rinsed very carefully to avoid this problem.

6.0 APPARATUS AND MATERIALS

6.1 Continuous liquid-liquid extractor - Equipped with Teflon or glass connecting joints and stopcocks requiring no lubrication (Hershberg-Wolf Extractor--Ace Glass Company, Vineland, New Jersey, P/N 6841-1-, or equivalent).
6.2 Drying column - 20 mm ID Pyrex chromatographic column with Pyrex glass wool at bottom, with Teflon stopcock.

[NOTE: Fritted glass discs are difficult to decontaminate after highly contaminated extracts have been passed through. Columns without frits may be purchased. Use a small pad of Pyrex glass wool to retain the adsorbent. Prewash the glass wool pad with 50 mL of acetone followed by 50 mL of elution solvent prior to packing the column with adsorbent.]

6.3 Kuderna-Danish (K-D) apparatus

6.3.1 Concentrator tube -10 mL graduated (Kontes K-570050-1025 or equivalent).
6.3.2 Evaporation flask - 500 mL (Kontes K-570001-500 or equivalent).
6.3.3 Snyder column - Three ball macro (Kontes K-503000-0121 or equivalent).
6.3.4 Snyder column - Two ball micro (Kontes K-569001-0219 or equivalent).

6.4 Boiling chips - Solvent extracted, approximately 10/40 mesh (silicon carbide or equivalent).
6.5 Water bath - Heated, with concentric ring cover, capable of temperature control (+/- 5-C). The bath should be used in a hood.
6.6 Vials - 2 mL, glass with Teflon lined screw-caps or crimp tops.
6.7 pH indicator paper - pH range including the desired extraction pH.
6.8 Heating mantle - Rheostat controlled.
6.9 Syringe - 5 mL.

7.0 SAFETY

The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; however, each chemical compounds should be treated as a potential health hazard, and exposure to these chemicals must be reduced to the lowest possible level by whatever means available. The laboratory maintains a reference file of Material Safety Data Sheets (MSDS) for the chemicals specified in this method. Additional information on laboratory safety is available in the Laboratory Safety Plan and from the Laboratory Safety Officer (LSO).

8.0 REAGENTS

8.1 Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening theaccuracy of the determination. Reagents should be stored in glass to prevent the leaching of contaminants from plastic containers.
8.2 Organic-free reagent water - All references to water in this method refer to organic-free reagent water, defined as

water in which no target analyte is observed at the Reporting Limit of the compounds of interest. EA Laboratories uses a Culligan reverse osmosis (R/O) water purification system to generate organic-free deionized water which meets or exceeds the specifications of ASTM Type II water.
8.3 Sodium hydroxide solution (10N), NaOH. Dissolve 40 g NaOH in organic-free reagent water and dilute to 100 mL.
8.4 Sodium sulfate (granular, anhydrous), Na_2SO_4. Purify by heating at 400-C for 4 hours in a shallow tray, or by precleaning the sodium sulfate with methylene chloride. If the sodium sulfate is precleaned with methylene chloride, a method blank must be analyzed, demonstrating that there is no interference from the sodium sulfate.
8.5 Sulfuric acid solution (1:1 v/v), H_2SO_4. Slowly add 50 mL of H_2SO_4 (sp. gr. 1.84) to 50 mL of organic-free reagent water.
8.6 Extraction/exchange solvents

8.6.1 Methylene chloride, CH_2Cl_2 - Pesticide quality or equivalent.
8.6.2 Hexane, C_6H_{14} - Pesticide quality or equivalent.
8.6.3 2-Propanol, $(CH_3)_2CHOH$ - Pesticide quality or equivalent.
8.6.4 Cyclohexane, C_6H_2 - Pesticide quality or equivalent.
8.6.5 Acetonitrile, CH_3CN - Pesticide quality or equivalent.

8.7 Stock Solutions: Refer to EAL-SOP-331; *Organic LCS, MS, MSD, and Surrogate Stock Solution Preparation.*

9.0 PROCEDURE

9.1 The extraction analyst reviews the sample chain-of-custody upon receipt in the laboratory and prepares the appropriate electronic extraction sheet.
9.2 The samples are retrieved and returned according to EAL-SOP-039; *Internal Custody Transfer of Samples.*
9.3 The samples of interest are then organized into analytical batches containing the required quality control samples consisting of one method blank, one LCS, and one MS/MSD for every 20 samples.
9.4 If high concentrations are anticipated, a smaller volume may be used and then diluted with organic-free reagent water to 1 liter alternatively. Mark the meniscus on each sample bottle. Transfer the entire sample (from 1 liter capacity containers only) to the continous extractor. Pour 100 ml of methylene chloride into the sample container. Replace the lid and shake for 15-20 seconds. Pour the methylene chloride into the continous extractor (taking care to remove the lid carefully due to the possiblity of vapor pressure build up from the shaking process). Fill the sample container to the meniscus with deionized water. Quantitatively transfer the deionized water to a 1000 ml graduated cylinder, measure amd record the sample volume. Using a graduated cylinder for samples, measure out 1 liter (nominal) of sample and transfer it quantitatively to the continuous extractor. Check the pH of the sample with wide-range pH paper and adjust the pH, if necessary, to the pH indicated in Table 2. Pipet 1.0 mL of the surrogate standard spiking solution into each sample into the extractor and mix well. (See Method 3500 and the determinative method to be used, for details on the surrogate standard solution and the matrix spike solution.) For the sample in each analytical batch selected for spiking, add 1.0 mL of the matrix spiking standard. For base/neutral-acid analysis, the amount of the surrogates and matrix spiking compounds added to the sample should result in a final concentration of 100 ng/uL of each base/neutral analyze and 200 ng/uL of each acid analyze in the extract to be analyzed (assuming a 1 uL injection). If Method 3640, Gel-Permeation Cleanup, is to be used, add twice the volume of surrogates and matrix spiking compounds since half the extract is lost due to loading of the GPC column.
9.5 Add 300-500 mL of methylene chloride to the distilling flask. Add several boiling chips to the flask.
9.6 Add sufficient water to the extractor to ensure proper operation and extract for 18-24 hours.
9.7 Allow to cool; then detach the boiling flask. If extraction at a secondary pH is not required (see Table 2), the extract is dried and concentrated using one of the techniques referred to in Section 9.9.
9.8 Carefully, while stirring, adjust the pH of the aqueous phase to <2 with sulfuric acid(1:1). Attach a clean distilling flask containing 500 mL of methylene chloride to the continuous extractor. Extract for 18-24 hours, allow to cool, and detach the distilling flask.
9.9 If performing GC/MS analysis (Method 8270), the acid and neutral/base extracts may be combined prior to concentration. However, in some situations, separate concentration and analysis of the acid and neutral/base extracts may be preferable (e.g. if for regulatory purposes the presence or absence of specific acid and neutral/base compounds at low

concentrations must be determined, separate extract analyses may be warranted).
9.10 Assemble a Kuderna-Danish (K-D) concentrator by attaching a 10-mL concentrator tube to a 500-mL evaporation tube.
9.11 Dry the extract by passing it through a drying column containing about 10 cm of anhydrous sodium sulfate. Collect the dried extract in a K-D concentrator. Rinse the flask which contained the solvent extract with 20-30 mL of methylene chloride and add it to the column to complete the quantitative transfer.
9.12 Add one or two clean boiling chips to the flask and attach a three ball Snyder column. Prewet the Snyder column by adding about 1 mL of methylene chloride to the top of the column. Place the K-D apparatus on a hot water bath (15-20-C above the boiling point of the solvent) so that the concentrator tube is partially immersed in the hot water and the entire lower rounded surface of the flask is bathed with hot vapor. Adjust the vertical position of the apparatus and the water temperature, as required, to complete the concentration in 10-20 minutes. At the proper rate of distillation the balls of the column will actively chatter, but the chambers will not flood. When the apparent volume of liquid reaches 1 mL, remove the K-D apparatus from the water bath and allow it to drain and cool for at least 10 minutes. Remove the Snyder column and rinse the flask and its lower joints into the concentrator tube with 1-2 mL of extraction solvent.
9.13 If a solvent exchange is required (as indicated in Table 2), momentarily remove the Snyder column, add 50 mL of the exchange solvent, a new boiling chip, and reattach the Snyder column. Concentrate the extract, as described in Section 9.12, raising the temperature of the water bath, if necessary, to maintain proper distillation.
9.13 Remove the Snyder column and rinse the flask and its lower joints into the concentrator tube with 1-2 mL of methylene chloride or exchange solvent. If sulfur crystals are a problem, proceed to Method 3660 for cleanup. The extract may be further concentrated by using the techniques outlined in Section 9.12 or adjusted to 10.0 mL with the solvent last used.
9.14 Remove the concentrator tube and place in a nitrogen evaporator apparatus. Attach a Pasteur pipet to the evaporator and adjust its height and the nitrogen flow so as to just ripple the surface of the extract in each concentrator tube. Evaporate the solvent until 2.0 mL remains in the tube.
9.15 The extracts obtained may now be analyzed for analyte content using a variety of organic techniques. If analysis of the extract will not be performed immediately, stopper the concentrator tube and store refrigerated. If the extract will be stored longer than 2 days it should be transferred to a vial with a Teflon-lined screw cap and labeled appropriately.
9.16 At the completion of the sample extraction process, the analyst completes and prints the electronic extraction sheet which is then reviewed by the Group Leader/Supervisor before the internal custody of the samples is transferred to the instrumentation laboratory.

9.16.1 The extraction analyst relinquishes custody of the extracted samples to the instrumentation laboratory for analysis by signing and dating the extraction sheet after the instrumentation analyst inspects the sample extracts. The receiving instrumentation analyst then signs and dates the extraction sheet and a copy is made to accompany the sample extracts in the instrumentation laboratory. The orginial copy of the extraction sheet is kept in the Organic Extraction Laboratory

10.0 QUALITY CONTROL

10.1 Method blanks, LCS, and MS/MSD samples are be subjected to exactly the same procedures as those used upon actual samples.
10.2 Initial Demonstration of Precision and Accuracy: Prior to the extraction of samples, each analyst is required to demonstrate the ability to generate data of acceptable bias and precision by meeting the acceptance criteria specified in the determinative method for the method blank and LCS in three extraction batches. The documentation of this demonstration is included in the analyst's Training Record.

11.0 REFERENCES

11.1 U.S. EPA 40 CFR Part 136, "Guidelines Establishing Test Procedures for the Analysis of Pollutant Under the Clean Water Act; Final Rule and Interim Final Rule and Proposed Rule," October 26, 1984.

11.2 U.S. EPA, "Test Methods for Evaluating Solid Waste," SW-846, 1995, third edition, Office of Solid Waste and Emergency Response, Washington, D.C.
11.3 EA Laboratories, EA Laboratories Standard Operating Procedure Manual. EAL-SOP-33; "Organic LCS, MS, MSD, and Surrogate Stock Solution Preparation,", 1996. EA Laboratories, Sparks, MD 21152

TABLE 2. SPECIFIC EXTRACTION CONDITIONS FOR VARIOUS DETERMINATIVE METHODS

Determinative Method	Initial Extraction pH	Secondary Extraction pH	Exchange solvent required for analysis	Exchange solvent required for cleanup	Volume of Extract required for cleanup (mL)	Final extract volume for analysis (mL)
8040	<=2	none	2-propanol	hexane	1.0	1.0,10.0(a)
8060	as received	none	hexane	hexane	2.0	10.0
8080	5-9	none	hexane	hexane	10.0	10.0
8100	as received	none	none	cyclohexane	2.0	1.0
8140	6-8	none	hexane	hexane	10.0	10.0
8141	as received	none	hexane	hexane	10.0	10.0
8250(b)	>11	<2	none	-	-	1.0
8270(b)	>11	<2	none	-	-	1.0
8310	as received	none	acetonitrile	-	-	1.0

(a) Phenols may be analyzed, by Method 8040, using a 1.0 mL 2-propanol extract by GC/FID. Method 8040 also contains an optional derivatization procedure for phenols which results in a 10 mL hexane extract to be analyzed by GC/ECD.
(b) The specificity of GC/MS may make cleanup of the extracts unnecessary. Refer to Method 3600 for guidance on the clean-up procedures available if required.

PAHs
SW 8310

EA Engineering, Science, and Technology, Inc.

EA Laboratories

Method

Number: 8310 **Rev. No.:** 0

Title: Polynuclear Aromatic Hydrocarbons by High Performance Liquid Chromatography

Prepared By: C.T. Gilles, HPLC Supervisor — 02 June 1993

Revised By: C.T. Gilles, HPLC Supervisor — 16 Aug 96 Date

Approved By: M.M. Uhlfelder, Quality Services Manager — 16 Aug 96 Date

Approved By: P.A. Christopher, Operations Manager — 16 Aug 96 Date

WHEN STAMPED IN RED, THIS IS A CONTROLLED COPY. IF STAMP IS NOT RED, VERIFY THAT YOU ARE USING THE CURRENT REVISION BEFORE PROCEEDING WITH WORK UNDER THIS PROCEDURE

1.0 SCOPE AND APPLICATION

This method is intended for the trace analysis of Polynuclear Aromatic Hydrocarbons (PAHs) by high performance liquid chromatography using a UV and a Fluorescence detector. Table 1 lists the compounds that are routinely determined by this method, and the laboratory Reporting Limit for each analyte. Modifications to the analyte list or procedural changes to reach lower Reporting Limits are allowed if required by client, project or program. Any changes in the analytical procedures must be approved by theOperations Manager and the Quality Services Manager before samples can be analyzed.

TABLE 1. 8310 Analyte List and Reporting Limits

Analyte:	CAS #(a)	Reporting Limit (µg/L)	Reporting Limit (µg/kg)
Acenaphthene	83-32-9	1.0	40
Acenaphthylene	208-96-8	2.0	70
Anthracene	120-12-7	0.20	5.0
Benzo(a)anthracene	56-55-3	0.10	2.0
Benzo(a)pyrene	50-32-8	0.10	2.0
Benzo(b)fluoranthene	205-99-2	0.15	2.0
Benzo(g,h,i)perylene	191-24-2	0.20	2.0
Benzo(k)fluoranthene	207-08-9	0.10	2.0
Chrysene	218-01-9	0.10	5.0
Dibenzo(a,h)anthracene	53-70-3	0.20	2.0
Fluoranthene	206-44-0	0.20	7.0
Fluorene	86-73-7	0.20	7.0
Indeno(1,2,3-cd)pyrene	193-39-5	0.10	2.0
Naphthalene	91-20-3	1.0	40
Phenanthrene	85-01-8	0.20	5.0
Pyrene	129-00-0	0.20	9.0

(a) = Chemical Abstract Services Registry Number

Sample reporting limits are highly matrix-dependent. These are provided for guidance and may not always be achievable.

2.0 SUMMARY OF METHOD

This method provides high performance liquid chromatography (HPLC) conditions for the detection of PAH compounds. Prior to use of this method, appropriate sample extraction techniques must be used. The sample extract is injected into a HPLC using an autosampler, and the compounds in the LC effluent are detected by an ultraviolet (UV) and a Fluorescence detector.

3.0 DEFINITIONS

3.1 **Organic-free reagent water** refers to water in which no target analyte is observed at the Reporting Limit of the compounds of interest. EA Laboratories uses a Culligan reverse osmosis (R/O) water purification system to generate organic-free deionized water.

3.2 **Initial Calibration Verification (ICV)** is a second source calibration standard used to verify the initial calibration and evaluate method performance. It contains all of the analytes listed in Table 1. The stock used to prepare the must be from a source that is different from the stocks used to prepare the calibration standards.

3.3 **Continuing Calibration Verification(CCV)** is a mid level calibration standard used to verify the initial calibration throughout the analytical sequence at a frequency of 1/20 injections.

3.4 **Laboratory Control Samples (LCS)** consist of laboratory pure water or solvent and a measured concentration of the analyte(s) being tested. Laboratory control samples are treated like samples and subjected to all steps of the

analytical procedure.

3.5 **Matrix Spike/Matrix Spike Duplicate (MS/MSD)** are two sample duplicates spiked with a representative subset of the analytes of interest and taken through the entire analytical procedure.

3.6 **Method Blank** is a reagent water or standard solid spiked with all surrogates of interest and taken through the entire analytical procedure.

3.7 **Surrogate** is a non-target compound spiked into all samples and QC samples and taken through the entire analytical procedure to determine purging efficiency, and any possible matrix bias.

4.0 SAMPLE HANDLING, PRESERVATION, AND HOLDING TIME

Sample container, preservation and holding time requirements are given in Table 2. While sample extracts are in the custody of the laboratory, they are stored in the HPLC laboratory at 4 °C ± 2 °C prior to analysis. After analysis is completed, samples are stored by the Sample Management Office in laboratory walk-ins until disposal.

Table 2. Recommended Sample Containers, Preservation Techniques, and Holding Times

Matrix	Container	Preservative	Holding Time
Concentrated Waste Samples	8-oz. wide mouth glass with Teflon liner	None	Samples must be extracted within 14 days of sample collection and extracts analyzed within 40 days following extraction.
Liquid Samples	1-gal. or 2 x 0.5 gal. or 2 x 1L amber glass with Teflon liner	Cool, 4 °C	Samples must be extracted within 7 days of sample collection and extracts analyzed within 40 days following extraction.
Soil/Solid Samples	8 oz wide mouth glass with Teflon liner	Cool to 4 °C	Samples must be extracted within 14 days of sample collection and extracts analyzed within 40 days following extraction.

5.0 INTERFERENCES

5.1 Contamination by carryover can occur. To reduce carryover, the injection loop is rinsed between samples with mobile phase. Whenever an unusually concentrated sample is encountered, the following sample(s) may need to be reanalyzed to determine if carryover contamination had occurred.

5.2 Analytical interferences may be caused by contaminants in solvents, reagents, glassware, and other sample processing hardware. All of these materials must be routinely demonstrated to be free of interferences, under the conditions of the analysis, by running laboratory method blanks.

5.3 Interferences coextracted from the samples will vary considerably from source to source. If analysis of an extracted sample is prevented due to interferences, further cleanup of the sample extract may be necessary. If additional cleanups do not help, the analyst should advise theLaboratory Supervisor and the Laboratory Project Manager. GC/MS analysis of the extract may be necessary.

6.0 APPARATUS AND MATERIALS

6.1 HPLC System

6.1.1 HPLC - Hewlett-Packard Model 1090 equipped with a pump capable of achieving 4000 psi, a 250 µL variable injector and a 254 nm UV detector coupled to the fluorescence detector.

6.1.2 Column: SUPELCOSIL LC-PAH, 5-micron particle size diameter, in a 150-mm x 4.6-mm I.D. stainless steel column.

6.1.3 Data system.

6.2 Materials

6.2.1 High pressure injection syringe - 25 µL.

6.2.2 Disposable cartridge filters - 0.45 um. (PVDF or equivalent)
6.2.3 Micro syringes - Class A, glass, Appropriate sizes.
6.2.4 Pasteur pipets.
6.2.5 Vials - 15 mL, glass, Teflon-lined cap.
6.2.6 Vials- 40 mL, glass, Teflon-lined cap.
6.2.7 Disposable syringes - Plastipak, 3 mL and 10 mL or equivalent.
6.2.8 Volumetric flasks - Appropriate sizes with ground glass stoppers, Class A.
6.2.9 Graduated cylinders - Appropriate sizes.

7.0 SAFETY AND CHEMICAL HYGIENE

The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; however, each chemical compound should be treated as a potential health hazard, and exposure to these chemicals must be reduced to the lowest possible level by whatever means available. The laboratory maintains a reference file of Material Safety Data Sheets (MSDS) for the chemicals specified in this method. Additional information on laboratory safety is available in the Laboratory Safety Plan and from the Laboratory Safety Officer.

8.0 REAGENTS

8.1 Acetonitrile, CH_3CN - HPLC grade.
8.2 Stock standard solutions are purchased as certified solutions.
8.3 Standard Solutions are prepared from the stock standard solution(s) and must include any surrogates used. Table 3 lists the concentrations of the standard solutions used in the initial calibration. All stock standards must be replaced after 1 year or sooner if comparison with check standards indicates a problem.
8.4 Surrogate standards: The analyst should monitor the performance of the extraction, cleanup (when used), analytical system and the effectiveness of the method in dealing with each sample matrix by spiking each sample, standard, and reagent water blank with the surrogates 4,4'-dibromooctafluorobiphenyl, benzo(e)pyrene, and p-terphenyl..
8.5 HPLC Mobile Phase: approximate gradient as follows:

TIME (min)	% WATER	% ACETONITRILE
0	60	40
5	60	40
30	0	100
35	0	100
40	60	40

TABLE 3. Standard Concentrations (µg/L)

	STANDARD (ug/L)				
ANALYTE	1	2	3	4	5
p-Terphenyl (surrogate)	200	500	2000	5000	10000
4,4'-Dibromooctafluorobiphenyl (surrogate)	400	2000	5000	10000	20000
Benzo(e)pyrene (surrogate)	400	2000	5000	10000	20000
Acenaphthene	500	2000	10000	25000	37500
Acenaphthylene	1000	4000	20000	50000	75000
Anthracene	50	200	1000	2500	3750
Benzo(a)anthracene	50	200	1000	2500	3750
Benzo(a)pyrene	50	200	1000	2500	3750
Benzo(b)fluoranthene	100	400	2000	5000	7500
Benzo(g,h,i)perylene	100	400	2000	5000	7500

Benzo(k)fluoranthene	50	200	1000	2500	3750
Chrysene	50	200	1000	2500	3750
Dibenzo(a,h)anthracene	100	400	2000	5000	7500
Fluoranthene	100	400	2000	5000	7500
Fluorene	100	400	2000	5000	7500
Indeno(1,2,3-cd)pyrene	50	200	1000	2500	3750
Naphthalene	500	2000	10000	25000	37500
Phenanthrene	50	200	1000	2500	3750
Pyrene	50	200	1000	2500	3750

8.6 Different standards concentrations may be used if linearity (regression coefficient≥ 0.990) is achieved with a minimum of five points. Refer to Table 4. for standard and surrogate preparation.

9.0 PROCEDURE

9.1 Calibration of HPLC:

9.1.1 Samples are analyzed in a set referred to as an analytical sequence. The sequence begins with instrument calibration or calibration verification followed by samples or extracts. To validate the HPLC system qualitative performance, a continuing calibration standard is analyzed at the end of every sequence and, at a minimum, at a frequency of 1 per 20 samples for this method. If the continuing calibration fails the acceptance criterion the standard should be reanalyzed immediately. If the standard fails again the analysis is stopped, instrument maintenance and/or a new initial calibration is performed. The impact to data usability must be determined for the samples bracketed by the failing standard. If data usability is impacted, all samples after the last successful calibration are to be reanalyzed. The sequence ends when the set of samples has been injected or when qualitative and/or quantitative QC criteria are exceeded. If any analyte in any of the standards falls outside their retention time window, the system is out of control. The cause of the problem must be determined and corrected.
9.1.2 HPLC conditions must be set to ensure acceptable method performance:
9.1.3 Calibration: Calibration curves are required for all target compounds, including surrogate compounds.

9.1.3.1 Initial calibration: For each analyte of interest, prepare calibration standards at the concentrations listed in Table 3 by adding volumes of one or more stock standards to a volumetric flask and diluting to volume with an appropriate solvent.

Inject each calibration standard using the technique that will be used to introduce the actual samples into the HPLC. A regression analysis ($r^2 \geq 0.990$) is used to establish the curve and used for quantitation. If the correlation coefficient criteria is not met, the system is out-of-control. Corrective action must be taken (i.e. instrument maintenance) and a new initial calibration performed.
9.1.3.2 Daily Calibration: The working calibration curve must be verified on each working day. If the calculated result for any analyte varies from the predicted response by more than +/- 15%, reanalyze or reprepare the standard. If the analyte responses are still more than +/- 15%, instrument maintenance and/or a new calibration curve must be prepared for that analyte. The retention time windows are updated based on the daily calibration standard retention time.

9.2 Retention Time Window

9.2.1 Each analyte will be ±0.15 minutes from the average retention time of the initial calibration. Confirm that all calibration standards are within the windows. If not, calibration fails and the initial calibration is performed again.

TABLE 4. Standard Preparation

Calibration Stock Standard Concentration	Analyte(s)	Volume of Stock Standard (uL)	Acetonitrile Final Volume (mL)	Final Concen. (ug/mL)	Calibration Stock Standard Concentration	Analyte(s)	Volume of Stock Standard (uL)	Acetonitrile Final Volume (mL)	Final Concen. (ug/mL)
400 ug/mL	Acenaphthylene	100	10	*4.00*	60 ug/mL	1-Methylnapthylene (surrogate) 2-Methylnapthylene (surrogate)	100	10	*0.60*
		187.5	1.0	*75.0*			187.5	1.0	*11.25*
		250	1.0	*100*			250	1.0	*15.0*
		500	10.0	*20.0*			500	10.0	*3.00*
		625	5.0	*50.0*			625	5.0	*7.50*
200 ug/mL	Acenaphthene Naphthalene	100	10	*2.00*	40 ug/mL	Benzo[b]fluoranthene Benzo[g,h,i]perylene Dibenzo[a,h]anthracene Fluoranthene Fluorene p-Terphenyl (surrogate)	100	10	*0.400*
		187.5	1.0	*37.5*			187.5	1.0	*7.50*
		250	1.0	*50.0*			250	1.0	*10.0*
		500	10.0	*10.0*			500	10.0	*2.00*
		625	5.0	*25.0*			625	5.0	*5.00*
100 ug/mL	Benzo[e]pyrene (surrogate) 4,4'-Dibromooctofluorobiphenol (surrogate)	100	10	*1.00*	20 ug/mL	Anthracene Benzo[a]anthracene Benzo[k]fluoranthene Benzo[a]pyrene Chrysene Indeno[1,2,3-cd]pyrene Phenanthrene Pyrene	100	10	*0.20*
		187.5	1.0	*18.75*			187.5	1.0	*3.75*
		250	1.0	*25.0*			250	1.0	*5.00*
		500	10.0	*5.00*			500	10.0	*1.00*
		625	5.0	*12.5*			625	5.0	*2.50*

9.3 Sample analysis:

9.3.1 If the sample extract responses exceed the calibration range of the system, dilute the extract and reanalyze.
9.3.2 Each sample analysis must be monitored for retention time shifts by evaluating the retention time of the surrogates. If either of the surrogate retention times is outside of the retention time window established during the initial calibration or daily calibration, the sample must be reanalyzed. If the same problem occurs and the standards bracketing the sample do not exhibit the same shift, matrix interferences can be assumed. Notify the laboratory supervisor or manager before proceeding any further.
9.3.3 Tentative identification of an analyte occurs when a peak from a sample extract falls within the retention time window of a target analyte. The peak response of both the UV and the fluorescence detector should be compared to the responses in the analytical standards to determine if the presence of a target analyte is confirmed. Note: the analytes acenaphthylene and 4,4'-dibromooctafluorobiphenyl (surrogate) will not respond with the fluorescence detector. Unless otherwise required by client, project or program, additional confirmation is not required.
9.3.4 The analytical sequence sample volume injected (10 µL), dilutions and standards are identified in the instrument injection log (EAL-SOP-100).
9.3.5 Using the calibration procedure (Section 9.2), identity and quantitate each component peak (using equations in Section 10.0) in the sample chromatogram which corresponds to the compounds used for calibration purposes. Only target compounds that quantitate above the reporting limit will be reported as being present

10.0 CALCULATIONS:

10.1 The concentrations of the target analytes in the sample extracts are calculated by using least squares linear regression from the initial calibration curve. See Table 5 to determine which detector the target analyte is quantitated from. The sample concentration is calculated from the following equations:

10.1.1 Water:

$$\text{Concentration } (\mu g/L) = ((Ce)(Vf)(Df)) / ((Vo))$$

where:
Ce = Concentration of analyte in extract (µg/L).
Vo = Volume of water extracted in liters (L)
Vf = Volume of the concentrated extract in liters (L)
Df = Dilution Factor. The dilution factor for analysis of water samples by this method is defined as follows:

$$\frac{\mu L \text{ extract used to make dilution} + \mu L \text{ clean solvent}}{\mu L \text{ extract used to make dilution}}$$

If no dilution is performed, Df = 1.0.

10.1.2 Soil/Sediment:

$$\text{Concentration } (\mu g/kg) = ((Ce)(Vf)(Df)) / ((Ws)(D))$$

where
Ce, Vf, and Df are as given for water, above.
D = (100 - % moisture) / 100
Ws = Weight of sample extracted in grams (g)

TABLE 5. 8310 ANALYTE LIST AND QUANTITATION DETECTOR	
ANALYTE:	DETECTOR:
4,4'-Dibromooctafluorobiphenyl (surrogate)	UV
Benzo(e)pyrene (surrogate)	Fluor.
Acenaphthene	Fluor.
Acenaphthylene	UV
Anthracene	Fluor.
Benzo(a)anthracene	Fluor.
Benzo(a)pyrene	Fluor.
Benzo(b)fluoranthene	Fluor.
Benzo(g,h,i)perylene	Fluor.
Benzo(k)fluoranthene	Fluor.
Chrysene	Fluor.
Dibenzo(a,h)anthrracene	Fluor.
Fluoranthene	Fluor.
Fluorene	Fluor.
Indeno(1,2,3-cd)pyrene	Fluor.
Naphthalene	Fluor.
Phenanthrene	Fluor.
Pyrene	Fluor.

11.0 QUALITY CONTROL

11.1 Before processing any samples, the analyst must demonstrate the ability to generate acceptable accuracy and precision, the analyst must perform the following operations.

11.1.1 Analyze four LCSs according to the method beginning with preparation of the samples.
11.1.2 Calculate the average recovery (x) in ug/L, and the standard deviation of the recovery (s) in ug/L, for each analyte of interest using the four results.
11.1.3 For each analyte compare s and x with the LCS laboratory control limits for precision and accuracy, established from historical data. If s and x for all analytes meet the acceptance criteria as specified in Table 6., the system performance is acceptable and analysis of actual samples can begin. If any individual s exceeds the precision limit or any individual falls outside the range for accuracy, then the system performance is unacceptable for that analyte.

NOTE: The large number of method analytes represent a substantial probability that one or more will fail at least one of the acceptance criteria when all analytes of a given method are analyzed.

11.1.4 When one or more of the analytes tested fail at least one of the acceptance criteria, the analyst must locate and correct the source of the problem and ***repeat the test for those analytes that failed to meet criteria***. Repeated failure, however, will confirm a general problem with the measurement system. If this occurs, locate and correct the source of the problem and repeat the test for all method compounds beginning with the procedures in Section 11.1

11.3 Laboratory Control Sample: Calculate the spiked compound recoveries in the LCS and determine if they are within the laboratory determined limits.
11.4 If surrogate recoveries or spike recoveries are outside QC limits in the method blank and/or LCS, the impact to data usability needs to be determined. The entire sample batch may need to be reextracted if a significant impact to data useability is determined.
11.5 The MS/MSD recoveries must be determined and compared against the LCS or established MS/MSD QC limits.

11.5.1 If the MS/MSD recoveries are outside the QC limits and the LCS recovery was acceptable, the method is

considered in-control and a matrix bias is assumed.

11.5.2 If the MS/MSD and LCS recoveries are outside the QC limits, the method is considered out-of-control. The entire extraction batch should be re-extracted. Note: High recoveries, indicating a positive bias, may yield useful data if the specific target analyte(s) is not detected above the reporting limits. Notify the Department Supervisor or Division Manager if this situation should occur.

12.0 GC/MS CONFIRMATION

12.1 All positive results must be confirmed by quantitative second column analysis.

12.2 Any confirmation results which differs form the primary column result by more than 25% must be evaluated by the analyst for integration error and/or measurement bias due to the sample matrix. The analyst will report the results form the column with the lowest overall %RSD for initial calibration, or %D for calibration verification, as applicable.

13.0 HPLC CONFIRMATION

13.1 Positive results are confirmed by the secondary (UV) detector, except for acenaphthylene, for which no confirmation is performed. Confirmed results should not differ by more than 25%, but are dependent on analyst judgement and are subject to the following considerations:

13.1.1 Extremely low level results will not appear on the UV detector due to its lesser sensitivity.

13.1.2 The two detectors respond differently to matrix interferences.

13.2 Only primary detector results are reported.

14.0 REFERENCES

United States Environmental Protection Agency. 1986. Revised July 1992. Test Methods for Evaluating Solid Waste. Physical/Chemical Methods. EPA SW-846, 3rd edition, including Update II. U.S. EPA, Washington, D.C.

TABLE 6. QC Acceptance Criteria

Parameter	Test conc. (µg/L)	Limit for s (µg/L) (b)	Range for x (µg/L) (a)	Range P, Ps (%) (b)
Acenaphthene	6.4	40.3	D-6.8	D-124
Acenaphthylene	12.8	45.1	2.8-14.3	D-139
Anthracene	0.64	28.7	0.072-0.72	D-126
Benzo(a)anthracene	0.64	4.0	0.20-0.74	12-135
Benzo(a)pyrene	0.64	4.0	0.013-0.70	D-128
Benzo(b)fluoranthene	1.28	3.1	0.23-1.8	6-150
Benzo(g,h,i)perylene	1.28	2.3	D-1.4	D-116
Benzo(k)fluoranthene	0.64	2.5	D-0.90	D-159
Chrysene	0.64	4.2	D-1.1	D-199
Dibenzo(a,h)anthracene	1.28	2.0	0.038-1.3	D-110
Fluoranthene	1.28	3.0	0.35-1.4	14-123
Fluorene	1.28	43.0	D-1.5	D-142
Indeno(1,2,3-cd)pyrene	0.64	3.0	0.077-0.64	D-116
Naphthalene	6.4	40.7	1.4-6.4	D-122
Phenanthrene	0.64	37.7	0.054-0.86	D-155
Pyrene	0.64	3.4	0.090-0.77	D-140

s = Standard deviation of four recovery measurements, in µg/L.

x = Average recovery for four recovery measurements, in µg/L.

P, P(s) = Percent recovery measured.
D = Detected; result must be greater than zero.

(a) Criteria from 40 CFR Part 136 for Method 610 adjusted for the concentration added.
(b) Criteria from 40 CFR Part 136 for Method 610.

TABLE 7. SUMMARY OF LABORATORY QUALITY CONTROL REQUIREMENTS AND CORRECTIVE ACTION PROCEDURES FOR SW-846 METHODS [A]

QC Check	Frequency	Acceptance Criteria	Laboratory Corrective Action
SW8310 Polynuclear Aromatic Hydrocarbons by HPLC			
Holding time	Water: extract within 7 days of samping; analyze within 40 days of extraction. Solid: extract within 14 days of sampling; analyze within 40 days of extraction.	Extraction and analysis are completed within holding time.	Notify client, determine if laboratory to proceed or if client will resample.
Calibration curve	Initial 5 point calibration, verified daily at mid level and at end of sequence.	1. Initial calibration curve ($r \geq 0.990$) 2. Continuing calibration %D from initial calibration no greater than +/-15 percent.	1. Reanalyze check standard. 2. If similar results are obtained, recalibrate instrument. 3. Evaluate data useability. Reanalyze if data useability is impacted. 4. Document actions taken in a Nonconformance Record, and in the analytical report
Method Blank	1 per analytical batch	Concentration is less than the MDL of the analyte.	1. Determine source of contamination, i.e. instrument, blank water, reagents. 2. Take appropriate corrective action and document. 3. If preparation in error reanalyze or prepare analytical batch. 4. If samples cannot be reanalyzed or reprepared, qualify data. 5. Document actions taken in a Nonconformance Record, and in the analytical report
LCS	1 per analytical batch	Control analyte values are within three SD of mean historical values or method control limits of precision and accuracy. Acceptance criteria are in Table 6.	1. Validate instrument parameters, sensitivity and linearity. Correct problems and document. 2. Validate standard and LCS preparation. Correct any problems and document. 3. Evaluate against project specific DQOs and report data if there is not impact on data usability. 4. If data is not usable, reprepare and reanalyze the method blank, LCS and all field samples in the batch. 5. If repreparation of samples is not possible, qualify data, and note in the report narrative. 6. Document all actions taken in a Nonconformance Record and in the report narrative.
Surrogate spike	All field and QC samples	Surrogate spiking compounds spike concentrations and control limits are in Table 6.	1. Examine all QC (including but not limited to LCS, MB). 2. If surrogate in LCS and/or MB is out-of-control, check quantitation. If quantitation is correct reanalyze. 3. If similar results are obtained from reanalysis, obtain fresh, verified surrogate solution and reanalyze the analytical batch. 4. If samples cannot be reprepared, qualify data. 5. If surrogate spike in LCS and MB are acceptable but out-of-control for samples, validate preparation of samples. If no errors or problems are discovered for sample preparation, qualify data. 6. If errors are discovered in preparation of samples, reprepare QC samples and all affected samples. 7. Document actions taken in a Nonconformance Record and in the report narrative..
MS/MSD	1 set per 20 samples	Matrix spiking compounds, spike concentrations, and control limits are in Table 6.	1. Analyze spiking solution. 2. Verify that correct spiking solutions and amounts were used. 3. Check method blanks and LCS recovery. 4. Reanalyze samples if laboratory error is suspected. 5. Document actions taken.

(a) Abbreviations: **CCV**, continuing calibration verification standard; **F**, graphite furnace atomic absorption; **ICV**, initial calibration verification standard; **LCS**, laboratory control sample; **MDL**, method detection limit; **MS/MSD**, matrix spike/matrix spike duplicate; **NCR**, Nonconformance Record; **IS**, internal standard; **QC**, quality control; **RPD**, relative percent difference; **SD**, standard deviation;

PCBs
SW 8082

EA Engineering, Science, and Technology, Inc.

EA Laboratories

METHOD STANDARD OPERATING PROCEDURE

Number: EAL-M- 8081A/8082 **Rev. No.:** 0

Title: Organochlorine Pesticides and PCBs, and PCB Congeners by Gas Chromatography

Approved By: G. Gregory
Glen Gregory, Section Chief — Date 8/5/98

Approved By: M M Uhlfelder
M. M. Uhlfelder, Quality Services Manager — Date 8/5/98

Approved By: R. Karimi
A. R. Karimi, Laboratory Director — Date 8/5/98

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EA Engineering, Science, and Technology, Inc.

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METHOD STANDARD OPERATING PROCEDURE

Number: EAL-8081A/8082 **Rev. No.:** 0

Revisions		
Rev. No.	**Date of Release**	**Description**
0		Initial distribution for updated/new methods

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METHOD STANDARD OPERATING PROCEDURE

Number: EAL-8081A/8082 **Rev. No.:** 0

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1.0 SCOPE AND APPLICATION

1.1 This SOP is used to determine the concentration of various organochlorine pesticides and polychlorinated biphenyls (PCBs). Tables 1a and 1b list the compounds that are routinely determined by this method, and the laboratory Reporting Limit for each analyte. Analytes with [ ] are only reported when requested by the client. These are provided for guidance and may not always be achievable.

1.2 Modifications to the analyte list or procedural changes to reach lower Reporting Limits are allowed if required by client, project or program. Any changes in the analytical procedures must be approved by the Section Chief and the Quality Services Manager before samples can be analyzed. Sample reporting limits are highly matrix-dependent.

TABLE 1a. METHOD 8081 ANALYTE LIST

ANALYTE:	CAS NUMBER	REPORTING LIMIT (ug/L)	REPORTING LIMIT (ug/kg)
Aldrin	309-00-2	0.05	1.7
alpha-BHC	319-84-6	0.05	1.7
beta-BHC	319-85-7	0.05	1.7
delta-BHC	58-89-9	0.05	1.7
gamma-BHC (Lindane)	319-86-8	0.05	1.7
[Chlordane (technical)]	57-74-9*	1.0	33
α-Chlordane	5103-71-9	0.05	1.7
γ-Chlordane	5103-74-2	0.05	1.7
[Chlorobenzilate]	510-15-6	0.10	3.3
[Diallate]	2303-16-4	0.10	3.3
[DBCP (1,2-Dibromo-3-chloropropane)]	96-12-8	0.10	3.3
4,4'-DDD	72-54-8	0.10	3.3
4,4'-DDE	72-55-9	0.10	3.3
4,4'-DDT	50-29-3	0.10	3.3
Dieldrin	60-57-1	0.10	3.3
Endosulfan I	959-98-8	0.05	1.7
Endosulfan II	33213-65-9	0.10	3.3
Endosulfan sulfate	1031-07-8	0.10	3.3
Endrin	72-20-8	0.10	3.3
Endrin aldehyde	7421-93-4	0.10	3.3
Endrin ketone	53494-70-5	0.10	3.3
Heptachlor	76-44-8	0.05	1.7
Heptachlor epoxide	1024-57-3	0.05	1.7
[Hexachlorobenzene]	118-74-1	0.10	3.3
[Hexachlorocyclopentadiene]	96-12-8	0.10	3.3
[Isodrin]	465-73-6	0.10	3.3
Methoxychlor	72-43-5	0.50	17
Toxaphene	8001-35-2	5.0	170

TABLE 1b. METHOD 8082 ANALYTE LIST				
ANALYTE	BZ #	CAS NUMBER	REPORTING LIMIT (ug/L)	REPORTING LIMIT (ug/kg)
PCB-1016	NA	12674-11-2	1.0	33
PCB-1221	NA	11104-28-2	2.0	67
PCB-1232	NA	11141-16-5	1.0	33
PCB-1242	NA	53469-21-9	1.0	33
PCB-1248	NA	12672-29-6	1.0	33
PCB-1254	NA	11097-69-1	1.0	33
PCB-1260	NA	11096-82-5	1.0	33
2,4'-Dichlorobiphenyl	8		0.1	3.3
2,2',5-Trichlorobiphenyl	18	37680-65-2	0.1	3.3
2,4,4'-Trichlorobiphenyl	28		0.1	3.3
2,2',3,5'-Tetrachlorobiphenyl	44	41464-39-5	0.1	3.3
2,2',4,5'-Tetrachlorobiphenyl	49		0.1	3.3
2,2',5,5'-Tetrachlorobiphenyl	52	35693-99-3	0.1	3.3
2,3',4,4'-Tetrachlorobiphenyl	66	32598-10-0	0.1	3.3
3,3',4,4'-Tetrachlorobiphenyl	69		0.1	3.3
2,2',3,4,5'-Pentachlorobiphenyl	87	38380-02-8	0.1	3.3
2,2',4,5,5'-Pentachlorobiphenyl	101	37680-73-2	0.1	3.3
2,3,3',4,4'-Pentachlorobiphenyl	105		0.1	3.3
2,3',4,4,5'-Pentachlorobiphenyl	118		0.1	3.3
3,3',4,4,5'-Pentachlorobiphenyl	126		0.1	3.3
2,2',3,3',4,4'-Hexachlorobiphenyl	128		0.1	3.3
2,2',3,4,4',5'-Hexachlorobiphenyl	138	35065-28-2	0.1	3.3
2,2',4,4',5,5'-Hexachlorobiphenyl	153	35065-27-1	0.1	3.3
2,3,3',4,4',5-Hexachlorobiphenyl	156		0.1	3.3
3,3',4,4',5,5'-Hexachlorobiphenyl	169		0.1	3.3
2,2',3,3',4,4',5-Heptachlorobiphenyl	170	35065-30-6	0.1	3.3
2,2',3,4,4',5,5'-Heptachlorobiphenyl	180	35065-29-3	0.1	3.3
2,2',3,4,4',5',6-Heptachlorobiphenyl	183	52663-69-1	0.1	3.3
2,2',3,4,4',6,6'-Heptachlorobiphenyl	184		0.1	3.3
2,2',3,4',5,5',6-Heptachlorobiphenyl	187	52663-68-0	0.1	3.3
2,2'3,3',4,4',5,6-Octachlorobiphenyl	195		0.1	3.3
2,2',3,3',4,4',5,5',6-Nonachlorobiphenyl	206	40186-72-9	0.1	3.3
2,2',3,3',4,4',5,5',6'6'-Decachlorobiphenyl	209		0.1	3.3

2.0 SUMMARY OF METHOD

2.1 This SOP provides gas chromatographic conditions for the detection of ppb levels of certain organochlorine pesticides and PCBs.

2.1.1 Prior to the use of this method, appropriate sample extraction techniques must be used.

2.1.1.1 A single extraction is done if no clean-up are required.

2.1.1.2 Should a clean-up be required, the sample extract is split prior to any clean up steps. One portion is processed for organochlorine pesticides and the other for PCB analysis.

2.1.2 The sample extract is injected into a gas chromatograph (GC) using an autosampler, and compounds in the GC effluent are detected by an electron capture detector (ECD).

2.2 The sensitivity of this method usually depends on the level of interferences rather than on instrumental limitations.

2.2.1 If interferences prevent detection of the pesticide target analytes, this method may also be performed on samples that have undergone cleanups such as EAL-M-3620 (Florisil Column Cleanup) and for EAL-M-3660 (Sulfur Cleanup).

2.2.2 Extracts for PCB analysis may be subjected to a modified sulfuric acid cleanup designed specifically for these analyses. This cleanup technique will remove (destroy) many of the single component organochlorine or organophosphorous pesticides.

2.3 Quantitation of PCBs as Aroclors is appropriate for many regulatory compliance determinations, but is particularly difficult when the Aroclors have been weathered by long exposure in the environment.

2.3.1 Due to these difficulties, provisions are outlined for the determination of selected individual PCB congeners. The PCB congener approach potentially affords greater quantitative accuracy when PCBs are know to be present, especially in determining weathered Aroclors.

2.3.2 The laboratory must exercise caution using the congener method when regulatory requirements are based on Aroclor concentrations.

3.0 DEFINITIONS

3.1 **Organic-free reagent water** refers to water in which no target analyte is observed at the Reporting Limit of the compounds of interest. EA Laboratories uses a Culligan reverse osmosis (R/O) water purification system to generate organic-free deionized water.

3.2 **Initial Calibration Verification (ICV)** is a second source calibration standard used to verify the initial calibration and evaluate method performance. It contains all the analytes listed in Table 1. The stock used to prepare the ICV must be from a source that is different from the stocks used to prepare the calibration standards.

3.3 **Continuing Calibration Verification(CCV)** is a mid-level calibration standard used to verify the initial calibration throughout the analytical sequence.

3.4 **Method Blank** is a reagent water or standard solid matrix spiked with all surrogates of interest and taken through the entire analytical procedure.

3.5 **Surrogate** is a non-target compound spiked into all samples and QC samples and taken through the entire analytical procedure to determine purging efficiency, and any possible matrix bias.

3.6 **Laboratory Control Sample (LCS)** is an aliquot of reagent water or a standard solid matrix, e.g. Na_2SO_4 or sand, spiked with a representative subset of the analytes of interest and taken through

the entire analytical procedure. It is used to monitor the analytical process and recoveries of target analytes are compared to laboratory or project specified control limits for precision and accuracy.

3.7 **Matrix Spike/Matrix Spike Duplicate (MS/MSD)** are two sample duplicates spiked with a representative subset of the analytes of interest and taken through the entire analytical procedure. Results are used to evaluate measurement bias due to the sample matrix. Recoveries of target analytes are compared to LCS control limits.

3.8 **Reference** the terminology used to identify the native sample used for matrix spiking purposes.

4.0 SAFETY AND CHEMICAL HYGIENE

4.1 The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; however, each chemical compound should be treated as a potential health hazard, and exposure to these chemicals must be reduced to the lowest possible level by whatever means available. The laboratory maintains a reference file of material safety data sheets for the chemicals specified in this method. Additional information on general laboratory safety is available from the Laboratory Safety Officer.

4.2 The following parameters covered by this method have been tentatively classified as known or suspected, human or mammalian carcinogens: 4,4'-DDT, 4.4'-DDD, the BHCs, and the PCBs. Primary standards of these toxic compounds should be prepared in a hood. A NIOSH/MESA approved toxic gas respirator should be worn when the analyst handles high concentrations of these toxic compounds

4.3 Good laboratory technique dictates the use of appropriate dermal protection. A laboratory coat, eye protection, and gloves are the minimum requirements.

4.4 All wastes must be disposed of following the procedure outlined in EAL-SOP-018.

5.0 SAMPLE HANDLING AND PRESERVATION

5.1 Sample container, preservation and holding time requirements are given in Table 3.

5.2 While sample extracts are in the custody of the laboratory, they are stored in the semivolatiles laboratory at 4°C ± 2°C prior to and during analysis.

5.3 After analysis is completed, samples are stored by the Sample Management Office in laboratory walk-ins until disposal.

Table 3. RECOMMENDED SAMPLE CONTAINERS, PRESERVATION TECHNIQUES, AND HOLDING TIMES

Matrix	Container	Preservative	Holding Time
Concentrated Waste Samples:	8-oz. wide mouth glass with Teflon liner	None	Extract samples within 14 days; analyze extracts within 40 days of extraction.
Liquid Samples: No Residual Chlorine Present	Amber glass , Teflon liner	Cool to 4°C	Extract samples within 7 days; analyze extracts within 40 days of extraction.

Liquid Samples: Residual Chlorine Present	Amber glass , Teflon liner	Add 3 mL of 10% $Na_2S_2O_3$/gal. Cool to 4°C	Extract samples within 7 days; analyze extracts within 40 days of extraction.
Soil/Sediments and Sludges:	8 oz wide mouth glass with Teflon liner	Cool to 4°C	Extract samples within 14 days; analyze extracts within 40 days of extraction.

6.0 INTERFERENCES

6.1 Contamination by carryover can occur whenever high-level and low-level samples are sequentially analyzed. To reduce carryover, the injection syringe must be rinsed between samples with solvent. Whenever an unusually concentrated sample is encountered, the following sample(s) may need to be reanalyzed to determine if carryover contamination had occurred.

6.2 Analytical interferences may be caused by contaminants in solvents, reagents, glassware, and other sample processing hardware. All of these materials must be routinely demonstrated to be free of interferences, under the conditions of the analysis, by running laboratory reagent blanks.

6.3 Interferences coextracted from the samples will vary considerably from source to source. If analysis of an extracted sample is prevented due to interferences, further cleanup of the sample extract may be necessary.

6.4 Interferences by phthalate esters introduced during in sample preparation can pose majors problems in pesticide determinations. These materials may be removed prior to analysis using EAL-M-3640 (Gel Permeation Cleanup).

6.5 The presence of elemental sulfur will result in broad peaks that interfere with the detection of early-eluting organochlorine pesticides.

6.5.1 Sulfur contamination should be expected with sediment samples. EAL-M-3660 is suggested for the removal of sulfur.

6.5.2 Since the recovery of Endrin aldehyde (using the TBA procedure) is drastically reduced, this compound must be determined prior to sulfur cleanup.

6.6 Waxes, lipids, and other high molecular weight materials can be removed by EAL-M-3640 (Gel Permeation Cleanup).

6.7 Other halogenated pesticides or industrial chemicals may interfere with the analysis of pesticides. Certain co-eluting organophosphourus pesticides are eliminated by EAL-M-3640. Co-eluting chlorophenols may be eliminated by using EAL-M-3620 (Florisil cleanup).

6.8 PCBs may also interfere with the analysis of organochlorine pesticides. This problem may be most severe for the analysis of multi-component analysis such as Chlordane, Toxaphene, and Strobane.

6.9 Glassware contamination

6.9.1 Clean all glassware as soon as possible after use by detergent washing with hot water, and rinses with tap water and organic-free reagent water. The glassware is rinsed with

isopropanol, drained and dried in an oven at 400°C for several hours.

6.9.2 Glassware contamination resulting in analyte degradation includes soap residue on glassware may cause degradation of certain analytes. Specifically, aldrin, heptachlor, and most organophosphorous pesticides will degrade in this situation. This problem is especially pronounced with glassware that may be difficult to rinse (e.g., 500-mL K-D flask). These items should be hand-rinsed very carefully to avoid this problem.

NOTE: Oven-drying of glassware used for PCB analyses can increase contamination because PCBs are readily volatilized in the oven and spread to other glassware. It is important that glassware from samples containing high concentration of PCBs are not dried with glassware that may be used for trace analyses.

7.0 APPARATUS/INSTRUMENTATION

7.1 Gas chromatograph: Analytical system complete with gas chromatograph suitable for on-column injections and all required accessories, including detectors, column supplies, data system, gases, and syringes.

7.2 Columns: Wide bore capillary column (30m x 0.53 mm ID). Examples of phases include RTX-5 and RTX-35.

7.3 Detectors: Electron capture (ECD).

7.4 Microsyringe: various sizes

7.5 Vials: Glass, 2-, 10-, and 20-mL capacity with Teflon-lined screw cap.

8.0 STANDARDS AND REAGENTS

8.1 Reagents

8.1.1 Hexane (pesticide quality or equivalent).

8.1.2 Isooctane (2,2,4-trimethylpentane) (pesticide quality or equivalent).

8.2 Standards

8.2.1 The Standards Log Book must be filled out completely following EA-SOP-299.

8.2.2 Stock standard solutions are purchased as certified solutions. These solutions must be replaced after six months or sooner if routine checks indicate a problem.

8.2.3 All standards must be replaced after 6 months, or sooner if comparison with check standards indicates a problem.

8.2.4 Certificates of analysis for all purchased standards will kept on file.

8.2.5 Pesticide Calibration Standards:

8.2.5.1 Pesticide calibration standards are prepared at a minimum of five concentration levels and are prepared through dilution of the stock standards with hexane. One of the concentration levels should be at a concentration at the laboratory Reporting Limit. The remaining concentration levels should correspond to the expected range of concentrations found in real samples or should define the working range of the GC.

8.2.5.2 Stock Standards: Commercially prepared stock standards are purchased pre-mixed at various levels (8-80 µg/mL - Restek Pest Mix A and Pest Mix B or equivalent).

8.2.5.3 Intermediate standards are prepared by diluting the stock standards 500 µL to 100 mL (resulting in concentrations between 0.04 - 0.08 µg/mL).

8.2.5.4 Instrument calibration standards are prepared by diluting the working standards to the concentrations identified in Table 4.

8.2.5.5 Surrogates are added to the standards at the levels indicated in Table 4.

8.2.6 Multi-component Pesticide Standards.

8.2.6.1 Some Toxaphene components, particularly the more heavily chlorinated, are subject to dechlorination reactions. As a result, standards from different vendors may exhibit marked differences which could lead to possible false negative results or to large differences in quantitative results.

8.2.6.2 Stock Standards: Commercially prepared stock standards are purchased pre-mixed.

8.2.6.3 Toxaphene and (technical) Chlordane working standards are prepared individually at 0.100 µg/mL for chlordane and at 0.500 µg/mL for Toxaphene All multi-component standards must contain the surrogates at 20.0 ng/mL.

8.2.6.4 Instrument calibration standards are prepared by diluting the working standards to the concentrations identified in Table 4 (50 µL → 100 µL to a final volume of 100 mL).

8.2.7 Aroclor Calibration Standards

8.2.7.1 A standard containing a mixture of Aroclor 1016 and Aroclor 1260 will include many of the peaks represented in the other five Aroclor mixtures. Therefore, a five point initial calibration using a mixture of Aroclors 1016 and 1260 are sufficient to demonstrate the linearity of the detector response without having to perform initial calibration for each of the remaining Aroclors. This mixture is also used to demonstrate that a sample does not contain peaks that represent any one of the Aroclors.

8.2.7.2 Single standards of each of the other Aroclors are required to aid in pattern recognition. Once linearity is demonstrated by 8.2.7.1, a single standard

corresponding to the point of the linear range of the detector is prepared for the other Aroclors.

8.2.7.3 Stock Standards: Commercially prepared stock standards are purchased pre-mixed at a concentration of 1000 µg/mL..

8.2.7.4 Instrument calibration standards are prepared by diluting the stock standards to the concentrations identified in Table 4 (10 µL →200 µL to 100 mL final volume)..

8.2.8 PCB Congener Calibration Standards

8.2.8.1 If the samples are to be quantitated as individual PCB congeners, standards for the pure congeners are prepared.

8.2.8.2 Stock standards are purchased as commercially-prepared solutions.

8.2.8.3 Calibration standards are prepared at a minimum of five concentrations by dilution the stock standard with Isooctane or hexane at the concentrations specified in Table 4.

Table 4. Concentration of Calibration Standards (ug/mL)

Compound	CON1	CON2	CON3	CON4	CON5
tetrachloro-m-xylene (TCX) - surrogate	0.005	0.020	0.040	0.060	0.080
decachlorobiphenyl (DCB) - surrogate	0.010	0.040	0.080	0.120	0.160
alpha-BHC	0.005	0.020	0.040	0.060	0.080
beta-BHC	0.005	0.020	0.040	0.060	0.080
delta-BHC	0.005	0.020	0.040	0.060	0.080
gamma-BHC (lindane)	0.005	0.020	0.040	0.060	0.080
aldrin	0.005	0.020	0.040	0.060	0.080
heptachlor	0.005	0.020	0.040	0.060	0.080
heptachlor epoxide	0.005	0.020	0.040	0.060	0.080
alpha-chlordane	0.005	0.020	0.040	0.060	0.080
gamma-chlordane	0.005	0.020	0.400	0.060	0.080
dieldrin	0.010	0.040	0.080	0.120	0.160
endosulfan I	0.005	0.020	0.040	0.060	0.080
endosulfan II	0.010	0.040	0.080	0.120	0.160
endosulfan sulfate	0.010	0.040	0.080	0.120	0.160
endrin	0.010	0.040	0.080	0.120	0.160
endrin aldehyde	0.010	0.040	0.080	0.120	0.160
endrin ketone	0.010	0.040	0.080	0.120	0.160
DDT	0.010	0.040	0.080	0.120	0.160
DDE	0.010	0.040	0.080	0.120	0.160
DDD	0.010	0.040	0.080	0.120	0.160
methoxychlor	0.050	0.200	0.400	0.600	0.800
technical chlordane (TCHLOR)	0.100	0.200	0.400	0.800	1.600
toxaphene (TOXAPH)	0.500	1.000	2.000	4.000	8.000
PCB-1016 (AR1016)*	0.100	0.400	0.800	1.200	1.600
PCB-1221 (AR1221)	0.200	0.800	1.600	2.400	3.200
PCB-1232 (AR1232)	0.100	0.400	0.800	1.200	1.600
PCB-1242 (AR1242)	0.100	0.400	0.800	1.200	1.600

Table 4. Concentration of Calibration Standards (ug/mL)					
Compound	CON1	CON2	CON3	CON4	CON5
PCB-1248 (AR1248)	0.100	0.400	0.800	1.200	1.600
PCB-1254 (AR1254)	0.100	0.400	0.800	1.200	1.600
PCB-1260 (AR1260)*	0.100	0.400	0.800	1.200	1.600

*Multi-point initial calibration includes Aroclors 1016 and 1260 only. For all other multi-component analytes, a single point representing the reporting limit is used.

8.2.9 ICV/CCV Standards: Prepared from a separate stock as the calibration standards and at the mid-point of the calibration curve. (See Table 4.)

8.2.10 Matrix Spike Standards:

8.2.10.1 Pesticide stock standards are purchased as commercially-prepared solutions.

8.2.10.1.1 Working standards are prepared from the stock standard. The standard contains the entire target analyte list (not including Toxaphene and technical chlordane) at 0.25, 0.5, and 1,25 µg/mL.

8.2.10.1.2 2.0 mL of matrix spike working stock solution are added to every sample, resulting in 0.05, 0.1, and 0.25 µg/mL in the extract.

8.2.11 Surrogate standards: The analyst should monitor the performance of the extraction, cleanup (when used), analytical system and the effectiveness of the method in dealing with each sample matrix by spiking each sample, standard, and reagent water blank with the pesticide surrogates tetrachloro-m-xylene (TCMX) and decachlorobiphenyl (DCB).

8.2.11.1 Pesticide and Aroclor surrogate stock standards are purchased as commercially-prepared solutions at 200 µg/mL.

8.2.11.1.1 The intermediate surrogate standard is prepared from a stock standard and contains both tetrachloro-m-xylene (TCMX) and decachlorobiphenyl (DCB) by diluting 0.0750 µL if the stock solution to a final volume of 250 mL, resulting in a concentration of 0.6 µg/mL.

8.2.11.1.2 1.0 mL of surrogate stock are added to every sample, resulting in 0.06 µg/mL in the extract.

8.2.12 Column Degradation Check Standard:

8.2.12.1 Stock standards are purchased as commercially-prepared solutions.

8.2.12.2 The standard contains the following compounds at the specified concentrations:

Compound	Conc (µg/mL)
Endrin	0.050
4,4'-DDT	0.10

9.0 PROCEDURES

9.1 Sample Extraction

9.1.1 Samples may be extracted by several different methods for soil, water, and waste matrices:

Matrix	SW846 Method	EA SOP Number
aqueous	Method 3510 (Sep Funnel)	EAL-M3510
aqueous	Method 3520 (Cont Liq-Liq)	EAL-M-3520
soil	Method 3540 (Soxhlet)	EAL-M-3540
soil	Method 3550 (Ultrasonic)	EAL-M-3550
waste	Method 3580 (Waste Dilution)	EAL-M-3580

9.1.2 Hexane-acetone (1:1) may be more effective as an extraction solvent for OCP in some environmental and waste matrices than Methylene Chloride:Actone. The use of Hexane:Acetone generally reduces that amount of interferences that are extracted and improve signal-to-noise.

9.2 Extract Cleanup

9.2.1 Extracts may be "cleaned" using several different techniques depending upon the analytes of interest.

9.2.2 Option for extract cleanup include:

SW846 Cleanup Method	EA SOP Number
Method 3620 (Florisil Cleanup)	EAL-M-3602
Method 3640 (Gel Permeation Cleanup)	EAL-M-3640
Method 3660 (Sulfur Cleanup)	EAL-M-3660
Method 3665 (Sulfuric Acid [mod] Cleanup)	EAL-M-3665

9.3 INSTRUMENTATION

9.3.1 Gas chromatograph: Analytical system complete with gas chromatograph suitable for on-column injection. Hewlett Packard 5890.

9.3.2 Gas chromatography conditions. The chromatographic columns and operating condition for the instrument are:

9.3.2.1 Temperature program for columns 1 and 2:

Injector temperature:	240°C
Carrier gas (He) flow rate:	4-5 mL/min
Initial temperature:	140°C, hold for 5 minutes
Temperature program:	15°C/min to 210°C, then 6°C/min to 240°C, then 15°C/min to 300°C
Final temperature:	300°C, hold until all expected compounds have eluted.
Detector temperature:	325°C

9.3.3 Automated Data Acquisition System:

9.3.3.1 HP3365/Enviroquant. The system allows for the continuous acquisition and storage of all mass spectra obtained throughout the duration of the chromatographic program.

9.3.3.2 The data system has the capability to search any GC/MS data file for ions of a specific mass and plot such ion abundances versus time or scan number (Extracted Ion Current Profile (EICP). The most recent version of the EPA/NIH Mass Spectral Library is used.

9.4 Column Conditioning

9.4.1 Because of the low concentration of pesticide standards injected on a GC/ECD, column adsorption may be a problem when the GC has not been used for a day.

9.4.2 Therefore, the GC column(s) must be primed or deactivated by injecting a PCB or pesticide standard mixture approximately 20 times more concentrated than the mid-level standard. Inject this prior to beginning initial or daily calibration.

9.4.3 Several analytes, including Aldrin, may be observed in the injection just following this system priming. An acceptable blank must be achieved prior to analysis of any subsequent standards and samples.

9.5 Retention Time Windows : Default values of ±0.05 and ±0.07 minutes are used.

9.6 Initial Calibration

9.6.1 Pesticides

9.6.2 Introduce 2-5 μL of each calibration standard into the gas chromatograph using the same technique that will be used to introduce the actual samples.

9.6.3 Using peak responses and mass injected, the software calculates a response factor (RF) for each compound for each concentration in the standard curve, and calculate the percent relative standard deviation (%RSD)of the five responses for each compound.

9.6.4 If the percent relative standard deviation (%RSD) of the response factors is less than 20% over the working range, linearity through the origin can be assumed, and the average

response factor can be used in place of a calibration curve.

9.6.4.1 Should the %RSD fail to meet the 20% criteria, the following may apply:

9.6.4.1.1 Review the results (area counts, response factors) for those analytes which failed the %RSD acceptance criteria to determine if the problem is with just one of the standards. Should this be the case, the analyst has the option to reanalyze and replace the standard in question.

9.6.4.1.2 The analyst also has the option of narrowing the calibration range by replacing one or more of the standards with standards of different concentrations.

9.6.4.1.3 It is important to remember that this may cause samples to be diluted at the high end of the curve.

9.6.4.1.4 In addition, the analyst must verify that changing the standard concentration would not effect any client DQO's (that the new quantitation level is at least as low as any required regulatory limits or action levels).

9.6.4.2 In instances where the RSD for one or more analytes exceed 20%, the initial calibration may still be acceptable if the following conditions are met:

9.6.4.2.1 The mean of the RSD values for <u>all</u> analytes in the calibration is less than or equal to 20%.

9.6.4.2.2 The mean RSD is calculated by summing RSD values for each analyte and dividing by the total number of analytes.

9.6.4.2.3 the mean RSD criteria applies to **all analytes in the standards**, regardless of whether or not they are of interest for a specific project. (If the target analyte is part of the cal standard, its RSD value is included in the evaluation.)

9.6.4.2.4 The lab must provide either a summary of the ICAL data or a specific list of those compounds for which the RSD is exceeded and the results of the mean RSD calculation.

9.6.4.3 If the %RSD criteria for any compound in the initial calibration is exceeded, the lab may opt to use a regression analysis to establish the curve for that compound and used for quantitation.

9.6.4.3.1 The correlation coefficient must be $r \geq 0.990$ ($r^2 \geq 0.980$).

9.6.4.3.2 If the r^2 value does not meet the acceptance criteria, remake the curve (or outlier points) and reanalyze.

9.6.4.3.3 The line must not be forced through the origin. Do not include the origin (0,0) as the sixth point.

9.6.4.3.4 If the y-intercept is higher than the reporting limit.

9.6.4.3.4.1 The best approach is to raise the reporting limit to above the y-intercept (or at least meet the y-intercept) if it would not change required client reporting limits. **This must be approved by the QC Chemist, QSM, and the LPM prior to reporting of the data package.**

9.6.4.3.4.2 *If the y-intercept is above the reporting limits and using average or mean RSD criteria is not an option (due to client DQOs), the initial calibration is not acceptable for that analyte and must be repeated.*

9.6.5 Aroclor Calibrations

9.6.5.1 When PCBs are to be determined as Aroclors, the following calibration scheme is used:

9.6.5.1.1 A 5 level calibration curve will be performed for PCB 1016 and 1260 (see Table 4).

9.6.5.1.2 A single standard corresponding to the reporting level is prepared for the other Aroclors.

9.6.5.1.3 In situations (i.e., client DQOs) where only a few Aroclors are of interest for a specific project, a five-points curve for individual Aroclors may be analyzed along with the 1016/1260 standard described above. (See Table 4.)

9.6.5.1.4 A minimum of 3 peaks must be chosen for quantitation of each Aroclor. The peaks must be characteristic of the Aroclor in question. Choose peaks in the Aroclor standards that are at least 24% of the height of the largest Aroclor peak. For each Aroclor, the chosen peaks should include at least one peak that is unique to that Aroclor. Use at least five peaks for the Aroclor 1016/1260 mix, none of which should be found in both of these Aroclors.

9.6.5.1.5 Aroclor 1016/1260 is used to demonstrate the detector response; therefore this mixture must be applied to the other five Aroclors for which only single standards are analyzed.

9.6.5.1.6 The response factors or calibration factors from the initial calibration are used to evaluate the linearity of the initial calibration.

9.6.5.1.7 Alternately, a 3-5 point multi-point calibration may be performed for

each individual Aroclor. When the multi point calibration approach is chosen, use the calibration factors from these standards to evaluate linearity.

9.6.5.2 When PCBs are to be quantitatively determined as congeners, and initial five-point curve is performed that in includes standards for all of the target congeners.

9.6.5.2.1 This involves the calculation of the mean response or calibration factor, the standard deviation, and the relative standard deviation for each congener or Aroclor peak.

9.7 Initial Calibration Verification

9.7.1 The initial calibration curve must be verified immediately after the calibration is performed using a second source stock.

9.7.2 The % R calculated from the initial calibration curve must be ±15% of the true value.

$$\%Difference = \frac{CF - \overline{CF}}{CF} \times 100$$

where:

$\underline{CF}$	=	Calibration factor from the analysis of the verification standard
$\overline{CF}$	=	Mean calibration factor from the initial calibration

9.7.3 If this standard fails, the standard should be remade immediately and the curve verified.

9.7.4 Should it fail a second time, the analysis is stopped and a new initial calibration curve is prepared.

9.8 Endrin/DDT Breakdown Check

9.8.1 Because Endrin and DDT are easily degraded if the injection port is contaminated with high boiling residue from sample injections or when the injector contains metal fittings, check for degradation by injecting a standard containing on 4,4'-DDT and Endrin. The presence of DDE, DDD, Endrin ketone, or Endrin aldehyde indicates breakdown is occurring.

9.8.2 If the degradation of either DDT or Endrin exceed 15%, corrective action must be taken before proceeding with the calibration.

9.8.3 The breakdown of DDT and Endrin must be measured before samples are analyzed and at the beginning of each 12 hour shift or every 20 samples, whichever is sooner.

9.8.4 Injector maintenance and recalibraton should be completed if the breakdown is greater than 15% of either compound.

$$\%Breakdown\ of\ DDT = \frac{total\ of\ degradtion\ peak\ area\ (DDD + DDE)}{total\ of\ all\ peak\ areas\ DDT + DDE + DDD)} \times 100$$

$$\%\ Breakdown\ of\ Endrin = \frac{total\ of\ degradation\ peak\ areas\ (Endrin\ Aldehyde + Endrin\ ketone\ )}{total\ of\ all\ peak\ area\ (Endrin + Endrin\ aldehyde + Endrin\ ketone)} \times 100$$

9.9 Continuing Calibration Verification

9.9.1 The calibration is verified every 12 hours by injecting the calibration verification standards prior to conducting any sample analyses.

9.9.1.1 A calibration standard must also be injected at intervals of not less than once every 20 samples [every 10 samples may cut down on repeat analysis] and at the end of the analysis sequence.

9.9.1.2 The high and low concentration mixtures of single component analyses and multi-component analyses are alternated from calibration verification.

9.9.2 The calibration factor for each analyte must not exceed a ±15% difference from the nominal (or true) value from the initial calibration.

$$\%Difference = \frac{CF - \overline{CF}}{CF} \times 100$$

where:

9.9.3 Alternatively, if the average of the responses for <u>all</u> analytes is within ±15% (absolute value), then the calibration has been verified. However, the average must include all analytes in the calibration, whether or not they are targets of interest for a specific project. Information regarding the compounds which exceed the 15% criteria must be provided to the client.

9.9.4 If the CCV fails to meet acceptance criteria, the standard should be reanalyzed immediately.

9.9.5 Should the standard fail a second time, the analysis is stopped and a new initial curve is analyzed.

9.9.6 All samples since the previous acceptable verification standard must be rerun. <u>This includes the ending verification standard.</u>

9.10 Matrix Spike (Matrix Spike Duplicate)

9.10.1 With each analytical batch of 20 or fewer client samples an MS/MSD pair must be analyzed.

9.10.2 Spike each aliquot and analyze in the same manner as the reference sample.

9.10.3 Report the results of the both the %R in the MS and MSD samples and the %RPD between the MS and MSD. Note results in the case narrative.

9.10.3.1 Spike Recovery

$$\%\ Spike\ Recovery\ (\%R) = \frac{SSR - SR}{SA} \times 100$$

where:
SSR = Spiked Sample Result
SR = Sample Result
SA = Spike Added

9.10.3.2 %RPD

$$\%\ RPD = \frac{|MSR - MSRD|}{(MSR + MSRD)/2} \times 100$$

where:
MSR = Matrix spike recovery
MSRD = Matrix spike duplicate recovery

(The vertical bars in the formula above indicate the absolute value of the difference, therefore, RSD is always expressed as a positive value.)

10.0 IDENTIFICATION/QUANTITATION/CALCULATIONS

10.1 Quantitation of Pesticides

10.1.1 Analytes are quantitated by comparing the measured response in the sample against the initial standard curve.

10.1.2 The concentration in the extract for each analyte is taken directly from the standard curve.

10.2 Quantitation of Multi-component Analytes

10.2.1 Multi-component analyses present problems in measurement. Suggestions are offered in the following sections for handing Toxaphene and Chlordane.

10.2.1.1 Identification of mixtures (i.e. Chlordane and Toxaphene) is based on the characteristic "fingerprint" retention time and shape of the indicator peak(s); and quantitation is based on the area under the characteristic peaks as compared to the area under the corresponding calibration peak(s) of the same retention time and shape generated using either internal or external calibration procedures. If compound identification or quantitation is precluded due to interference (e.g., broad, rounded peaks or ill-defined baselines are present) cleanup of the extract or replacement of the capillary column or detector is warranted. Rerun the sample on another instrument to determine if the problem results from analytical hardware or the sample matrix. Refer to Method 3600 for the procedures to be followed in sample cleanup.

10.2.1.2 Toxaphene - Toxaphene is manufactured by the chlorination of camphenes. Quantitation of Toxaphene is difficult, but reasonable accuracy can be obtained.

10.2.1.2.1 Quantitate Toxaphene using the total area of the Toxaphene pattern or using 3 to 5 major peaks.

10.2.1.2.2 While Toxaphene contains a large number of compounds that will produce well resolved peaks in a GC/ECD chromatogram, it also contains many other components that are not chromatographically resolved. This unresolved complex mixture results in the "hump" in the chromatogram that is characteristic of this mixture. Although the resolved peaks are important for the identification of the mixture, the area of the unresolved complex mixture contributes a significant portion of the area of the total response.

10.2.1.2.3 To measure total area, construct the baseline of Toxaphene in the sample chromatogram between the retention times of the first and last eluting Toxaphene components in the standard. In order to use the total area approach, the pattern in the sample chromatogram must be compared to that of the standard to ensure that all of the major components in the standard are present in the sample. Otherwise, the sample concentration may be significantly underestimated.

10.2.1.2.4 When Toxaphene is determined using the 3 to 5 peaks approach, the analyst must take care to evaluate the relative areas of the peaks chosen in the sample and standard chromatograms. It is highly unlikely that the peaks will match exactly, but the analyst should not employ peaks from the sample chromatogram whose relative sizes or areas appear to be disproportionally larger or smaller in the sample compared to the standard.

10.2.1.2.5 The heights or areas of the 3 to 5 peaks should be summed together and used to determine the Toxaphene concentration. Alternatively, use each peak in the standard to calculate a

calibration factor for that peak, using the total mass of Toxaphene in the standard. These calibration factors are then used to calculate the concentration of each corresponding peak in the sample chromatogram and the 4 to 6 resulting concentrations are averaged to provide the final result for the sample.

10.2.1.3 Chlordane - Technical Chlordane is a mixture of at least 11 major components and 30 or more minor components that is used to prepare specific pesticide formulations. The CAS Registry number for Technical Chlordane is properly given as 12789-03-6. Trans-Chlordane (or a-Chlordane, CAS RN 5103-71-9) and cares-Chlordane (y-Chlordane, CAS RN 5103-74-2), are the two most prevalent major components of Technical Chlordane.

10.2.1.3.1 The exact percentage of each in the technical material is not completely defined, and is not consistent from batch to batch. Moreover, changes may occur when the technical material is used to prepare specific pesticide formulations. The approach used for evaluating and reporting Chlordane results will often depend on the end use of the results and the analyst's skill in interpreting this multi-component pesticide residue.

10.2.1.3.2 EA Laboratories uses two options for reporting Chlordane: reporting Chlordane (not otherwise specified, 57-74-9), and reporting the individual α- and γ- isomers that can be identified under their individual CAS numbers.

10.2.1.3.3 When the GC pattern of the residue resembles that of Technical Chlordane, EA quantitates Chlordane residues by comparing the total area of the Chlordane chromatogram using three to five major peaks or the total area. If the Heptachlor epoxide peak is relatively small, include it as part of the total Chlordane area for calculation of the residue. If Heptachlor and/or Heptachlor epoxide are much out of proportion, calculate these separately and subtract their areas from the total area to give a corrected Chlordane area.

NOTE: Octachloro epoxide, a metabolite of Chlordane, can easily be mistaken for Heptachlor epoxide on a nonpolar GC column.

10.2.1.3.4 The GC pattern of a Chlordane residue in a sample may differ considerably from that of the Technical Chlordane standard. In such instances, it may not be practical to relate a sample chromatogram back to the pesticide active ingredient Technical Chlordane. Therefore, depending on the objectives of the analysis, the analyst may choose to report the sum of all the identifiable Chlordane components as "Chlordane (n.o.s.)" under the CAS number 57-74-9.

10.2.1.3.5 The second option is to quantitate the peaks of α-Chlordane, γ-Chlordane, and Heptachlor separately against the appropriate reference materials, and report these individual components under their respective CAS numbers. To measure the total area of the Chlordane chromatogram, inject an amount of a Technical Chlordane standard which will produce a chromatogram in which the major peaks are approximately the same size as those in the sample chromatograms.

10.3 Polychlorinated biphenyls (PCBs)

10.3.1 Quantitation of residues of PCB involves problems similar to those encountered in the quantitation of toxaphene, Strobane, and chlordane. In each case, the chemical is made up of numerous compounds. So the chromatograms are multi-peak. Also in each case, the chromatogram of the residue may not match that of the standard.

10.3.2 Mixtures of PCBs of various chlorine contents were sold for many years in the U.S. by the Monsanto Co. under the trade name Aroclor (1200 series and 1016). Though these Aroclors are no longer marketed, the PCBs remain in the environment and are sometimes found as residues in foods, especially fish.

10.3.3 PCB residues are quantitated by comparison to one or more of the Aroclor materials, depending on the chromatographic pattern of the residue.

10.3.3.1 A choice must be made as to which Aroclor or mixture of Aroclors will produce a chromatogram most similar to that of the residue.

10.3.3.2 This may also involve judgment about what proportion of the different Aroclors to combine to produce the appropriate reference material.

10.3.4 Quantitate PCB residues by comparing total area or height of residue peaks to total area of height of peaks from appropriate Aroclor(s) reference materials.

10.3.4.1 Measure total area or height response from the common baseline under all peaks. Use only those peaks from the sample that can be attributed to chlorobiphenyls.

10.3.4.2 These peaks must also be present in the chromatogram of the reference materials. Mixtures of Aroclors may be required to provide the best match of the GC patterns of the sample and reference.

10.4 Calculations

10.4.1 Water

$$Conc_{sample} = \frac{A_x \times V_t \times DF}{\overline{CF} \times V_i \times V_s}$$

where:

$Conc_{sample}$	=	Sample concentration (ug/L)
A_x	=	Area or height of the peak for the compound to be measured.
CF	=	Calibration factor from the initial calibration (area per pg)
V_t	=	Volume of the concentrated extract in milliliters (mL)
V_i	=	Volume of extract injected in microliter (uL). If a single injection is made onto two columns, use one half the volume in the syringe as the volume injected onto each column.
V_s	=	Volume of sample extracted in milliliters (mL)
DF	=	Dilution Factor. If no dilution is performed, DF = 1.0. The dilution factor is defined as follows:

$$DF = \frac{V_e + V_s}{V_e}$$

where:

V_e = Volume of extract used to make dilution (uL)
V_s = Volume of clean solvent (uL)

10.4.2 Soil/Sediment

$$Conc_{sample} = \frac{A_x \times V_t \times DF \times F_{clean}}{\overline{CF} \times V_i \times W_s}$$

where:

$Conc_{sample}$	=	Sample concentration on wet weight basis (ug/kg)
A_x	=	Area or height of the peak for the compound to be measured.
CF	=	Calibration factor from the initial calibration (area per pg)
V_t	=	Volume of the concentrated extract in milliliters (uL)
V_i	=	Volume of extract injected in microliter (uL). If a single injection is made onto two columns, use one half the volume in the syringe as the volume injected onto each column.
W_s	=	Weight of sample extracted in grams (g)
F_{clean}	=	Volume correction factor for cleanup procedures. When GPC cleanup is used the factor is 2.
DF	=	Dilution Factor. If no dilution is performed, DF = 1.0. The dilution factor is defined as follows:

$$DF = \frac{V_e + V_s}{V_e}$$

where:

V_e = Volume of extract used to make dilution (uL)
V_s = Volume of clean solvent (uL)

10.4.3 If sample concentrations are to be reported on a dry weight basis, the following equation is used:

$$Conc_{dry} = \frac{Conc_{sample}}{\%Solid} \times 100$$

where:

$Conc_{dry}$ = Concentration of the sample on a dry weight basis (ug/kg)
$Conc_{sample}$ = Concentration of the sample on a wet weight, or "as received" basis (ug/kg)
%Solid = Percent solids determined gravimetrically.

11.0 QUALITY CONTROL

11.1 Quality Control Acceptance Criteria for this method, including the frequency and corrective actions are shown in Table 9.

11.2 Initial Calibration

11.2.1 In order to produce acceptable sample results, the response of the instrument must be within the working range established by the initial calibration.

11.2.2 The extrapolation of the calibration to concentrations above or below those of the actual calibration standards is not permitted.

11.3 The standard curve is verified using a second source standard (ICV).

11.4 Method Blank: A method blank is analyzed once per analytical batch of 20 or fewer samples to determine whether or not the analysis has introduced any contamination to the samples.

11.5 Laboratory Control Sample: Analyzed once per analytical batch of 20 or fewer samples.

11.6 Matrix Spike/Matrix Spike Duplicate: Analyze one MS/MSD pair once per analytical batch of every 20 or fewer samples.

11.7 Confirmation

11.7.1 Second column confirmation is required for all reported results above the RL. .

11.7.2 All QC criteria must me met in order to use a column for second column confirmation.

11.7.3 If confirmed by GC/MS, a minimum concentration of 10 ng/uL in the final extract for each single-component compound is required.

12.0 REFERENCES

12.1 United States Environmental Protection Agency. 1995. Test Methods for Evaluating Solid Waste. Physical/Chemical Methods. EPA SW-846, 3rd edition, including Update III. U.S. EPA, Washington, D.C.

12.2 United States Environmental Protection Agency. 1995. Contract Lab Program , Statement of Work OLM03.2. U.S. EPA, Washington, D.C.

TABLE 9. ORGANOCHLORINE PESTICIDES AND PCBS BY GC/ECD: SW846 METHODS 8081A and 8082

EAL-M-8081A / 8082 Organochlorine Pesticides and Polychlorinated Biphenyls

QC CHECK	FREQUENCY	ACCEPTANCE CRITERIA	LABORATORY CORRECTIVE ACTION
Holding time	Aqueous samples within 7 days; Solid and waste samples within 14 days; Analyze all extracts within 40 days	Extraction and analysis must be completed within holding time.	Notify client to determine if laboratory is to proceed with analysis or if client will resample
Initial Calibration	Initial 5 point calibration Low standard ≤PQL.	Initial calibration for all single peak target analytes, and Aroclors 1016 and 1260: <20 %RSD; or mean %RSD for all analytes in the standard ≤20%; or use calibration curve ($r \geq 0.990$).	Verify standard preparation and instrument operations, correct problem. Recalibrate instrument. Document actions taken.
Initial Calibration Verification (ICV)	Second source mid-level standard following initial calibration	%D from initial calibration no greater than ±15%.	Verify standard preparation, if incorrect reprepare ICV Verify preparation of calibration standards if incorrect reprepare standards, and recalibrate instrument. Document actions taken.
Continuing Calibration Verification (CCV)	Beginning of each 12 hour analytical shift and after every 20 samples (10 recommended)	%D from initial calibration no greater than ±15%.	If %D>15 and no target analytes are found, no further action is required. Verify standard preparation and instrument operations, correct problem. Repeat initial calibration if problem cannot be identified. Document actions taken.
Degradation Standard	Beginning of analytical sequence	Breakdown of Endrin : <15% Breakdown of DDT : <15%	Evaluate system perform system maintenance. Recalibrate as necessary.
Method Blank	1 per analytical batch Must be analyzed on each instrument used to analyze samples	All target compound concentrations must be <PQL.	Determine source of contamination, i.e. instrument, reagent water, reagents. Take appropriate corrective action and document. If preparation in error reanalyze or prepare analytical batch. If samples cannot be reanalyzed or reprepared, qualify data. document actions taken.

TABLE 9. ORGANOCHLORINE PESTICIDES AND PCBS BY GC/ECD: SW846 METHODS 8081A and 8082

EAL-M-8081A / 8082 Organochlorine Pesticides and Polychlorinated Biphenyls

QC CHECK	FREQUENCY	ACCEPTANCE CRITERIA	LABORATORY CORRECTIVE ACTION
Laboratory Control Sample (LCS)	1 per analytical batch	Values are within project specified control limits (or lab determined) for precision and accuracy.	Reanalyze the LCS. Check instrument parameters, sensitivity and linearity. Correct any problems. Validate LCS preparation. If error is found, reprepare the LCS, and reanalyze the method blank, LCS and all field samples in the batch. If LCS is valid, evaluate against project specific DQOs and report data if there is not impact on data usability. If data is not usable, reprepare and reanalyze the method blank, LCS and all field samples in the batch. If repreparation of samples is not possible, qualify data, and note in the report narrative. Document all actions taken in a NCR and in the report narrative.
Surrogate spike	All field and QC samples	Evaluate %R for primary surrogate. If low, evaluate secondary surrogate compound. Secondary surrogate values must be within project specified control limits (or lba detremined) for precision and accuracy.	Examine all QC (including but not limited to LCS, MB). IF surrogate in LCS and/or MB is out-of-control, check quantitation. If quantitation is correct reanalyze. If similar results are obtained from reanalysis, obtain fresh, verified surrogate solution, and reprepare and reanalyze the analytical batch. If samples cannot be reprepared, qualify data. If surrogate recoveries in LCS and MB are acceptable but below the control limit for any sample, validate sample preparation. Correct any problem then restrict and reanalyze the sample If surrogate recoveries in LCS and MB are acceptable but above the upper control limit for any sample and no target analytes are detected, data is reported with discussion in the analytical narrative.

TABLE 9. ORGANOCHLORINE PESTICIDES AND PCBS BY GC/ECD: SW846 METHODS 8081A and 8082

EAL-M-8081A / 8082 Organochlorine Pesticides and Polychlorinated Biphenyls

QC CHECK	FREQUENCY	ACCEPTANCE CRITERIA	LABORATORY CORRECTIVE ACTION
MS/MSD	1 set per analytical batch	Values are within project specified control limits (or lab determined) for precision and accuracy	I analyte recovery is outside control limits in LCS and data is judged unusable, reanalyze the analytical batch. If LCS acceptable but recovery is outside control limits in MS/MSD, validate preparation of samples. If no errors or problems are discovered for sample preparation, data is reported with discussion in analytical narrative.
2nd Column Confirmation	100% for all positive results	Same acceptance criteria as for primary column.	Same corrective actions as for primary column.

Metals Digestion (Solids)
SW 3050A

EA Engineering, Science, and Technology, Inc.

EA Laboratories

Method

Number: EAL-M-3050A **Rev. No.:** 3

Title: Acid Digestion Of Sediments, Sludges, Paint Chips, Biological Tissues, Wipes, and Soil For Total Metals Determination By ICP and GFAA Spectroscopy

Approved By: _____ 8/19/98
P.A. Christopher, Inorganics Section Chief Date

Approved By: _____ 8/20/98
for M.M. Uhlfelder, Quality Services Manager Date

Approved By: _____ 8/20/98
A.R. Karimi, Laboratory Director Date

EA Engineering, Science, and Technology, Inc.

EA Laboratories

Method

Procedure No.: 3050A **Revision No.:** 3

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EA Engineering, Science, and Technology, Inc.

EA Laboratories

Method

Procedure No.: 3050A **Revision No.:** 3

Revisions		
Rev. No.	**Date of Release**	**Description**
0	NA	Method SOP revised before distribution.
1	5/19/97	Initial distribution.
2	1/28/98	Corrected tape colors in sections 7.1 and 8.1 per internal audit finding LAB-97-002-010. A2LA audit findings concerning paint lead samples.
3		Revised to reflect use of distinct graduated cylinder per sample for USACE projects.

1.0 SCOPE AND APPLICATION - This method is an acid digestion procedure used to prepare sediments, sludges, paint chips, biological tissues, wipes, and soil samples for total metals determinations by inductively coupled argon plasma spectroscopy (ICP) or by furnace atomic absorption spectroscopy (GFAA). Tables 1 and 2 list the method analytes.

Table 1. Method Analytes by ICP Analysis

Analyte:	Symbol	CAS #	Digestion	Initial Reflux Acid	Final Reflux Acid	Final Digestate Matrix
Aluminum	Al	7440-36-0	HNO_3/H_2O_2	HCl	dilute HCl	5%v/v HNO_3/HCl
Antimony	Sb	7740-36-0	HNO_3/H_2O_2	HCl	dilute HCl	5%v/v HNO_3/HCl
Arsenic	As	7440-38-2	HNO_3/H_2O_2	HCl	dilute HCl	5%v/v HNO_3/HCl
Barium	Ba	7440-39-3	HNO_3/H_2O_2	HCl	dilute HCl	5%v/v HNO_3/HCl
Beryllium	Be	7440-41-7	HNO_3/H_2O_2	HCl	dilute HCl	5%v/v HNO_3/HCl
Boron	B	7440-42-8	HNO_3/H_2O_2	HCl	dilute HCl	5%v/v HNO_3/HCl
Cadmium	Cd	7440-43-9	HNO_3/H_2O_2	HCl	dilute HCl	5%v/v HNO_3/HCl
Calcium	Ca	7440-70-2	HNO_3/H_2O_2	HCl	dilute HCl	5%v/v HNO_3/HCl
Chromium	Cr	7440-43-9	HNO_3/H_2O_2	HCl	dilute HCl	5%v/v HNO_3/HCl
Cobalt	Co	7440-48-4	HNO_3/H_2O_2	HCl	dilute HCl	5%v/v HNO_3/HCl
Copper	Cu	7440-50-8	HNO_3/H_2O_2	HCl	dilute HCl	5%v/v HNO_3/HCl
Iron	Fe	7439-89-6	HNO_3/H_2O_2	HCl	dilute HCl	5%v/v HNO_3/HCl
Lead	Pb	7439-92-1	HNO_3/H_2O_2	HCl	dilute HCl	5%v/v HNO_3/HCl
Magnesium	Mg	7439-95-4	HNO_3/H_2O_2	HCl	dilute HCl	5%v/v HNO_3/HCl
Manganese	Mn	7439-96-5	HNO_3/H_2O_2	HCl	dilute HCl	5%v/v HNO_3/HCl
Molybdenum	Mo	7439-98-7	HNO_3/H_2O_2	HCl	dilute HCl	5%v/v HNO_3/HCl
Nickel	Ni	7440-02-0	HNO_3/H_2O_2	HCl	dilute HCl	5%v/v HNO_3/HCl
Potassium	K	7440-09-7	HNO_3/H_2O_2	HCl	dilute HCl	5%v/v HNO_3/HCl
Selenium	Se	7782-49-2	HNO_3/H_2O_2	HCl	dilute HCl	5%v/v HNO_3/HCl
Silica	SiO_2	7631-86-9	HNO_3/H_2O_2	HCl	dilute HCl	5%v/v HNO_3/HCl
Silver	Ag	7440-22-4	HNO_3/H_2O_2	HCl	dilute HCl	5%v/v HNO_3/HCl
Sodium	Na	7440-23-5	HNO_3/H_2O_2	HCl	dilute HCl	5%v/v HNO_3/HCl
Thallium	Tl	7440-28-0	HNO_3/H_2O_2	HCl	dilute HCl	5%v/v HNO_3/HCl
Tin	Sn	7440-31-5	HNO_3/H_2O_2	HCl	dilute HCl	5%v/v HNO_3/HCl
Vanadium	V	7440-62-2	HNO_3/H_2O_2	HCl	dilute HCl	5%v/v HNO_3/HCl
Zinc	Zn	7440-66-6	HNO_3/H_2O_2	HCl	dilute HCl	5%v/v HNO_3/HCl

Table 2. Method Analytes by GFAA Analysis

Analyte:	Symbol	CAS #	Digestion	Initial Reflux Acid	Final Reflux Acid	Final Digestate
Antimony	Sb	7440-36-0	HNO_3/H_2O_2	HNO_3	dilute HNO_3	5% v/v HNO_3
Arsenic	As	7440-38-2	HNO_3/H_2O_2	HNO_3	dilute HNO_3	5% v/v HNO_3
Beryllium	Be	7440-41-7	HNO_3/H_2O_2	HNO_3	dilute HNO_3	5% v/v HNO_3
Cadmium	Cd	7440-43-9	HNO_3/H_2O_2	HNO_3	dilute HNO_3	5% v/v HNO_3
Chromium	Cr	7440-43-9	HNO_3/H_2O_2	HNO_3	dilute HNO_3	5% v/v HNO_3
Lead	Pb	7439-92-1	HNO_3/H_2O_2	HNO_3	dilute HNO_3	5% v/v HNO_3
Selenium	Se	7782-49-2	HNO_3/H_2O_2	HNO_3	dilute HNO_3	5% v/v HNO_3
Silver	Ag	7440-22-4	HNO_3/H_2O_2	HNO_3	dilute HNO_3	5% v/v HNO_3
Thallium	Tl	7440-28-0	HNO_3/H_2O_2	HNO_3	dilute HNO_3	5% v/v HNO_3

2.0 SUMMARY OF METHOD - A representative 1- to 2-g (wet weight) sample is digested in nitric acid and hydrogen peroxide. The digestate is then refluxed with either nitric acid or hydrochloric acid (Tables 1, 2).

3.0 INTERFERENCES - Interferences are discussed in the determinative methods

4.0 APPARATUS AND MATERIALS

4.1 Griffin beakers - 250-mL
4.2 Watch glasses - ribbed
4.3 Drying ovens - That can be maintained at 30°C.
4.4 Whatman cellulose nitrate membrane filters (0.45 µm)
4.5 Vacuum filtration apparatus.
4.6 Graduated cylinders or equivalent - 100 mL, Class A
4.7 250-mL plastic bottles for digestates.
4.8 Electric Hot Plate - Adjustable and capable of maintaining a temperature of 90-95°C.

5.0 REAGENTS

5.1 High purity reagent grade chemicals shall be used
5.2 DI Water. De-ionized reagent water supplied through the laboratory's reverse osmosis system.
5.3 Nitric acid (1:1), HNO_3.
5.4 Hydrochloric acid (concentrated), HCl.
5.5 Hydrogen peroxide (30%), H_2O_2.

6.0 SAMPLES PRESERVATION AND HANDLING - Refer to the applicable determinative EAL Method SOP for sample preservation and handling.

7.0 PROCEDURE FOR ICP ANALYSIS

7.1 Using ***PURPLE*** tape, label a 250-mL Griffin beaker (beakers are segregated and labeled for lead paint analysis only) for each sample with the EA sample number, the final volume of the digestate and the

date of the digestion.

7 2. Mix the sample thoroughly to achieve homogeneity. Separate paint samples from substrate, homogenize with mortar and pestle. Soil samples are stirred and whole wipe samples are consumed for analysis. Tissue samples are homogenized according to EAL SOP-289 prior to digestion. Weigh 1.00-2.00 g of sample to the nearest 0.01 g and transfer to a Griffin 250 mL beaker.

7.3 Add 10 mL of 1:1 HNO_3, mix the slurry, and cover with a ribbed watch glass. Heat the sample to 95°C and reflux for 10 to 15 minutes without boiling. Cool the sample (5 - 10 minutes), add 5 mL of concentrated HNO_3, replace the watch glass, and reflux for 30 minutes. Repeat this last step to ensure complete oxidation. Using a ribbed watch glass, allow the solution to evaporate to 5 mL without boiling, while maintaining a covering of solution over the bottom of the beaker.

7.4 Cool the sample, add 2 mL DI water and 3 mL of 30% H_2O_2. Cover the beaker with a watch glass and return the covered beaker to the hot plate for warming and to start the peroxide reaction. Care must be taken to ensure that losses do not occur due to excessively vigorous effervescence. Heat until effervescence subsides and cool.

7.5 Continue to add 30% H_2O_2 in 1-mL aliquots with warming until the effervescence is minimal or until the general sample appearance is unchanged.

DO NOT ADD MORE THAN A TOTAL OF 10 ML 30% H_2O_2

7.6 Add 5 mL of concentrated HCl and 10 mL of DI water, cover the beaker with a ribbed watch glass, and return to the hot plate. Reflux for an additional 15 minutes without boiling.

7.7 Set up filtration apparatus using a vacuum filtration apparatus with a Whatman cellulose nitrate membrane filter (0.45 µm). Rinse the filter and filter apparatus with DI water and discard the rinsate.

7.8 Wash down the beaker walls and watch glass with DI water, and filter the sample. Transfer the filtered digestate to a 100 mL graduated cylinder and adjust the final volume to 100 mL with DI water.

7.9 Transfer the digestate to a 250 mL plastic digestate bottle. Transfer the beaker label to the digestate bottle. **NOTE: A DISTINCT GRADUATED CYLINDER MUST BE USED FOR EACH SAMPLE FOR USACE PROJECTS.**

7.10 The digestion chemist completes all documentation on the Digestion Log for the samples, signs the log sheet and delivers the digestates to the instrument laboratory where the log sheet is signed and dated by the receiving chemist to document the custody transfer.

8.0 PROCEDURE FOR GFAA ANALYSIS

8.1 Using ***GREEN*** tape, label a 250-mL Griffin beaker for each sample with the EA sample number, the final volume of the digestate and the date of the digestion.

8.2 Mix the sample thoroughly to achieve homogeneity. Separate paint samples from substrate, homogenize with mortar and pestle, soil samples are stirred and whole wipe samples are consumed for analysis. Weigh 1.00-2.00 g of sample to the nearest 0.01 g and transfer to a Griffin 250 mL beaker.

8.3 Add 10 mL of 1:1 HNO_3, mix the slurry, and cover with a ribbed watch glass. Heat the sample to 95°C and reflux for 10 to 15 minutes without boiling. Cool the sample (5 - 10 minutes), add 5 mL of concentrated HNO_3, replace the watch glass, and reflux for 30 minutes. Repeat this last step to ensure complete oxidation. Using a ribbed watch glass, allow the solution to evaporate to 5 mL without boiling, while maintaining a covering of solution over the bottom of the beaker.

8.3 Cool the sample, add 2 mL DI water and 3 mL of 30% H_2O_2. Cover the beaker with a watch glass and return the covered beaker to the hot plate for warming and to start the peroxide reaction. Care must be taken to ensure that losses do not occur due to excessively vigorous effervescence. Heat until effervescence subsides and cool.

8.4 Continue to add 30% H_2O_2 in 1-mL aliquots with warming until the effervescence is minimal or until the

general sample appearance is unchanged.

8.5 Cover the sample with a ribbed watch glass and continue heating the acid-peroxide digestate until the volume has been reduced to approximately 5 mL.

DO NOT ADD MORE THAN A TOTAL OF 10 ML 30% H_2O_2

8.6 Set up filtration apparatus using a vacuum filtration apparatus with a Whatman cellulose nitrate membrane filter (0.45 µm). Rinse the filter and filter apparatus with DI water and discard the rinsate.

8.7 Wash down the beaker walls and watch glass with DI water, and filter the sample Transfer the filtered digestate to a 100 mL graduated cylinder and adjust the final volume to 100 mL with DI water. **NOTE: A DISTINCT GRADUATED CYLINDER MUST BE USED FOR EACH SAMPLE FOR USACE PROJECTS.**

8.8 Transfer the digestate to a 250 mL plastic digestate bottle. Transfer the beaker label to the digestate bottle.

8.9 The digestion chemist completes all documentation on the Digestion Log for the samples, signs the log sheet and delivers the digestates to the instrument laboratory where the log sheet is signed and dated by the receiving chemist to document the custody transfer.

9.0 QUALITY CONTROL

9.1 Samples are prepared in a batch of up to 20 samples. QC required for each digestion batch:

9.1.1 Prep Blank (PBW) or Method Blank - Prepare a method blank with each batch of samples analyzed or a minimum of one method blank per 20 samples. For the wipe analysis, a clean unused wipe is used for the prep blank sample (beakers/watch glasses are segregated and labeled for lead paint analysis only).

9.1.2 Reagent Blank: Prepared for wipe samples only, per each analytical batch to verify glassware cleaning acid baths are free from contamination.

9.1.3 Laboratory Control Sample (LCSW): Prepare and analyze a laboratory control sample (LCS) with each batch samples analyzed. Prepare a minimum of one LCS per 20 samples. The percent recovery must fall within EA Laboratories established control limits or the samples must be reprepared and reanalyzed.

9.1.3.1 Solid Laboratory Control Sample (LCSS): One of the following are employed for the respective solid matrices; (1) NIST Solid LCS, (2) NIST Tissue SRM, (3) Lead paint SRM, and (4) Wipes; clean unused wipe with powdered paint.

9.1.4 Duplicate (DUP) - If sufficient sample is available, duplicate one sample per batch or one for every 20 samples in a batch. Duplicate RPD should be <20%. Reanalyze for verification if laboratory error is suspected. For wipe samples, the duplicate sample must be provided by the client.

9.1.5 Matrix Spike (SPK) - If sufficient sample is available, prepare and analyze a spiked sample once every 20 samples. If the analyzed spike sample is not recovered within the specified limits, reanalyze for verification particularly if laboratory error is suspected. Evaluate bias associated with recovery, flag data as appropriate and address in the narrative of the final report. For wipe samples , the samples can be spiked only when a duplicate sample aliquot has been collected.

9.1.6 Laboratory Background Wipe Test: Perform quarterly monitoring of background levels of lead by preparing and analyzing background wipes. Refer to EAL-M-287; *Preparing and Collecting Wipe Samples.*

9.2 For quality control samples preparation refer to *Preparation of Matrix Spike and LCS Solutions for SW-846, 200 Series CLP Methods and AFCEE Protocols* (EAL-SOP-337).

10.0 REFERENCES

10.1 Rohrbough, W.G.; et al. Reagent Chemicals, American Chemical Society Specifications, 7th ed.; American Chemical Society: Washington, DC, 1986.

10.2 1985 Annual Book of ASTM Standards, Vol. 11.01; "Standard Specification for Reagent Water"; ASTM: Philadelphia, PA, 1985; D1193-77.

10.3 Edgell, K.; USEPA Method Study 37 - SW-846 Method 3050 Acid Digestion of Sediments, Sludges, and Soils. EPA Contract No. 68-03-3254, November 1988.

10.4 United States Environmental Protection Agency. June 1997. Test Methods for Evaluating Solid Waste. Physical/Chemical Methods. EPA SW-846, 3rd edition. including UPDATE III. U.S. EPA, Washington, D.C.

Metals Digestion (ICP)
SW 3010A

EA Engineering, Science, and Technology, Inc.

EA Laboratories

Method

Number: EAL-M-3010A **Rev. No.:** 2

Title: Acid Digestion Of Aqueous Samples For Total Metals For Analysis By ICP Spectroscopy

Approved By: _____ 8/19/98
P.A. Christopher, Inorganics Section Chief Date

Approved By: _____ 8/20/98
gcr M.M. Uhlfelder, Quality Services Manager Date

Approved By: _____ 8/20/98
A. R. Karimi, Laboratory Director Date

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EA Engineering, Science, and Technology, Inc.

EA Laboratories

Method

Procedure No.: 3010A **Revision No.:** 2

Revisions		
Rev. No.	**Date of Release**	**Description**
0	06 Dec 1996	Initial distribution.
1	5/23/97	Revision of Section 6.0
2		Revised for use of distinct graduated cylinder per sample for USACE projects.

EA Engineering, Science, and Technology, Inc.

EA Laboratories

Method

Procedure No.: 3010A **Revision No.:** 2

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1.0 SCOPE AND APPLICATION

1.1 This digestion procedure is used for the preparation of aqueous samples that contain suspended solids for analysis, by inductively coupled argon plasma spectroscopy (ICP). The procedure is used to determine total metals. Samples prepared by Method 3010A may be analyzed by ICP for the following:

ANALYTE:	SYMBOL	CAS #
Aluminum	Al	7429-90-5
Antimony	Sb	7440-36-0
Arsenic	As	7440-38-2
Barium	Ba	7440-39-3
Beryllium	Be	7440-41-7
Cadmium	Cd	7440-43-9
Calcium	Ca	7440-70-2
Chromium	Cr	7440-47-3
Cobalt	Co	7440-48-4
Copper	Cu	7440-50-8
Iron	Fe	7439-89-6
Lead	Pb	7439-92-1
Magnesium	Mg	7439-95-4
Manganese	Mn	7439-96-5
Molybdenum	Mo	7439-98-7
Nickel	Ni	7440-02-0
Potassium	K	7440-09-7
Selenium	Se	7782-49-2
Silver	Ag	7740-22-4
Sodium	Na	7440-23-5
Thallium	Tl	7440-28-0
Vanadium	V	7440-62-2
Zinc	Zn	7440-66-6

1.2 This digestion procedure is not suitable for samples which will be analyzed by graphite furnace atomic absorption spectroscopy except antimony because hydrochloric acid can cause interferences during furnace atomization. Consult Method 3020A for samples requiring graphite furnace analysis.

1.3 Silver is only slightly soluble in the presence of chloride unless there is a sufficient chloride concentration to form the soluble chloride complex. Therefore, low recoveries of silver may occur in field and laboratory quality control samples. This method is suitable for the determination of silver in samples containing concentrations $\leq$100 ug/L.

1.4 Antimony is easily lost through volatilization during digestion. The analyst is cautioned not to boil the samples.

2.0 SUMMARY OF METHOD - A mixture of nitric acid and the material to be analyzed is refluxed in a covered Griffin beaker. This step is repeated with additional portions of nitric acid until the digestate is light in color or until its color has stabilized. After the digestate has been brought to a low volume, it is refluxed with hydrochloric acid and brought up to volume. If sample should go to dryness, it must be discarded and the sample reprepared.

3.0 INTERFERENCES - Interferences are discussed in EAL-M-6010B

4.0 APPARATUS AND MATERIALS

4.1 Griffin beakers - 250-mL
4.2 Watch glasses - Ribbed
4.3 Whatman cellulose nitrate membrane filters (0.45 µm)
4.4 Vacuum filtration apparatus.
4.4 Graduated cylinders or equivalent - 100mL, Class A.
4.5 250 mL plastic bottles for digestates.
4.6 Hot plate or equivalent heating source - adjustable and capable of maintaining a temperature of 90-95-C.

5.0 REAGENTS

5.1 High purity reagent grade chemicals shall be used
5.2 Reagent Water. Reagent water will be interference free.
5.3 Nitric acid (concentrated), HNO3.
5.4 Hydrochloric acid (1:1).

6.0 SAMPLE PRESERVATION AND HANDLING

6.1 Aqueous samples must be acidified to a pH of <2 with nitric acid.
6.2 The maximum holding time is six months from the date of collection.

7.0 PROCEDURE

7.1 Using RED tape, label a 250-mL Griffin beaker for each sample with the EA sample number, the final volume of the digestate and the date of the digestion.
7.2 Using a graduated cylinder, transfer 100-mL of the well-mixed sample to a 250-mL Griffin beaker and add 3 mL of concentrated HNO3. **NOTE: A DISTINCT GRADUATED CYLINDER MUST BE USED FOR EACH SAMPLE FOR USACE PROJECTS.** Cover the beaker with a ribbed watch glass. Place the beaker on a hot plate (90°C - 95°C)) and cautiously evaporate to a low volume (~5 mL.).

Make certain that the sample does not boil.
Make certain that no portion of the bottom of the beaker is allowed to go dry.
Low recoveries of metals, especially antimony, will result.
Should this occur, discard the sample and reprepare.

7.3 Cool the beaker (5 - 10 minutes) and add another 3-mL portion of concentrated HNO3. Cover the beaker with a ribbed watch glass and return to the hot plate. Maintain the temperature of the hot plate so that a gentle reflux action occurs.
7.4 Continue heating, adding additional acid as necessary, until the digestion is complete (25 - 30 min). This is (generally indicated when the digestate is light in color or does not change in appearance with continued

refluxing.
7.5 Again, uncover the beaker or use a ribbed watch glass, and evaporate to a low volume (3 mL), not allowing any portion of the bottom of the beaker to go dry. Cool the beaker. Add a small quantity of 1:1 HCl (10 mL/100 mL of final solution), cover the beaker, and reflux for an additional 15 minutes to dissolve any precipitate or residue resulting from evaporation.
7.6 Set up filtration apparatus using a vacuum filtration apparatus with a Whatman cellulose nitrate membrane filter (0.45 µm). Rinse the filter and filter apparatus with 10% nitric acid and discard the rinsate.
7.7 Wash down the beaker walls and watch glass with 10% nitric acid, and filter the sample Transfer the filtered digestate to a 100 mL graduated cylinder and adjust the final volume to 100 mL with deionized water so that the final acid concentration is 10%. **NOTE: A DISTINCT GRADUATED CYLINDER MUST BE USED FOR EACH SAMPLE FOR USACE PROJECTS.**
7.8 Transfer the digestate to a 250 mL plastic digestate bottle. Transfer the beaker label to the digestate bottle.
7.9 The digestion chemist completes all documentation on the Digestion Log for the samples, signs the log sheet and delivers the digestates to the instrument laboratory where the log sheet is signed and dated by the receiving chemist to document the custody transfer.

8.0 QUALITY CONTROL

8.1 Samples are prepared in a batch of up to 20 samples and includes a Prep Blank, (PBW), a Laboratory Control Sample (LCSW), a Duplicate (DUP), a Matrix Spike (SPK), and a Matrix Spike Duplicate (MSD).
8.2 Refer to EAL-SOP-337; *Preparation of Matrix Spike and LCS Solutions for SW-846, 200 Series CLP Methods and AFCEE Protocols,* for preparation of the required quality control samples.

9.0 REFERENCES

9.1 Rohrbough, W.G.; et al. Reagent Chemicals. American Chemical Society Specifications, 7th ed.; American Chemical Society: Washington, DC,1986.
9.2 1985 Annual Book of ASTM Standards, Vol. 11.01; "Standard Specification for Reagent Water"; ASTM: Philadelphia, PA, 1985; D1193-77.
9.3 United States Environmental Protection Agency. 1995. Test Methods for Evaluating Solid Waste. Physical/Chemical Methods. EPA SW-846, 3rd edition. including UPDATE II. U.S. EPA, Washington, D.C.

Metals by ICP
SW 6010B

EA Engineering, Science, and Technology, Inc.

EA Laboratories

Method

Number: 6010B **Rev. No.:** 0

Title: Determination of Metals for Inductively Coupled Plasma Atomic Emission Spectrometry

Approved By: _____ 3/19/98
P.A. Christopher, Inorganic Section Chief Date

Approved By: _____ 3/20/98
for M.M. Uhlfelder, Quality Services Manager Date

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Method

Procedure No.: 6010B **Revision No.:** 0

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EA Engineering, Science, and Technology, Inc.

EA Laboratories

Method

Procedure No.: 6010B **Revision No.:** 0

Revisions		
Rev. No.	**Date of Release**	**Description**
0		

1.0 SCOPE AND APPLICATION

1.1 This method may be used for the determination of dissolved, suspended or total elements in all matrices, including ground water, aqueous samples, TCLP extracts, industrial and organic wastes, soils, sludge, sediments, and other solid wastes. All matrices require digestion prior to analysis.
1.2 Table 1 lists elements for which this method applies along with recommended wavelengths and laboratory reporting limits using conventional ICP and TRACE ICP. Actual working method detection limits are determined annually and are matrix dependent. Other elements may be determined under this method as needed following the instrument manufacturers specifications. Modifications to the analyte list or procedural changes to reach lower Reporting Limits are allowed if required by client, project or program. Any changes in the analytical procedures must be approved by the Inorganic Division Manager and the Quality Services Manager before samples can be analyzed.

2.0 SUMMARY OF METHOD

2.1 Prior to analysis, samples must be solubilized or digested using appropriate sample preparation methods.
2.2 The method describes a technique for the simultaneous, or sequential, multi-element determination of trace elements in solution. The basis of the method is the measurement of atomic emission by an optical spectroscopic technique. Samples are nebulized and the aerosol that is produced is transported to the plasma torch where excitation occurs. Characteristic atomic-line emission spectra are produced by a radio-frequency inductively coupled plasma (ICP). The spectra are dispersed by a grating spectrometer, and the intensities of the lines are monitored by photomultiplier tubes. The photocurrents from the photomultiplier tubes are processed and controlled by a computer system. A background correction technique is required to compensate for variable background contribution to the determination of trace elements. Background must be measured adjacent to analyte lines on samples during analysis. The position selected for the background intensity measurement, on either or both sides of the analytical line, will be determined by the complexity of the spectrum adjacent to the analyte line. The position used must be free of spectral interference and reflect the same change in background intensity as occurs at the analyte wavelength measured. Background correction is not required in cases of line broadening where a background correction measurement would actually degrade the analytical result. The possibility of additional interferences named in 5.1-5.3 (and tests for their presence are described in 5.4) should also be recognized and appropriate corrections made.

TABLE 1. ANALYTES (ICAPAES TECHNIQUE)

	Reporting Limits						
Element	**Liquid (µg/L)**		**Solid (mg/kg)**		**Wavelength**		
	Conventional	**Trace**	**Conventional**	**Trace**	**JY24**	**JY50**	**TRACE**
Al	200		20		308.215	396.152	
Sb	60	6.0	6.0	0.6	206.833	206.833	206.831 206.832
As	100	10.0	10.0	1.0	188.983 193.699	193.695	189.042
Ba	200		20		455.403	233.527	
Be	5.0		0.5		313.042	313.042	
Cd	5.0	5.0	0.5	0.5	214.438	226.502	226.502
Ca	1000		100		317.933	317.933	
Cr	10.0		1.0		267.716	267.716	
Co	50		5.0		228.616	228.616	
Cu	10		1.0		324.754	324.754	
Fe	100		10.0		259.940 310.050	259.940	
Pb	100	3.0	10.0	0.3	220.353	220.353	220.351 220.352
Li	2.0				670.783		
Mg	1000		100		279.553	279.079	
Mn	15		1.5		257.610	257.610	
Mo	50		5.0		202.030	202.032	
Ni	40		4.0		231.604	231.604	
P	100				213.618		
K	1000		100		766.490	766.490	
Se	100	5.0	10	0.5	196.030	196.026	196.021 196.022
Ag	10		1.0		328.068	328.068	
Na	1000		100		588.995	589.592	
Sr	100				407.771		
Tl	100	10	10	1.0	190.804	190.800	190.864
V	50		5.0		292.402	292.402	
Zn	20		2.0		213.856	213.856	
Sn	25		2.5		189.930	189.926	
Ti	10		1.0		334.940		
B	100		10.0		208.959	249.678	
Si	200		20.0		251.611	251.611	

3.0 DEFINITIONS

3.1 Dissolved--Those elements which will pass through a 0.45 µm membrane filter.

3.2 Suspended--Those elements which are retained by a 0.45 µm membrane filter.
3.3 Total--The concentration determined on a unfiltered sample following vigorous digestion, or the sum of the dissolved plus suspended concentrations.
3.4 Total recoverable--The concentration determined on an unfiltered sample following treatment with hot, dilute mineral acid.
3.5 Instrumental detection limit--The concentration equivalent to a signal, due to the analyte, which is equal to three times the standard deviation of a series of seven replicate measurements of a blank spiked at 3-5 times the expected detection limit measured on three non-consecutive days.
3.6 Sensitivity--The slope of the analytical curve, i.e. functional relationship between emission intensity and concentration.
3.7 Instrument check standard -- Multi-element standard of known concentrations prepared by the analyst to monitor and verify instrument performance on a daily basis.
3.8 Interference check sample--A solution containing both interfering and analyte elements of known concentration that can be used to verify background and interelement correction factors.
3.9 Quality control sample --A solution obtained from an outside source having known concentration values to be used to verify the calibration standards.
3.10 Calibration standards--A series of known standard solutions used by the analyst for calibration of the instrument (i.e., preparation of the analytical curve).
3.11 Linear dynamic range--The concentration range over which the analytical curve remains linear.
3.12 Reagent blank--A volume of deionized, distilled water containing the same acid matrix as the calibration standards carried through the entire analytical scheme.
3.13 Calibration blank--A volume of deionized, distilled water acidified with the same acid matrix as the calibration standards.
3.14 Method of standard addition--The standard addition technique involves the use of the unknown and the unknown plus a known amount of standard.
3.15 Reagent Water -- All references to water in the method refer to reagent water unless otherwise specified. Reagent water will be interference free. Reagent water refers to water that has been generated by any method which would achieve the performance specifications for ASTM Type II water or better.

4.0 SAMPLE HANDLING, PRESERVATION AND HOLDING TIME

4.1 For the determination of trace elements, contamination and loss are of prime concern. Dust in the laboratory environment, impurities in reagents and impurities on laboratory apparatus which the sample contacts are all sources of potential contamination. Sample containers can introduce either positive or negative errors in the measurement of trace elements by (a) contributing contaminants through leaching or surface desorption and (b) by depleting concentrations through adsorption. Thus the collection and treatment of the sample prior to analysis requires particular attention.
4.2 Sample holding times, preservation and suggested collection volumes are listed in Table 2.

TABLE 2. SAMPLE HOLDING TIMES, REQUIRED DIGESTION VOLUMES AND RECOMMENDED COLLECTION VOLUMES FOR METALS DETERMINATIONS

Measurement	Digestion Vol. Req. (a) (mL)	Collection Volume (b) (mL)	Preservative/ Holding Time
Metals (except hexavalent chromium and mercury):			
Total recoverable	100	1000	HNO_3 to pH < 2 6 months
Dissolved	100	1000	Filter on site and preserve; 6 months
Suspended	100	1000	Filter on site; 6 months
Total	100	1000	HNO_3 to pH < 2 6 months

(a) Solid samples should be at least 200 g and usually require no preservation other than storing at 4°C until analyzed.
(b) Either plastic or glass containers may be used.

5.0 INTERFERENCES

There are three types of phenomena that may interfere with the analysis of metals by ICAPAES.

5.1 Spectral interferences. These can be categorized as:

(a) overlap of a spectral line from another element,
(b) unresolved overlap of molecular band spectra;
© background contribution from continuum or recombination phenomena, and
(d) background contribution from stray light from the line emission of high concentration elements.

5.2 Physical interferences. These are generally considered to be transport processes. These include viscosity, surface tensions and high dissolved solids.
5.3 Chemical interferences. These are characterized by molecular compound formation, ionization effects and solute vaporization effects. Normally these effects are not pronounced with ICAPAES techniques, however, if observed they can be minimized by careful selection of operating conditions.
5.4 The following techniques can be used to detect and/or correct for interferences.

5.4.1 Serial dilution--If the analyte concentration is sufficiently high (minimally a factor of 10 above the instrumental detection limit after dilution), an analysis of a dilution should agree within 10% of the original determination. If not, a chemical or physical interference effect should be suspected.
5.4.2 Spike addition--The recovery of a spike addition added at a minimum level of 10X the instrumental detection limit to the original determination should be recovered to within 80 to 120 percent or within the established control limit for that matrix. If not, a matrix effect should be suspected. The use of a standard

addition analysis procedure can usually compensate for this effect. Caution: The standard addition technique does not detect coincidental spectral overlap. If suspected, use of computerized compensation, an alternate wavelength, or comparison with an alternate method is recommended.
5.4.3 Comparison with alternate method of analysis--When investigating a new sample matrix, comparison tests may be performed with other analytical techniques such as atomic absorption spectrometry, or other approved methodology.
5.4.4 Wavelength scanning of analyte line region--If the appropriate equipment is available, wavelength scanning can be performed to detect potential spectral interferences.

6.0 INSTRUMENTATION & OPERATING CONDITIONS

6.1 Instruments, S.A. JY50 Inductively Coupled Argon Plasma Atomic Emission Spectrometer with IBM personal system/2 computer with printer for data reduction and reporting. Nitrogen purge system for analysis of elements with analytical wavelengths in far ultraviolet. The JY50 ICP is operated as follows:

- Sheath gas argon flow rate: 0.2 Liter/min.
- 100 PSI argon pressure to ICP reducer and regulator
- Sample uptake through peristaltic pump
- Plasma argon flow rate: 12-15 Liter/min.
- Pressure of argon within the system: 2.9 Bars

6.2 Instruments, S.A. JY24 Inductively Coupled Argon Plasma Atomic Emission Spectrometer with IBM compatible computer with printer for data reduction and reporting. Nitrogen purge system for analysis of elements with analytical wavelengths in far ultraviolet. The JY24 ICP is operated as follows:

- Sheath gas argon flow rate: 0.2 Liter/min.
- 100 PSI argon pressure to ICP reducer and regulator
- Sample uptake through peristaltic pump
- Plasma argon flow rate: 12-15 Liter/min.
- Pressure of argon within the system: 2.9 Bars

6.3 Thermal Jarrell Ash Trace 61E Inductively Coupled Plasma Emission Spectrometer with IBM compatible computer and printer for data reduction and reporting. The TJA Trace is operated as follows:

- Torch gas - High flow
- Aux gas - Med 1.0 L/min
- Neb gas - 15 psi
- RF power -950W
- Pump rate - 2.4 mL/min

7.0 SAFETY INFORMATION

The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; however, each chemical compound should be treated as a potential health hazard. As such, protective gloves should be worn when handling samples or reagents. Additionally, the Laboratory Safety Office maintains a reference file of material safety data sheets which are available to all personnel involved in the analysis.

8.0 REAGENTS AND STANDARD REFERENCE MATERIAL (SRM)

8.1 Nitric acid (HNO_3); ultra high purity or equivalent.
8.2 Hydrochloric acid (HCl); ultra high purity or equivalent.
8.3 Deionized reagent water: Prepared by a reverse osmosis system by McNew Culligan with an E-Pure polishing system including an ultrafilter assembly. This system passes all ASTM Type I water requirements. Use deionized water for the preparation of all reagents, calibration standards and as dilution water.
8.4. Calibration Stock SRMs, 1,000 mg/L - 10,000 mg/L: The trace metals SRMs are available as commercially prepared stock solutions for each analyte. All parent compounds will either be obtained from NIST or must be traceable to NIST standards. All standard stock solutions are warranted stable for up to a year from the date received. The Calibration Stock SRM solutions are used to prepare calibration substock solutions. For SRMs not obtained from NIST, traceability documentation will be maintained on file in the laboratory.
8.5 Continuing Calibration Check Standard Stock SRMs, 1,000 mg/L - 10,000 mg/L: QC SRMs must meet the same specifications as the Calibration SRMs Stock solutions but must come from a different source than those used for Calibration SRMs. The QC SRM stock solutions are used to prepare the check standards. Traceability documentation for SRMs will be maintained on file at the laboratory.
8.6 The interference check solution is purchased prepared to contain known concentrations of interfering elements that will provide an adequate test of the correction factors.
8.7 Mixed calibration and calibration check standard solutions: Commercially prepared mixed calibration standard solutions are purchased as certified stock solutions. The metals standards obtained from private sources are certified traceable to NIST standards. Traceability documentation is maintained on file at the laboratory. All secondary standards, traceable to NIST SRMs, used for certification and calibration will be checked against existing SRMs the first time a bottle is used and every six months thereafter to confirm its concentration. All results must be within 10 percent of the certified value, thus certifying the integrity of the initial calibration. Records for solution verification are kept on file in the metals office.

9.0 PROCEDURE

9.1 Sample preparation. Preliminary treatment of most matrices is necessary because of the complexity and variability of sample matrices. Solubilization and digestion procedures are presented in Sample Preparation Methods (Methods 3005A-3050A).
9.2 Standard preparation. Working calibration and QC verification standards (initial and continuing) are prepared by dilution of commercially prepared mixed stock solutions or NIST traceable 1000 or 5000 ppm single element stocks. (Refer to EAL-SOP-332 and EAL-SOP-333.) Dilutions are made in volumetric flasks using volumetric pipets or calibrated micropipets. Preparation of calibration, QC standards, and interference checks are recorded in the appropriate standards logbook. Working standards must be verified prior to use and are discarded every three months; QC verification standards are prepared fresh weekly. Standards are prepared in the same acid matrix as the sample digestates. Calibration standards and QC calibration verification (initial and continuing) standards must be prepared from different calibration verification lots of stock solutions.
9.3 Instrument Set-up and Daily Maintenance

9.3.1 Peristaltic pump tubing should be checked at the beginning of each analytical day and changed if excessive wear is evident.
9.3.2 The waste container should be checked and emptied if necessary.
9.3.3 Establish the operating conditions detailed in Section 6.0. With the gases on at specified flows, deionized water should be aspirated and the nebulizer spray chamber should be examined to ensure the sample is being nebulized properly. Light the torch according to manufacturing instructions and allow at least 30 minutes for complete warm-up before continuing with analysis.

9.4 Calibration and Sample Analysis

9.4.1 When the instrument has warmed up sufficiently conduct an autosearch (wavelength search) using the appropriate procedure for each ICP. After the autosearch is completed, select the method for analysis, place the appropriate standards in the autosampler and calibrate the instrument. Use the average intensity of multiple exposures for both standardization and sample analysis to reduce random error. Calibration curves must be composed of a minimum of a blank and three standards. The correlation coefficient of the curve must be 0.995 or better to continue analysis.
The lowest standard should be at or near the reporting limit or PQL. If the calibration curve does not include the PQL standard, then a PQL standard must be run directly after calibration and agree within 10%, in order to show accuracy at the reporting limit.

NOTE : For boron concentrations greater than 500 µg/L extended flush times of 1 to 2 min may be required.

9.4.2 Once calibration is complete, select "analyze" from the menu, create an ID file for the samples to be run, place samples in the autosampler and proceed with analysis according to the following run sequence:

Calibration Blank
Standard 1
Standard 2
Standard 3
CKS (Calibration standard 3)
ICV
ICB
ICSAI (interference check sample)
ICSABI (interference check sample)
CCV1
CCB1
PB (preparation blank)
LCS (laboratory control sample)
SAMPLE 1
SAMPLE 1 DUPLICATE
SAMPLE 1 MATRIX SPIKE
SAMPLE 1 MATRIX SPIKE DUPLICATE
SAMPLE 1 POST DIGESTION SPIKE
SAMPLE 2
SAMPLE 3
SAMPLE 3 SERIAL DILUTION
CCV2
CCB2
ICSAF
ICSABF
CCV3
CCB3

9.4.3 Before beginning the sample run, reanalyze the high mixed calibration standard as if it were a sample. Concentration values obtained should not deviate from the actual values by more than ± 5 percent

9.4.4 Flush the system with the calibration blank solution for at least 1 minute before the analysis of each sample. Analyze the calibration verification standard and the calibration blank after each 10 samples.
9.4.5 Dilute and reanalyze samples that are more concentrated than the highest calibration standard.

10.0 CALCULATIONS

10.1 The microprocessor on the ICP will read will calibration standards in emission counts. Following calibration, the microprocessor will calculate the concentration of each sample in µg/L.
10.2 If dilutions were performed, the appropriate factor must be applied to sample values.
10.3 Data should be rounded to the thousandth place and all results should be reported in µg/L or mg/kg up to three significant figures.

11.0 QUALITY CONTROL

11.1 Employ a minimum of one preparation blank per sample batch to determine if contamination or any memory effects are occurring. A preparation blank is a volume of reagent water acidified with the same amounts of acids as were the standards and samples, and prepared in the same manner as the samples. All analytes must be below the RL as required by the project plan or QAPjP or the samples must be redigested or reanalyzed. In addition, if the method blank concentration of a particular element is above the limit and the samples are greater than 20 times the blank concentration, the data is considered acceptable with no further corrective action. In the event of all other blanks the analyte concentration must be less than 5% of the regulatory limit.
11.2 Employ a minimum of one Laboratory Control Sample (LCS) per batch to determine if the method performance is acceptable. A LCS is a check standard which has been processed through the entire method beginning with sample preparation. LCS recoveries for all elements must be within EA Laboratories established control limits or the samples must be redigested and reanalyzed (possible exception: high LCS recovery and no analyte detected in the sample). Solid samples require the preparation of a solid matrix LCS. A tissue (NIST SRM) matrix LCS is prepared for the analysis of tissue samples in addition to a liquid LCS.
11.3 Initial and Continuing Calibration Verification. Analyze an appropriate instrument check standard containing the elements of interest at a frequency of 10%. This check standard is used to determine instrument drift. If agreement is not within ± 10% of the expected values or within the established control limits, whichever is lower, the analysis is out of control. The analysis should be terminated, the problem corrected, and the instrument recalibrated. All samples following the last successful CCV must be reanalyzed after recalibration.
11.4 Analyze the calibration blank at a frequency of 10%. The result should be within the ± 3X the MDL or less than the reporting limit. If not, terminate the analysis, correct the problem and recalibrate the instrument. All samples following the last successful CCB must be reanalyzed after recalibration.
11.5 Analyze duplicate samples at the frequency of 5% (1 per 20 or per batch). A duplicate sample is a sample brought through the whole sample preparation and analytical process in duplicate. Duplicate RPD should be <20% or matrix effects should be suspected. Reanalyze for verification and redigest and reanalyze if lab error is suspected.
11.6 Serial dilution: If the analyte concentration is sufficiently high (minimally, a factor of 10 above the instrumental detection limit after dilution), an analysis of a 1:4 dilution should agree within ±10% of the original determination. If not, a chemical or physical interference effect should be suspected.
11.7 Analyze one spike sample for every 20 samples or analytical batch. A matrix spike is a sample brought through the entire sample preparation and analytical process. The recovery of the matrix spike should be recovered to within 75-125% of the known value. If the spike is not recovered within the specified limits, a matrix effect should be suspected. Reanalyze for verification and redigest and reanalyze

if lab error is suspected. Evaluate bias associated with recovery, flag data as appropriate and address in the narrative of the final report.

11.8 Analyze one spike duplicate sample for every 20 samples or analytical batch. A matrix spike duplicate is a sample brought through the entire sample preparation and analytical process. The recovery of the matrix spike should be recovered to within 75-125% of the known value. If the spike is not recovered within the specified limits, a matrix effect should be suspected. Reanalyze for verification and redigest and reanalyze if lab error is suspected. Evaluate bias associated with recovery, flag data as appropriate and address in the narrative of the final report. The RPD between MS and MSD should be within +/-20%.

11.9 To verify the inter-element and background correction factors, analyze the interference check sample at the beginning and end of the sample run. Results should fall within the established control limits of ± 20%. If not, terminate the analysis, correct the problem and recalibrate the instrument.

11.10 Standard Addition (Analytical/Post Digestion) Spike. Refer to Section 5.4.2. If the spike recovery does not meet the 75-125% criteria, a matrix effect should be suspected.

11.11 In addition, for TCLP extracts the method of standard addition is required if the matrix spike recovery is <50%, the sample concentration is less than the regulatory limit, but within 20% of the regulatory limit.

12.0 REFERENCES

United States Environmental Protection Agency. 1986. Update I, July 1992. Test Methods for Evaluating Solid Waste. Physical/Chemical Methods. EPA SW-846, 3rd edition. U.S. EPA, Washington, D.C.

Metals Digestion (GFAA)
SW 3020A

EA Engineering, Science, and Technology, Inc.

EA Laboratories

Method

Number: EAL-M-3020A **Rev. No.:** 3

Title: Acid Digestion Of Aqueous Samples For Total Metals For Analysis By GFAA Spectroscopy

Approved By: _____ 8/19/98
P.A. Christopher, Inorganics Section Chief Date

Approved By: _____ 8/20/98
for M.M. Uhlfelder, Quality Services Manager Date

Approved By: _____ 8/20/98
A. R. Karimi, Laboratory Director Date

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EA Engineering, Science, and Technology, Inc.

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Method

Procedure No.: 3020A **Revision No.:** 3

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EA Engineering, Science, and Technology, Inc.

EA Laboratories

Method

Procedure No.: 3020A **Revision No.:** 3

Revisions		
Rev. No.	**Date of Release**	**Description**
0	N/A	Revised before release
1	5/19/97	Initial distribution
2	2/27/98	Revised note to reflect use of reduced volumes. Added note about using increased volumes where necessary. Per internal audit finding LAB-97-002-009
3		Revised to reflect use of distinct graduated cylinder per sample for USACE projects.

1.0 SCOPE AND APPLICATION

1.1 This digestion procedure is used for the preparation of aqueous samples, mobility-procedure extracts, and wastes that contain suspended solids for analysis by furnace atomic absorption spectroscopy (GFAA) for the metals listed below. The procedure is used to determine the total amount of the metal in the sample.
1.2. Samples prepared for analysis by GFAA for the metals listed in Table 1.

Table 1. Method Analytes

Analyte	Symbol	CAS #
Arsenic	As	7440-38-2
Cadmium	Cd	7440-43-9
Chromium	Cr	7440-47-3
Lead	Pb	7439-92-1
Silver	Ag	7440-22-4
Thallium	Tl	7440-28-0
Selenium	Se	7782-49-2

1.3 For an alternative digestion and GFAA analysis of arsenic and selenium, see Methods 7060 and 7740. For an alternative digestion and GFAA analysis of silver, see Method 7761.

2.0 SUMMARY OF METHOD

A mixture of nitric acid and the material to be analyzed is refluxed in a covered Griffin beaker. This step is repeated with additional portions of nitric acid until the digestate is light in color or until its color has stabilized. After the digestate has been brought to a low volume, it is cooled and brought up in dilute nitric acid such that the final dilution contains 3% (v/v) nitric acid. This percentage will vary depending on the amount of acid used to complete the digestion.

3.0 INTERFERENCES - Interferences are discussed in the determinative method.

4.0 APPARATUS AND MATERIALS

4.1 Griffin beakers - 250-mL
4.2 Watch glasses - ribbed
4.3 Whatman cellulose nitrate membrane filters (0.45 µm)
4.4 Vacuum filtration apparatus.
4.5 Graduated cylinders or equivalent - 100 mL, Class A.
4.6 250 mL plastic bottles for digestates.
4.7 Hot plate - adjustable and capable of maintaining a temperature of 90-95°C.

5.0 REAGENTS

5.1 High purity reagent grade chemicals shall be used
5.2 Reagent Water - Deionized water supplied by the laboratory's reverse osmosis treatment system.
5.3 Nitric acid (concentrated), HNO_3.

6.0 SAMPLE PRESERVATION AND HANDLING

6.1 Aqueous samples must be acidified to a pH of <2 with nitric acid.
6.2 The maximum holding time is six months from the date of collection.

7.0 PROCEDURE

7.1 Using ***YELLOW*** tape, label a 250-mL Griffin beaker for each sample with the EA sample number, the final volume of the digestate and the date of the digestion.
7.2 Using a graduated cylinder, transfer 50 mL* of the well-mixed sample to a 250-mL Griffin beaker and add 1.5 mL of concentrated HNO_3. **NOTE: A DISTINCT GRADUATED CYLINDER MUST BE USED FOR EACH SAMPLE FOR USACE PROJECTS.** Cover the beaker with a ribbed watch glass. Place the beaker on a hot plate (90 - 95°C) and cautiously evaporate to a low volume (5 mL), making certain that the sample does not boil and that no portion of the bottom of the beaker is allowed to go dry. Cool the beaker (5 -10 minutes) and add another 1.5-mL portion of concentrated HNO_3. Cover the beaker with a ribbed watch glass and return to the hot plate. Increase the temperature of the hot plate so that a gentle reflux action occurs.
7.3 Continue heating, adding additional acid as necessary, until the digestion is complete (25-30 minutes). This is generally indicated when the digestate is light in color or does not change in appearance with continued refluxing.
7.4 When the digestion is complete, use a ribbed watch glass and evaporate to a low volume (3 mL). Add approximately 10 mL of DI water, mix, and continue warming the beaker for 10 to 15 minutes.

Do not allow any portion of the bottom of the beaker to go dry.

7.5 Set up filtration apparatus using a vacuum filtration apparatus with a Whatman cellulose nitrate membrane filter (0.45 µm). Rinse the filter and filter apparatus with DI water and discard the rinsate.
7.6 Wash down the beaker walls and watch glass with DI water, and filter the sample. Transfer the filtered digestate to a 100 mL graduated cylinder and adjust the final volume to 50 mL* with DI water. **NOTE: A DISTINCT GRADUATED CYLINDER MUST BE USED FOR EACH SAMPLE FOR USACE PROJECTS.**
7.7 Transfer the digestate to a 250 mL plastic digestate bottle. Transfer the beaker label to the digestate bottle.
7.8 The digestion chemist completes all documentation on the Digestion Log for the samples, signs the log sheet and delivers the digestates to the instrument laboratory where the log sheet is signed and dated by the receiving chemist to document the custody transfer.

* Where project specific requirements dictate (noted on the digestion assignment page) that a 100 mL initial volume is used, bring to final volume of 100 mL and double all reagent volumes.

8.0 QUALITY CONTROL

8.1 Samples are prepared in an analytical batch (20 samples or fewer). Included in the batch are a Prep Blank, (PBW), a Laboratory Control Sample (LCSW), a Duplicate (DUP), a Matrix Spike (SPK), and a Matrix Spike Duplicate (MSD).
8.2 Refer to EAL-SOP-337; *Preparation of Matrix Spike and LCS Solutions for SW-846, 200 Series CLP Methods and AFCEE Protocols* for the preparation of the required quality control samples.

9.0 REFERENCES

9.1 Rohrbough, W.G.; et al. Reagent Chemicals. American Chemical Society Specifications, 7th ed.; American Chemical Society: Washington, DC, 1986.
9.2 1995 Annual Book of ASTM Standards, Vol. 11.01; "Standard specification for Reagent Water"; ASTM: Philadelphia, PA, 1995; D1193-91.
9.3 United States Environmental Protection Agency. 1995. Test Methods for Evaluating Solid Waste. Physical/Chemical Methods. EPA SW-846, 3rd edition. including UPDATE II. U.S. EPA, Washington, D.C.

Metals by GFAA
SW 7000 series

EA Engineering, Science, and Technology, Inc.

EA Laboratories

Method

Number: 7000S **Rev. No.:** 1

Title: Determination of Metals in Water, Wastes and Solids by Graphite Furnace Atomic Absorption (GFAA) Spectrometry

Prepared By:	P.A. Christopher, Operations Manager	20 November 95
Revised By:	A.M. Thacker, QC Specialist	12/6/96 Date
Approved By:	M.M. Uhlfelder, Quality Services Manager	12/6/96 Date
Approved By:	P.A. Christopher, Operations Manager	12/6/96 Date

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EA Engineering, Science, and Technology, Inc.

EA Laboratories

Method

Procedure No.: 7000S **Revision No.:** 1

Revisions		
Rev. No.	**Date of Release**	**Description**
0	not released	Initial distribution. Format revised.
1		Added SOP reference, editorial revisions.

EA Engineering, Science, and Technology, Inc.

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Method

Procedure No.: 7000S **Revision No.:** 1

Controlled Distribution

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1.0 SCOPE AND APPLICATION

1.1 Liquid. This method is approved for determining dissolved, suspended or total metals in TCLP extracts, aqueous, wastes, groundwater, drinking, surface and saline water, domestic and industrial wastes.

1.2 Solid or Semisolid. This method is approved for measuring all metals in soils, sediments, bottom deposits and sludge type materials.

1.3 Analytes, reporting limits and recommended wavelengths are detailed in Table 1. Other elements may be determined under the method following the instrument manufacturer's specifications. Modifications to the analyte list or procedural changes to reach lower reporting limits are allowed if required by client, project or program. Any changes in the analytical procedures must be approved by the Inorganic Division Manager and the Quality Services Manager before samples can be analyzed.

TABLE 1. ANALYTES (AA, FURNACE TECHNIQUE)

ANALYTE:	CAS #	REPORTING LIMIT		WAVELENGTH
		(µg/L)	(mg/kg)	
Antimony (Sb)	7440-36-0	6	0.60	217.6
Arsenic (As)	7440-38-2	10	1.0	193.7
Beryllium (Be)	7440-41-7	1	0.1	234.9
Cadmium (Cd)	7440-[illegible]-9	5	0.50	228.8
Chromium (Cr)	7440-47-3	10	1.0	357.9
Copper (Cu)	7440-50-8	10	1.0	324.8
Lead (Pb)	7439-92-1	3	0.30	283.3
Nickel (Ni)	7440-02-0	10	1.0	232.0
Silver (Ag)	7440-22-4	10	1.0	328.1
Thallium (Tl)	7440-28-0	10	1.0	276.8
Selenium (Se)	7782-49-2	5.0	0.50	196.0

Actual working method detection limits are determined annually and are matrix dependent.

2.0 SUMMARY OF METHOD

2.1 Drinking water free of particulate matter may be analyzed directly.

2.2 Preliminary treatment of waste water, ground water, TCLP extracts, and industrial waste is always necessary because of the complexity and variability of sample matrix. Solids, slurries, and suspended material must be subjected to a solubilization process before analysis. This process may vary because of the metals to be determined and the nature of the sample being analyzed.

2.3 When using the furnace technique in conjunction with an atomic absorption spectrophotometer, a representative aliquot of a sample is placed in the graphite tube in the furnace, evaporated to dryness, charred, and atomized. As a greater percentage of available analyte atoms is vaporized and dissociated for absorption in the tube rather than the flame, the use of smaller sample volumes or detection of lower concentrations of elements is possible. The principle is essentially the same as with direct aspiration atomic absorption, except that a furnace, rather than a flame, is used to atomize the sample. Radiation from a given excited element is passed through the vapor containing ground-state atoms of that element. The intensity of the transmitted radiation decreases in proportion to the amount of the ground state element in the vapor. The metal atoms to be measured are placed in the beam of radiation by increasing the temperature of the furnace, thereby causing the injected specimen to be volatilized. A monochromator isolates the characteristic radiation from the hollow cathode lamp or electrodeless discharge lamp, and a photosensitive device measures the attenuated transmitted radiation. When using furnace

techniques, however, the analyst should be cautioned as to possible chemical reactions occurring at elevated temperatures which may result in either suppression or enhancement of the analysis element.

3.0 DEFINITIONS

3.1 Dissolved--Those elements which will pass through a 0.45 µm membrane filter.

3.2 Suspended-Those elements which are retained by a 0.45 µm membrane filter.

3.3 Total--The concentration determined on a unfiltered sample following vigorous digestion, or the sum of the dissolved plus suspended concentrations. (See Section 9.1)

3.4 Total recoverable--The concentration determined on an unfiltered sample following treatment with hot, dilute mineral acid.

3.5 Instrumental detection limit--The concentration equivalent to a signal, due to the analyte, which is equal to three times the standard deviation of a series of seven replicate measurements of a reagent blank spiked at 3-5 times the expected detection limit measured on three non-consecutive days.

3.6 Sensitivity--The slope of the analytical survey, i.e. functional relationship between emission intensity and concentration.

3.7 Instrument check standard -- Multi-element standard of known concentrations prepared by the analyst to monitor and verify instrument performance on a daily basis.

3.9 Quality control sample--A solution obtained from an outside source having known, concentration values to be used to verify the calibration standards.

3.10 Calibration standards--a series of known standard solutions used by the analyst for calibration of the instrument (i.e., preparation of the analytical curve).

3.11 Linear dynamic range--The concentration range over which the analytical curve remains linear.

3.12 Reagent blank--A volume of deionized, distilled water containing the same acid matrix as the calibration standards carried through the entire analytical scheme.

3.13 Calibration blank--A volume of deionized, distilled water acidified with the same acid matrix as the calibration standards.

3.14 Method of standard addition--The standard addition technique involves the use of the unknown and the unknown plus a known amount of standard.

3.15 Reagent Water -- All references to water in the method refer to reagent water unless otherwise specified. Reagent water will be interference free. Reagent water refers to water that has been generated by any method which would achieve the performance specifications for ASTM Type II water.

4.0 SAMPLE HANDLING, PRESERVATION, AND HOLDING TIME

4.1 All sample containers must be prewashed with detergents, acids, and Type II water. Plastic and glass containers are both suitable.

4.2 Special containers (e.g., containers used for volatile organic analysis) may have to be used if very volatile arsenic compounds are to be analyzed.

4.3 Aqueous samples must be acidified to a pH of <2 with nitric acid, and analyzed within 6 months of collection.

4.4 Nonaqueous samples shall be refrigerated, and analyzed within 6 months of collection.

4.5 Silver standards and samples should be stored in the dark, in brown bottles, and refrigerated, when possible.

5.0 INTERFERENCES

5.1 Although the problem of oxide formation is greatly reduced with furnace procedures because atomization occurs in an inert atmosphere, the technique is still subject to chemical interferences. The composition of the sample matrix can have a major effect on the analysis. It is those effects which must be determined and taken into consideration in the analysis of each different matrix encountered. To help verify the absence of matrix or chemical interference, the serial dilution technique may be used. Those samples which indicate the presence of interference should be treated in one or more of the following ways:

- Successively dilute and reanalyze the samples to eliminate interferences.
- Modify the sample matrix either to remove interferences or to stabilize the analyte. Examples are the addition of ammonium nitrate to remove alkali chlorides and the addition of ammonium phosphate to retain cadmium. Refer to the following table for a list of recommended matrix modifiers:

Element	Matrix Modifier
As	Nickel Nitrate
Se	Nickel Nitrate
Pb	Magnesium Nitrate/Ammonium Phosphate
Tl	H_2SO_4
Sb	Nickel Nitrate
Cd	Magnesium Nitrate/Ammonium Phosphate
Be	Magnesium Nitrate
Ag, Cr, Cu, Ni	None

- Analyze the sample by method of standard additions while noticing the precautions and limitations of its use.

5.2 Gases generated in the furnace during atomization may have molecular absorption bands encompassing the analytical wavelength. When this occurs, use either background correction or choose an alternate wavelength. Background correction may also compensate for nonspecific broad-band absorption interference.

5.3 Continuum background correction cannot correct for all types of background interference. When the background interference cannot be compensated for, chemically remove the analyte or use an alternate form of background correction, e.g., Zeeman background correction.

5.4 Interference from a smoke-producing sample matrix can sometimes be reduced by extending the charring time at a higher temperature or utilizing an ashing cycle in the presence of air. Care must be taken, however, to prevent loss of the analyte.

5.5 Samples containing large amounts of organic materials should be oxidized by conventional acid digestion before being placed in the furnace. In this way, broad-band absorption will be minimized.

5.6 Anion interference studies in the graphite furnace indicate that, under conditions other than isothermal, the nitrate anion is preferred. Therefore, nitric acid is preferable for any digestion or solubilization step. If another acid in addition to HNO_3 is required, a minimum amount should be used. This applies particularly to hydrochloric and, to a lesser extent, to sulfuric and phosphoric acids.

5.7 Carbide formation resulting from the chemical environment of the furnace has been observed. Carbide formation is greatly reduced and the sensitivity increased with the use of pyrolytically coated graphite.

5.8 Cross-contamination and contamination of the sample can be major sources of error because of the extreme sensitivities achieved with the furnace. The sample preparation work area should be kept scrupulously clean. All glassware should be cleaned as directed in EAL-SOP-062. Special attention should be given to reagent blanks in both analysis and in the correction of analytical results. Lastly, pyrolytic graphite, because of the production process and handling, can become contaminated. As many as five to ten high- temperature burns may be required to clean the tube before use.

5.9 In addition to the normal interferences experienced during graphite furnace analysis, arsenic, cadmium, selenium, and silver analyses can suffer from severe nonspecific absorption and light scattering caused by matrix components during atomization. Arsenic and selenium analyses are particularly susceptible to these problems because of their low analytical wavelength (193.7 nm, arsenic) (196.0 nm, selenium). Simultaneous background correction must be employed to avoid erroneously high results. Aluminum is a severe positive interferent in the analysis of arsenic, especially using D2 arc background correction. For selenium analysis, high iron levels can give overcorrection with deuterium background. Zeeman background correction is very useful in these situations.

5.10 If the analyte is not completely volatilized and removed from the furnace during atomization, memory effects will occur. If this situation is detected by means of blank burns, the tube should be cleaned by operating the furnace at full power at regular intervals in the analytical scheme.

6.0 INSTRUMENTATION AND OPERATING CONDITIONS

6.1 Perkin Elmer Model 5100 Zeeman Atomic Absorption Spectrophotometer, equipped with a model HGA-600 Graphite Furnace and a Model AS-60/70 Autosampler or equivalent. Zeeman background correction is applied for all analytes and Argon is used as the purge gas in the graphite furnace.

6.2 Perkin Elmer Model 5000 Atomic Absorption Spectrophotometer, equipped with a Model HGA-500 Graphite Furnace and an AS-40 Autosampler or equivalent. Deutorium Arc or Tungsten Background wavelengths are applied for all analytes and Argon is used as the purge gas in graphite furnace.

6.3 Instrument Operating Parameters. The operating conditions used for each specified analytes are listed in Table 2 below.

TABLE 2. WAVELENGTHS, SLIT SIZE, SAMPLE ALIQUOT, AND LAMP CONDITIONS

Analyte	Wavelength (nm)	Slit Size (L)	Sample Aliquot (uL)	Lamp
Antimony (Sb)	217.6	0.7	20	EDL or HCl
Arsenic (As)	193.7	0.7	20	EDL
Beryllium (Be)	234.9	0.7	15	HCl
Cadmium (Cd)	228.8	0.7	10	EDL or HCl
Chromium (Cr)	357.9	0.7	10	HCl
Copper (Cu)	324.8	0.7	20	HCl
Nickel (Ni)	232.0	0.20	20	HCl
Lead (Pb)	283.3	0.7	15	EDL or HCl
Selenium (Se)	196.0	2.0	20	EDL

Silver (Ag)	328.1	0.7	20	HCl
Thallium (Tl)	276.8	0.7	20	EDL or HCl

6.4 Graphite Furnace Conditions. The following graphite furnace conditions are used for each specified analyte:

ANTIMONY				
Step #	Temp (°C)	Ramp	Hold	Gas Flow
1	140	1	50	300
2	700	1	30	300
3	2400	0	6	0
4	2600	1	5	300
5	20	1	5	300

ARSENIC				
Step #	Temp (°C)	Ramp	Hold	Gas Flow
1	120	1	50	300
2	1300	1	30	300
3	20	1	15	300
4	2300	0	5	0
5	2600	1	5	300

BERYLLIUM				
Step #	Temp (°C)	Ramp	Hold	Gas Flow
1	120	15	15	300
2	1500	15	15	300
3	2500	0	5	0
4	2650	1	5	300
5	20	1	5	300

CADMIUM				
Step #	Temp (°C)	Ramp	Hold	Gas Flow
1	120	1	50	300
2	900	1	30	300
3	20	1	15	300
4	1650	0	5	20
5	2600	1	5	300

CHROMIUM				
Step #	Temp (°C)	Ramp	Hold	Gas Flow
1	140	1	50	300
2	1650	5	30	300
3	20	1	15	300
4	2500	0	5	0

5	2650	1	10	300

COPPER				
Step #	**Temp (°C)**	**Ramp**	**Hold**	**Gas Flow**
1	140	1	60	300
2	1300	1	30	300
3	20	1	15	300
4	2500	0	5	0
5	2650	1	5	300

NICKEL				
Step #	**Temp (°C)**	**Ramp**	**Hold**	**Gas Flow**
1	120	1	70	300
2	1400	1	30	300
3	20	1	15	300
4	2400	0	5	0
5	2600	1	5	300

LEAD				
Step #	**Temp (°C)**	**Ramp**	**Hold**	**Gas Flow**
1	120	5	50	300
2	900	1	30	300
3	20	1	15	300
4	1800	0	5	0
5	2600	1	5	300

SELENIUM				
Step #	**Temp (°C)**	**Ramp**	**Hold**	**Gas Flow**
1	120	1	50	300
2	1100	1	30	300
3	20	1	15	300
4	2100	0	5	0
5	2600	1	5	300

SILVER				
Step #	**Temp (°C)**	**Ramp**	**Hold**	**Gas Flow**
1	120	15	30	300
2	500	15	15	300
3	1600	0	5	60
4	20	1	5	300
5	2600	1	5	300

THALLIUM				
Step #	Temp (°C)	Ramp	Hold	Gas Flow
1	120	5	50	300
2	600	5	30	300
3	20	1	15	300
4	1400	0	5	0
5	2600	1	5	300

During routine use of this method, slight modifications may be necessary to compensate for sample matrices, conditions of graphite contact rings, or other environmental factors that may affect the performance of the program.

7.0 SAFETY AND CHEMICAL HYGIENE

The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; however, each chemical compound should be treated as a potential health hazard, and exposure to these chemicals must be reduced to the lowest possible level by whatever means available. The laboratory maintains a reference file of Material Safety Data Sheets (MSDS) for the chemicals specified in this method. Additional information on laboratory safety is available in the Laboratory Safety Plan and from the Laboratory Safety Officer.

8.0 REAGENTS AND STANDARD REFERENCE MATERIALS (SRMs)

8.1 HNO_3, conc., ultra high purity or equivalent.
8.2 HNO_3 1:1 (v:v), prepared from ultra high purity HNO3.
8.3 HNO_3 1%, prepared from ultra high purity HNO3.
8.4 Hydrogen peroxide, 30%, reagent grade.
8.5 Deionized water: Prepared by a reverse osmosis system by McNew Culligan with an E-Pure polishing system including an ultrafilter assembly. This system passes all ASTM Type I water requirements. Use deionized water for the preparation of all reagents, calibration standards and as dilution water.
8.6 Matrix Modifiers
8.6.1 Nickel Nitrate Hexahydrate, reagent grade
8.6.2 Nickel Nitrate stock: dissolve 24.780 g of Nickel Nitrate Hexahydrate in deionized water and dilute to 50 mL. Add 10 μL to 1 mL of sample. Use for the analysis of arsenic, selenium and antimony on the Perkin Elmer Model 5000.
8.6.3 Nickel Nitrate matrix modifier: dilute 2 mL nickel nitrate substock to 50 mL with deionized water. Use for the analysis of arsenic, selenium, and antimony on the Perkin Elmer 5100.
8.6.4 Magnesium Nitrate, reagent grade.
8.6.5 Ammonium Phosphate, monobasic, reagent grade.
8.6.6 Ammonium Phosphate/Magnesium Nitrate substock: Dissolve 5.2 g $Mg(NO_3)_2*H_2O$) and 20 g $NH_4H_2PO_4$ in deionized water and dilute to 100 mL. Use for the analyses of cadmium (10 μL/ 1 mL) and lead (20 μL/ 1 mL) on the Perkin Elmer Model 5000.
8.6.7 Ammonium Phosphate/Magnesium Nitrate solution: Dilute 2 mL of ammonium phosphate/magnesium nitrate solution at 50 mL with deionized water. Use for the analysis of lead and cadmium on the Perkin Elmer Model 5100.
8.6.8 Sulfuric Acid (H_2SO_4), conc.

8.6.9 Sulfuric Acid (H_2SO_4) 25%. Cautiously add 25 mL of concentrated H_2SO_4 to 50 mL deionized water. Mix frequently during addition and then dilute to 100 mL. Use 10 μL/ 1 mL for the analysis of thallium on the Perkin Elmer Model 5000.

8.6.10 Sulfuric Acid, 4%. Dilute 4 mL of conc. sulfuric acid to 100 mL with deionized water. Use 10 μL/ 1 mL for the analysis of thallium on the Perkin Elmer Model 5100.

8.7 Calibration Stock Solutions, SRMs, 1,000 mg/L - 10, 000 mg/L: The trace metal SRMs are available as commercially prepared stock solutions for each analyte. All parent compounds will either be obtained from NIST or must be traceable to NIST standards. All standard stock solutions are warranted stable for up to a year from the date of receipt. The Calibration Stock SRM solutions are used to prepare calibration substock solutions. Traceability documentation of SRMs not obtained from NIST will be maintained on file at the laboratory.

8.8 Calibration Check Standard Stock SRMs, 1,000 mg/L - 10,000 mg/L: Check Standard SRMs must meet the same specifications as the Calibration SRM Stock solutions but must come from a different source than those used for Calibration SRMs. The Check Standard stock solutions are used to prepare the Check Standard Substocks. Traceability documentation of SRMs will be maintained on file at the laboratory.

8.9 Standard substock (1 mg/L): Prepared by diluting appropriate volumes of stock solution with deionized water including nitric acid to result in a 1% HNO_3 matrix. Refer to EAL-SOP-334 for preparation instructions.

8.10 Working standards: Prepare dilutions of the stock solution to be used as calibration standards at the time of the analysis. Refer to EAL-SOP-334 for preparation instructions.

9.0 PROCEDURE

9.1 Furnace devices (flameless atomization) are a most useful means of extending detection limits. Because of differences between various makes and models of satisfactory instruments, no detailed operating instructions can be given for each instrument. Instead, the analyst should follow the instructions provided by the manufacturer of a particular instrument.

9.2 Background correction is important when using flameless atomization, especially below 350 nm. Certain samples, when atomized, may absorb or scatter light from the lamp. This can be caused by the presence of gaseous molecular species, salt particles, or smoke in the sample beam. If no correction is made, sample absorbance will be greater than it should be, and the analytical result will be erroneously high. Zeeman background correction is effective in overcoming composition or structured background interferences. It is particularly useful when analyzing for As in the presence of Al and when analyzing for Se in the presence of Fe.

9.3 Memory effects occur when the analyte is not totally volatilized during atomization. This condition depends on several factors: volatility of the element and its chemical form, whether pyrolytic graphite is used, the rate of atomization, and furnace design. This situation is detected through blank burns. The tube should be cleaned by operating the furnace at full power for the required time period, as needed, at regular intervals during the series of determinations.

9.4 Furnace parameters suggested in Section 6.4 should be employed as guidelines. Because temperature-sensing mechanisms and temperature controllers can vary between instruments or with time, the validity of the furnace parameters must be periodically confirmed by systematically altering the furnace parameters while analyzing a standard. In this manner, losses of analyte due to overly high temperature settings or losses in sensitivity due to less than optimum settings can be minimized. Similar verification of furnace parameters may be required for complex sample matrices.

9.5 An autosampler is used to inject the sample into the furnace and atomize. If the concentration found is greater than the highest standard, the sample should be diluted in the same acid matrix and reanalyzed. The use of multiple injections can improve accuracy and help detect furnace pipetting errors.

9.6 All samples that suffer from matrix interferences should be analyzed by the method of standard additions.

9.7 Run a check standard after every 20 injections of samples. Standards are run in part to monitor the life and performance of the graphite tube. Lack of reproducibility or significant change in the signal for the standard indicates that the tube should be replaced.

9.8 Calculate metal concentrations by (1) the method of standard additions, or (2) from a calibration curve, or (3) directly from the instrument's concentration readout. All dilution or concentration factors must be taken into account. Concentrations reported for multiphased samples must be appropriately qualified (e.g., 5 ug/g aqueous phase).

9.9 Duplicates, spiked samples, and check standards should be routinely analyzed.

10.0 CALCULATIONS

10.1 For determination of metal concentration by furnace: Read the metal value in ug/L from the calibration curve or directly from the read-out system of the instrument.

10.2 If dilution of sample was required:

ug/L metal in sample = A((C+B)/C)

where:
A = ug/L of metal in diluted aliquot from calibration curve.
B = Acid blank matrix used for dilution, mL.
C = sample aliquot, mL.

10.3 For solid samples, report all concentrations as ug/kg based on wet weight. Hence:

ug/kg metal in sample = A (V/W)

where:
A = ug/L of metal in processed sample from calibration curve.
V = final volume of the processed sample, mL.
W = weight of sample, grams.

11.0 QUALITY CONTROL

11.1 Calibration curves must be composed of a minimum of a blank and three standards, verified by use of one standard at or near the mid-range. The mid-range standard must be within 5% of the orignial curve. The lowest standard should be at or near the reporting limit or PQL. The curve must be verified by a check standard analyzed approximately every 20 injections. Results of the check standard must be ± 10% or the analysis should be stopped, the problem corrected and the instrument recalibrated.

11.2 Dilute samples if they are more concentrated than the highest standard or if they fall on the plateau of a calibration curve.

11.3 Employ a minimum of one blank per sample batch to determine if contamination or any memory effects are occurring. A preparation blank is a volume of reagent water acidified with the same amounts of acids as were the standards and samples and prepared in the same manner as the

samples. All analytes must be below the RL, PQL or CRDL as required by the project plan or QAPjP; otherwise, the samples must be redigested or reanalyzed. In addition, if the method blank concentration of a particular element is above the limit and the samples are greater than 10 times the blank concentration, the data is considered acceptable with no further corrective action. .

11.4 Analyze the calibration blank at a frequency of 10%. The result should be within the +/- 3X the MDL or less than the reporting limit. If not, terminate the analysis, correct the problem and recalibrate the instrument. All samples following the last successful CCB must be reanalyzed after recalibration.

11.5 Verify calibration with an independently prepared check standard every 20 injections. Results of check standard must be +/- 10% or the analysis should be stopped, the problem corrected and the instrument recalibrated. All samples following the last successful CCV must be reanalyzed after recalibration.

11.6 Employ a minimum of one Laboratory Control Sample (LCS) per batch to determine if the method performance is acceptable. A LCS is a check standard which has been processed through the entire method beginning with sample preparation. LCS recoveries for all elements must be within EA Laboratories established control limits or the samples must be redigested and reanalyzed (possible exception: high LCS recovery and no analyte detected in the sample). Solid samples require the preparation of a solid matrix LCS. A tissue (NIST SRM) matrix LCS is prepared for the analysis of tissue samples in addition to a liquid LCS.

11.7 Run one duplicate sample for every 20 samples. Duplicate data should be qualified and relative percent difference should be +/- 20%. If the duplicate RPD exceeds 20%, reanalyze for verification and redigest and reanalyze if lab error is suspected.

11.8 Run one spike sample for every 20 samples or with each sample digestion group. The criteria for recovery of matrix spike is 75-125% or matrix effect should be suspected. Reanalyze for verification and redigest and reanalyze if lab error is suspected. Evaluate bias associated with recovery, flag data as appropriate and address in the narrative of the final report.

11.9 Run one spike duplicate sample for every 20 samples or with each sample digestion group. The criteria for recovery of matrix spike is 75-125% or matrix effect should be suspected. Reanalyze for verification and redigest and reanalyze if lab error is suspected. Evaluate bias associated with recovery, flag data as appropriate and address in the narrative of the final report.

11.10 The method of standard additions shall be used for the analysis, whenever the analytical spike recovery does not meet 85-115% criteria (per CLP protocol).

11.11 Serial dilution: The dilution volume should be based on the analysis of the undiluted sample. The dilution should be 1:4, while keeping in mind that the diluted value should be at least 5 times the instrument detection limit. The diluted aliquots should then be analyzed and the unspiked results, multiplied by the dilution factor, should be compared to the original determination. Agreement of the results (within 10%) indicates the absence of interference. Comparison of the actual signal from the spike with the expected response from the analyte in an aqueous standard should help confirm the finding from the dilution analysis.

12.0 REFERENCES

United States Environmental Protection Agency. 1986. Revised July 1992. Test Methods for Evaluating Solid Waste. Physical/Chemical Methods. EPA SW-846, 3rd edition, including Update II. U.S. EPA, Washington, D.C.

Mercury
SW 7470/7471

EA Engineering, Science, and Technology, Inc.

EA Laboratories

Method

Number: 7471A-P **Rev. No.:** 0

Title: Mercury Digestion Procedure for Soils

Prepared By: Clare M Kayal for — 1/10/96
M.A. Pendergast, Metals Group Leader — Date

Revised By: M.E. Wilcox — 03 Dec 95
M.E. Wilcox, Quality Control Specialist II — Date

Approved By: M.M. Uhlfelder — 19 Dec 95
M.M. Uhlfelder, Quality Services Manager — Date

Approved By: — 29 Dec 95
P.A. Christopher, Inorganic Manager — Date

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EA LABORATORIES ANALYTICAL METHOD	EAL-M-7471A-P	Group: Metals
Mercury Digestion Procedure for Soils by SW-846	Page: 1	Of: 1

Purpose: To describe the appropriate digestion procedure for soil, sediments, and sludge samples requiring mercury analysis by Methods SW-7471A.

Scope: EPA SW-846 Method is an approved procedure for determining the concentrations of mercury in soil, sediments, and sludge samples. All samples must be subjected to an appropriate dissolution step prior to analysis.

Procedure:

1. Soil Digestion

1.1 Weigh 0.2g of sample and transfer to a BOD bottle. Also transfer the appropriate volume of standards, blanks, and quality control samples to BOD bottles.
1.2 Add 5 ml concentrated H2SO4 and 2 ml concentrated HNO3, mixing after each addition.
1.3 Add 15 ml potassium permanganate solution. Swirl and cover each bottle with foil and place in the autoclave.
1.4 Autoclave at 121C and 15 psi for 15 minutes.
1.5 Remove from autoclave and allow to cool completely. Just prior to analysis add 6 ml hydroxylamine-hydrochloride, swirl, and then continue with the analysis. Soil Digestion

2. Quality Control:

The EPA SW- 846 Method 7471A specific quality control samples required for each digestion batch or for every 20 samples:

2.1 Method Preparation Blank (PBW) - An analyte-free matrix to which all reagents are added in the same volumes or proportion as used in sample processing. The method blank is carried though the complete sample preparation and analytical procedure The method blank is used to detect contamination resulting from the analytical process.
2.2 Laboratory Control Sample (LCSW) - A known matrix spiked with compound(s) representative of target analytes. Results of the LCS are used to monitor the method performance.
2.3 Duplicate (DUP) - An intra laboratory spilt sample which is used to monitor the method precision.
2.4 Matrix Spike (SPK) - An aliquot of sample spiked with a known concentration of target analyte(s). The spiking occurs prior to sample preparation and analysis. The matrix spike is used to monitor the sample matrix bias of a method.
2.5 Matrix Spike Duplicate (MSD) - Intra laboratory split sample spiked with identical concentrations of target analyte(s). The spiking occurs prior to sample preparation and analysis. The matrix spiked duplicate sample is used to monitor the sample matrix bias and precision of a method.
2.6 Serial Dilution (L) - A randomly selected sample within the analytical batch that is diluted 5:1 to detect sample matrix interference.

EA Engineering, Science, and Technology, Inc.

EA Laboratories

Method

Number: 7470A-P **Rev. No.:** 0

Title: Mercury Digestion Procedure for Waters

Prepared By:	Clare R.H. Kayal for — M.A. Pendergast, Metals Group Leader	1/10/96 Date
Revised By:	M.E. Wilcox — M.E. Wilcox, Quality Control Specialist II	03 Dec 95 Date
Approved By:	M.M. Uhlfelder — M.M. Uhlfelder, Quality Services Manager	19 Dec 95 Date
Approved By:	[signature] — P.A. Christopher, Inorganic Manager	29 Dec 95 Date

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EA LABORATORIES ANALYTICAL METHOD	EAL-M-7470A-P	Group: Metals
Mercury Digestion Procedure for Waters by SW-846	Page: 1	Of: 1

Purpose: To describe the appropriate digestion procedure for aqueous waste and ground waters requiring mercury analysis by Methods SW-7470A.

Scope: EPA SW-846 Method is an approved procedure for determining the concentrations of mercury in aqueous waste and ground waters. All samples must be subjected to an appropriate dissolution step prior to analysis.

Procedure:

1. Aqueous Digestion

1.1 Shake sample to mix and measure 100 ml into a BOD bottle. Also transfer 100 ml of all standards, blanks, and quality control samples to BOD bottles.
1.2 Add 5 ml concentrated H2SO4 and 2.5 ml concentrated HNO3, mixing after each addition.
1.3 Add 15 ml potassium permanganate solution. Swirl and add additional portions of permanganate, if necessary, until the purple color persists for at least 15 minutes.
1.4 Add 8 ml potassium persulfate solution.
1.5 Heat for two hours in a water bath at 80C.
1.6 Remove from water bath and allow to cool completely. Just prior to analysis add 6 ml hydroxylamine-hydrochloride, swirl, and then continue with the analysis.

2. Quality Control:

The EPA SW- 846 Method 7470A specific quality control samples required for each digestion batch or for every 20 samples:

2.1 Method Preparation Blank (PBW) - An analyte-free matrix to which all reagents are added in the same volumes or proportion as used in sample processing. The method blank is carried though the complete sample preparation and analytical procedure The method blank is used to detect contamination resulting from the analytical process.
2.2 Laboratory Control Sample (LCSW) - A known matrix spiked with compound(s) representative of target analytes. Results of the LCS are used to monitor the method performance.
2.3 Duplicate (DUP) - An intra laboratory spilt sample which is used to monitor the method precision.
2.4 Matrix Spike (SPK) - An aliquot of sample spiked with a known concentration of target analyte(s). The spiking occurs prior to sample preparation and analysis. The matrix spike is used to monitor the sample matrix bias of a method.
2.5 Matrix Spike Duplicate (MSD) - Intra laboratory split sample spiked with identical concentrations of target analyte(s). The spiking occurs prior to sample preparation and analysis. The matrix spiked duplicate sample is used to monitor the sample matrix bias and precision of a method.
2.6 Serial Dilution (L) - A randomly selected sample within the analytical batch that is diluted 5:1 to detect sample matrix interference.

EA Engineering, Science, and Technology, Inc.

EA Laboratories

Method

Number: 7470/1 **Rev. No.:** 0

Title: Mercury in Waste by Manual Cold-Vapor Atomic Absorption Spectroscopy

Prepared By: M.A. Pendergast, Metals Group Leader — 20 November 95

Revised By: M.E. Wilcox — 19 Mar 96
M.E. Wilcox, Quality Control Specialist II — Date

Approved By: M.M. Uhlfelder — 19 March 1996
M.M. Uhlfelder, Quality Services Manager — Date

Approved By: — 20 Mar 96
P.A. Christopher, Operations Manager — Date

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1.0 SCOPE AND APPLICATION

1.1 Liquid. This method is a cold-vapor atomic absorption procedure for determining the concentration of mercury in mobility-procedure extracts, aqueous wastes, and ground waters. (Method 7470 can also be used for analyzing certain solid and sludge-type wastes; however, Method 7471 is usually the method of choice for these waste types.) All samples must be subjected to an appropriate dissolution step prior to analysis.

1.2 Solid or Semisolid. This method is for measuring total mercury (organic and inorganic) in soils, sediments, bottom deposits, and sludge-type materials. All samples must be subjected to an appropriate dissolution step prior to analysis.

2.0 SUMMARY OF METHOD

2.1 Prior to analysis, all samples must be prepared according to the procedure discussed in this method.

2.2 This method, 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. Absorbance (peak area) is measured as a function of mercury concentration.

2.3 The Reporting Limits for this method are listed in Table 1.

Table 1. Mercury Reporting Limits

ANALYTE:	CAS #	MATRIX	RL
Mercury (Hg)	7439-97-6	W	0.0002 mg/L
Mercury (Hg)	"	S	0.10 mg/kg

3.0 DEFINITIONS

3.1 Dissolved--Those elements which will pass through a 0.45 μm membrane filter.

3.2 Suspended--Those elements which are retained by a 0.45 μm membrane filter.

3.3 Total--The concentration determined on an unfiltered sample following vigorous digestion, or the sum of the dissolved plus suspended concentrations. (See Section 9.1)

3.4 Total recoverable--The concentration determined on an unfiltered sample following treatment with hot, dilute mineral acid.

3.5 Instrumental detection limit (IDL)-- The concentration, which is equal to three times the standard deviation of a series of seven replicate measurements of a standard approximately 3-5 times the expected IDL. The average standard deviation of this analysis on three separate nonconcurrent days should be used.

3.6 Sensitivity--The slope of the analytical curve, i.e. functional relationship between emission intensity and concentration.

3.7 Instrument check standard -- Standard of known concentrations prepared by the analyst to monitor and verify instrument performance on a daily basis.

3.8 Quality control sample--A solution obtained from an outside source having known, concentration values to be used to verify the calibration standards.

3.9 Calibration standards--A series of known standard solutions used by the analyst for calibration of the instrument (i.e., preparation of the analytical curve).

3.10 Linear dynamic range--The concentration range over which the analytical curve remains linear.

3.11 Reagent blank--A volume of deionized, distilled water containing the same acid matrix as the calibration standards carried through the entire analytical scheme.

3.12 Calibration blank--A volume of deionized, distilled water acidified with the same acid matrix as the calibration standards.

3.13 Reagent water--All references to water in this method refer to reagent water unless otherwise specified. Reagent water will be interference free. Reagent water refers to water that has been generated by any method which would achieve the performance specifications for ASTM Type II water.

4.0 SAMPLE COLLECTION, PRESERVATION, AND HANDLING

4.1 All sample containers are purchased prewashed and certified. The lot certifications are maintained on file at the laboratory.

4.2 Plastic and glass containers are both suitable.

4.3 Aqueous samples must be acidified to a pH <2 with HNO_3. The maximum holding times for these samples is 28 days.

4.4 Nonaqueous samples shall be refrigerated at 4°C $\pm$ 2°C when possible, and analyzed as soon as possible.

5.0 INTERFERENCES

5.1 Potassium permanganate is added to eliminate possible interference from sulfide. Concentrations as high as 20 mg/L of sulfide as sodium sulfide do not interfere with the recovery of added inorganic mercury from Type II water.

5.2 Copper has also been reported to interfere; however, copper concentrations as high as 10 mg/L had no effect on recovery of mercury from spiked samples.

5.3 Sea waters, brines, and industrial effluents high in chlorides require additional permanganate (as much as 25 mL) because, during the oxidation step, chlorides are converted to free chlorine, which also absorbs radiation of 253.7 nm. Care must therefore be taken to ensure that free chlorine is absent before the mercury is reduced and swept into the cell. This may be accomplished by using an excess of hydroxylamine hydrochloride reagent (25 mL). Both inorganic and organic mercury spikes have been quantitatively recovered from seawater by using this technique.

6.0 APPARATUS AND MATERIALS

6.1 Glassware/Hardware

1. 300 mL BOD bottles
2. Graduated cylinders, 100 mL capacity
3. Glass pipettes and micropipettes
4. Volumetric Flasks: 50 mL, 100 mL, 1000 mL, and 2000 mL
5. Erlenmeyer Flask: > 300 mL capacity
6. Autoclave
7. Water bath

6.2 Instrumentation.

6.2.1 Perkin Elmer Model 5100 Atomic Absorption Spectrophotometer, equipped with a model FIAS-200 Flow Injection Atomic Spectroscopy unit and a Model AS-90 Autosampler or equivalent. Argon is used as the purge gas.

6.2.2 Instrument Operation Parameters. The operating conditions are:

Wavelength: 253.7 nm
Slit: 0.7 nm Low
Sample Volume: 1000μL
Lamp: EDL/4 watts, HCL/6 ma
Cell Temperature: 200°C
Integration Time: 30 sec
Measurement Type: Peak Area

6.2.3 FIAS-200 Operation Conditions are summarized in Table 2.

Table 2. FIAS-200 Conditions

STEP	TIME (sec)	PUMP SPEED (rpm)		VALVE POSITION
		PUMP #1	PUMP #2	FILL/INJECT
Prefill	30	100	120	Fill
1	15	100	120	Fill
2	12	0	100	Inject
3	1	0	100	Fill

6.3 Mercury hollow cathode lamp or electrodeless discharge lamp.

6.4 Absorption cell: Standard spectrophotometer cells 10 cm long with quartz end windows may be used. Suitable cells may be constructed from Plexiglas tubing, 1 in. O.D. x 6.5 in. The ends are ground perpendicular to the longitudinal axis, and quartz windows (1 in. diameter x 1/16 in. thickness) are cemented in place. The cell is trapped to a burner for support. The cell is then positioned and adjusted vertically and horizontally to give the maximum transmittance.

7.0 SAFETY AND CHEMICAL HYGIENE

7.1 The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; however, each chemical compound should be treated as a potential health hazard, and exposure to these chemicals must be reduced to the lowest possible level by whatever means available. The laboratory maintains a reference file of Material Safety Data Sheets (MSDS) for the chemicals specified in this method.

7.2 Because mercury vapor is toxic, precaution must be taken to avoid its inhalation. Therefore, a bypass has been included in the system to vent the mercury vapor into an exhaust hood.

7.3 Additional information on laboratory safety is available in theLab Safety Plan and from the Laboratory Safety Officer.

8.0 REAGENTS AND STANDARD REFERENCE MATERIALS (SRMS)

8.1 Nitric acid (HNO_3), conc., ultra high purity or equivalent.
8.2 HNO_3 1:1 (v:v), prepared from ultra high purity HNO_3.
8.3 Sulfuric acid (H_2SO_4), conc., ultra high purity or equivalent.
8.4 Hydrochloric acid (HCl), conc., ultra high purity or equivalent.
8.5 HCl. 10% (v:v), prepared from ultra high purity HCl.
8.6 Deionized water: Prepared by a reverse osmosis system by McNew Culligan with an E-Pure polishing system including an ultrafilter assembly. This system passes all ASTM Type I water requirements. Use deionized water for the preparation of all reagents, calibration standards and as dilution water.
8.7 Calibration Stock Solutions, SRMs, 1,000 mg/L - 10,000 mg/L: The mercury SRMs are available as commercially prepared stock solutions. All parent compounds will either be obtained from NIST or must be traceable to NIST standards. All standard stock solutions are warranted stable for up to a year from the date of receipt. The Calibration Stock SRM solutions are used to prepare calibration substock solutions. Traceability documentation of SRMs not obtained from NIST will be maintained on file at the laboratory.
8.8 Calibration Check Stock Solutions, SRMs, 1,000 mg/L - 10,000 mg/L: Check standards must meet the same specifications as the Calibration SRM Stock solutions but must come from a different source than those used for Calibration SRMs. The Check Standard Stock solutions are used to prepare the Check Standard Substocks. Traceability documentation of SRMs will be maintained on file at the laboratory.
8.9 Stannous Chloride solution: Transfer 200 mL of conc. HCl to a Erlenmeyer flask and heat on a hotplate. Add 100 g $SnCl_2$. Heat until $SnCl_2$ dissolves. Transfer to 2000 mL volumetric flask containing approximately 1000 mL of deionized water and dilute to volume with deionized water.
8.10 Potassium Permanganate, 5% solution, (w/v): Transfer 50 g $KMnO_4$ to a 1000 mL volumetric flask containing approximately 500 mL deionized water. Mix to dissolve and dilute to volume with deionized water.
8.11 Potassium Persulfate, 5% solution, (w/v): Transfer 50 g $K_2S_2O_4$ to a 1000 mL volumetric flask containing approximately 500 mL deionized water. Mix to dissolve and dilute to volume with deionized water.
8.12 Sodium Chloride - Hydroxylamine Hydrochloride solution: Transfer 120 g NH_2OHHCl and 120 g NaCl to a 1000 mL volumetric flask containing approximately 500 mL of deionized water. Mix to dissolve and dilute to volume with deionized water.
8.13 Mercury Intermediate solution, 10 mg/L. Prepared from stock solutions in a 1% HNO_3 matrix.
8.14 Working Mercury solution, 100 µg/L. Prepared from 10mg/L intermediate solution 1% HNO_3 matrix.

9.0 PROCEDURE

9.1 Liquid Sample Preparation.
9.1.1 Sample preparation: Transfer 100 mL, or an aliquot diluted to 100 mL, containing 1.0 g of mercury, to a 300-mL BOD bottle. Add 5 mL of H_2SO_4 and 2.5 mL of concentrated HNO_3, mixing after each addition. Add 15 mL of potassium permanganate solution to each sample bottle. Sewage samples may require additional permanganate. Ensure that equal amounts of permanganate are added to standards and blanks. Shake and add additional portions of potassium permanganate solution, if necessary, until the purple color persists for at least 15 min. Add 8 mL of potassium persulfate to each bottle. Cover the bottle with foil and heat in an autoclave for 15 minutes at 120°C and 15 psi.

NOTE: Alternatively, samples and standards must be heated in a water bath for 2 hrs maintained at 95°C for specific projects or regulatory requirements.

Cool and just prior to placing samples in the autosampler add 6 mL of sodium chloride-hydroxylamine hydrochloride to reduce the excess permanaganate. Continue as described in step 9.3.

9.1.2 Liquid Standard preparation: Transfer 0-, 0.5-, 1.0-, 2.0-, 5.0-, and 10.0-mL aliquots of the mercury working standard, containing 0-1.0 ug of mercury, to a series of 300-mL BOD bottles. Add enough Type II (or better) water to each bottle to make a total volume of 100 mL. Mix thoroughly and add 5 mL of concentrated H_2SO_4 and 2.5 mL of concentrated HNO_3 to each bottle. Add 15 mL of $KMnO_4$ solution to each bottle and allow to stand at least 15 min. Add 8 mL of potassium persulfate to each bottle. Cover the bottle with foil and heat in an autoclave for 15 min at 120°C and 15 psi.

NOTE: Alternatively samples and standards must be heated in a water bath for 2 hrs maintained at 95°C for specific projects on Regulatory Requirements.

Cool and just prior to placing standards in the autosampler add 6 mL of sodium chloride-hydroxylamine hydrochloride to reduce the excess permanaganate. Continue as described in step 9.4.

9.2 Solids and Semi-solids Sample Preparation.

9.2.1 Weigh 0.2 g portions of untreated sample and place in the bottom of a 300mL BOD bottle. Triplicate portions will be performed on a projected-by-project basis. Add 5 mL of concentrated H_2SO_4 and 2 mL of concentrated HNO_3. Add 5 mL of $KMnO_4$ solution and cover the bottle with aluminum foil. Place in the autoclave for 15 min at 120°C and 15 psi. Cool and dilute to a volume of 100 mL with Type II water. Just prior to placing in the autosampler add 6 mL of sodium chloride/hydroxylamine hydrochloride to reduce the excess permanganate. Continue to step 9.3.

9.2.2 Transfer 0-, 0.5-, 1.0-, 2.0-, 5.0- and 10 mL aliquots of mercury working standard containing 0-1.0 μg of mercury to a series of 300 mL BOD bottles. Add deionized water to make a total volume of 10 mL. Add 5.0 mL of concentrated H_2SO_4 and 2.0 mL of concentrated HNO_3. Add 5.0 mL of $KMnO_4$ solution and cover the bottle with aluminum foil. Place in the autoclave for 15 min at 120°C and 15 psi. Cool and dilute to a volume of 100 mL with Type II (or better) water. Just prior to placing in the autosampler, add 6.0 mL of sodium chloride/hydroxylamine hydrochloride solution to reduce the excess permanganate. Continue to step 9.3.

9.3 Analyze all EP extracts, all samples analyzed as part of a delisting petition, and all samples that suffer from matrix interferences by the method of standard additions (see Section 11.7).

9.4 Instrument Setup and Daily Maintenance

9.4.1 Set the instrument operating conditions as described in section 6.2.

9.4.2 Peristaltic pump tubing should be checked at the beginning of each analytical run and changed if excessive wear is evident. Fill the two reagent bottles with 10% HCl (v:v) and stannous chloride solution respectively.

9.4.3 15 mL plastic centrifuge vials are filled with standards or sample digestates. The 5100 software is programmed for the autosampler and FIAS pumps to deliver the appropriate sample aliquot and necessary reagents for release of mercury vapor. Argon carrier gas carries the mercury vapor to the cell where atomic absorption is measured.

10.0 CALCULATIONS

10.1 The computer system software automatically stores the successful calibration data and will generate the sample concentrations in units of μg/L.

10.2 If any sample response exceeds that of the highest calibration standard aliquots of the digestates are diluted with a calibration blank and reanalyzed.

10.3 Calculate metal concentrations (1) by the method of standard additions, or (2) from a calibration curve. All dilution or concentration factors must be taken into account. Concentrations reported for multiphased or wet samples must be appropriately qualified (e.g., 5 μg/g dry weight).

11.0 QUALITY CONTROL

11.1 Employ a minimum of one blank per sample batch to determine if contamination or any memory effects are occurring. The mercury concentration must be below the RL, PQL or CRDL as required by the project plan or QAPjP; otherwise, the samples must be redigested and reanalyzed. In addition, if the method blank concentration is above the limit and the samples are greater than 20 times the blank concentration, the data is considered accceptable with no further corrective action.

11.2 Verify calibration with an independently prepared check standard immediately after calibration and after every 10 samples. The recovery of the initial calibration verification check standard must be within the control limits 90-110%, and all subsequent check standards must be within the control limits 80-120%. If the check standards are not within the control limits the analysis should be terminated, the problem corrected and the instrument recalibrated. All samples following the last successful CCV must be reanalyzed after recalibration.

11.3 Run one duplicate sample for every 20 samples or analytical batch. A duplicate sample is a sample brought through the entire sample preparation and analytical process. The relative percent difference should be +/- 20% or matrix effects should be suspected. Reanalyze for verification; redigest and reanalyze if lab error is suspected.

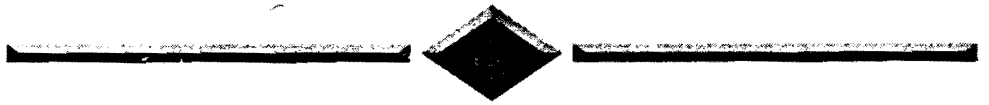
11.4 Run one spike sample for every 20 samples or analytical batch. A matrix spike sample is a sample brought through the entire sample preparation and analytical process. The recovery of the matrix spike should be within 75-125% or matrix effects should be suspected. Reanalyze for verification and redigest and reanalyze if lab error is suspected. Evaluate bias associate with recovery, flag data as appropriate and address in the narrative of the final report.

11.5 Run one spike duplicate sample for every 20 samples or analytical batch. A matrix spike duplicate sample is a spiked sample brought through the entire sample preparation and analytical process in two separate aliquots. The recovery of the MSD should be within 75-125% or matrix effects should be suspected. The RPD between the MS and MSD should be with +/- 20%.

11.6 Calibration curves must be composed of a minimum of a blank and three standards. The correlation efficient of the curve must be 0.995 or better to continue analysis.

12.0 REFERENCES

United States Environmental Protection Agency. 1986. Revised July 1992. Test Methods for Evaluating Solid Waste. Physical/Chemical Methods. EPA SW-846 (Methods 7470, 7471), 3rd edition, including Update I. U.S. EPA, Washington, D.C.



AVS/SEM

EA Engineering, Science, and Technology, Inc.

EA Laboratories

Method

Number: AVS **Rev. No.:** 1

Title: Acid Volatile Sulfide (AVS) and Simultaneously Extractable Metals

Prepared By:	L.C. Quinn, Senior Chemist	30 January 1995
Revised By:	J.S. Dembowski, Inorganic Section Chief	9/8/97 Date
Approved By:	M.M. Uhlfelder, Quality Services Manager	9/10/97 Date
Approved By:	P.A. Christopher, Inorganic QC Chemist	9/5/97 Date

1.0 SCOPE AND APPLICATION

1.1 This method is used to process soil and sediment samples for Acid Volatile Sulfide (AVS) and Simultaneously Extractable Metals (SEM).
1.2 The concentration of AVS is indicative of the amount of sulfide available in the sample for binding trace metals. The SEM procedure determines the concentration of trace metals that are solublized under the given conditions and available for binding to the sulfide.

2.0 SUMMARY OF METHOD

An aliquot of the sample is acidified in a solution of 1.2 M hydrochloric acid in a closed system. Any hydrogen sulfide gas generated is purged from the reaction vessel with nitrogen gas into an absorber solution of sodium hydroxide for a period of one hour. The reaction mixture is stirred but not heated during the one hour reaction period. The absorber solution is titrated to determine the acid volatile sulfide concentration and the acidified contents of the flask is filtered and analyzed for various trace metals to determine the simultaneously extractable metals concentrations.

3.0 DEFINITIONS

3.1 Acid Volatile Sulfide (AVS): Amorphous, moderately crystalline monosulfides, and other sulfides that form hydrogen sulfide under the conditions of this test.
3.2 Simultaneously Extractable Metals (SEM): Metals, commonly cadmium, copper, lead, mercury, nickel, lead, and zinc, which form less soluble sulfides than do iron, or manganese, and which are at least partially soluble under the conditions of this test.

4.0 SAMPLE HANDLING AND PRESERVATION

Samples should be collected with a minimum of aeration. The sample bottle should be filled as completely as possible, excluding head space, and stoppered. Analysis should commence as soon as possible. Samples should be kept in a cool, dark place until analysis.

5.0 INTERFERENCES

5.1 Oxygen in reagents and apparatus is the primary interference reported. Purging of the apparatus with oxygen-free nitrogen or argon will remove oxygen.
5.2 The pH of the sample must be below 3 after the addition of the acid during the AVS generation procedure or the generation of hydrogen sulfide gas may hindered.

6.0 APPARATUS AND MATERIALS

6.1 Flask, 500 mL, two- or three-necked, Ŧ 24/40 ground-glass joints.
6.2 Dropping (addition) funnel, 250 mL, with pressure-equalizing tube, stopcock, and Ŧ 24/40 ground-glass joint and Teflon sleeve.
6.3 Absorber tube.
6.4 Stirring bars.
6.5 Rotometer, capable of measuring 40 mL/min of nitrogen.
6.6 Stir plate.

7.0 SAFETY AND CHEMICAL HYGIENE

The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; however, each chemical compound should be treated as a potential health hazard, and exposure to these chemicals must be reduced to the lowest possible level by whatever means available. The laboratory maintains a reference file of material safety data sheets for the chemicals specified in this method. Additional information on general laboratory safety is available in the

EA Laboratories' Safety Plan (EAL-004) and from the Laboratory Safety Officer.

8.0 REAGENTS

8.1 Sulfide quality control solution, ~1,000 mg S^{2-}/L: Dissolve 0.8 g of sodium sulfide, $Na_2S{\cdot}9H_2O$, in 100 mL of deionized water. Sulfide solutions are very unstable and must be titrated against iodine daily prior to use to determine the exact concentration. Prepare fresh monthly or when titrated sulfide concentration falls below 800 mg/L. The QC solution is used to prepare the laboratory control sample (LCS).
8.2 Hydrochloric acid solution, 6 M: Cautiously add 500 mL of concentrated HCl to 500 mL of deionized water.
8.3 Sodium hydroxide solution, 0.1 N: Dissolve 4.0 g of NaOH in deionized water and dilute to 1000 mL.

9.0 PROCEDURE

9.1 Assemble the apparatus as shown in Figure 1. A two-necked flask and magnetic stirrer can be used instead of the arrangement in Figure 1.
9.2 Add 50 mL of 0.1N NaOH solution to the absorber tube and dilute with deionized water to obtain an adequate depth of liquid.
9.3 With addition funnel stopcock closed, add 20 mL of 6 M hydrochloric acid to the funnel.
9.4 Add 10 g of the soil or sediment to be tested to the flask. Record the weight of waste to nearest 0.01 g. Add 100 mL of deionized water to the sample in the flask.
9.5 Close the system and adjust the nitrogen flow rate to 40 mL/min.
9.6 With the nitrogen flowing, open the dropping funnel stopcock and add the 20 mL 6 M HCl solution to the 500-mL flask. The resulting HCl concentration in the flask will be approximately 1.2 M HCl. Begin stirring the mixture (do not create a vortex) while the acid is entering the flask, and start the 1 hour test period.
9.7 After 60 min, turn off the nitrogen and disconnect the absorber. Record the time interval used.
9.8 Record the final absorber solution volume. Analyze the absorber solution for sulfide using method EAL-M-376.1, Sulfide (Titrimetric).
9.9 Filter the acidified contents of the 500-mL flask through an acid washed 0.45 um membrane filter. Record the final volume of the filtrate. Do not digest the filtrate. Analyze the filtrate directly by Inductively Coupled Plasma (ICP) or Atomic Absorption Spectroscopy (AA) for Cadmium, Copper, Nickel, Lead, and Zinc.
9.10 Preparation of method blank: Place 10 g (10 mL) of deionized water in a flask in place of sample. Proceed with steps 9.1 through 9.10.
9.11 Preparation of sulfide laboratory control sample (LCS): Pipet 2.0 mL of ~1,000 mg/L sulfide QC stock into a the 500-mL flask in place of sample. Process according to steps 9.1 to 9.9 to a final absorber volume of 50 mL. The target concentration of the LCS in 50 mL of absorber solution is ~50 mg/L. The exact target concentration is calculated from the titrated concentrations of the QC stocks prior to use.

10.0 CALCULATION

10.1 Calculate the concentration of Acid Releasable Sulfide as S_2. in the waste sample as follows:

$$\text{AVS as } S_2\text{. (mg/kg-dry)} = \frac{C\,V}{W\,D}$$

where:

C = titrated concentration of S_2. in absorber solution (mg/L)
V = total volume of absorber solution (mL)
W = as-received weight of sample used (g)
D = percent solids as decimal fraction

10.2 Convert from units of mg S_2./kg-(dry) to umole H_2S/g-(dry):

AVS as umole H_2S/g-(dry) = C/32.06

where: C = concentration of AVS as mg S S_2/ kg-(dry)

10.3 Simultaneously Extractable Metals (SEM): Convert the concentration of each trace metal from mg/kg-dry to umole/g-dry:

The general formula for conversion for any metal determined by the SEM procedure is:

$$\text{Metal umole/g-dry} = \frac{1}{\text{atomic weight}} (C)$$

Where C = concentration of metal as mg/kg-dry

The specific conversion for Cd, Cu, Ni, Pb, Zn is as follows:

Cd umole/g-dry = 0.008896 (C)
Cu umole/g-dry = 0.01574 (C)
Ni umole/g-dry = 0.01704 (C)
Pb umole/g-dry = 0.004827 (C)
Zn umole/g-dry = 0.01529 (C)

10.4 Calculate the Total SEM molar concentration as the sum of the individual trace metals concentrations in units of umole/g-dry.

10.5 Calculate ratio of AVS to Total SEM as follows:

$$\text{AVS/SEM molar ratio} = \frac{A}{B}$$

Where:
A = AVS molar concentration as umole H_2S/g-dry
B = Total SEM molar concentration as umole/g-dry

11.0 QUALITY CONTROL

Calculate the AVS percent recovery of the LCS from the measured analytical concentration of the absorber solution and the calculated target concentration:

$$\text{AVS Percent recovery} = \frac{\text{analytical concentration (mg/L)}}{\text{target concentration (mg/L)}} \times 100$$

12.0 OPERATING NOTES

The original draft method suggests three possible analytical methods for determining the sulfide concentration of the final AVS impinger solution: gravimetric - generation of silver sulfide, colorimetric - diamine color development, and sulfide ion selective electrode. This method uses direct titration of the impinger solutions by EPA method 376.1.

13.0 REFERENCES

13.1 Allen, H.E. and F. Gongmin et al. 1991. Determination of Acid Volatile Sulfide and Simultaneously Extractable Metals in Sediment, April 1991 (Draft Analytical Method for the Determination of Acid Volatile Sulfide in Sediment, U.S. EPA Office of Water and Office of Science and Technology, Health and Ecological Criteria Division, Washington, D.C., August 1991).

13.2 Allen, H.E. and F. Gongmin, et al. "Analysis of Acid Volatile Sulfide (AVS) and Simultaneously Extracted Metals (SEM) for the Estimation of Potential Toxicity in Aquatic Sediments". 1990. Environmental Toxicology and Chemistry, vol. 12, pp 1441-1453.

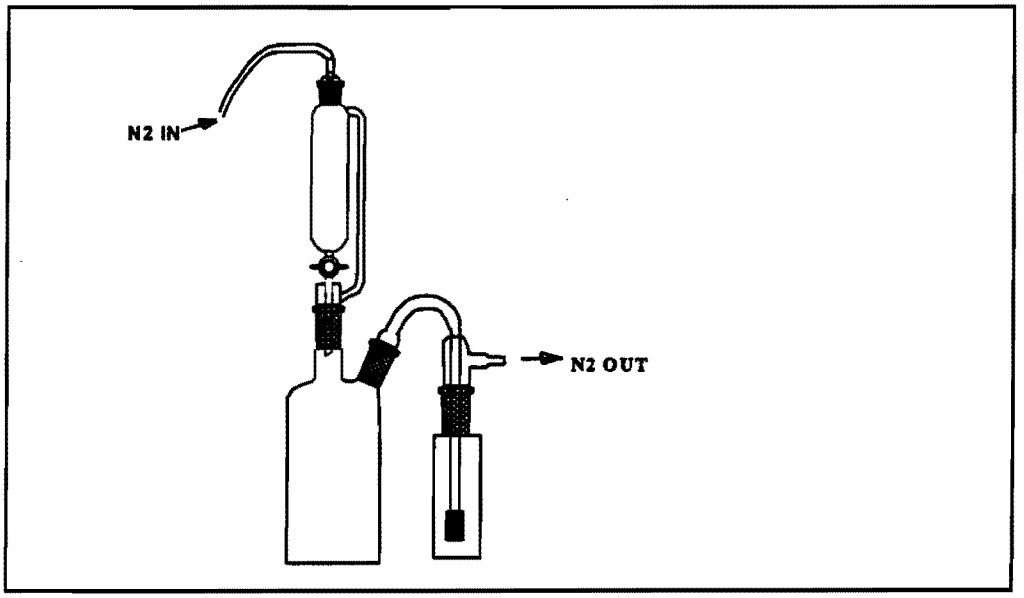


Figure 1: Apparatus for the determination of AVS/SEM.

Hexavalent Chromium
SW 7196A

EA Engineering, Science, and Technology, Inc.

EA Laboratories

Analytical Method

Number: 7196A **Rev. No.:** 0

Title: Hexavalent Chromium (Colorimetric, Diphenylcarbazide)

Approved By: _____ _____
P.A. Christopher, Inorganics Section Chief Date

Approved By: _____ _____
M.M. Uhlfelder, Quality Services Manager Date

Approved By: _____ _____
A.R. Karimi, Laboratory Director Date

EA Engineering, Science, and Technology, Inc.

EA Laboratories

Method

Procedure No.: 7196A **Revision No.:** 0

Controlled Distribution

Name	Manual No.
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Steve Kirschnick	11
Phyllis Christopher	13
Reza Karimi	18
Mohammed Haq	19

EA Engineering, Science, and Technology, Inc.

EA Laboratories

Method

Procedure No.: 7196A **Revision No.:** 0

Revisions		
Rev. No.	**Date of Release**	**Description**
0		Initial distribution

1. Scope and Application

1.1 This method may be used to determine the concentration of dissolved hexavalent chromium, Cr(VI), in ground and surface waters and toxicity extracts. This method may also be applicable to certain domestic and industrial wastes provided that no interfering substances are present.
1.2 The applicable concentration range is 0.02 to 1 mg of Cr(VI) per liter.
1.3 This method is approved for NPDES compliance monitoring.

2. Summary of Method

2.1 Dissolved hexavalent chromium, in the absence of interfering amounts of substances such as molybdenum, vanadium, and mercury, may be determined colorimetrically by reaction with diphenylcarbazide in acid solution. A red-violet product is produced, and its absorbance is measured photometrically at 540 nm.

3. Definitions

3.1 Chromium can exist in aqueous samples in two oxidation states: the trivalent, Cr(III), and the hexavalent, Cr(VI).
3.2 Trivalent chromium is the more stable of the two oxidation states and is generally hydrated or complexed with other anions in solution. At an acidic pH, Cr(III) forms a complex with the anions of the acid; at a neutral or high pH, Cr(III) can form a chromic hydroxide precipitate. Chromic oxide and other chromites are insoluble in water and would be found in the suspended material of a sample.
3.3 Hexavalent chromium forms several different water soluble species depending upon the pH of the water. At a pH of greater than 6, the chromate ion, CrO_4^{2-}, predominates. If the pH is between 2 and 6, the dichromate ion, $Cr_2O_7^{2-}$, is in equilibrium with the chromate ion. The dichromate ion predominates at pH <2 and is fairly stable. However, it is a strong oxidizer and is easily reduced to Cr(III) in the presence of oxidizable materials that may be present in the sample.

4. Sample Handling and Preservation

4.1 Water samples must be filtered through a 0.45-um membrane filter as soon as possible after collection.
4.2 Water samples must not be preserved by acidification, but instead transported and stored at 4 C until time of analysis.
4.3 The stability of hexavalent chromium in environmental samples is not completely understood. The chemical nature of the sample matrix can have a definite effect on the chemistry of chromium; therefore, sample analysis should be carried out as soon as possible. The NPDES holding time is 24 hours.
4.4 Soil and sediment samples should be processed by the alkaline digestion procedure as soon as possible.

5. Interferences

5.1 The chromium reaction with diphenylcarbazide is usually free from interferences. However, certain substances may interfere if the chromium concentration is relatively low. Hexavalent molybdenum and mercury also react to color with the reagents; however, the red-violet intensities produced are much lower than those for chromium at the specified pH. Concentrations of up to 100 mg/L of molybdenum and mercury can be tolerated.
5.2 Vanadium should not be present in concentrations exceeding 4 mg/L. The chromium color develops almost instantly and is stable, whereas vanadium color develops instantly and the fades rapidly. If the original

vanadium concentration is less than 10 mg/L, no vanadium color persists after 10 min.
5.3 Iron in concentrations greater than 1 mg/L may produce a yellow color, but the ferric iron color is not strong and no difficulty is normally encountered if the absorbance is measured photometrically at the appropriate wavelength.
5.4 The effect of water color is small, and color up to 50 platinum-cobalt units can be tolerated.
5.5 If the sample contains materials capable of reducing hexavalent chromium to the trivalent state on acidification (e.g., cyanides, thiosulfate, organic matter), an incorrect result will be obtained. Under such conditions the rigorous determination of hexavalent chromium becomes very difficult. However, this reduction frequently occurs slowly and usable results can be obtained if the analysis is completed immediately after acidification.

6. Apparatus and Materials

6.1 Spectrophotometer, for use at 540 nm, with a light path of 1 cm or longer.
6.2 Volumetric flasks, 1-L, 500-mL, and 100-mL capacity.
6.3 Tubes, 50-mL capacity, graduated at 50, 35, 25, and 12.5 mL (Kimax No. 47125 or equivalent).
6.4 Vortex mixer.

7. Safety and Chemical Hygiene

7.1 The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; however, each chemical compound should be treated as a potential health hazard, and exposure to these chemicals must be reduced to the lowest possible level by whatever means available. The laboratory maintains a reference file of material safety data sheets for the chemicals specified in this method. Additional information on general laboratory safety is available in the laboratory safety and chemical hygiene manuals.

8. Reagents

8.1 Calibration solutions

8.1.1 Hexavalent chromium stock solution, 500 mg/L: Dissolve 1.414 ± 0.0005 g of dried potassium dichromate, $K_2Cr_2O_7$, in deionized water in a 1000-mL volumetric flask and dilute to the mark.
8.1.2 Hexavalent chromium substock solution, 10 mg/L: Pipet 10.0 mL of the 500 mg/L stock solution into a 500-mL volumetric flask and make up to volume with deionized water.
8.1.3 Calibration standards: Prepare a blank and calibration standards by pipetting the indicated amounts of substock solution into 100-mL volumetric flasks and diluting to volume with deionized water. Prepare fresh on the day of use.

Substock Volume (mL)	Final Volume (mL)	Final Concentration (mg/L)
10.0	100	1.0
5.0	100	0.5
1.0	100	0.1
5.0 of 1.0 mg/L	100	0.05
10.0 of 0.1 mg/L	100	0.01

8.1.4 Quality control (QC) stock, 500 mg/L: Prepare in same manner as standard stock (8.1.1), but use a different lot of $K_2Cr_2O_7$.
8.1.5 Laboratory control sample (LCS): Prepare a hexavalent chromium solution of appropriate concentration from the QC stock (8.1.4), or use a commercially prepared quality control solution.

8.2 Sulfuric acid, 3.0 M: Dilute 16.3 mL of reagent grade H_2SO_4 to 100 mL with deionized water.
8.3 1,5-Diphenylcarbazide (1,5-diphenylcarbohydrazide), $C_6H_5NHNHCONHNHC_6H_5$, reagent grade, Aldrich 25,922-5 or equivalent.
8.4 NPDES-approved reagents (see Note 12.1):

8.4.1 Ethanol, 95% (190-proof grain alcohol).
8.4.2 Phthalic anhydride, C_6H_4-1,2-$(CO)_2O$, Aldrich 32,006-4 or equivalent.
8.4.3 Diphenylcarbazide reagent: Add 0.5 g of phthalic anhydride to 80 mL of 95% ethanol and shake for a few minutes. Add 0.1 g of 1,5-diphenylcarbazide and dilute to 100 mL with ethanol. Shake occasionally to dissolve the anhydride. Store in a brown bottle. This reagent is stable for several weeks; slight discoloration may be noted, but this does not impair the usefulness of the reagent.

8.5 RCRA-approved reagents (see Note 12.1):

8.5.1 Acetone, analytical reagent grade.
8.5.2 Diphenylcarbazide reagent: Dissolved 250 mg of 1,5-diphenylcarbazide into 50 mL of acetone. Store in brown bottle. Discard solution when discolored.

9. Procedure

9.1 Sample preparation:

9.1.1 Water samples must be filtered through a 0.45-um membrane filter prior to colorimetric analysis.
9.1.2 Soil and sediment samples must be prepared by a soluble salts extraction.

9.2 Sample screening: High concentration of Cr(VI) will destroy the diphenylcarbazide reagent so that no

color will develop. Samples with such high concentrations will be colored from yellow through orange to burnt orange. The darker the orange color, the higher the level. Depending upon the oxidation state of chromium present, blue or green samples are also possible. To avoid reagent destruction do the following:

9.2.1 Record the original color of the sample before reagent addition, such as no color, light or dark yellow, orange, burnt orange.
9.2.2 If the sample develops an initial purple color upon the addition of reagents but then fades quickly, reagent destruction is taking place, and the sample must be diluted until a stable color is developed.
9.2.3 If the original sample is colored and no color develops, dilute the sample until a faint yellow color persists and add the reagents. If no color develops, either the sample has been diluted too much, or the sample is still destroying the reagent. Therefore, run the sample at a lesser dilution and at a higher dilution to see if color develops at either dilution. It has been found necessary to dilute a dark burnt-orange colored sample as much as 1:10,000 to avoid reagent destruction.
9.2.4 Record all dilutions attempted on a sample and the results of the dilutions (e.g., state if no color developed, if color developed and then faded, if the color was too dark, etc.).

9.3 Color development and measurement:

9.3.1 Set spectrophotometer wavelength to 540 nm. Using a 1-cm cell zero the instrument with deionized water.
9.3.2 Prepare a calibration curve consisting of a minimum of six standards and a calibration blank. Develop the color and measure the absorbances of the standards and blank as described in steps 9.3.3 through 9.3.7.
9.3.3 Pour a 25.0-mL aliquot of each standard, sample, or diluted sample into a separate graduated test tube.
9.3.4 Add 1.0 mL of 3.0 M sulfuric acid to each tube and mix.
9.3.5 To each tube add 1.5 mL of diphenylcarbazide reagent (either that in 8.4.3 or in 8.5.2, depending on the program). Mix; the pink color should begin to develop immediately.
9.3.5 Allow tubes to stand for a minimum of 10 minutes for full color development.
9.3.6 Transfer a portion of each to a 1-cm absorption cell and measure its absorbance at 540 nm.
9.3.7 Any sample that yields an absorbance higher than the highest standard must be diluted and prepared again.

9.4 Color correction: Some waste samples , even after filtering, may be very turbid or dark brown in color and, therefore, present problems for the colorimetric analysis. It may be possible to overcome interferences from turbidity and color by performing the following procedure (Friedman and Erdmann 1982):

9.4.1 Without adding the colorimetric reagents, measure the absorbance of the sample at 540 nm.

9.4.1.1 If the absorbance reading is less than ~0.010 absorbance units, proceed with color development as described in steps 9.3.1 to 9.3.6.
9.4.1.2 If the absorbance reading is greater than ~0.010 but less than 0.050 absorbance units, proceed to step 9.4.2.
9.4.1.3 If the absorbance reading is greater than ~0.050, dilute an aliquot of the sample until the absorbance reading is $\leq$ 0.050. Record the dilution used. Proceed to step 9.4.2.

9.4.2 Proceed with color development as described in steps 9.3.1 to 9.3.6 using two aliquots of the sample or the dilution. Add 1.0 mL of sulfuric acid to both aliquots. Add 1.5 mL of the diphenylcarbazide reagent to one aliquot and 1.5 mL of the diphenylcarbazide solvent only to the other. Record the absorbance readings

of the both aliquots.
9.4.3 Calculate the sample concentrations of both aliquots from the calibration curve, using a dilution factor, if any.
9.4.4 Subtract the apparent concentration of the aliquot without diphenylcarbazide from the concentration of that with diphenylcarbazide to obtain a corrected sample concentration.

NOTE: If a dilution is required in this procedure, the detection limit will be increased by the dilution factor used. Highly turbid or colored samples may require dilutions that produce unacceptably high detection limits. In this case an alternate method, such as Hexavalent Chromium (Lead Coprecipitation, Furnace AAS), should be used.

10. Calculations

10.1 Prepare a calibration curve by regressing the instrument responses (peak heights, areas, or absorbances) of the standards against their concentrations.

10.1.1 Use a degree one (1) for a linear fit, with at least four levels of standards plus the blank.
10.1.2 Proceed if the correlation coefficient of the regression is greater than 0.995.
10.1.3 Back-calculate the concentration of the standards from the regression curve.
10.1.4 Compare the calculated blank concentration against the instrument detection limit.

10.2 Back-calculate the analytical concentrations from the sample instrument responses using the regression curve. Use any dilution and sample preparation factors to calculate the sample concentrations.
10.3 Calculate the concentration of solid samples (in mg/kg dry) from the analytical concentration using the following formula:

$$Concentration(mg/kg\ dry) = \frac{C\ V}{W\ S}$$

where:

C = analytical concentration of digested solid sample (mg/L)
V = volume of digested sample (mL)
W = weight of solid sample digested (g)
S = decimal percent solids of the sample = percent solids/100

11. Quality Control

11.1 Perform the quality control specified in EAL-SOP-185 (Quality Control for Colorimetric Analyses). Additional quality control procedures that apply to this method are specified in the following sections.

11.1.1 Laboratory control sample (LCS): For this method, the initial calibration verification (ICV) serves as the LCS for water sample analyses.

11.2 For soil and sediment samples prepare the following additional quality control samples during the alkaline digestion procedure:

11.2.1 Laboratory control sample (LCS): Preparation of the LCS is specified in method EAL-M-183 (Alkaline Digestion for Hexavalent Chromium).
11.2.2 Method matrix spike: Preparation of a method matrix spike specified in method EAL-M-183 (Alkaline Digestion for Hexavalent Chromium).
11.2.3 Method blank: Preparation of the method blank specified in method EAL-M-183 (Alkaline Digestion for Hexavalent Chromium).

12. Operating Notes

12.1 The procedure approved for NPDES compliance monitoring is based on Methods I-1230-78 (U.S. GS 1979) and 307B (APHA 1976). The RCRA Method 7196 (U.S. EPA 1986) and Method 312B (APHA 1985), which eliminate the phthalic anhydride and use acetone in place of ethanol, are not NPDES approved.
12.2 A lower detection limit and working range can be achieved by using a 5-cm cell instead of the 1-cm in the spectrophotometer. The concentration of the standards would have to altered appropriately.

13. References

13.1 American Public Health Association, American Water Works Association, Water Pollution Control Federation. 1992. Method 3500-Cr D - Chromium (Colorimetric Method), in Standard Methods for the Examination of Water and Wastewater, 18th edition. APHA, Washington, D.C.
13.2 Friedman, L.C. and D.E. Erdmann. 1982. Correction for color interference, in Quality Assurance Practices for the Chemical and Biological Analyses of Water and Fluvial Sediments: Book 5, Chapter A6 of Techniques of Water-Resources Investigations of the United States Geological Survey. USGS, Washington, D.C. p. 23.
13.3 United States Environmental Protection Agency. 1997. Method 7196A - Chromium, Hexavalent (Colorimetric), in Test Methods for Evaluating Solid Wastes. Physical/Chemical Methods. EPA SW-846, 3rd edition, Update III. U.S. EPA, Washington, D.C.
13.4 United States Geological Survey. 1979. Method I-1230-78, in Methods for Determination of Inorganic Substances in Water and Fluvial Sediments. Chapter A1, Book 5 in Techniques of Water-Resources Investigations. USGS, Washington, D.C.

TOC
SW 9060

EA Engineering, Science, and Technology, Inc.

EA Laboratories

Method

Number: 9060 **Rev. No.:** 0

Title: Total Organic Carbon (Catalytic Combustion, Infrared)

Prepared By: L.C. Quinn, Senior Chemist — 30 January 1995

Revised By: for M.E. Wilcox — 13 Mar 96
M.E. Wilcox, Quality Control Specialist II — Date

Approved By: M.M. Uhlfelder — 19 Mar 96
M.M. Uhlfelder, Quality Services Manager — Date

Approved By: — 20 Mar 96
P.A. Christopher, Operations Manager — Date

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1.0 SCOPE AND APPLICATION

1.1 This method is applicable to soils, sediments, and other solid wastes.
1.2 Sludges or other waste liquids can also be analyzed by this methods.

2.0 SUMMARY OF METHOD

2.1 Organic carbon in the sample is converted to carbon dioxide, CO_2, by high temperature oxidation. The oxidation of the sample occurs in a quartz furnace tube, packed with an oxidation promoter compound, under an oxygen atmosphere at approximately 800 C. The CO_2 produced is measured directly by a nondispersive infrared (IR) analyzer.
2.2 Many instrument configurations are available to carry out the high temperature oxidation procedure. The Dohrman Model DC-80 TOC analyzer and PRG-1 furnace unit described in this method perform as follows. A small aliquot of solid (or liquid) sample is placed in a clean platinum boat. The sample is treated with phosphoric acid to remove inorganic carbon. The platinum boat containing the treated sample is placed in the sealed, oxygen-purged PRG-1 unit and pushed into the heated quartz furnace tube containing the oxidation promoter. The oxygen gas flow then carries the CO_2 generated from the oxidized sample to the DC-80 infrared (IR) analyzer.

3.0 DEFINITIONS

3.1 Total carbon (TC): The total of inorganic and organically bound carbon in a sample.
3.2 Total organic carbon (TOC): All carbon atoms covalently bonded in organic molecules.
3.3 Inorganic carbon (IC): Carbonate, bicarbonate, and dissolved carbon dioxide in the sample. In most samples the IC concentration greatly exceeds the TOC concentration. IC is usually removed from the sample prior to determining the TOC concentration. See section 5.1.

4.0 SAMPLE HANDLING AND PRESERVATION

4.1 Soil, sediments, and other solid waste samples are stored in glass jars at 4 C until analysis. Samples should be analyzed within 28 days of collection.

5.0 INTERFERENCES

5.1 In most samples the IC concentration greatly exceeds the TOC concentration. IC is usually removed from the sample prior to determining the TOC concentration. IC interference is removed by acidify the sample to pH 2 or less to convert the IC to CO_2 and heating the acidified sample to remove the CO_2.

6.0 APPARATUS AND MATERIALS

6.1 Dohrman DC-80 total organic carbon analyzer: A schematic of the instrument is shown in Figure 1.
6.2 Dohrman PRG-1 furnace unit (Figure 2).
6.3 Platinum boats.

7.0 SAFETY AND CHEMICAL HYGIENE

7.1 The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; however, each chemical compound should be treated as a potential health hazard, and exposure to these chemicals must be

reduced to the lowest possible level by whatever means available. The laboratory maintains a reference file of material safety data sheets for the chemicals specified in this method. Additional information on general laboratory safety is available in the laboratory safety and chemical hygiene manuals.

8.0 REAGENTS

8.1 Calibration solutions:

8.1.1 TOC standard stock, 2,000 mg TOC/L: Dissolve 4.250 g of dried, primary standard grade potassium hydrogen phthalate (KHP), $C_8H_5O_4K$, in 800 mL deionized water, add 1 mL of phosphoric acid, and dilute to final volume of 1,000 mL. Refrigerate at 4 C. Discard after 6 months.
8.1.2 TOC calibration standards (Table 1):

Because most soil and sediment samples are typically very high in TOC, the least sensitive setting (40 uL) of the DC-80 is calibrated and used to analyze soil and sediment samples. The calibration standards specified below are used with the 40-uL sensitivity setting. Prepare 1000-uL volumes of the calibration standards using the TOC stock and digital micropipettors. Only 40 uL of the calibration standards will be required to calibrate the instrument. Prepare fresh the day of use.

Table 1. CALIBRATION STANDARD PREPARATION

Substock Volume (mL)	Final Volume (10 mL)	Final Concentration of 1000 uL (mg TOC/l)
5.0 of 2000	10	1000
5.0 of 1000	10	500
5.0 of 500	10	250
1.0 of 1000	10	100
1.0 of 500	10	50
1.0 of 250	10	25

8.1.3 Quality control (QC) stock, 2,000 mg TOC/L: Prepare in same manner as standard stock (8.1.1), but use a different lot of KHP.
8.1.4 Laboratory control sample (LCS): Prepare a TOC solution of appropriate concentration from the QC stock (8.1.3), or use a commercially prepared quality control solution.

8.2 Nitric acid solution: Acidify approximately 100 mL of deionized water to a pH less than 2 using concentrated nitric acid. Use the solution to fill the sparger and humidifier vessels of the PRG-1 unit.
8.3 Nitric acid, 1+1: Cautiously add 100 mL of concentrated nitric acid to 100 mL of deionized water.

9.0 PROCEDURE

9.1 PRG-1 furnace operating conditions:

9.1.1 Make sure the quartz furnace tube is intact and is packed with cobalt oxide. (Refer to the TOC System Manual, Figure 2.)
9.1.2 Conditioning the Oxidation Promoter - Conditioning the catalyst helps trap any toxic fumes which may be released by the cobalt oxide during its initial heating. Excercise caution when handling parts. If necessary, wait until heated components have cooled to ensure the greatest level of safety. Proceed as follows:

9.1.2.1 Remove the Teflon line at the exit end of the combustion tube. In its place, install a 15" length of 1/8" OD Teflon tubing with cored grey septum.
9.1.2.2 Place the free end of the Teflon line into a flask containing basic sodium hydroxide solution.
9.1.2.3 Flip the toggle between the sparger and mist trap to the up position. Oserve the brisk bubbling of gas through the flask (at about 200 cc/minute).
9.1.2.4 Power up the Boat Sampling Module using the POWER switch on the front panel of the Boat Sampling Module. (This provides power to the combustion furnance.) Observe that the red light on the rocker switch becomes lit.
9.1.2.5 Allow the furnace to get hot. Then, allow the gas to flow and the catalyst to condition for about 1 hour. The furnace will require about 15 minutes to become fully stablized at 800 C . When the furnace reaches this temperature, the green LED on the front panel will turn on.
9.1.2.6 Carefully remove the Teflon line installed in Step 9.1.2.2. (The furnace will be hot!)
9.1.2.7 Reinstall the teflon line removed in Step 9.1.2.1.

9.1.3 Turn on oxygen carrier flow. Set oxygen pressure to 30 psi. Turn on furnace unit. Allow furnace to heat to 800 C, as indicated by a reddish glow of the furnace tube.
9.1.4 Place fresh nitric acid water in sparge and humidifier vessels on front of PRG-1. Do not use unacidified deionized water in the vessels or the generated CO_2 will dissolve in the water instead of passing on to the DC-80 unit. Never use hydrochloric acid to acidify any solutions used in the TOC unit or damage to the instrument could occur. Oxygen flow is indicated by the presence of sparger bubbles.

Note: For soils/furnace boat application of the PRG-1 unit it is not necessary to have the oxygen flow coming from the furnace unit to the acidified sparge and humidiier vessel. The exit line from the furnace can be directed in to the proper inlet bulkhead of the DC-80 unit.

9.1.5 The toggle switches on the front of the PRG-1 unit are at the following settings for soils/furnace boat applications:

9.2 DC-80 Instrument Operating Conditions:

9.2.1 Refer to Figure 1 for a diagram of the DC-80 unit. Note that the persulfate reactor vessel and UV light source are not used for the Combustion-IR method of TOC determination.
9.2.2 Connect the gas line exiting the humidifier of the PRG-1 unit to the DC-80 bulkhead inlet port of the DC-80. If the sparge and humidifier of the PRG-1 unit are bypassed, connect the gas line exiting the PRG-1 furnace tube to the DC-80 inlet bulkhead.
9.2.3 Connect the DC-80 bulkhead gas line directly to the top port of the U-tube inside the DC-80, bypassing the reaction vessel.
9.2.4 Turn on power to the DC-80 unit. The UV light source and reactor vessel are not required. Adjust front panel controls to the following settings:

9.3 Calibration procedure:

9.3.1 Clean platinum boats as follows:

9.3.1.1 Soak in 10% nitric acid overnight.
9.3.1.2 Allow to dry.

9.3.2 Analysis of standards

9.3.3 Calibration of DC-80

9.4 Preparation of soils and sediment samples:

9.4.1 Weigh 0.0015 ± 0.0005 g of sample in to a tared, clean platinum boat. Record the weight. Place the weighboat on a watchglass. (Note: Aluminum weigh dishes will react witht ehnitric acid, causing errors in the TOC operation.)
9.4.2 Add 1 drop of 1+1 nitric acid to the sample. If effervescence occurs, add additional acid until bubbling ceases. Place acidified sample in an oven at 70°C for 1 hour to remove inorganic carbon from the sample.
9.4.3 The treated sample can be stored in a desiccator until analysis.
9.4.4 Using the magnet and wire basket assembly of the PRG-1 unit, place the platinum boat into the wire basket and slide the sample into the furnace unit. Push the "START" button, and remove sample boat when the "READY" light comes on.
9.4.5 If the TOC concentration of the sample aliquot exceeds the calibration range of the system, prepare a smaller aliquot of sample for analysis.
9.4.6 If the response of the aliquot exceeds the calibration range of the instrument, the TOC concentration will have to be reported as a "greater than" concentration, which is calculated from the formula in 10.3 using the concentration of the highest calibration standard as the analytical concentration.
9.4.7 Prepare a sample spike as follows: Add 40 ul of a 400 mg/L solution to the prepared sample.

10.0 CALCULATIONS

10.1 Prepare a calibration curve by regressing the instrument responses (peak heights or areas) of the standards against their concentrations.

10.1.1 Use a degree one (1) for a linear fit or a degree two (2) for a parabolic fit, with at least four levels of standards plus the blank.
10.1.2 Proceed if the correlation coefficient of the regression is greater than 0.995.
10.1.3 Back-calculate the concentration of the standards from the regression curve.
10.1.4 Compare the calculated blank concentration against the instrument detection limit.

10.2 Back-calculate the analytical concentrations from the sample instrument responses using the regression curve.
10.3 Calculate the concentration of solid samples (in mg/kg dry) from the analytical concentration using the following formula:

$$\text{Concentration (mg/kg dry)} = \frac{C\,V}{W\,S}$$

where:

C = analytical concentration of analyzed solid sample (mg/L)
V = volume of sample (mL) = 0.04 mL (see Note 12.1 below)
W = weight of solid sample analyzed (g)
S = decimal percent solids of the sample = percent solids/100

11.0 QUALITY CONTROL

11.1 Perform the quality control specified in EAL-SOP-185 (Quality Control for Colorimetric Analyses). Additional quality control procedures that apply to this method are specified in the following sections.

11.1.1 Laboratory control sample (LCS): For this method, the initial calibration verification (ICV) serves as the LCS.
11.1.2 Method matrix spike: Preparation of method matrix spike specified in section 9.4.7. Calculate the matrix spike recovery as follows:

$$\text{Percent Matrix Spike Recovery} = \frac{\text{Spiked Sample Concentration (mg/Kg)} - \text{Sample Concentration (mg/Kg)}}{\text{Spike Amount (mg/Kg)}} \times 100$$

12.0 OPERATING NOTES

12.1 For the purposes of the calculation, the volume of the analyzed sample is considered to be 0.040 mL or 40 uL, which is the volume of standard used in the calibration. The use of a volume in the calculation is necessary because the concentrations of the standards, rather than the weights of carbon added to the boat, are used in preparing the calibration curve and, therefore, the analytical concentrations of the samples are expressed in mg/L.

13.0 REFERENCES

13.1 Rosemont Analytical - Dohrman Division. February 1989. DC-80 Automated Laboratory Total Organic Carbon Analyzer Manual, Edition 13. Santa Clara, Calif.
13.2 United States Environmental Protection Agency. 1986. Method 9060 - Total Organic Carbon in Test Methods for Evaluating Solid Wastes. Physical/Chemical Methods. EPA SW-846, 3rd edition. U.S. EPA, Washington, D.C.
13.3 Annual Book of ASTM Standards, Part 31, "Water," Standard D 2574-79, p. 469 (1976).
13.4 Standard Methods for the Examination of Water and Wastewater, 14th Edition, p.532, Method 505 (1975).

Tissue Homoginization

EA Engineering, Science, and Technology, Inc.

EA Laboratories

Standard Operating Procedures

Number: 289 **Rev. No.:** 1

Title: Preparation of Animal Tissue for Analysis

Approved By: _____ 3/5/98
G.M. Gregory, Semivolatiles Section Chief — Date

Approved By: _____ 3/4/98
W.E. Miller, Semivolatiles QC Chemist — Date

Approved By: _____ 3/9/98
M.M. Uhlfelder, Quality Services Manager — Date

1.0 SCOPE AND APPLICATION

This method is a procedure for the preparation of animal tissue for preparation methods or instrumental analysis.

2.0 SUMMARY OF METHOD

The frozen whole body animal or selected part of animal is prepared for analysis by being chopped into small pieces, and homogenized in a blender or other homogenization device (i.e., HOBART).

3.0 PROCEDURE

3.1 Sample Collection

3.1.1 A sufficient number of organisms or parts of organisms should be combined by sampling site location and species to obtain the minimum weight (250 grams).
3.1.2 The collected samples are wrapped in aluminum foil and frozen for transport to the lab.

3.2 Sample Homogenization

3.2.1 At the laboratory, unwrap the organisms or parts of organisms and chop into small pieces suitable for the homogenization device. Small portions of the frozen organism are blended until tissue is completely pulverized. Repeated additions of small portions of the organism are blended until all of the tissue is completely pulverized. Repeated additions of small portions of the organism aids in maintaining a powdery fluff that will mix more uniformly.

3.2.2 Weigh two 10.0 g portions of the sample into separate VOA vials, and store in a freezer. These sample aliquots are ready for volatile organic analysis.
3.2.3 Divide the remaining homogenized tissue sample equally between two liter-size glass containers, cap with Teflon-lined lids and store in a freezer.
3.2.4 One aliquot will be used for extractable organic determinations (e.g., semivolatile and pesticide/PCB analysis), and the second aliquot for the determination of metals and general chemistry parameters.

4.0 QUALITY CONTROL

4.1 Standard clam tissues should be used for the method blank and laboratory control sample, and should be processed through all the sample preparation steps including blending for the same length of time as the samples.
4.2 For analysis, the QA/QC requirements in the method or project must be satisfied.

5.0 REFERENCES

5.1 "Interim Methods for the Sampling and Analysis of Priority Pollutants in Sediments and Fish Tissue". EPA 600/4-81-055. U.S. Environmental Protection Agency, Cincinnati, Ohio, October 1980.

Percent Lipids

EA Engineering, Science, and Technology, Inc.

EA Laboratories

Method

Number: LIPID **Rev. No.:** 2

Title: Determination of Percent Lipids (Gravimetric)

Prepared By: L.C. Quinn, Senior Chemist 13 December 95

Revised By: M.E. Wilcox 09/12/96
M.E. Wilcox, QC Specialist II Date

Approved By: M.M. Uhlfelder 9/12/97
M.M. Uhlfelder, Quality Services Manager Date

Approved By: Walter E Miller 9/12/97
W.E. Miller, Organic QC Chemist Date

WHEN STAMPED IN RED, THIS IS A CONTROLLED COPY. IF STAMP IS NOT RED, VERIFY THAT YOU ARE USING THE CURRENT REVISION BEFORE PROCEEDING WITH WORK UNDER THIS PROCEDURE

1.0 SCOPE AND APPLICATION

This method is used to determine the concentration of the lipid content in animal tissue such as fish/invertebrates. Various organic environmental pollutants are lipid-soluble in biota samples. This method provides integral analytical information for further biological assessments.

2.0 SUMMARY OF METHOD

Homogenized tissue is extracted with methylene chloride by vortexing. The extract is concentrated by heating. Percent lipids is determined gravimetrically.

3.0 DEFINITIONS

Laboratory Control Sample (LCS) is extracted with each analytical batch to determine if the spiked compound recoveries are within the laboratory limits.

Method Blank is extracted with each analytical batch to determine whether or not the extraction and/or analysis introduced any target analytes into the samples. If the Method Blank contains any target analytes, the analytical batch is considered contaminated and should be re-extracted.

4.0 SAMPLE HANDLING, PRESERVATION, AND HOLDING TIME

4.1 Samples are received in coolers packed with ice to preserve the animal tissue. The tissue samples are contained in plastic bags or glass jars.
4.2 While samples are in the custody of the laboratory they are stored in the sample walk-in freezer by the Sample Management Office.
4.3 Holding times have not been established at this time by any regulatory agencies for the percent lipid determination.

5.0 INTERFERENCES

All glassware used for the lipid determination must be free from all types of animal fat. Rinsing glassware with methylene chloride will significantly eliminate most grease or fat residues.

6.0 APPARATUS AND MATERIALS

6.1 Analytical Balance: (± 0.0001 g. Accuracy)
6.2 Tissuemiser, Hobart food chopper or other mechanical device.
6.3 Filter paper: Whatman PS
6.4 VOA vials: 40-ml, glass, Teflon-lined cap.
6.5 Vortex mixer.
6.6 100 ml beaker, glass-powder funnel.
6.7 Hot plate: capable of maintaining constant temperatures.

7.0 SAFETY AND CHEMICAL HYGIENE

The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; however, each chemical compound should be treated as a potential hazard, and exposure to these chemicals must be reduced to the lowest possible level by whatever means available. The laboratory maintains a reference file of Material Safety Data Sheets (MSDS) for the chemicals specified in this method. Additional information concerning laboratory safety is available in theLaboratory Safety Plan and from the Laboratory Health and Safety Officer.

8.0 REAGENTS

8.1 Methylene Chloride, CH_2Cl_2 - supplied by laboratory vendor.
8.2 Anhydrous Sodium Sulfate, $NaSO_4$ - supplied by laboratory vendor.
8.3 Cod Liver Oil - commerical source.

9.0 PROCEDURE

9.1 Sample Preparation: Tissue homogenization is performed by tissuemiser, Hobart food chopper, or other mechanical means.
9.2 Weigh 3.0 g of the homogenized tissue into a VOA vial. Record the sample weight onto the EA Laboratories % Lipids Extraction Sheet to the nearest 0.0001 g. (FIGURE 1).

9.2.1 The method blank consists of a clean VOA vial.
9.2.2 For the LCS, add approximately 30-60 mg (0.0300-0.0600 grams) of cod liver oil to a clean VOA vial (this represents a 1-2% lipid content based on a 3 gram sample).

9.3 Add 25 mL of methylene chloride to the sample in the VOA vial. Vortex for one minute.
9.4 Weigh a clean, dry 100 mL beaker on analytical balance, record the tare weight to nearest 0.0001 g.
9.5 Set beaker on clean surface. Place glass funnel in mouth of beaker, place folded phase-separating filter paper (Whatman PS) in funnel.
9.6 Quantitatively transfer the vortexed contents of the VOA vial to the filter paper-lined funnel in the beaker. Add 5 mL of methylene chloride to the empty VOA vial to rinse the vial, transfer rinseate to funnel. Repeat rinse with another 5 mL of methylene chloride.

NOTE: If sample contains a significant amount of free water, anhydrous sodium sulfate may be placed in the phase-separator paper to absorb excess water.

9.7 Remove funnel from beaker, place beaker on clean hot plate set at medium (50%) heat and evaporate sample to near dryness. Care must be taken not to splatter sample during boiling nor burn residual oil. Allow beaker to cool for five minutes. Remaining solvent will evaporate during cooling.
9.8 Using analytical balance, weigh beaker and lipid residue. Record weight to nearest 0.0001 g.

10. CALCULATIONS - Calculation of the Lipid Content as follows:

$$\text{Percent Lipid} = \frac{(A-B)}{C} \times 100$$

Where:
A = weight of beaker and dried residue (g)
B = tare weight of beaker (g)
C = weight of tissue sample (g)

11.0 QUALITY CONTROL - To establish the ability to generate data of acceptable bias and precision, the analysis must meet the following quality control criteria:

11.1 The method blank is acceptable if the final weight deviates from the initial weight by less than 0.0030 grams (which is equivalent to less than 0.1% lipid based on a 3 gram sample). If the weights deviate by 0.0030 grams or more, the entire batch must be reprepared and %lipid determination redone.
11.2 The percent recovery for the cod liver oil added to the LCS is determined and evaluated against laboratory established limits.

11.2.1 If the recovery is above the upper QC limit, determine if any samples had a percent lipid value above 0.1% (based on 3 gram sample). If any associated samples have lipid values above 0.1%, then those samples must be re-extracted and %lipid determination re-reanalyzed.
11.2.2 If the recovery is below the lower QC limit, the entire batch must be re-extracted and %lipid determination re-analyzed.

12.0 REFERENCE

12.1 Bligh, E.G., and Dyer, W.J., 1959. A Rapid Method of Total Lipid Extraction and Purification: Canadian Journal of Biochemistry and Physiology, Vol 37, pp 911-917.
12.2 Gardner, W.S., Frez, W.A., Chichocki, E.D. and Parrish, C.C., 1985. Micro Method for Lipid Analyses in Aquatic Invertebrates. Limnology and Oceanography, Vol 30, pp 1099-1105.
12.3 National Oceanic and Atmospheric Administration, July 1993. Tissue Lipid Determination Method in Sampling and Analysis Methods of the National Bethie Surveillance and Mussel Water Projects. 1984-1992. Volume II. comprehensive Descriptions of Complementary Measurements. NOAA, Silver Spring, Maryland.
12.4 Randall, R.C., Lee II, H., Ozretich, R., Lake, J.L., and Pruell, R.J., 1991. Evaluation of Selected Lipid Methods for Normalizing Bioaccumulation: Environmental Toxicology and Chemistry. Vol 10, pp 1431-1436.
12.5 United States Army Engineer Waterways Experiment Station. May 1995. A Comparison of Three Lipid Extraction Methods. Technical Note EEDP-01-35. 3909 Halls Ferry Road, Vicksburg, Mississippi 39180-6199.

EA LABORATORIES PERCENT LIPIDS EXTRACTION SHEET

CLIENT:	EXTRACTION CHEMIST:	METHOD: EA IN HOUSE
BATCH #: LIP-	CONCENTRATION CHEM	EXTRACTION SOLVENT:
	DATE:	SOLVENT LOT:

EA NUMBER	CLIENT ID	FRACTION	MATRIX	MASS BEAKER	MASS SAMPLE	MASS BEAKER PLUS RESIDUE	% LIPIDS	COMMENTS
LPBLK		LIPIDS	TISSUE					
LPLCS		LIPIDS	TISSUE					
		LIPIDS	TISSUE					
		LIPIDS	TISSUE					
		LIPIDS	TISSUE					
		LIPIDS	TISSUE					
		LIPIDS	TISSUE					
		LIPIDS	TISSUE					
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FIGURE 1.
EA Laboratories Percent Lipids Extraction Sheet

28-day Toxicity Test
Leptocheirus plumulosus

SEDIMENT 28-DAY TOXICITY TEST WITH *Leptocheirus plumulosus*

1. TEST OBJECTIVE

To assess the toxicity of a whole sediment sample to estuarine amphipod *Leptocheirus plumulosus* and determine the effects on survival, growth (determined by dry weight and/or length) and reproduction of the test organisms compared to controls.

2. TEST ARTICLE

2.1 Description/Identification

Unless otherwise specified, the test material is supplied by the client. The test article is a whole sediment sample. Adequate chemical specifications with special reference to hazardous properties and storage conditions are also supplied by the client. When available, information on the stability, composition, or other characteristics which define the test article are on the file with the client.

2.2 Sample Preparation

Depending upon the project, the sediment may be screened through a suitably sized sieve to remove large particles and indigenous organisms, and then homogenized before being placed in the test chambers. Sediment and overlying water may be added to test vessels 24 hours prior to introduction of test organisms in order to allow suspended sediments to settle.

3. EXPERIMENTAL DESIGN

3.1 Test Organisms

3.1.1 Species

The test species is the estuarine amphipod *Leptocheirus plumulosus*.

3.1.2 Source

Leptocheirus plumulosus used for toxicity tests are obtained from in-house cultures, or from a scientific organism vendor as specified in the report.

3.1.3 Culturing and Holding Conditions

Stocks obtained from a scientific vendor are transferred into holding tanks with clean control sediment and overlying water. The *Leptocheirus plumulosus* are maintained in an environmentally controlled laboratory at 20±2°C with a 16-hour light/8-hour dark photoperiod cycle. During holding, the amphipods are fed finely ground Tetramin flake food. The organisms are gradually acclimated to testing conditions prior to use in testing. Certain regulatory or project specific objectives may require organism acclimation to the dilution water when it is different from the holding/culture water.

3.1.4 Size of Test Organisms at Test Initiation

Immature amphipods which pass through a 500 μm screen, but are retained on a 250 μm screen, are used to initiate toxicity tests.

3.2 Test Concentration Series

L. plumulosus are exposed in replicate chambers to whole sediment samples and to a laboratory or reference sediment control.

3.3 Overlying Water

Overlying water for sediment tests is either GP-2 artificial seawater made with deionized water, dechlorinated tap water mixed with commercially available synthetic sea salts, or an appropriate receiving water. Batches of GP-2 at the appropriate salinity are made for the chronic testing using the formulation in US EPA (1993).

The source of the dechlorinated tap water is the City of Baltimore municipal water system. Upon entry to the laboratory, the water passes through a high-capacity, activated carbon filtration system to remove chlorine and possible organic contaminants. This water source has proven safe for aquatic organism toxicity testing at EA, as evidenced by maintenance of multigeneration *Daphnia* sp. and fathead minnow cultures with no evident loss of fecundity. The dechlorinated tap water or deionized tap water is mixed with commercially available synthetic sea salts to a salinity of approximately 20±2 ppt or a salinity specified by the project study plan.

3.4 Test Vessels and Test Volume

Test vessels are 1-L exposure beakers containing 100-200 ml of sediment and 400-800 ml of

overlying water (typically 1:4 ratio of sediment to overlying water). The size of the test vessels, and the volume of sediment and overlying water may be changed depending on the study requirements.

3.5 Test Organism Number

Tests are conducted using at least three replicate chambers per sediment sample, with 20 organisms per replicate chamber.

3.6 Test Environment

The test vessels are maintained in an environmentally controlled laboratory with a 16-hour light, 8-hour dark photoperiod. Temperature within the environmental room is monitored continuously using temperature recorders and is maintained at 20 ± 1 °C (unless a different project-specific temperature is required).

3.7 Test Observations

Water quality measurements (temperature, pH, dissolved oxygen, and salinity) are recorded at test initiation, test termination, and prior to each renewal from a minimum of one replicate of each sediment, reference, and control treatment. Renewal of the overlying water is performed three times per week. The overlying water may be gently aerated, if necessary, to maintain dissolved oxygen levels at ≥4.0 mg/L. Analytical determinations are conducted according to APHA et al. (1995) and EPA (1979).

The study terminates after 28 days of exposure to the sediment sample. At test termination, the sediment from each replicate is carefully sieved to retrieve all adult organisms and offspring. The number of surviving adults and offspring is recorded for each replicate. The reproductive response for the 28-day exposure test is the average number of offspring produced per surviving female. Surviving adults are then examined under a dissecting microscope to determine gender and in the case of females, the presence of eggs or embryos in the brood pouch.

At the end of the test period, a growth endpoint may be determined through length measurement and/or dry weight determination. For length measurements, surviving organisms are preserved in 70 percent ethanol, and body length from the base of the first antenna to the base of the third pleon segment along the dorsal surface is measured using an ocular micrometer. Length measurements are performed to 0.1 mm. For dry weight determinations, surviving amphipods may be placed in pre-weighed, oven-dried aluminum pans (one replicate

per pan). Organisms are dried in an oven for a minimum of six hours at 100°C after which each pan is weighed. Mean dry weight of the amphipods in each replicate is calculated by subtracting the weight of the pan from the combined weight of the pan and organisms, then dividing by the number of organisms per replicate.

3.8 Data Analysis

Statistical analyses can be performed on percent survival and on dry weight data. An analysis of variance (ANOVA) and t-Test are used to analyze significance of effects between the control sediment and a single test sediment (US EPA 1994). Alternatively, Tukey's Test may be performed to check the difference between all pairs of treatments. Other appropriate statistical analyses may be performed. The statistical methods used are specified in the final report.

4. FINAL REPORT

The final report is prepared to contain, at a minimum, the following information:

- Objectives and procedures stated in the approved protocol, including any changes made to the original protocol
- Identity of the test article(s) by name or code number and a description of any pretreatment
- Source of the overlying water, its chemical characteristics, and a description of any pretreatment
- Test concentration series used and duration of the assay
- Mean dry weights and/or lengths of test organisms with the respective standard deviations
- Average number of offspring produced per surviving female with respective standard deviations
- Water quality characteristics (pH, dissolved oxygen, temperature, etc.) of overlying water from reference, control and test sediment treatments

- Any unforeseen circumstances that may have affected the quality or integrity of the study
- Signature of the project manager, senior technical reviewer, and quality control officer authorizing release of the report
- Location of all archived data and the original copy of the final report at EA

Items of data to be included in the report consist of experimental design and test performance, effects on general appearance of test organisms (if applicable), morbidity and mortality, tabular presentation and appropriate statistical evaluation of water quality characteristics, survival, weight and/or length data.

5. QUALITY ASSURANCE

5.1 Amendments to Protocol

Amendments to the authorized protocol established by EA or by the client are made only after proper authorization. Such authorization is achieved by completion of the Protocol Amendment Form by EA after consultation with the client.

5.2 Standard Operating Procedures

Unless otherwise specified, all procedures given in the protocol are subject to detailed Standard Operating Procedures (SOPs) which are contained in the SOP manuals of the participating departments. These SOPs and protocols generally follow the type of requirements in the U.S. EPA's Good Laboratory Practice Standards (GLPs) (US EPA 1989).

5.3 Reference Toxicant

A reference toxicant test, utilizing sodium dodecyl sulfate (SDS), cadmium chloride, or another appropriate chemical is used as an internal quality check of the sensitivity of the test organisms. Testing is conducted on each population of organisms purchased for testing from an outside source if reference toxicant data are not available from the supplier on the acquired lot. The results of each test are compared with historical, species-specific toxicological information from reference toxicant tests performed at EA, to determine if the results are within acceptable limits. Limits are established using the control charts outlined in US EPA (1993).

5.4 Quality Assurance Evaluation

Studies conducted under this protocol may be subject to internal audit by EA's Quality Assurance Unit. A quality control officer is responsible for monitoring each study to assure the client that the facilities, equipment, personnel, methods, practices, records, and controls are in conformance with EA's QC program and, if applicable, EPA's GLPs.

5.5 Inspection by Regulatory Authorities

In the event of an inspection of EA by an outside authority during the course of the study, the client whose study is being inspected will be consulted before examiners are permitted access to any of the project records or the experimental areas.

5.6 Archives

Copies of project-specific records shall be transferred to the client promptly after the project is completed or as negotiated and budgeted. Original primary data are retained at EA for 5 years. Primary data include chain-of-custody records, laboratory data sheets, records, memoranda, notes, photographs, microfilm, and computer printouts that are a result of the original observations and activities of the study and which are necessary for the reconstruction and evaluation of the study report.

5.7 Location

Studies are conducted at the Ecotoxicology Laboratory of EA Engineering, Science, and Technology, Inc. at the Loveton Office in Sparks, Maryland.

6. SPECIFICATIONS OF THE *Leptocheirus plumulosus* SEDIMENT TOXICITY TEST

6.1 Basic References

American Public Health Association (APHA) American Water Works Association, Water Environment Federation. 1995. Standard Methods for Examination of Water and Wastewater, 19th or most recent version. APHA, Washington, D.C.

American Society for Testing and Materials (ASTM). 1995. Standard Guide for Conducting 10-day Static Sediment Toxicity Tests with Marine and Estuarine Amphipods. ASTM Designation E 1367-92, Philadelphia, Pennsylvania.

American Society for Testing and Materials (ASTM). 1995. Standard Practice for Conducting Acute Tests with Fishes, Macroinvertebrates, and Amphibians. ASTM Designation: E729-80, Philadelphia, Pennsylvania.

American Society for Testing and Materials (ASTM). 1995. Standard Guide for Conducting Sediment Toxicity Tests with Freshwater Invertebrates. ASTM Designation: E1383-94, Philadelphia, Pennsylvania.

EA. 1996. Quality Control and Standard Operating Procedures Manual for EA's Ecotoxicology Laboratory. Fifth Revision. EA Manual ATS-102. Internal document prepared by EA's Ecotoxicology Laboratory, EA Engineering, Science, and Technology, Inc., Sparks, Maryland.

McGee, B.L., C.E. Schlekat and E. Reinharz. 1993. Assessing Sublethal Levels of Sediment Contamination Using the Estuarine Amphipod *Leptocheirus plumulosus*. Environ. Toxicol. Chem. 12: 577-587.

US EPA. 1979. Methods for Chemical Analysis of Water and Wastes. EPA/600/4-79/020. Environmental Monitoring and Support Laboratory, Cincinnati, Ohio.

US EPA. 1988. Short-term Methods for Estimating the Chronic Toxicity of Effluents and Receiving Waters to Marine and Estuarine Organisms. EPA/600/4-87/028. U.S. Environmental Protection Agency. Environmental Monitoring and Support Laboratory, Cincinnati, Ohio.

US EPA. 1989. Toxic Substances Control Act (TSCA); Good Laboratory Practice Standards. Title 40 CFR Part 792. Fed. Regist. 54(158): 34034-34074.

US EPA. 1991. Methods for Measuring the Acute Toxicity of Effluents and Receiving Waters to Freshwater and Marine Organisms. Fourth Edition. EPA/600/4-90/027. U.S. Environmental Protection Agency. Environmental Monitoring Systems Laboratory, Cincinnati, Ohio.

US EPA. 1993. Methods for Measuring the Acute Toxicity of Effluents and Receiving Waters to Freshwater and Marine Organisms. Fourth Edition. EPA/600/4-90/027F. U.S. Environmental Protection Agency, Environmental Monitoring Systems Laboratory, Cincinnati, Ohio.

6.2 Test Specifications

Test organism:	*Leptocheirus plumulosus*
Organism age/size:	Immature amphipods which pass through a 500 μm screen but are retained on a 250 μm screen
Aeration:	Gentle aeration may be provided through a 1-ml glass pipet, placed no closer than 2 cm above the sediment surface
Temperature:	20±1°C (or as specified by project study plan)
Light quality:	Wide-spectrum fluorescent light
Light intensity:	50-100 f.c.
Photoperiod:	16-hour light, 8-hour dark
Overlying water:	Synthetic seawater or appropriate receiving water
Test container:	1-L beakers
Test volume:	100-200 ml of sediment with 400-800 ml of overlying water (typically 1:4 ratio of sediment to overlying water)
No. of replicates:	Minimum of three
No. organisms per replicate:	20
Feeding regime:	Finely ground Tetramin flake food, three times per week, after renewal of overlying water
Test duration:	28 days
Endpoints:	Survival, growth, and reproduction
Test acceptability:	May vary depending on regulatory requirements; one criterion is ≥70 percent control survival

Toxicity Test
Neanthes arenaceodentata

SEDIMENT TOXICITY TEST WITH *Neanthes arenaceodentata*

1. TEST OBJECTIVE

To assess the toxicity of a whole sediment sample to polychaetous annelid *N. arenaceodentata* and determine the effects on survival and growth (determined by dry weight) of the test organisms compared to controls.

2. TEST ARTICLE

2.1 Description/Identification

Unless otherwise specified, the test material is supplied by the client. The test article is a whole sediment sample. Adequate chemical specifications with special reference to hazardous properties and storage conditions are also supplied by the client. When available, information on the stability, composition, or other characteristics which define the test article are on the file with the client.

2.2 Sample Preparation

Depending upon the project, the sediment may be screened through a suitably sized sieve to remove large particles and indigenous organisms, and then homogenized before being placed in the test chambers. Sediment and overlying water may be added to test vessels 24 hours prior to introduction of test organisms in order to allow suspended sediments to settle.

3. EXPERIMENTAL DESIGN

3.1 Test Organisms

3.1.1 Species

The test species is the marine polychaete *Neanthes arenaceodentata*.

3.1.2 Source

Neanthes arenaceodentata used for toxicity tests are obtained from a scientific organism vendor as specified in the report.

3.1.3 Culturing and Holding Conditions

Stocks obtained from a scientific vendor are transferred into a glass aquaria containing the marine alga *Enteromorpha* sp. The *N. arenaceodentata* are gradually acclimated to testing conditions and to the overlying water used in testing if appropriate, and are maintained in an environmentally controlled laboratory at 20±2°C, with a 16-hour light, 8-hour dark photoperiod cycle. The polychaetes are fed *Enteromorpha* sp. or ground alfalfa. Certain regulatory or project specific objectives may require organism acclimation to the dilution water when it is different from the holding/culture water.

3.1.4 Size of Test Organisms at Test Initiation

Use recently emerged *N. arenaceodentata* larvae having approximately 18-21 setigerous segments.

3.2 Test Concentration Series

N. arenaceodentata are exposed in replicate chambers to whole sediment samples and to a laboratory or reference sediment control.

3.3 Overlying Water

Overlying water for sediment tests is either GP-2 artificial seawater made with deionized water, dechlorinated tap water mixed with commercially available synthetic sea salts, or an appropriate receiving water. Batches of GP-2 at the appropriate salinity are made for the chronic testing using the formulation in US EPA (1993).

The source of the dechlorinated tap water is the City of Baltimore municipal water system. Upon entry to the laboratory, the water passes through a high-capacity, activated carbon filtration system to remove chlorine and possible organic contaminants. This water source has proven safe for aquatic organism toxicity testing at EA, as evidenced by maintenance of multigeneration *Daphnia* sp. and fathead minnow cultures with no evident loss of fecundity. The dechlorinated tap water or deionized tap water is mixed with commercially available synthetic sea salts to a salinity of approximately 30±2 ppt or a salinity specified by the project study plan.

3.4 Test Vessels and Test Volume

Test vessels are 1-L exposure beakers containing 100-200 ml of sediment and 400-800 ml of

overlying water. The size of the test vessels, and the volume of sediment and overlying water may be changed depending on the study requirements.

3.5 Test Organism Number

Tests are conducted using at least three replicate chamber per sediment sample, with at least five organisms per replicate chamber.

3.6 Test Environment

The test vessels are maintained in an environmentally controlled laboratory with a 16-hour light, 8-hour dark photoperiod. Temperature within the environmental room is monitored continuously using temperature recorders and is maintained at 20 ± 1°C (unless a different project-specific temperature is required).

3.7 Test Observations

At test termination, test organisms are observed to record the number of surviving polychaetes. The study terminates after ten days of exposure to the sediment sample.

Measurements of water quality are taken at test initiation and daily thereafter for dissolved oxygen, pH, temperature, and conductivity from a minimum of one replicate of each sediment, reference, and control treatment. Renewal of the overlying water is performed every fourth day. Aliquots of overlying water may be gently aerated, if necessary. Analytical determinations are conducted according to APHA et al. (1995) and US EPA (1979).

At the end of the test period, surviving polychaetes may be placed in pre-weighed, oven dried aluminum pans (one replicate per pan). Organisms are oven dried for a minimum of six hours at 100°C after which each pan is weighed. Mean dry weight of the polychaetes (weight of pan and organisms minus weight of pan/number of organisms) is calculated.

3.8 Data Analysis

Statistical analyses are performed on percent survival and mean dry weight data. Analysis of variance (ANOVA) and either Bonferroni's T-Test or Dunnett's Mean Comparison Test are used to analyze significance of effects. Depending on the distributional characteristics of the data generated, it may be necessary to use Steel's Many-One Rank Test or the Wilcoxon Rank Sum Test instead (US EPA 1994). The methods used are specified in the final report.

4. FINAL REPORT

The final report is prepared to contain, at a minimum, the following information:

- Objectives and procedures stated in the approved protocol, including any changes made to the original protocol
- Identity of the test article(s) by name or code number and a description of any pretreatment
- Source of the overlying water, its chemical characteristics, and a description of any pretreatment
- Test concentration series used and duration of the assay
- Mean dry weights of test organisms with the respective standard deviations
- Water quality characteristics (pH, dissolved oxygen, temperature, etc.) of overlying water from reference, control and test sediment treatments.
- Any unforeseen circumstances that may have affected the quality or integrity of the study
- Signature of the project manager, senior technical reviewer, and quality control officer authorizing release of the report
- Location of all archived data and the original copy of the final report at EA

Items of data to be included in the report consist of experimental design and test performance, effects on general appearance of test organisms (if applicable), morbidity and mortality, tabular presentation and appropriate statistical evaluation of water quality characteristics, survival, and weight data.

5. QUALITY ASSURANCE

5.1 Amendments to Protocol

Amendments to the authorized protocol established by EA or by the client are made only after proper authorization. Such authorization is achieved by completion of the Protocol Amendment Form by EA after consultation with the client.

5.2 Standard Operating Procedures

Unless otherwise specified, all procedures given in the protocol are subject to detailed Standard Operating Procedures (SOPs) which are contained in the SOP manuals of the participating departments. These SOPs and protocols generally follow the type of requirements in the U.S. EPA's Good Laboratory Practice Standards (GLPs) (US EPA 1989).

5.3 Reference Toxicant

A reference toxicant test, utilizing sodium dodecyl sulfate (SDS), cadmium chloride, or another appropriate chemical is used as an internal quality check of the sensitivity of the test organisms. Testing is conducted on each population of organisms purchased for testing from an outside source if reference toxicant data are not available from the supplier on the acquired lot. The results of each test are compared with historical, species-specific toxicological information from reference toxicant tests performed at EA, to determine if the results are within acceptable limits. Limits are established using the control charts outlined in US EPA (1993).

5.4 Quality Assurance Evaluation

Studies conducted under this protocol may be subject to internal audit by EA's Quality Assurance Unit. A quality control officer is responsible for monitoring each study to assure the client that the facilities, equipment, personnel, methods, practices, records, and controls are in conformance with EA's QC program and, if applicable, EPA's GLPs.

5.5 Inspection by Regulatory Authorities

In the event of an inspection of EA by an outside authority during the course of the study, the client whose study is being inspected will be consulted before examiners are permitted access to any of the project records or the experimental areas.

5.6 Archives

Copies of project-specific records shall be transferred to the client promptly after the project is completed or as negotiated and budgeted. Original primary data are retained at EA for 5 years. Primary data include chain-of-custody records, laboratory data sheets, records, memoranda, notes, photographs, microfilm, and computer printouts that are a result of the original observations and activities of the study and which are necessary for the reconstruction and evaluation of the study report.

5.7 Location

Studies are conducted at the Ecotoxicology Laboratory of EA Engineering, Science, and Technology, Inc. at the Loveton Office in Sparks, Maryland.

6. SPECIFICATIONS OF THE *Neanthes arenaceodentata* SEDIMENT TOXICITY TEST

6.1 Basic References

American Public Health Association (APHA) American Water Works Association, Water Environment Federation. 1995. Standard Methods for Examination of Water and Wastewater, 19th or most recent version. Method 8510: Annelids. APHA, Washington, D.C.

American Society for Testing and Materials (ASTM). 1995. Standard Practice for Conducting Acute Tests with Fishes, Macroinvertebrates, and Amphibians. ASTM Designation: E 729-80, Philadelphia, Pennsylvania.

American Society for Testing and Materials (ASTM). 1995. Standard Guide for Conducting Sediment Toxicity Tests with Freshwater Invertebrates. ASTM Designation: E 1383-94, Philadelphia, Pennsylvania.

American Society for Testing and Materials (ASTM). 1995. Standard Guide for Conducting Acute, Chronic, and Life-Cycle Aquatic Toxicity Tests with Polychaetous Annelids. ASTM Designation: E1562-94, Philadelphia Pennsylvania.

EA. 1996. Quality Control and Standard Operating Procedures Manual for EA's Ecotoxicology Laboratory. Fifth Revision. EA Manual ATS-102. Internal document prepared by EA's Ecotoxicology Laboratory, EA Engineering, Science, and Technology, Inc., Sparks, Maryland.

US EPA. 1988. Short-term Methods for Estimating the Chronic Toxicity of Effluents and Receiving Waters to Marine and Estuarine Organisms. EPA/600/4-87/028. U.S. Environmental Protection Agency. Environmental Monitoring and Support Laboratory, Cincinnati, Ohio.

US EPA. 1989. Toxic Substances Control Act (TSCA); Good Laboratory Practice Standards. Title 40 CFR Part 792. Fed. Regist. 54(158): 34034-34074..

US EPA. 1991. Methods for Measuring the Acute Toxicity of Effluents and Receiving Waters to Freshwater and Marine Organisms. Fourth Edition. EPA/600/4-90/027. U.S. Environmental Protection Agency. Environmental Monitoring Systems Laboratory, Cincinnati, Ohio.

US EPA. 1993. Methods for Measuring the Acute Toxicity of Effluents and Receiving Waters to Freshwater and Marine Organisms. Fourth Edition. EPA/600/4-90/027F. U.S. Environmental Protection Agency, Environmental Monitoring Systems Laboratory, Cincinnati, Ohio.

6.2 Test Specifications

Test organism:	*Neanthes arenaceodentata*
Organism age:	Recently emerged larvae having approximately 18-21 setigerous segments.
Aeration:	Gentle aeration may be provided through a 1-ml glass pipet, placed no closer than 2 cm above the sediment surface.
Temperature:	20±1°C
Light quality:	Wide-spectrum fluorescent light
Light intensity:	50-100 f.c.

Photoperiod:	16-hour light, 8-hour dark
Overlying water:	Synthetic seawater or appropriate receiving water
Test container:	1-L beakers
Test volume:	100-200 ml of sediment with 400-800 ml of overlying water
No. of replicates:	Minimum of three
No. organisms per replicate:	5
Feeding regime:	175 mg of ground cereal leaves or powdered alfalfa in 700 ml deionized water: approximately 10 ml of suspension, daily per test chamber.
Test duration:	10 days
Endpoints:	Survival and growth
Test acceptability:	May vary depending on regulatory requirements; one criterion is 70 percent or greater survival in all control replicates.

Whole-Sediment Bioaccumulation

MD-1 GUIDELINES FOR PERFORMING WHOLE-SEDIMENT BIOASSAYS AND WHOLE-SEDIMENT BIOACCUMULATION STUDIES

1. OVERVIEW

Whole-sediment bioassays and whole sediment bioaccumulation studies are required for evaluation of dredged material proposed for disposal in ocean waters. These evaluations are required in response to Section 103 of PL 95-532 (Marine Protection, Research, and Sanctuaries Act of 1972) and must follow guidelines established jointly by the Environmental Protection Agency and the Corps of Engineers (COE).

The methods presented generally follow those given in "Evaluation of Dredged Material Proposed for Ocean Disposal-Testing Manual," compiled by the U.S. Environmental Protection Agency and U.S. Army Corps of Engineers (1991) and Regional Implementation Manual, Requirements and Procedures for Evaluation of the Ocean Disposal of Dredged Material in Southeastern Atlantic and Gulf Coastal Waters (USACESAD/EPA Region IV 1993).

2. DREDGED MATERIAL COLLECTION AND PRESERVATION

2.1 Sampling Stations

Sediment and water samples to be collected from designated sampling stations within the dredging area will be defined in advance by the COE District. Reference and control sediments will be collected at locations specified by the District or EPA Region.

2.2 Maximum Holding Time

Dredged material will be used within 14 days of collection.

2.3 Equipment List

- Noncontaminating (stainless steel) sediment grab or core sampler
- Noncontaminating water sampler
- Acid-rinsed and solvent-rinsed linear polyethylene or polypropylene bottles for water samples
- Acid-rinsed and solvent-rinsed glass bottles with Teflon-lined, screw-type lids for water samples

- Polypropylene buckets with lids for collection of dredged material samples
- Ice chests for preservation and shipping of dredged material

2.4 Dredged Material Sample Collection for Bioassays and Bioaccumulation Studies

1. Sediment samples will be taken with a corer or grab sampler at designated stations. The larger the proposed dredging site, generally the more samples will be required for characterization.
2. Samples will be placed in air-tight polypropylene containers and stored on ice at 4°C. All containers must be completely full and void of any air bubbles.
3. The samples must never be frozen or dried.

3. PREPARATION OF DREDGED MATERIALS FOR WHOLE-SEDIMENT BIOASSAYS AND BIOACCUMULATION STUDIES

For preparing sediment samples for whole sediment bioassay and bioaccumulation studies, the following procedures will be used.

1. Remove any live organisms by dry sieving the sediment through a 1.0-mm screen. All material retained on the sieve is discarded.
2. Combine and thoroughly mix the reference sediment samples. Combine and thoroughly mix the test sediment sample. Combine and thoroughly mix the control sediment sample.
3. Place sediment into test aquaria.
4. Sediment can be used immediately or stored (at 4°C in an air-tight container) until needed.

4. PHYSICAL AND CHEMICAL TEST CONDITIONS

4.1 Test Temperatures

Test temperatures must be held stable at 20 ± 1°C for the *N. virens* and *A. abdita* tests and 15 ± 1°C for *M. nasuta*.

4.2 Salinity

The salinity in all test vessels will be maintained at 30 ppt ±10% for the duration of the test.

4.3 pH

The pH of dilution water will be maintained at 8.0±0.2.

4.4 Dissolved Oxygen Concentrations

Dissolved oxygen must not fall below 4 mg/L.

4.5 Air Flow

Air flow should not exceed 100 bubbles of air per minute unless required to maintain dissolved oxygen at ≥4.0 mg/L. To avoid undue loss of volatile toxicants, diffuser stones will not be used in test aquaria. Instead, glass tubing (3-mm ID) will be used to deliver air to test tanks.

4.6 Recording Frequency

For whole sediment testing, temperature, salinity, dissolved oxygen, and pH in the test tanks will be measured and recorded on the day of test initiation and at 24-hour intervals throughout the test. Daily records will also be kept of obvious organism mortalities, formation of tubes or burrows, and unusual behavior patterns. Measurements will be made before and after solutions are renewed.

4.7 Light Duration

Lighting will be automatically regulated to provide 16 hours of light and 8 hours of dark every 24 hours.

5. TEST ORGANISMS

5.1 Organism Handling and Acclimation

Organisms for testing will be obtained from a biological supplier. All organisms to be used in toxicity testing will be gradually acclimated to testing conditions. Selection of test species is dependent on test type and purpose.

5.2 Holding

Organisms obtained from biological suppliers usually will be held in the laboratory at least 24 hours before use in testing. This will allow acclimation to the test temperature and artificial seawater and provide an opportunity to assess the general health of the test populations.

5.3 Feeding

For the solid phase testing, organisms will be fed on a daily basis at a rate of approximately one percent of the total weight of all animals in each tank. Tetramin flake food or conditioning food is adequate for *N. virens* and *M. nastuta*.

5.4 Size

Every attempt will be made to test animals of approximately equal size.

5.5 Number

Twenty organisms of each species are used in each test chamber for whole sediment bioassays.

5.6 Organism Transfer

Organisms will be randomly assigned to the test chambers.

6. REFERENCE TOXICANT TESTING

Reference (standard) toxicant tests will be performed on each acquired lot of organisms used in the solid phase testing to evaluate the sensitivity and general health of the organisms. Sodium dodecyl sulfate will be used as the toxicant. Modifications to the reference toxicant testing methods can be implemented to comply with the specific requirements set forth by the COE and the EPA.

7. CONDUCTING WHOLE-SEDIMENT BIOASSAYS

7.1 Equipment and Materials

- 110-volt circulating pump
- 10-gal aquarium test tanks
- 20-gal aquarium holding tanks
- 350-gal saltwater mixing tanks
- Aquarium filters for holding tanks
- Organism loading trays

- Walk-in test chamber
- Air blower and air lines
- Aeration pipets
- 5-gal carboys
- glass renewal plates
- 5-gal buckets
- Synthetic sea salt mixture
- Calibrated thermometer
- Salinity/conductivity meter
- 50-gal. saltwater holding barrel
- Tygon tubing
- Sediment scoops
- Meter stick
- Sieve screens
- Dechlorinated water
- Refrigeration
- pH meter
- Dissolved oxygen meter

7.2 Test Organisms

1. Whole-sediment 10-day bioassays will be conducted with appropriately sensitive benthic marine organisms. RIM Guidelines recommend use of *Nereis virens* and *Ampelisca abdita*.

2. Each test tank will contain a minimum of 20 organisms of each species.

7.3 Glassware Cleaning

Glassware to be used in the tests will be cleaned according to the general toxicology laboratory procedures for cleaning test containers and equipment, as recommended by U.S. EPA.

7.4 Test Procedures

Each whole-sediment bioassay will consist of five replicates (e.g., 10 gallon aquaria) for each test sediment, reference sediment, and control sediment.

1. Add enough sediment to provide an even layer of approximately 50-mm on the bottom of each aquaria.

2. Add 10 L of artificial seawater to each aquaria.

3. Let sediment settle overnight.

4. Place organisms into sorting trays and randomly distribute to all of the test tanks, until 20 organisms are placed in each tank.

5. Renew solutions in each test tank daily. This is accomplished by siphoning solution from the tank to a level approximately 2 cm above the sediment surface. Artificial seawater is slowly introduced to each tank so as not to disturb the sediment surface unnecessarily. Pouring water alongside a glass plate fitted to the tank dimensions reduces turbulence in the tank.

7.5 Observations

The following observations will be made and recorded initially and at 24-hour intervals on solutions before and after renewal:

- Salinity
- Temperature
- Dissolved oxygen
- pH
- Observations of obvious mortalities and unusual behavior patterns.

7.6 Test Duration

Whole sediment bioassays will be conducted for 10 days.

7.7 Organism Recovery

At the end of the testing period, organisms will be recovered in the following manner:

1. Siphon solutions from test tanks.

2. Remove large organisms (clams, polychaetes) by gently hand-combing sediment. Smaller organisms should be recovered by sieving sediments through a screen of appropriate mesh size.

    a. Consider organisms alive if they show any response to gentle probing.

    b. Sublethal effects such as partial paralysis, inability to burrow, or inability to excavate burrows will be recorded.

    c. Specimens not recovered must be considered dead.

d. Count and record living organisms at the end of the test.

7.8 Data Recording

Each set of data recorded must be initialed by the person making that entry. The following items will be recorded on data sheet for whole-sediment bioassays.

- Date
- Test organism
- Treatment—reference, control or test site
- Replicate number
- Time of day
- Temperature
- Salinity
- Dissolved oxygen
- pH
- Number of any dead test organisms observed daily
- Species lot number
- QC test number

7.9 Reference Toxicant Testing

Reference toxicant tests will be performed on acquired lots of organisms used in the whole sediment bioassays to evaluate the sensitivity and general health of the organisms. Modifications to the reference toxicant testing methods can be implemented to comply with the specific requirements set forth by the COE and EPA.

7.10 Data Presentation

The data will be summarized in tables which includes the scientific names of the test species, the number of animal tested, and the percentage of animals recovered alive from each test chamber.

7.11 Statistical Analysis

The hypothesis will be tested that responses observed for organisms exposed to test sediments are not statistically different from the responses observed for organisms exposed to reference sediments. Specific methods for calculating significant differences are given in the 1991 EPA/COE Testing Manual referenced above in Section 1.0.

8. CONDUCTING WHOLE-SEDIMENT BIOACCUMULATION STUDIES

8.1 Equipment and Materials

Refer to Section 7.1 (Equipment and Materials).

8.2 Test Organisms

Consideration must be given to the size and number of test organisms tested. There must be enough tissue in the resulting sample for the number and kinds of chemical analyses to be performed. Relatively immobile species that are fairly large, such as bivalves or large polychaetes, usually are the most desirable organisms for bioaccumulation testing.

8.3 Glassware Cleaning

Glassware to be used in the tests will be cleaned according to the general toxicology laboratory procedures for cleaning test containers and equipment, as recommended by U.S. EPA.

8.4 Test Procedure

Five replicates of test sediment, control sediment, and reference sediment will be tested. The test tanks are prepared as described for whole sediment bioassays (Section 7.4), including the addition of the organisms to the test tanks.

8.5 Observations

The following observations will be made and recorded initially and at 24-hour intervals before and after solution renewals.

- Salinity
- Temperature
- Dissolved oxygen
- pH
- Observations of obvious mortalities and unusual behavior patterns.

8.6 Duration

Organisms will be tested for either 10 or 28 days, depending on the classes of chemicals (i.e., metals, organics) that must be analyzed in tissue samples.

8.7 Organism Recovery

1. Transfer organisms to clean salt water-holding tanks with no sediment to purge their digestive tracts at least 24-hours.

2. Do not feed the organisms.

3. Siphon the fecal material at least once during the depuration period.

4. As observed, record, remove, and discard any organisms that die.

5. Organisms are now ready for preparation and analysis of tissues. Define analysis desired, preparation required, and proceed accordingly.

8.8 Reference Toxicant Testing

Reference toxicant tests will be performed on each acquired lot of organisms used in the whole-sediment bioaccumulation studies to evaluate the sensitivity and general health of the organisms. Modifications to the reference toxicant testing methods can be implemented to comply with the specific requirements set forth by the COE and EPA.

8.9 Data Presentation

Data will be summarized in tables that include concentrations of chemicals analyzed in tissues of test organisms in each reference and test replicate.

8.10 Statistical Analysis

The hypothesis will be tested that chemical concentrations analyzed in organisms exposed to test sediments are not statistically different from chemical concentrations analyzed in organisms exposed to reference sediments. Specific statistical procedures are given in the 1991 EPA/COE Testing Manual referenced in Section 1.0.

9. QUALITY ASSURANCE

The following is a brief synopsis of the Quality Assurance Program followed by EA's Aquatic Toxicology Laboratory. A more detailed overview of EA's Quality Assurance Program can be

found in the Aquatic Toxicological Studies Quality Control and Standard Operating Procedures Manual (SOP's) (EA 1996).

9.1 Quality Assurance Program Applicability

Amendments to the authorized protocol established by EA or by the client will only be made after proper authorization. Such authorization is achieved by completion of the Amendment to Protocol Form by EA after consultation with the client.

9.2 Reference Toxicant

A reference toxicant test, utilizing sodium dodecyl sulfate (SDS), cadmium chloride, or other appropriate chemical is used as an internal quality check of the sensitivity of the test organisms. Testing is conducted at least once monthly on organisms which are cultured in-house and on each population of organisms purchased for testing from an outside supplier if reference toxicant data is not available on the acquired lot. The results of each test are compared with historical, species-specific toxicological information from reference toxicant tests performed at EA, to determine if the results are within acceptable limits. Limits are established using the control charts outlined in US EPA (1993).

9.3 Quality Assurance Evaluation

Studies conducted under this protocol are subject to internal audit by EA's Quality Assurance Unit. A quality assurance officer is responsible for monitoring each study to assure the client that the facilities, equipment, personnel, methods, practices, records, and controls are in conformance with EA's QA program.

9.4 Inspection By Regulatory Authorities

In the event of an inspection of EA by an outside authority during the course of the study, the client will be consulted before inspectors are permitted access to any of the project records or the experimental areas.

9.5 Archives

Copies of project-specific records shall be transferred to the client promptly after the project is completed or as negotiated and budgeted with each client. Original primary data will be retained at EA for 5 years. Primary data include laboratory data sheets, records, memoranda, notes, photographs, microfilm, and computer printouts that are a result of the original

observations and activities of the study and which are necessary for the reconstruction and evaluation of the report of the study.

9.6 Location

This study will be conducted by the Ecotoxicology Laboratory, EA Engineering, Science, and Technology at the Loveton Office, Sparks, Maryland.

COMPREHENSIVE QUALITY ASSURANCE PLAN

for

BROOKS RAND, LTD. (BRL)
3950 6th Ave. N.W.
Seattle, WA 98107
(206) 632-6206

CRAIG

Date: April 1, 1998

Richard J. Brooks - BRL President	4/1/98 Date
R. Paul Swift - Project Manager	04/01/98 Date
Colin Davies - QA Manager	4/1/98 Date

2.0 TABLE OF CONTENTS

QA Plan Elements

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3.0 Statement of Policy

The Brooks Rand, Ltd. (BRL) Analytical Services Division is committed to sound and useful Quality Assurance/Quality Control management practices resulting in the production of quality analytical data for environmental samples. The principal focus of the analytical laboratory is to provide specialized analytical services for trace metals in environmental samples with an emphasis on ultra-low detection limits, metals speciation and unusual or non-routine matrices.

We recognize that the achievement of obtaining quality data depends on an effective and consistent quality control program. Our quality control program is implemented through a team effort across the entire laboratory group, from management to laboratory analysts. A listing of the general considerations and objectives of the overall program are as follows:

- *Sample integrity must be preserved.* Integrity is preserved by following documented and accepted sample handling procedures for the preservation, custody, storage, labeling, and record keeping associated with samples received by the laboratory.

- *Approved analytical methods must be followed.* Analytical methods and related procedures are readily available. These are read and followed by all analysts. The results from quality control tests and from sample analyses are regularly evaluated to identify method weaknesses and/or to detect a need for further analyst training.

- *The analytical instrumentation must be in proper working order.* Determination of instrument performance through the use of calibration and other performance evaluation samples, and through preventive maintenance, is documented on a real-time basis. Calibration of the instruments is performed as part of the analytical procedure.

- *Raw data must be properly reduced and accurately transcribed to the proper reporting format.* Various levels of data review from acquisition to the final report are incorporated in order to reduce possibilities of error.

- *The laboratory-specific accuracy (precision and bias) of analytical methods must be documented and monitored on a continuing basis.* Data from analyses are monitored using control charts to assess continuing performance and to detect trends.

All of the above considerations are documented to determine the quality of the data. Subsequent sections of this manual will detail the various elements of the quality assurance (QA) program developed and practiced by the laboratory.

4.0 Organization and Responsibility

The success of the Quality Assurance/Quality Control management practices is achieved through the efforts of the entire staff at BRL.

4.1 Capabilities of Organization

BRL is a chemical analytical laboratory primarily providing analytical services for trace metals in environmental samples. BRL specializes in providing the lowest detection limits commercially available, metals speciation, and analysis of unusual or non-routine matrices. Analytical capabilities include*:

aluminum (Al_{Total})
antimony (Sb_{Total})
arsenic (As_{Total}, As^{III}, As^{V}, $As_{organics}$)
beryllium (Be_{Total})
cadmium (Cd_{Total})
chromium (Cr_{Total}, Cr^{III}, Cr^{VI})
cobalt (Co_{Total})
copper (Cu_{Total})
iron (Fe_{Total}, Fe^{II}, Fe^{III})
lead (Pb_{Total})
manganese (Mn_{Total}, Mn^{II}, Mn^{IV})
mercury (Hg_{Total}, CH_3Hg^+, HgS, Hg^{II}, Hg^0)
nickel (Ni_{Total})
selenium (Se_{Total}, Se^{IV}, Se^{VI})
silver (Ag_{Total})
thallium (Tl_{Total})
tin (Sn_{Total}, $Sn_{organics}$)
zinc (Zn_{Total})

All metals can also be analyzed for the total recoverable and dissolved fractions

Alkalinity
Conductivity
Cyanide (total, amenable)
Hardness (total , temporary)
Lab pH
Solids - Total
- Suspended
- Dissolved

*Florida State Dept. of Environmental Protection approves only those methods listed in section 5.

4.2 Duties and Responsibilities of Personnel

The laboratory staff are organized in such a way that all analytical personnel are trained in a variety of laboratory duties. While each person is familiar with many aspects of laboratory work, individuals are specialized in their area of primary responsibility. The laboratory staff, supporting personnel and their responsibilities are listed in Table 4.2. The QA Officer of BRL functions in other positions, but his role as QA Officer is separate and distinct from all other responsibilities for any specific project.

4.3 Organizational Chart

An organizational chart showing lines of responsibility is shown as Figure 4.3.

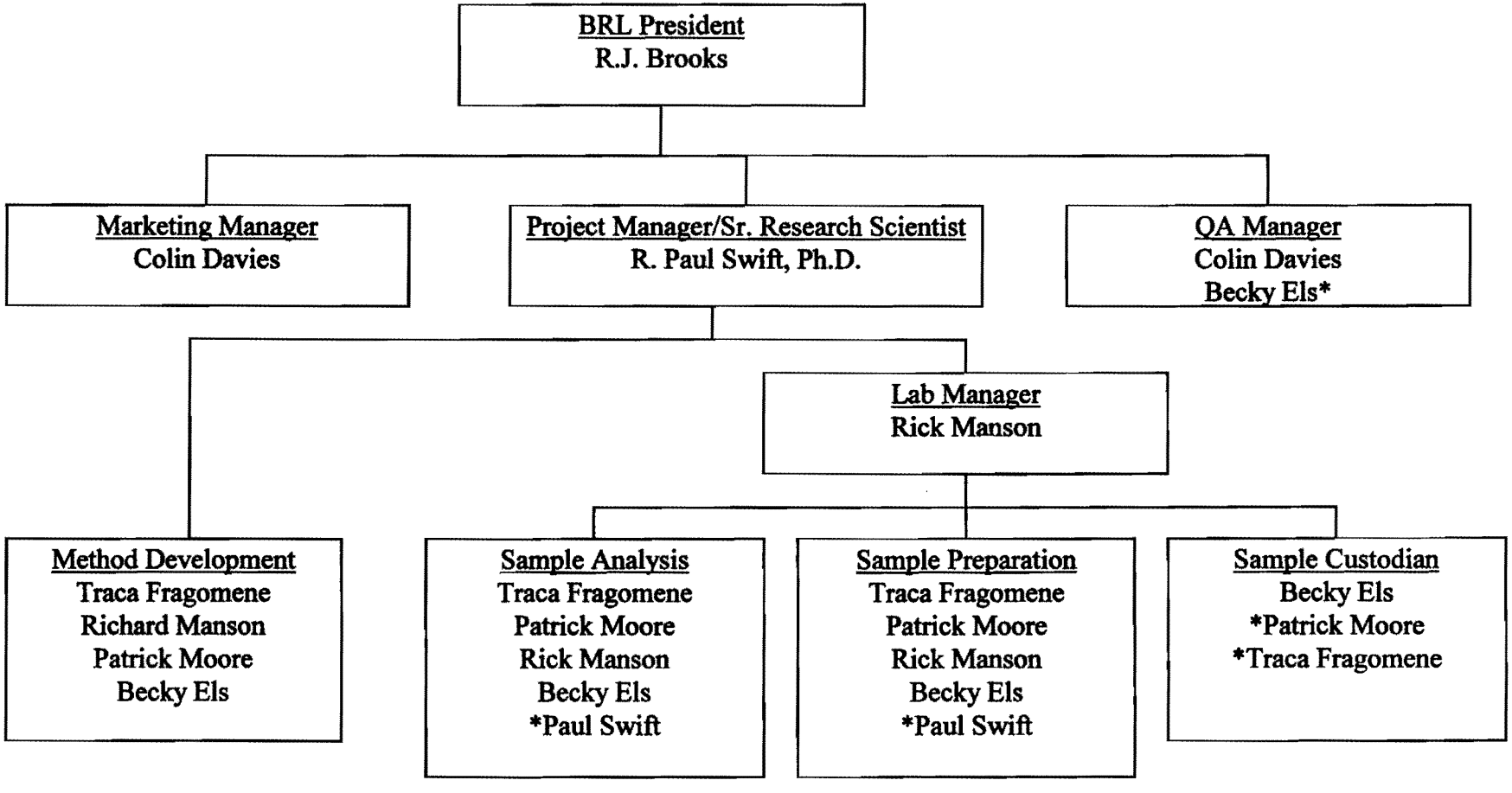
Table 4.2
MAIN RESPONSIBILITIES OF BRL LAB STAFF

Title	Responsibilities	Name
Senior Research Scientist	Method Development	R. Paul Swift, Ph.D.
	Method Improvement and Troubleshooting	
QA Management	Compile and Submit QA Plans	Colin Davies
	Data Review	Becky Els*
	Control Charts	
	Internal Laboratory Audits	
	Training of Personnel	
	Document Control	
	Lab Accreditations	
Project Management	Manage Client Projects	R. Paul Swift, Ph.D.
	Ensure client requirements and expectations are met	
Lab Manager	Sample Analysis Scheduling	Rick Manson
	General Management of Lab and Employees	
	Sample Tracking	
Analytical Technicians	Trace Metals Analysis	Traca Fragomene
	Sample Preparation	Patrick Moore
	Quality Control Analysis	Rick Manson
	Instrument Calibration and Documentation	Becky Els
	Reagent and Standard Prep. and Documentation	Paul Swift*
Sample Custodian	Sample Receipt	Becky Els
	Sample Disposal	Patrick Moore*
	Sample Storage	Traca Fragomene*
	Bottle Decontamination	
	Bottle Shipping	
Marketing	Proposal Coordination	Colin Davies
	Contract Review	
	Manage all Marketing Efforts	
BRL Consultants to Lab Division		
	Chemistry	Craig Brown, Ph.D. R. J. Brooks, M.S.
	Lab and Project Management	Colin Davies

*Alternate

Figure 4.3

ANALYTICAL SERVICES ORGANIZATIONAL CHART



*Denotes Alternate

5.0 Quality Assurance Objectives

5.1 Sample Preparation Methods

Table 5.1
Sample Preparation Methods

Sample Prep. Method Number	Description	Matrix	For these Analytical Methods:
EPA 200.2	HNO_3, HCl	SA/DW/SW/GW/SED/EFF	EPA 200.9, 220.1, 236.1, 239.1, 249.1, 289.1
EPA 200.3	HNO_3, H_2O_2	BIO	EPA 200.9, 220.1, 236.1, 239.1, 249.1, 289.1

BRL primarily utilizes the preparation methods listed above; other sample preparation methods may be used upon request for specific enforcement or compliance-based contracts. The Sample preparations listed above may also be used for analytical methods EPA 220.1, 236.1, 239.1, 249.1 and 289.1 for research applications only.

5.2 Analysis Methods

All analytical method capabilities of BRL are listed in Table 5.2 along with the QA objectives for each analyte and method. Acronyms used in Table 5.2 are as follows:

- EPA - refers to methods found in any EPA accepted source
- SM - Standard Methods
- NA - Not Applicable
- MDL - Method Detection Limit
- PQL - Practical Quantitation Limit
- MOD - Modified Method
- DW - Drinking Water
- SW - Surface Water
- GW - Ground Water
- SED - Sediments (includes domestic sludges)
- S - Solid
- EFF - Effluent
- BIO - Sample from biological matrices (tissues, muscle, shellfish etc.)
- SA - Saline Waters
- W - Water including surface water, groundwater and wastewater
- BR - Brooks Rand Procedure
- ALL - All matrices including DW, SW, GW, SED, S, EFF, BIO, SA

Table 5.2
Quality Assurance Objectives for Analytical Methods

Method No.	Matrix	Analyte	Precision % RPD	Conc.* Range	Accuracy %	Water MDL in µg/L	Solids MDL in µg/g
EPA 200.9	SA/DW/SW/GW/EFF	Ag	20%[2]	M	80-120%[2]	0.085	-
EPA 200.9	SED/S/BIO	Ag	35%[2]	M	65-135%[2]	-	0.05[2]
EPA 200.9	SA/DW/SW/GW/EFF	Al	20%[2]	M	80-120%[2]	7.8[2]	-
EPA 200.9	SED/S/BIO	Al	35%[2]	M	65-135%[2]	-	0.78[2]
EPA 200.9	SA/DW/SW/GW/EFF	As	20%[2]	M	80-120%[2]	0.5[2]	-
EPA 200.9	SED/S/BIO	As	22%	M	65-135%[2]	-	0.05[2]
EPA 1632	SA/DW/SW/GW	As, Inorganic	20%[2]	M	55-146%[2]	0.002[2]	-
EPA 200.9	SA/DW/SW/GW/EFF	Be	20%[2]	M	80-120%[2]	0.02[2]	-
EPA 200.9	SED/S/BIO	Be	35%[2]	M	65-135%[2]	-	0.002[2]
EPA 1637	SA/DW/SW/GW/EFF	Cd	20%[2]	M	70-116%[2]	0.0074[2]	-
EPA 1639[3]	SA/DW/SW/GW/EFF	Cd	20%[2]	M	64-145%[2]	0.023[2]	-
EPA 200.9	SA/DW/SW/GW/EFF	Cd	20%[2]	M	90-120%	0.05	-
EPA 200.9	SED/S/BIO	Cd	35%[2]	M	65-135%[2]	-	0.005
EPA 200.9	SA/DW/SW/GW/EFF	Co	20%[2]	M	80-120%[2]	0.7[2]	-
EPA 200.9	SED/S/BIO	Co	35%[2]	M	65-135%[2]	-	0.07[2]
EPA 200.9	SA/DW/SW/GW/EFF	Cr	20%[2]	M	80-120%[2]	0.1[2]	-
EPA 200.9	SED/S/BIO	Cr	35%[2]	M	65-135%[2]	-	0.01[2]
EPA 1639[3]	SA/DW/SW/GW/EFF	Cr^{III}	20%[2]	M	74-131%[2]	0.1[2]	-
EPA 218.5	SA/DW/SW/GW/EFF	Cr^{VI}	20%[2]	M	80-120[2]	2.3[2]	
EPA 220.1	SA/DW/SW/GW/EFF	Cu	20%[2]	M	80-120%[2]	100[2]	-
EPA 220.1	SED/S/BIO	Cu	35%[2]	M	65-135%[2]	-	10[2]
EPA 200.9	SA/DW/SW/GW/EFF	Cu	20%[2]	M	80-120%[2]	0.7[2]	-
EPA 200.9	SED/S/BIO	Cu	35%[2]	M	65-135%[2]	-	0.07[2]
EPA 236.1	SA/DW/SW/GW/EFF	Fe	20%[2]	M	80-120%[2]	120[2]	-
EPA 236.1	SED/S/BIO	Fe	35%[2]	M	65-135%[2]	-	12[2]
EPA 200.9	SA/DW/SW/GW/EFF	Fe	20%[2]	M	80-120%[2]	1[2]	-
EPA 200.9	SED/S/BIO	Fe	35%[2]	M	65-135%[2]	-	0.1[2]
EPA 245.1	SA/DW/SW/GW/EFF	Hg	11%	M	74-123%	0.2	-
EPA 245.5	SED/S	Hg	11%	M	65-135%[2]	-	0.2
EPA 1631 (MOD)	SA/DW/SW/GW/EFF	Hg	20%	M	80-115%	0.0001[1]	-
EPA 1631 (MOD)	SED/S/BIO	Hg	23%	M	83-121%	-	0.0001[1]
BR-0011	SA/DW/SW/GW/EFF	Hg, Monomethyl	24%	M	53-121%	0.00005[1]	-
BR-0011	SED/S	Hg, Monomethyl	23%	M	71-115%	-	0.0001[1]
BR-0011	BIO	Hg, Monomethyl	22%	M	73-117%	-	0.001[1]
EPA 200.9	SA/DW/SW/GW/EFF	Mn	20%[2]	M	80-120%[2]	0.3[2]	-
EPA 200.9	SED/S/BIO	Mn	35%[2]	M	65-135%[2]	-	0.03[2]
EPA 1639[3]	SA/DW/SW/GW/EFF	Ni	20%[2]	M	65-145%[2]	0.65[2]	-
EPA 249.1	SA/DW/SW/GW/EFF	Ni	20%[2]	M	80-120%[2]	40	-
EPA 249.1	SED/S/BIO	Ni	35%[2]	M	65-135%[2]	-	4
EPA 200.9	SA/DW/SW/GW/EFF	Ni	20%[2]	M	80-120%[2]	0.6	-
EPA 200.9	SED/S/BIO	Ni	35%[2]	M	65-135%[2]	-	0.06
EPA 1637	SA/DW/SW/GW/EFF	Pb	20%[2]	M	60-120%[2]	0.031	-
EPA 239.1	SA/DW/SW/GW/EFF	Pb	20%[2]	M	80-120%[2]	100[2]	-
EPA 239.1	SED/S/BIO	Pb	35%[2]	M	65-135%[2]	-	10[2]

Table 5.2 (continued)
Quality Assurance Objectives for Analytical Methods

Method No.	Matrix	Analyte	Precision % RPD	Conc.* Range	Accuracy %	Water MDL in µg/L	Solids MDL in µg/g
EPA 200.9	SA/DW/SW/GW/EFF	Pb	22%	M	70-123%	0.6	-
EPA 200.9	SED/S/BIO	Pb	35%[2]	M	65-135%[2]	-	0.07[2]
EPA 1639[3]	SA/DW/SW/GW/EFF	Sb	20%[2]	M	18-150%[2]	1.9[2]	-
EPA 200.9	SA/DW/SW/GW/EFF	Sb	20%[2]	M	80-120%[2]	0.8	-
EPA 200.9	SED/S/BIO	Sb	35%[2]	M	65-135%[2]	-	0.08
EPA 1639[3]	SA/DW/SW/GW/EFF	Se	20%[2]	M	56-131%[2]	0.83[2]	-
EPA 200.9	SA/DW/SW/GW/EFF	Se	20%[2]	M	80-120%	0.6	-
EPA 200.9	SED/S/BIO	Se	35%[2]	M	80-115%	-	0.13
EPA 200.9	SA/DW/SW/GW/EFF	Sn	20%[2]	M	80-120%[2]	1.7[2]	-
EPA 200.9	SED/S/BIO	Sn	35%[2]	M	65-135%[2]	-	0.17[2]
EPA 200.9	SA/DW/SW/GW/EFF	Tl	20%[2]	M	80-120%[2]	0.7[2]	-
EPA 200.9	SED/S/BIO	Tl	35%[2]	M	65-135%[2]	-	0.07[2]
EPA 1639[3]	SA/DW/SW/GW/EFF	Zn	20%[2]	M	67-142%[2]	0.14[2]	-
EPA 289.1	SA/DW/SW/GW/EFF	Zn	20%[2]	M	80-120%[2]	5	-
EPA 289.1	SED/S/BIO	Zn	35%[2]	M	65-135%[2]	-	0.5
EPA 200.9	SA/DW/SW/GW/EFF	Zn	20%[2]	M	80-120%[2]	0.3	-
EPA 200.9	SED/S/BIO	Zn	35%[2]	M	65-135%[2]	-	0.03
EPA 310.2	SA/DW/SW/GW/EFF	Alkalinity	14%[2]	M	90-100%[2]	2 mg/L[2]	-
EPA 120.1	SA/DW/SW/GW/EFF	Conductivity	13%[2]	M	85-105%[2]	1 µmho/cm[2]	-
EPA 130.2	DW/SW/SA	Hardness	13%[2]	M	90-100%[2]	2 mg/L[2]	-
EPA 150.1	DW/SW/SA/EFF	Lab pH	5%[2]	M	96-102%[2]	0.1 pH unit[2]	-
SM 2540 D	DW/SW/SA/GW/EFF	T.S.S.	12%	M	90-100%	1 mg/L	-
SM 2540 B	S/SED/BIO	% Solids	5%	M	95-105%	-	0.01 %

* Concentration range is for both precision and accuracy.

[1] MDLs have been calculated based on the 40 CFR 136, app. b protocol of determining method detection limits unless noted otherwise.

[2] Values are published method values due to insufficient data for in-house criteria determination. In-House targets will be provided when available.

[3] This method is functionally the same analytically as EPA Method 200.9, but method 1639 is specific for ambient water and includes protocols for trace level sample handling.

All values for in-house determined precision and accuracy criteria are control limits set at 2x standard deviation.

For all GFAA and FLAA methods:

1) Precision criteria are only valid if the sample result is > 5 times the MDL (or CRDL).
2) If sample result is < 5 times the MDL, precision criteria for waters is that the duplicate precision must be < than the value of the MDL, and for solids the duplicate precision must be less than 2 times the MDL
3) Precision criteria for duplicate injections are ± 20% for all matrices
4) Accuracy criteria are only valid if the sample result is less than 4 times the spike added.

6.0 Sampling Procedures

6.1 Sampling Capabilities

BRL does not normally conduct field sampling; therefore, the following Sampling Procedure topics are not addressed: Sampling Equipment, Sampling Protocols, Field Sample Documentation, Sample Dispatch, Field Reagent and Standard Storage, and Field Waste Disposal.

6.2 List of Equipment Provided for Sampling

Table 6.1
Equipment Provided for Sampling

Equipment	Construction	Use	Parameter Groups
Sample Container	Teflon - FEP, PFA	Sampling/Storage	Mercury in soil/water
	LDPE, HDPE, PPE	Sampling/Storage	Trace Metals, except Hg
Sample Coolers	Plastic	Sample Transport	all parameter groups
Gloves, Clean Room	Vinyl, non-powdered	Sampling	Trace Metals

BRL may include the preservative solution in the sample containers, in which case a proper volume of the appropriate preservative will be added using a pipette and acids that have been pretested for the analytes of concern. Sample containers to be shipped to clients (samplers) are all double bagged in ziploc bags and packaged in a clean cooler.

6.3 Decontamination Procedures

All metals except Hg

BRL only supplies sample containers for mercury analyses and iron speciation. For all other parameters, it is the responsibility of the field sampling organization to provide their own sample containers.

Cooler/shipping containers

All coolers must be washed with DI water and alconox , triple rinsed with DI water, and allowed to air dry prior to use for storing and/or shipping samples or sample containers. Appropriate sample containers are individually bagged and placed in the coolers to make a sampling kit. Sampling kits are sent via freight carrier (UPS, Airborne, FedEx) to the sampling team.

For mercury

Due to the historical problems of false positive results from mercury contamination, it is extremely important that all water samples are collected in rigorously acid-cleaned Teflon® containers. The method for decontamination of Teflon® bottles follows:

1. DESCRIPTION
   A. Definition: Removal of any trace element contaminate in sample vessels to ultra-trace levels.
   B. Scope: One of the most important tasks in the laboratory is bottle decontamination. The bottles are used for a variety of tasks, including sample collection, storage of samples for laboratory analysis, and preparation and storage of reagents. This protocol will outline the bottle cleaning process, and point out the basic precautions to ensure bottle cleanliness to ultra-trace levels.
   C. Summary: Once identified as requiring cleaning (SOP BR-0303), the sample container is emptied of its contents, rinsed and cleaned, rerinsed, and acidified for storage until use.

2. EQUIPMENT AND MATERIALS
   Concentrated Nitric Acid, 70-71%
   Concentrated HCl, 35-37%
   Teflon® cylindrical vats (for Nitric), and rectangular polypropylene vats (for HCl).
   Safety Gear (See section 3C "Safety Gear" for safety equipment)

3. PROCEDURE
   A. Preparation of dirty bottles

   After sample disposal is authorized (See BR-0303), the contents of the sample container can be disposed and then the sample container can begin the decontamination process.

Safety Note: Several of the reagents used in the mercury analysis are hazardous, and produce fumes which may be harmful, causing such immediate health problems as headaches and sore throats. One of these reagents, bromine monochloride, can be identified by its yellow color and distinctive odor and it must be reduced with hydroxylamine hydrochloride before further disposal. Sample disposal procedures are located in BR-0303 and must be followed. Goggles and gloves should be worn at all times.

After the sample has been disposed, rinse out the vessel and cap in order to remove any chemical residue. Sample vials must be washed with a brush and soap and water to remove any residue. Water in sample bottles will bead-up if there is no residue; when rinsing sample bottles, if the water does not bead-up, the bottle(s) must be washed with a brush and soap and water. The vessel(s) can then be placed in a dirty bottle bin.

B. The acid vats:

Of all the steps involved in the decontamination of vessels, the most potentially dangerous are those where the vessels are placed in and removed from the acid vats. Although serious injury could possibly occur, the probability of injury can be greatly reduced by following common sense precautions as discussed below. There are three Teflon® vats and two polypropylene vat which are currently used for bottle washing. All acid vats are kept in the fume hoods inside the decontamination room. The Teflon® vats contain 8N nitric acid (HNO_3), the large rectangular vat contains 6N HCl and the small rectangular vat contains 1.2N HCl.

Safety Note: In case of skin contact, wash the exposed area with copious amounts of water. Sodium bicarbonate ($NaHCO_3$) is kept near the acid room for neutralizing any acid which may contact the skin. Soda ash is also be kept nearby to neutralize any acid spills.

The vats are the most dangerous when the acid is hot. The chance of a severe burn is greatly increased, and the gloves cannot withstand prolonged exposure to hot acid. Therefore, NEVER STICK YOUR HAND INTO A HOT ACID VAT!!! Always allow the vats to cool. The nitric vats and the 1.2N HCl vat usually take approximately 5-6 hours to cool. Due to the large size of the 6N HCl vat, it should be allowed to cool overnight. The cooling process can be speeded up by removing the lid and/or by removing the heating element from the vat. Removing the lid, however, will release a tremendous amount of acid fumes. Although these fumes are efficiently vented to the outside, removal of the hot acid vat lids should only be done when sample container needs are urgent.

When the acid vats are properly cooled, they may be emptied of clean sample containers and filled with dirty or new containers.

C. Safety Gear:

As with all lab work, a lab coat must be worn at all times. A safety shower is located next to the acid vat room. All personnel must be aware of the safety shower location and operation. The following minimum amount of gear must be worn at all times while working with the Nitric acid vats: "Silver Shield" chemical resistant gloves (as liners), "Silver Shield" chemical resistant sleeves, "Playtex" gloves as an outer layer glove, a green rubber apron, eye glasses or goggles, and a face shield.. Protective eye glasses or goggles are not a substitute for the face shield--both must be worn!

While working with the HCl vat, a lab coat, rubber apron, face shield and eye glasses or goggles, and "Viton" black extra-long gloves must be worn, to ensure safety and compliance with BRL safety regulations (see the BRL Chemical Hygiene Plan for further details). Due to the venting of fumes, a respirator mask is not required.

D. Filling the acid vats:

Only items which are made of Teflon® or glass can be cleaned in the vats. Teflon® bottles can be identified by the blue hue or by the identification stamp "FEP" on the bottom of the bottle. Any other materials such as polypropylene (marked with "PP") will degrade in the cleaning process. All vials used for sediments must be cleaned in the nitric acid vats. In addition, all bottles used for high level standards and samples that exceed the analyte limits for disposal down the drain set in SOP BR-0303 must be cleaned in the nitric vats.

Each cylindrical vat (nitric) should be filled approximately 2/3 to 3/4 full with acid. If the vats are filled any deeper there is a risk of acid running over into the user's gloves or onto the counter when bottles are placed into the vats. Each vat has the capacity to hold approximately 17-19 (125 mL) bottles, 8-10 (250 mL) bottles, or 3 (1L) bottles. All bottles and caps should be carefully placed and arranged in the vat so that acid covers the entire bottle inside and out.

All new Teflon® bottles must remain in a vat with heated hydrochloric acid for no less than 48 hours at a temperature of 70 ± 10° C. On the third day, the vat may be turned off, cooled and later emptied. All old or dirty bottles should be cleaned in a vat containing either nitric or hydrochloric acid for no less than 24 hours. If the bottles are extremely

dirty (i.e. stained or used for high level samples) they should be cleaned in nitric acid for 24 to 48 hours. Bottles that contained water samples higher than 200ng/L but below the disposal limit of 200µg/L should be heated for 48 hours in the 50% hydrochloric acid vat, unless the sample custodian or lab manager determines that extended cleaning in nitric acid is more appropriate.

If time permits, it is best to load sample containers into the vats and allow them to heat up overnight. Vats should be left on the entire next day and turned off just prior to leaving work. Vats are then allowed to cool overnight, and can be emptied and reloaded the following day. See SOP BR-0303 for further requirements for cleaning based on sample analyte concentrations.

Caution: Watch for acidic condensation on the lid of the vat and drippings on the floor. These can be cleaned up with rags.

Note: All **new** bottles, tubing, or other Teflon® materials must be cleaned for 48 hours--bottles in the HCl vat, and tubing, vials and other Teflon® items in the nitric vats.

E. Emptying the acid vats:

After the vats have been heated for the appropriate length of time, they should be turned off and allowed to cool as stated above. For nitric vats, after turning off the power, remove the heating pads from the vats. After allowing an appropriate amount of time for cooling, place the rectangular rinsing vat (containing clean DI H_2O) into the fume hood next to the vat to be emptied. Rinse the nitric vat lids in the rinsing vat to remove any condensed nitric acid. The water in the rinsing vat should changed at least every week to ensure cleanliness (to avoid contamination) and to ensure that the water does not get too acidic.

Safety Note: As always, wear the proper safety gear when working with the acid vats!!!

To retrieve a vessel from the vat, stick your gloved hand into the acid and carefully remove it. Be careful not to splash the acid while removing the vessel because it will ruin equipment and damage clothing. Pour the acid from inside the vessel back into the vat, and place the empty bottle into the rinsing vat. When removing items from the vat be careful to prevent acid from spilling over into your glove. Bottle lids can be cleaned by placing them in a glass beaker inside the vat. This makes removal of the lids both safer and faster. The beaker can then be removed and the acid carefully drained back into the acid vat. Repeat this procedure for each vat and for both types of acids, nitric or hydrochloric. Once the vats are empty they may now be filled again with dirty or new vessels. If the vats are not to be used they should be left in the fume hoods in the acid room with lids on and the heat off.

F. Rinsing the bottles:

Remove the bottles from the rectangular rinsing vat and wash them under the de-ionized water faucet. Rinse them three times inside and out, filling the vessel and allowing it to overflow the rim each time. After rinsing the vessel the third time, fill the vessel with water up to the inside rim just below the threads on the vessel neck. Place the filled vessel in the clean air hood. Also, rinse the lids to each bottle and place them in the clean air hood to dry.

Precautionary Note: Always wear vinyl clean-room lab gloves when rinsing the bottles for two reasons: 1) the rinsing vat, which contains water, will become acidic after repeated

use and will cause irritation to the skin, and 2) clean-room gloves will prevent contamination of the bottles.

G. Storing the clean bottles:

All bottles should be acidified by adding 1.0 mL of hydrochloric acid per 125 mL. Bottles cleaned in nitric acid should be acidified with HCl that contains 0.8% hydoxylamine HCl. These (capped) bottles should then be placed into the drying oven over night at a temperature of about 55° C. The next day the oven should be turned off and the bottles removed. Bottles should then be triple rinsed again, and acidified with 0.5mL HCl per 125mL. At this stage, all bottles receive the same HCl for acidification. After they have dried in the clean air hood, containers should be bagged by size with the Julian date preceded by the last two digits of the year (i.e. 94-236) marked with permanent ink on the bag.

4. Quality Assurance

A. Bottle blanks:

Two bottles from every cleaning batch are analyzed for Hg contamination and recorded in the bottle blank logbook. Before analyzing bottle blanks, each cleaning batch is quarantined in the storage bins and no bottles are put into regular service. Bottles must be under 0.2ng/L for the cleaning batch to be put into service. (Teflon® vials and jars, as well as any glassware, are generally put into service without analyzing bottle blanks.) Bottles are then stored in the bottle storage bins and vials are stored in the upstairs clean lab.

B. Acid vat testing:

As an added safeguard to ensure that bottles will be decontaminated sufficiently, all acid vats are tested for mercury once a month. For gathering acid vat samples for testing, 5 mL from each vat should be pipetted into sample bottles which are then diluted to 100 mL. These samples are then logged in under project # BRL003. If acid vat concentrations exceed 100 µg/L then the acid from that vat should be removed into a neutralizing vessel, the acid neutralized and then disposed of according to sample disposal guidelines set in SOP BR-0303.

Nitric vats also should have 25 mL of BrCl added to them once per month to ensure complete oxidation of methylmercury. It has been demonstrated that methylmercury does not completely oxidize in hot nitric acid, and therefore this step ensures that containers will not be contaminated for methylmercury.

6.4 Sample Preservation, Holding Times, Container Types, Required Sample Volumes

All preservation reagents used by BRL are analytical grade or better. For mercury, samples should be sent to BRL on ice via overnight delivery where they will be preserved with pretested ultratrace HCl. For all other parameters, the field sampling crew can add the appropriate preservative (See Table 6.2) or should send samples overnight to be preserved at BRL. Sample bottles for mercury analysis are all documented for cleaning and sample bottles shipped to clients are tracked for each project. For projects where ultra-trace contaminants are not of concern, BRL recommends immediate field preservation using appropriate preservation reagents (sampling company is responsible for these preservation supplies).

Table 6.2
Summary of Sample Containers & Preservatives

Parameter	Min.* Vol.	Container	Means of Preservation	Holding Time
A. Tissues				
Total Hg, MMHg, As, Se	1 gram each	Teflon® or Glass with Teflon® lined lids	A) 4°C B) <-5°C	A) 28 days B) 1 year
B. Sediments/Soils				
Total Hg, MMHg, As, Se	5 grams each	Teflon® or Glass with Teflon® lined lids	A) 4°C B) <-5°C	A) 28 days B) 1 year
C. Water				
Total Hg, Hg(II), MMHg, As, Se	125 mL each	Teflon®	1mL HCl/125mL H_2O (pH<2)	28 days
Dimethyl Hg	300 mL	Teflon®	4°C, Dark	48 hours
Elemental Hg	300 mL	Teflon®	4°C, Dark	48 hours
D. Air[1]				
Total Hg	10 L	Gold trap	Store in Ziploc® Bag	6 months
MMHg	10 L	Carbotrap	1°C, Dark	7 days
Dimethyl Hg	10 L	Carbotrap	1°C, Dark	48 hours
Elemental Hg	10 L	Gold trap	Store in Ziploc® Bag	6 months
E. Seds./Soils/Tissues**				
Ag, Al, Be, Cd, Co, Cr, Cu, Fe, Mn, Ni, Pb, Sb, Sn, Tl, Zn	2 grams each	4 oz. CWM[2]	4°C, Dark	6 months
F. Water, Total				
Ag, Al, Ba, Be, Cd, Co, Cr, Cu, Fe, Mn, Ni, Pb, Sb, Sn, Tl, Zn	100 mL	250 mL HDPE[3]	pH < 2 HNO_3	6 months
G. Solids, Suspended				
Ag, Al, Ba, Be, Cd, Co, Cr, Cu, Fe, Mn, Ni, Pb, Sb, Sn, Tl, Zn	200 mL		Filter on site and refrigeration	6 months[4]

*Minimum volumes are to be used as guidelines only as they are dependent on the need for replicate analysis, the concentration of analytes in the sample and the project required detection or reporting limits.
**Holding times and handling may vary for speciation
[1] Air is not a matrix reviewed by Florida DEP QA section
[2] Clear Wide Mouth Glass
[3] High Density Polyethylene
[4] Samples should be filtered immediately on site before adding preservatives for dissolved metals

Table 6.2 (Continued)
Summary of Sample Containers & Preservatives

Parameter	Min.* Vol.	Container	Means of Preservation	Holding Time
H. Solids, Dissolved				
Ag, Al, Ba, Be, Cd, Co, Cr, Cu, Fe, Mn, Ni, Pb, Sb, Sn, Tl, Zn	200 mL	500 mL HDPE	Filter on site, pH < 2 HNO_3	6 months
I. Soil[5]				
Cr^{VI}	2 grams	4 oz. CWM	Cool 4° C	24 hours
J. Water[5]				
Cr^{VI}	200 mL	500 mL HDPE	Cool 4° C	24 hours
K. Water				
Alkalinity	100 mL	250 mL HDPE	Cool 4° C	14 days
L. Water				
Conductivity, specific	100 mL	250 mL HDPE	Cool 4° C	28 days
M. Water				
Hardness	100 mL	250 mL HDPE	pH<2 HNO_3/H_2SO_4	6 months
N. Water				
Lab pH, hydrogen ion	25 mL	60 mL HDPE	None Required	Analyze immed.
O. Water				
Total Suspended Solids (TSS)	100 mL	250 mL HDPE	Cool 4° C	7 Days
P. Water				
% Solids	100 mL	250 mL HDPE	Cool 4° C	7 Days

[5] Cr^{VI} not reviewed by Florida DEP QA section

7.0 Sample Custody

7.1 Field Custody/Considerations

For mercury work, formal custody requirements begin at BRL by documenting the shipment of cleaned sample (Teflon®) containers to the field. If needed, BRL provides Chain of Custody (COC) forms with the containers. See next page for a copy of the BRL COC. In addition, all sample container shipments are documented to track container information such as cleaning batch numbers, quantity of containers shipped and their date of shipment. Teflon® sample bottles are engraved with a unique bottle identification number, and the decontamination date is documented for all sample containers.

While BRL does not provide field services we do recommend that certain issues be taken into account during sampling. Special consideration should be given to the procurement, transportation, preservation, and storage of samples to be analyzed. These procedures are intended to ensure that any analyte originally present in the sample matrix has not degraded or been lost, and that contaminants that might interfere with the analysis have not been added. For mercury work, only rigorously acid-cleaned Teflon® containers may be used for any water samples. Depending on filtration requirements and analysis requested, water samples for mercury analysis may either be shipped on ice to Brooks Rand within 24 hours of collection and preserved upon receipt, or samples may be preserved in the field with preanalyzed HCl. Either preservation method for mercury is acceptable as stated in EPA Method 1631 (Section 8.5). See section 6.2 for container, preservation and holding time requirements. Tissues and other solid matrices may be stored in acid-cleaned glass containers with Teflon lined lids. Under no circumstances should polyethylene, polypropylene, or other non-approved plastics be used for ultra-trace mercury work. Solid samples are preserved by shipping on ice and storing in either a refrigerator or freezer. Water sample containers for ultra-trace mercury must be sealed inside two Ziploc® bags which are labeled by the customer/field sampling technician. See section 6.2 for container requirements for other analytes.

7.2 Laboratory Custody

7.2.1 Sample Receipt

All samples submitted to the laboratory are delivered to the laboratory's sample receiving area and are received by the sample custodian or designated alternate. Custody during transport of samples is documented by courier and can be obtained, if not provided upon delivery, from the carrier. Samples are generally sent via overnight carrier. COC requirements are fulfilled by having the sample custodian sign the receipt and the customer's COC form upon delivery.

BROOKS LAND, LTD.
3950 6th Ave. NW/ Seattle, WA, 98107 / phone (206) 632-6206 / fax (206) 632-6017

BRL Client:		Phone#:			Analysis Requested					TAT:
Client Contact:		PO#:								Deliverables (circle one)
Client Project #:		BRL Project #:								Standard / Full / Other
#	**Sample ID**	**Date**	**Time**	**Matrix**						**Comments**
1										
2										
3										
4										
5										
6										
7										
8										
9										
10										

Relinquished by: (signature)	Received by: (signature)	Relinquished by: (signature)	Received by: (signature)
Printed Name:	Printed Name:	Printed Name:	Printed Name:
Company:	Company:	Company:	Company:
Time/Date:	Time/Date:	Time/Date:	Time/Date:
Special Instructions/Comments:		Relinquished by: (signature)	Received by: (signature)
		Printed Name:	Printed Name:
		Company:	Company:
Carrier/Airbill #:		Time/Date:	Time/Date:

Please Fax COC to BRL on day of shipment & include in shipment

The sample custodian opens the cooler, which may or may not be sealed with a separate custody seal. Immediately after opening the cooler, the sample custodian checks whether ice is present and the condition of the samples (intact, broken, leaking, etc.) and notes any findings on the COC form. The sample custodian also verifies that each container is properly labeled and sealed, and compares the sample identification (ID) or field ID# against the COC form. If the sample ID and the COC do not match, or the seals on any of the containers are broken, the sample custodian notes the problem on the sample receiving log.

The Sample Custodian is also responsible for ensuring that all samples are properly preserved. Water samples for certain analytes do not require acidification and are stored in the refrigerator at 4°C. If water samples that do require acidification are preserved in the field, the Sample Custodian checks the pH and documents that it is less than 2. If it is not less than 2, the measured pH is documented on the sample receiving log and the client is notified. If samples were shipped overnight on ice and not preserved in the field (as acceptable per EPA Method 1631 for total Hg in water), the sample custodian shall preserve the samples to a pH of less than 2. The temperature of the samples at the time of receipt is estimated by inserting a non-mercury thermometer into the sample cooler, closing the cooler and allowing the temperature to equilibrate for approximately ten minutes. Any problems with the conditions of the samples is documented on the sample receiving log, and reported to both the lab manager and the client.

7.2.2 Sample Log In

Tag numbers, bottles numbers, sample matrix, bottle size and analysis requested should then be listed for each sample on the sample receiving log form. All information on sample bottles (or tags) should be checked against the Chain of Custody (COC) or other documentation provided by the client. If this information does not match or any other significant problems are observed (i.e. water samples received at room temperature), the client should be contacted immediately and the discrepancies resolved.

For each current project, the Lab Manager maintains an active file of information specific to the project on the BRL "Tracking" database. When logging in samples, the Sample Custodian must check the contract information against the samples received to ensure that the work has been authorized and to ensure that there are no discrepancies between the work contractually approved by the client's accounting department and the work requested by the client's sampling team. If any discrepancies are found, the Lab Manager should be immediately notified.

An additional sample log-in form (EPA form DC-1) must be filled out during receipt of EPA samples when required by the client. The Lab Manager will notify the Sample Custodian when this form is required. Each section is completed by either filling in the appropriate information where indicated, or circling the appropriate choice. If any of the items/descriptions marked with an "*" are circled the Sample Management Office (SMO) must be contacted and the discrepancy resolved. A record of resolution then must accompany this sample log-in sheet.

All samples are given a unique sample identification number at the time of receipt by the Sample Custodian. This number consists of a tracking number which is unique to each sample shipment received and a sample number for each sample within that particular shipment. Sample Tracking numbers consist of a 2-digit code followed by the letters "BR" followed by a 3-digit code. The first 2-digit code relates to the year in which the samples were received (for 1996 this code is "96"). The three digit code is a sequential numbering system starting with 001 for the

first sample shipment received in any given year. For example, the first sample shipment received in 1996 is given the tracking number 96BR001. The samples within a shipment are then each identified by a two digit sequential number. For example, if three samples were received in the 96BR001 shipment they would be given the sample numbers 96BR001-01, 96BR001-02 and 96BR001-03. On the sample receiving log the client's sample ID and the BRL ID numbers are listed for each sample. The in house BRL numbers (not the client's ID numbers) are referenced during all laboratory preparations and analyses.

Teflon sample bottles (for water samples) are all engraved with a bottle ID number which is used for sample identification purposes. Clients may wish to use additional sample identification numbers but the unique sample container numbers must be documented on the sample receiving log. When a bottle is removed, the number engraved on the bottle should be matched with the number written on the bag. Any discrepancies should be noted in the sample receiving log. Each bottle should be rinsed with clean DI water (for low level samples) and/or wiped with a clean cloth. Bottles are then placed in the clean hood, and labeled with the customer project number, the BRL sample number, the date of receipt and the analyses requested. An example of a BRL sample label is as follows:

Project ID: DER001
Sample ID: 96BR001-01
Date Recieved: 1/1/96
Analyses Requested: Hg, MMHg

Custody labels, sample seals and sample tags are not provided by BRL, and must be provided with the samples by the client if so desired.

If any filtration or volatile mercury analysis is required, this should be performed before preservation of samples. All samples must be preserved in accordance with the preservation instructions in each appropriate analytical methodology. For all water samples requiring acidification for preservation, the pH will be checked and documented. Any samples not originally adequately preserved by the client will have additional preservation reagent added and this addition will be documented. All preservation components should be documented including the lot number of any reagents used, the type of reagent and amount used and storage conditions and location.

After samples are all logged-in and stored, all sample information, including the COC and a copy of the BRL Sample Receiving Log, should be filed in the "Active Customer" file located in the Lab Manager's office. A copy of the BRL Sample Receiving Log is on page 6. In addition, an "Internal Custody for Original Samples" form is generated at the time of sample log-in (see page 7 of this section). This form documents the life cycle of each sample shipment from receipt to disposal, and is kept with the samples at all times. After samples have been disposed of and documented, the custody forms are stored in the Laboratory Manager's files.

Information for all current projects is kept in the "Active Customer" files and is sorted alphabetically by the project reference number. The Sample Custodian should check the customer summary page for instructions on faxing the COC and/or the BRL Sample Receiving Log. Sample Receiving Logs must be signed by the Sample Custodian (or designated alternate).

The original BRL Sample Receiving Log sheets should be kept in a three ring binder at the sample receiving desk. After Sample Receiving Log sheets accumulate up to 100 tracking

numbers, the Sample Custodian should bind these originals in the velo-binder and give the bound receiving sheets to the Lab Manager for archival.

Sample collection dates may be provided on the COC by the organization conducting the field sampling. If provided, the COC is used as documentation for sample collection dates.

7.2.3 Sample Storage

After the samples are logged in, the sample custodian stores them in the refrigerator, freezer or shelf space in the secure sample storage cabinets. Unless solid samples are to be prepared for analysis within one week of receipt they will be stored in the freezer until they are to be prepared.

- Samples requiring refrigeration or freezing are stored in a refrigerator or freezer dedicated to secured sample storage in sample storage room #1. The samples are removed from the shipping cooler and stored in their original containers, unless damaged.
- Samples not requiring refrigeration are stored on shelves in the secure sample storage cabinets, which helps to protect samples from UV radiation.

A sample is considered to be "in custody" in the laboratory if it meets the following criteria:

- It is actually in the Sample Custodian's possession
- It is actually in the analyst's possession
- It remains in the analyst's view, once the person has assumed physical possession of the sample
- It was in an analyst's possession and then locked or sealed to prevent tampering.
- It is in a secure area

Sample Receiving Log

Tracking #

Customer:
Contact:

Collection Date

QA Level

Sample Condition
Shipping container intact?

Shipping container type:
Shipping container temp:
Shipping container coolant:

Sample preservation:
Acid lab #
Hg Concentration:
Sample storage area:

28 day holding time:

Comments:

Received by:
Receiving Date:
Receiving Time:
Log-In Date:
Log-In Time:

Airbill present?
Airbill #
Carrier:

Custody seal present?
Custody seal intact?
COC Present?
COC Number:

Analysis request form?

Sample tag numbers?
Sample tags present?
COC/Sample log agree?

Tracking #	Lab ID:	Sample Tag #	Container #	Size:	pH	Matrix	Analysis	Comments:

Sample Custodian signature Date

INTERNAL CUSTODY FOR ORIGINAL SAMPLES

Tracking #: Project Reference #:

Activity								
Location								
Initials								
Date								
Time								
Sample ID								

A secure area is defined as a locked area within the premises of BRL where the samples are stored and with access restricted to sample custodian, sample analysts, lab director and QA manager. To satisfy these custody provisions, the laboratory implements the following procedures:

- Samples are stored in a secure area
- Access doors to the laboratory are kept locked, except during normal working hours
- Visitors are escorted while in the laboratory
- Samples remain in the secure area until they are removed for sample preparation or analysis

All standards are stored separately from samples.

7.2.4 Sample Distribution and Tracking

The system for tracking samples through preparation and analysis consists of a sample processing form, an "Internal Custody for Sample Preparations" form, laboratory worksheets, laboratory notebooks, instrument operation logbooks, instrument printouts (raw data), and final analytical reports.

7.2.4.A Sample Batching - After samples are received and logged-in, the samples are then batched by the Laboratory Manager. Batches are sequentially numbered starting with the last two digits of the year followed by a three digit sequential number. For example the first batch in 1996 is numbered 96-001. Batch numbers are assigned to each sample on the BRL "Tracking" data base. The Sample Processing Form (SPF) is then generated for each batch of samples (see the example on page 10 of this section). An "Internal Custody for Sample Preparations" form is also generated at this time (see page 11 of this section). This form accompanies the sample preparation batch at all times, documenting the batch from preparation to disposal

7.2.4.B Sample Preparation - The SPF and internal custody form are given to the scientist responsible for sample preparation. From the time the scientist removes samples from the storage area the forms must remain with the sample batch. All sample preparation details must be documented on a form or in a log book (see page 12 for an example). Copies of all preparation documentation, once complete, must accompany the SPF. The SPF must be signed and dated by the scientist performing the sample preparation, and any comments on unusual observances or deviations from the analytical SOP must be documented. After sample preparation is complete, the prepared samples, along with the SPF, internal custody form and preparation documentation, should be stored in the secured sample preparation storage room (BRL storage room #2).

7.2.4.C Sample Analysis - When a batch of samples is analyzed, the analyst must sign and date the SPF. Any comments on unusual observances or deviations from the analytical SOP must be documented and must be referenced on the SPF. All movement of the batch is documented on the internal custody form, which is kept with the batch in storage room #2 after analysis is complete. All data generated during analysis including the raw instrument printouts, the analyst bench sheets (see example on page 13 of this section), and the preparation notes must be attached to the SPF. This data package is then submitted to another analyst or to the Lab Manager for raw data review.

7.2.4.D Raw Data Review - When the data is reviewed, the reviewer must sign and date the SPF and comment on any unusual observances or deviations from appropriate. The data package is then returned to the analyst for Data Entry.

7.2.4.E Data Entry - After data entry is complete, the SPF is signed and dated. Comments on any unusual observances or deviations from appropriate SOPs should be documented on the SPF. The computer result pages are not printed out at this time, but instead are reviewed in electronic format during the final review. The data package is then submitted to the QA Manager for final review.

7.2.4.F Final Review - The QA Manager reviews the final data and prints out the computer result pages which are then included in the data package. After final review, the QA Manager must sign and date the SPF and include comments on any unusual observations and/or deviations from the appropriate SOPs. If the sample batch contains more than one customer, copies of the data package must be made for each additional client. The data packages are then stored in the "Active Client" file (located in the Lab Manager's office) until all data is complete for a particular set of samples (as defined by the client) at which time the report is prepared. The QA Manager should also update the batch status in the BRL "Tracking" database.

7.2.4.G Incomplete analysis of a batch - If all samples included on one SPF are not analyzed on the same day, then the samples not analyzed are crossed out with a single line, initialed and dated, and placed on a new SPF with a different Batch number. If a sample needs to be reanalyzed on another day, this should be noted on the SPF. The sample to be rerun should then be placed with another Batch of samples and the information for this sample recorded on both the SPF and internal custody form for that batch of samples.

7.2.4.H. Deviation Traceability - All documents are used to track samples. The main form for sample tracking is the SPF. This form should contain any mention of unusual events or occurrences or deviations from SOP's and should list where this information can be found if relevant. Examples include, but are not limited to the following:

- Out of Control calibration curve-see lab data sheet.
- Samples not cold when removed from refrigeration-see instrument log book.
- Samples over distilled-see distillation log sheet.
- Samples prepared differently from SOP-see prep. notes.

In this way all necessary information concerning samples and all sample handling steps can be traced and noted in the report to the customer.

SAMPLE PROCESSING FORM

Batch #: Analysis: QA:

Tracking #	Lab ID	Project Ref #	Date Received	Matrix	Comments

QA Comments:

Batched By: _____ Date: _____

Prepared By: _____ Date: _____
Comments: _____

Analyzed By: _____ Date: _____
Comments: _____

Raw Data Review By: _____ Date: _____
Comments: _____

Data Entry By: _____ Date: _____
Comments: _____

Final Review By: _____ Date: _____
Comments: _____

INTERNAL CUSTODY FOR SAMPLE PREPARATIONS

Batch #: Analysis: Method #:

Activity								
Location								
Initials								
Date								
Time								
Sample ID								

METHOD BR-0011 Distillation of Samples for Methylmercury Analysis

BATCH #_____ Preparation Date:_____

Tracking Number(s):_____ Distillation Technician:_____

Client(s):_____ Matrix:_____

Block Number	Sample #	Distillation Time (hh:mm)	Gas Flow Rate	
1				
2				Spiking Std:
3				
4				
5				
6				
7				QA:
8				
9				
10				
11				
12				
13				
14				
15				Comments:
16				
17				
18				
19				
20				
21				
22				
23				
24				
25				

Temperature (°C): 1-5:_____ 6-10:_____ 11-15:_____ 16-20:_____ 21-25:_____

Time started: 1-5:_____ 6-10:_____ 11-15:_____ 16-20:_____ 21-25:_____

Hg Analysis Sheet

Page 1 of___

☐Total Hg ☐Hg(II) ☐Other:_____ Batch:_____ Matrix:_____

Analyst:_____ Calibration Blank $\bar{X}$:_____ Blank Corr. Calib. Coef. $\bar{X}$:_____
Date:_____ Method Blank $\bar{X}$:_____ $SnCl_2$:_____ RSD:_____
Instrument ID #_____ Standards: 10 ng Hg/mL:_____ 30% NH_2OH-HCl:_____ N=:_____
QA: CLP-Type☐ Research☐ 1.0 ng Hg/mL:_____ BrCl Hg conc.:_____ r:_____

Run	Trap	Bubb.	Sample ID	Sample vol/wt. (mL/mg)	Dilution Volume (mL)	Analyzed volume (mL)	BrCl (mL)	PA	pg	Result

Calculation Equation:_____
Comments:_____

Calibration SRM:_____ Method SRM:_____

7.2.4.I Quality Assurance - Each person is responsible for filling-out the appropriate information for the task that they performed. The data and SPF will not be accepted by the next responsible person unless the previous section is complete.

To ensure that sample holding times are met, samples for all projects are assigned a holding time. This holding time is kept in the BRL "Tracking" database. The lab manager is responsible for ensuring that all holding times (or sample turn-around times) are met. This is done by checking the database daily to see what deliverables are approaching the due dates, which can be performed by running a simple query on our database. In addition, all sample processing forms have a due date recorded on them, which correlates to the date that the sample prep., analysis, data review and batch report need to be completed. This date is generally one week prior to the final report due date, to allow sufficient time for the final report generation, review and submittal.

On extremely rare occasions, BRL may submit samples to another lab. In such a case, the documentation to transfer samples includes collection date and time (if available from the field samplers), the Field ID#, the BRL Lab ID #, the date of preparation (if extracts are transferred), and the requested analyses.

7.2.5 Sample Disposal

7.2.5.A Sample Preparations - Once samples have been analyzed, and either the data has been reviewed and found to be acceptable or it has been otherwise determined that there is no value in reanalyzing the sample preparations, then the sample preparations may be disposed. The Lab Manager (or designee) will notify the sample custodian of any batches ready for disposal who shall then retrieve the appropriate batch(es) and dispose of the sample preparations accordingly. The disposal of each batch shall be documented on the "Internal Custody for Sample Preparations" form which shall then be given to the lab manager.

7.2.5.B Original Samples - After completion of a report for a particular set of samples (one or more tracking numbers), the original samples may be either transferred to long-term storage, or disposed of if approved by the Lab Manager or if the client gives approval. The lab director will notify the sample custodian which original samples to dispose of or transfer, and the sample custodian shall dispose (or transfer) the samples accordingly. Solid samples are generally transferred to BRL Freezers #2 or #3 for long term storage. If solid samples are in Teflon containers, and are to be transferred, the samples should first be removed from the Teflon containers and placed in acid cleaned sample jars or other appropriate non-Teflon containers. Before moving samples to Freezer #2 or #3, all of the samples for a particular project or tracking number should be placed in a bag, and the bag should be **marked clearly with the tracking number, name of client and the sample receiving date.** At least once a year, Freezers #2 and #3 should be cleared of any samples that are more than one year old. When solid samples are disposed of (generally after 1 year) then the "Internal Custody for Original Samples" log sheet should be signed off appropriately. Solid samples may be disposed of before 1 year at the discretion of the Lab Manager and/or upon approval from the client.

7.2.5.C Disposal Guidelines

1. Routine Disposal - Samples **without** Yellow Stickers (or other hazardous labels)
   a) Water Samples and Acid Digestions - Except for those samples having a yellow sticker, all water samples (including preparations) and acid digested solid samples

that are not otherwise hazardous may be disposed down the drain. All acidic samples must be neutralized with Soda-Ash prior to disposal.

b) Native Solid Samples and Dry Weights - All native solid samples (not sample preparations) without yellow stickers may be discarded directly into the garbage.

c) Solvent Extracts - All solvent extracts must be treated as hazardous waste. Solvent extracts may be consolidated in clearly marked containers near the hazardous waste fume hood, and disposed of as hazardous waste.

1. High Level Disposal - Samples **with** Yellow Stickers. All High Level metal sample waste (as well as other waste that is considered to be hazardous) must be recorded on the "High Level Metal Waste Disposal" sheets.

a) Water Samples and Acid Digestions - All water samples (including preparations) and acid digested solid samples with yellow stickers must be neutralized with Soda-Ash. If necessary, liquid samples may then be evaporated in the sample waste fume hood in a beaker labeled as high level metals waste. After evaporation (if that step is utilized), the sample material is placed directly into the high level metals waste storage container.

b) Native Solid Samples and Dry Weights - All native solid samples (not sample preparations) with yellow stickers may be disposed of directly into the hazardous waste container. Native solid samples with a high moisture content may be dried first in the sample disposal oven, prior to placement in the hazardous waste storage container.

c) Solvent Extracts - Solvent extracts must all be treated as hazardous waste, see section 1.c) above.

1. Non-Routine Disposal - Samples that are designated by the client to be high level in an analyte not performed by BRL shall be considered hazardous and treated as hazardous waste upon release for disposal. In certain cases, BRL may contract with a client to analyze samples that are known to be hazardous beyond the scope of our analysis (such as samples containing a high level of organic contaminants or dioxins), these samples will be flagged as requiring special disposal--either with a yellow sticker or by lab director's instruction--and disposed of through a licensed hazardous waste acceptance facility. BRL may also arrange with the client to return the leftover samples after analysis.
2. High Level Metals Waste Transport and Ultimate Disposal - Once a sufficient volume of waste is generated, warranting proper disposal, a waste disposal company should be contacted, and the waste scheduled for pick-up. It shall be the responsibility of the waste handling company to transport and dispose of the high metal level waste in a manner consistent with local and federal environmental laws and regulations.

7.2.5.D Low-level Radioactive Waste - All samples that are required to be disposed of as low-level radioactive waste, need to be disposed of in accordance with all local, state and federal regulations regardless of the concentrations of metals. All low-level radioactive samples at BRL are disposed in accordance with our radioactive materials handling license (from Washington State Department of Health)

7.2.5.E Documentation - The appropriate forms should be filled out when necessary to both document disposal of samples (and sample preparations) and to initiate disposal or transfer of samples. The forms should be filled out with sequentially numbered pages. All forms must be

signed and dated by appropriate personnel. All errors in documentation must be stricken with a single line, initialed, and dated. All documentation must be printed with non-erasable ink.
7.2.5.F Duration of Storage - Water samples are stored for one month from receipt, and frozen solid samples are stored for one year from receipt unless the client requests otherwise.

7.3 Electronic Data Records

7.3.1 Analytical Integration

All analytical instrumentation signal output is integrated by BRL-developed integration software, by manufacturer specific software (i.e. Perkin Elmer) or by a Spectra Physics integrator. All analytical runs are both printed out on hardcopy and stored electronically. All documentation of integrating software upgrades is maintained by the electronics department of BRL.

7.3.2 Data Entry

All analytical data is entered into a computer spreadsheet where the results are automatically recalculated, with the exception of GFAA data which is reduced automatically by the P.E./Gem software. After data entry, the data package is then compared by another lab technician or the lab manager to ensure consistency between all data points and results on the analyst's raw bench sheet and the computer generated spreadsheet.

7.3.3 Software Verification

For integration software, any software related problem affecting samples would also affect QA samples; therefore, as long as QA criteria are met, the software is presumed to be functioning properly. Software used for data entry is verified to be working properly by the data entry verification as described in 7.3.2. Any software problems or failures are documented as correspondence between the laboratory manager and the software programmer. All documentation/correspondence is kept on file by the lab manager.

7.3.4 Security

Only BRL personnel have access to electronic data records. All employees at BRL are authorized to access electronic information. No levels of accessibility exist.

7.3.5 Hardcopy Documentation

All hardcopy printouts of information stored electronically are filed in our Central Filing Cabinets. Information is stored and sorted by Project ID and then by BRL Tracking Number. All Deliverables are paginated, and a full copy of the full data package is stored on file in our Central Filing Cabinets.

7.3.6 Electronic Transfer

Electronic Files may be transferred on disk to a Client. All electronic files that are transferred to a Client are also printed and stored as mentioned in section 7.3.5.

8.0 Analytical Procedures

8.1 Method References

Refer to the tables in section 5.0 for method number references

8.2 Field Methods

Not Applicable

8.3 Analytical Method Modifications

Modification of EPA Method 1631 - EPA Method 1631 describes only for the determination of mercury in ambient water. Brooks Rand has modified the sample preparation in the procedure to allow for the determination of total mercury in all matrices. All solid (biological, sediments and soils) matrices are prepared in accordance with SOP BR-0002, *Determination of Total Mercury in Solid by Cold Vapor Atomic Fluorescence Spectrophotometry (CVAFS)*. The sample preparation for solids in BR-0002 is a HNO_3/H_2SO_4 acid digestion followed by a BrCl oxidation. All aqueous matrices are prepared in accordance with SOP BR-0003, *Determination of Total and "Acid-Labile" Mercury in Aqueous Samples by Cold Vapor Atomic Fluorescence Spectrophotometry (CVAFS)*. Please refer to the method validation package for SOPs BR-0002 and BR-0003 in Appendix A.

8.4 New Analytical Methods

Method BR-0011 - No standard method exists for methylmercury determinations at the ultratrace level. The SOP BR-0011, *Determination of Methyl Mercury by Aqueous Phase Ethylation, Trapping Pre-Collection, Isothermal GC Separation and CVAFS Detection*, is included in Appendix A. By this method all non-biological matrices (water, sediment, soil) are distilled to remove methyl mercury from the matrix. Biological samples are digested in a KOH/Methanol solution. After sample preparation all samples are ethylated with tetra-ethyl borate which forms ethyl derivatives of the mercury species. The ethylated mercury species are collected on a Tenax® trap which is then introduced into the GC oven, pyrolitic column and AFS detector. Under the flow of an inert carrier gas, the Tenax® trap is gently heated to release the mercury species which are then separated chromatographically prior to being decomposed to elemental mercury (Hg^0) in the pyrolitic column. Hg^0 is then detected by the AFS detector. Please refer to the method validation package for SOP BR-0011 in Appendix A.

8.5 Laboratory Operations

8.5.1 Laboratory Glassware

All glassware for ultra-trace metals analysis must be rigorously cleaned, in order to minimize possible contamination. Glassware is first cleaned by scrubbing with a glassware brush, alconox and DI water. After thorough rinsing with DI water, the glassware is immersed into a large rectangular vat which contains 6M HCl. Proper safety gear must be worn at all times when working in and around the acid vats. Safety gear consists of a lab coat, a rubber apron, face shield, and Viton black extra-long gloves. The HCl vat is equipped with a Teflon coated immersion heater, a temperature controller and thermocouple. After filling the vat with glassware, the temperature controller and heater are turned on to heat the vat to 75°C. The temperature must be set to maintain the temperature at 75°C, and heated overnight. The vat should then be allowed to adequately cool prior to removing the bottles. Once cool, all safety gear must be worn and glassware should be removed from the vat and placed into a DI water rinse tank. After removing all glassware from the vat, the rinseate tank should be moved to the sink and all glassware should be removed from the rinse tank, triple rinsed with DI water, and placed in the class-100 laminar flow hood to dry. Volumetric glassware should never be baked. Once dry, all glassware should be bagged in plastic Zip-loc® bags and stored in the appropriate location.

Glassware for certain parameters other than ultra-trace metals need only be cleaned with Alconox and DI water followed by copious DI rinsing. Some glassware will need to soak overnight in 30% HNO_3 following the Alconox cleaning. See lab director for more specific cleaning information.

8.5.2 Reagent Storage

All supplies (i.e., glassware, chemicals, reagents) are of the best possible quality to ensure proper instrument calibration and to avoid contamination. All purchased reagents are labelled with the date opened and date received. All reagents used are prepared from Analytical Reagent Grade (AR) chemicals or higher purity grades, unless such purity is not available. Reagent water is prepared by double deionization of tap water. Each prepared reagent is clearly labeled with the composition, concentration, date prepared, initials of preparer, expiration date, and special storage requirements, if any.

Reagent solutions are stored in appropriate glass or plastic containers, under conditions designed to maintain their integrity (refrigerated, dark, etc.). Shelf life is listed on the label, and the reagent is discarded after it has expired. Water used in the laboratory is deionized (ASTM Type I water) and periodically checked for purity. All acids used are either glass-distilled or special grade for trace metal analysis. Reagent solutions are checked for contamination by employing reagent blanks before use in any analysis. All reagents and reagent storage at BRL are listed in the following table:

Table 8.5.2
Reagent Storage

Chemical Name	Form	Location	MSDS	Hazard	Regulated?[1]
Acetic Acid	solution	back lab- cabinet #1	x	Corrosive	
Hydrochloric Acid	solution	back lab- cabinet #1	x	Corrosive	
Sulfuric Acid	solution	back lab- cabinet #1	x	Corrosive	
Nitric Acid	solution	back lab- cabinet #1	x	Corrosive	
pH 4 and 7 buffers	solution	back lab- cabinet #1	x	None	no
Rhodamine Indicator	solution	back lab- cabinet #1	x	None	
Ferrazine	solution	back lab- cabinet #1	x	None	
Potassium permagenate	sol & salt	back lab- cabinet #2	x	Oxidizer	
Potassium persulfate	sol & salt	back lab- cabinet #2	x	Oxidizer	
Potassium iodide	solution	back lab- cabinet #2	x	Ing, Irr	no
Calcium Hypochlorite	salt	back lab- cabinet #2	x	Oxidizer	
Potassium Chromate	salt	back lab- cabinet #2	x	Oxidizer	
silver nitrate	solution	back lab- cabinet #2	x	Irr	no

Inh - Inhalation
Ing - Ingestino
Con - Skin and/or eye contact
Irr - Irritating to skin

[1] NIOSH Pocket Guide to Chemical Hazards listing

Table 8.5.2 (Continued)
Reagent Storage

Chemical Name	Form	Location	MSDS	Hazard	Regulated?
Acid Back stock:					
acetic acid	liquid	bunker	x	Corrosive	
hydrochloric acid	liquid	bunker	x	Corrosive	
sulfuric acid	liquid	bunker	x	Corrosive	
nitric acid	liquid	bunker	x	Corrosive	

Chemical Name	Form	Location	MSDS	Hazard	Regulated?
activated alumina		chemical storeroom- shelf #1	x	None	
casein	powder	chemical storeroom- shelf #1	x	None	
L-cystine	powder	chemical storeroom- shelf #1	x	None	
soda lime	6-12 mesh	chemical storeroom- shelf #1	x	Caustic	
soda lime	4-8 mesh	chemical storeroom- shelf #1	x	Caustic	

Table 8.5.2 (Continued)
Reagent Storage

Chemical Name	Form	Location	MSDS	Hazard	Regulated?
(tetra)acetic acid disodium salt	salt	chemical storeroom- shelf #2	x	None	
aluminum oxide	salt	chemical storeroom- shelf #2	x	None	
aluminum sulfate	salt	chemical storeroom- shelf #2	x	None	
ammonium acetate	salt	chemical storeroom- shelf #2	x	None	
ammonium chloride	salt	chemical storeroom- shelf #2	x	None	
ammonium molybdate	salt	chemical storeroom- shelf #2	x	None	
ammonium phosphate, mono & dibasic	salt	chemical storeroom- shelf #2	x	None	
ammonium sulfate	salt	chemical storeroom- shelf #2	x	None	
ascorbic acid	salt	chemical storeroom- shelf #2	x	None	
barbituric acid	salt	chemical storeroom- shelf #2	x	Poison	
cacodylic acid	salt	chemical storeroom- shelf #2	x	None	
cadmium	salt	chemical storeroom- shelf #2	x	Poison	WAC 173.303-9905
calcium chloride	salt	chemical storeroom- shelf #2	x	None	
calcium hypochloride	salt	chemical storeroom- shelf #2	x	None	
calcium oxide	salt	chemical storeroom- shelf #2	x	None	
citric acid	salt	chemical storeroom- shelf #2	x	None	
cobalt chloride	salt	chemical storeroom- shelf #2	x	None	
dithizone	salt	chemical storeroom- shelf #2	x	None	
ferric ammonium sulfate	salt	chemical storeroom- shelf #2	x	None	
ferric chloride	salt	chemical storeroom- shelf #2	x	None	
ferrous ammonium sulfate	powder	chemical storeroom- shelf #2	x	None	
fluorescin, dibromo	powder	chemical storeroom- shelf #2	x	None	
hydroxylamin hydrochloride	salt	chemical storeroom- shelf #2	x	None	
glycerol	liquid	chemical storeroom- shelf #2	x	None	
lead carbonate	salt	chemical storeroom- shelf #2	x	Toxin	WAC 173.303-9905

Table 8.5.2 (Continued)
Reagent Storage

Chemical Name	Form	Location	MSDS	Hazard	Regulated?
magnesium chloride	salt	chemical storeroom- shelf #2	x	None	
magnesium nitrate	salt	chemical storeroom- shelf #3	x	None	
magnesium oxide	salt	chemical storeroom- shelf #3	x	None	
magnesium perchlorate	salt	chemical storeroom- shelf #3	x	None	
magnesium sulfate, anhydrous & monohydrate	salt	chemical storeroom- shelf #3	x	None	
phenanthroline	salt	chemical storeroom- shelf #3	x	None	
potassium acetate	salt	chemical storeroom- shelf #3	x	None	
potassium bromate	salt	chemical storeroom- shelf #3	x	None	
potassium bromide	salt	chemical storeroom- shelf #3	x	None	
potassium chloride	salt	chemical storeroom- shelf #3	x	None	
potassium chromate	salt	chemical storeroom- shelf #3	x	None	
potassium cyanide	salt	chemical storeroom- shelf #3	x	Poison	WAC 173.303-9905
potassium hydrogen phthalate	salt	chemical storeroom- shelf #3	x	None	
potassium hydroxide	salt	chemical storeroom- shelf #3	x	Caustic	
potassium iodide	salt	chemical storeroom- shelf #3	x	None	
potassium permanganate	salt	chemical storeroom- shelf #3	x	None	
potassium persulfate	salt	chemical storeroom- shelf #3	x	None	
potassium phosphate	salt	chemical storeroom- shelf #3	x	None	
rhodanine	salt	chemical storeroom- shelf #3	x	None	
selenoinosine	salt	chemical storeroom- shelf #3	x	None	WAC 173.303-9905
EDTA, bisodium salt	salt	chemical storeroom- shelf #3	x	None	

Table 8.5.2 (Continued)
Reagent Storage

Chemical Name	Form	Location	MSDS	Hazard	Regulated?
sodium acetate	salt	chemical storeroom- shelf #4	x	None	
sodium chloride	salt	chemical storeroom- shelf #4	x	None	
sodium citrate	salt	chemical storeroom- shelf #4	x	None	
sodium hydroxide	salt	chemical storeroom- shelf #4	x	Caustic	
sodium nitrite	salt	chemical storeroom- shelf #4	x	None	
sodium oxalate	salt	chemical storeroom- shelf #4	x	None	
sodium phosphate, mono and dibasic	salt	chemical storeroom- shelf #4	x	None	
sodium selenate	salt	chemical storeroom- shelf #4	x	None	
sodium sulfate	salt	chemical storeroom- shelf #4	x	None	
sodium sulfide	salt	chemical storeroom- shelf #4	x	None	
sodium bisulfate	salt	chemical storeroom- shelf #4	x	None	
sodium sulfite	salt	chemical storeroom- shelf #4	x	None	
sodium thiosulfite	salt	chemical storeroom- shelf #4	x	None	
stannous chloride	salt	chemical storeroom- shelf #4	x	Toxin	
sulfamic acid	salt	chemical storeroom- shelf #4	x	None	
sulfanilamide	salt	chemical storeroom- shelf #4	x	None	
sulfer, sublimed	salt	chemical storeroom- shelf #4	x	None	
tartaric acid	salt	chemical storeroom- shelf #4	x	None	
TRIS hydrochloride	salt	chemical storeroom- shelf #4	x	None	
urea	salt	chemical storeroom- shelf #4	x	None	
vanadium pentoxide	salt	chemical storeroom- shelf #4	x	None	WAC 173.303-9905
zinc acetate	salt	chemical storeroom- shelf #4	x	None	

Table 8.5.2 (Continued)
Reagent Storage

Chemical Name	Form	Location	MSDS	Hazard	Regulated?
methylene chloride	liquid	chemical storeroom- shelf #5	x	Carcinogen	

Chemical Name	Form	Location	MSDS	Hazard	Regulated?
dichloromethane	liquid	chemical storeroom- shelf #6	x	Carcinogen	
ion exchange medium ("Amberlite")	resin	chemical storeroom- shelf #6	x	None	

Chemical Name	Form	Location	MSDS	Hazard	Regulated?
Acids in Use:					
acetic acid	liquid	clean lab acid cabinet	x	Corrosive	
hydrochloric acid	liquid	clean lab acid cabinet	x	Corrosive	
sulfuric acid	liquid	clean lab acid cabinet	x	Corrosive	
sulfuric acid, ultrex	liquid	clean lab acid cabinet	x	Corrosive	
nitric acid	liquid	clean lab acid cabinet	x	Corrosive	
perchloric acid	liquid	clean lab acid cabinet	x	Corrosive	
hydrofluoric acid	liquid	clean lab acid cabinet	x	Corrosive	
phosphoric acid	liquid	clean lab acid cabinet	x	Corrosive	

Table 8.5.2 (Continued)
Reagent Storage

Chemical Name	Form	Location	MSDS	Hazard	Regulated?
elemental mercury	liquid	shop area Cabinet # 2	x	Toxin	
mercury(II) chloride	salt	shop area Cabinet # 2	x	Toxin	
mercury(II) iodide	salt	shop area Cabinet # 2	x	Toxin	
mercury(II) oxide	salt	shop area Cabinet # 2	x	Toxin	
dimethylmercury	salt	shop area Cabinet # 2	x	Toxin	
methylmercury	salt	shop area Cabinet # 2		Toxin	
phenylmethyl mercury	salt	shop area Cabinet # 2	x	Toxin	
elemental mercury in hexane	solution	shop area Cabinet # 2		Toxin	
elemental mercury saturated in water	solution	shop area Cabinet # 2		Toxin	
dimethylmercury in propanol	solution	shop area Cabinet # 2		Toxin	
dimethylmercury saturated in water	solution	shop area Cabinet # 2		Toxin	
High PurityStandards					
arsenic	1000 ug/L	shop area Cabinet # 2	requested	Toxin	
lead	1000 ug/L	shop area Cabinet # 2	requested	Toxin	
mercury	1000 ug/L	shop area Cabinet # 2	requested	Toxin	
selenium	1000 ug/L	shop area Cabinet # 2	requested	Toxin	
NIST					
Mercury in Water, 1641c, ampoules	1.47 mg/L	shop area Cabinet # 2	x	Toxin	
HSP					
Trace metals in Wastewater	500 ug/L	shop area Cabinet # 2		None	

Table 8.5.2 (Continued)
Reagent Storage

Chemical Name	Form	Location	MSDS	Hazard	Regulated?
hydrogen peroxide	liquid	clean lab refrigerator	x	None	
glutaraldehyde	liquid	clean lab refrigerator	x	None	
silanizing solution	liquid	clean lab refrigerator		None	
pentanedione	liquid	clean lab refrigerator	x	None	
methyl iodine	liquid	clean lab refrigerator	x	None	
pyrrolidinecarbodithioic acid		clean lab freezer		None	
sodium tetraethyl borate	powder	clean lab freezer	x	None	
methylcobalamin hydrate		clean lab freezer	x	None	

Table 8.5.2 (Continued)
Reagent Storage

Chemical Name	Amount	Location		Comments
Certified Reference Materials:				
International Atomic Energy Agency				
IAEA 034, MMHg in sediment	small amount	Clean lab desiccator		documentation on file
IAEA 791, MMHg in human hair	small amount	Clean lab desiccator		no documentation
IAEA 690, MMHg in human hair	small amount	Clean lab desiccator		no documentation
IAEA MA-M-2/OC, Mussle tissue lyophilized	small amount	Clean lab desiccator		no documentation
IAEA MA-B-3/TM, Fish tissue lyophilized	small amount	Clean lab desiccator		no documentation
IAEA H-9, Mixed human diet	small amount	Clean lab desiccator		no documentation
Tuna, 1.03 ug/g Hg	small amount	Clean lab desiccator		no documentation
Albacore tuna, 0.87 ug/g Hg	small amount	Clean lab desiccator		no documentation
NIES				
Sargasso, #9	small amount	Clean lab desiccator		no documentation
NBS(NIST)				
1648, Urban particles	small amount	Clean lab desiccator	not Hg certified	documentation on file
1566, Oyster tissue	small amount	Clean lab desiccator	0.0642 ug/g Hg	documentation on file
NIST				
1632b, Trace elements in coal	half gone	Clean lab desiccator	not Hg certified	documentation on file
1633a, Trace elements in fly ash	half gone	Clean lab desiccator	0.16 ug/g Hg	documentation on file
Brammer Standards				
SARM 20, Sasolburg Coal	full	Clean lab desiccator	0.25 ug/g Hg	documentation on file
BCR #181, Coking coal	small amount	Clean lab desiccator	0.13 ug/g Hg	documentation on file
BCR #40, Coal (60-90 mesh)	full	Clean lab desiccator	0.35 ug/g Hg	documentation on file
National Research Council Canada				
PACS-1, Marine sediment - mercury only	full	Clean lab desiccator	4.57 ug/g Hg	documentation on file
MESS-2, Marine sediment - trace metals	full	Clean lab desiccator	0.092 ug/g Hg	documentation on file
MESS-1, Marine sediment - trace metals	half full	Clean lab desiccator		use as back up to MESS-2
BCSS-1, Marine sediment - trace metals	full	Clean lab desiccator	not Hg certified	documentation on file
TORT-1, Lobster	unopened	Clean lab desiccator	0.33 ug/g Hg	documentation on file
DORM-1, Dogfish muscle	almost gone	Clean lab desiccator	0.798 ug/g Hg	documentation on file
DORM-2, Dogfish muscle	unopened	Clean lab desiccator	4.64 ug/g Hg	replaces DORM-1

8.5.3 Waste Disposal

Handling, storage and disposal of laboratory-related hazardous wastes are subject to the regulations contained in the Conservation and Recovery Act. BRL shall store, package, label, ship and dispose of hazardous wastes in a manner which ensures compliance with all federal, state and local laws. Potential hazardous wastes include all standards, reagent solutions, process wastes, solvents, native samples, sample extracts and digests.

A waste is considered hazardous if:

1. The waste material is listed as hazardous in 40 CFR Part 261.30-261.33.
2. The material exhibits any of the characteristics of hazardous waste: ignitability, corrosivity, reactivity or EP toxicity.
3. The waste is listed in 1 or 2 above and is not excluded by any provisions under the Resource Conservation and Recovery Act.

A waste is considered an acute hazardous waste if it is identified in 40 CFR Part 261.31, 261.32, 261.33 (e) as an acute hazardous waste.

BRL is categorized as a Conditionally Exempt Small Quantity Generator. This category is defined as: A generator who generates no more than 100 kilograms of hazardous waste or 1 kilogram of acute hazardous waste in a calendar month and accumulates no greater than 1000 kilograms of hazardous wastes (40 CFR Part 261.5).

BRL shall ensure delivery of hazardous waste to a treatment, storage or disposal facility which is:

1. Permitted under 40 CFR Part 270
2. In the interim status under 40 CFR Parts 270 and 265
3. Authorized to manage hazardous waste by a state with a hazardous waste management program approved under Part 271; or
4. Permitted, licensed, or registered by a state to manage municipal or industrial solid waste (subject to local regulations).

Hazardous waste solvents, as identified in the 40 CFR Part 261 may not be evaporated off in a fume hood. Solvents evaporated off during the extraction/testing process are exempt. Acidic and basic wastes may be neutralized and disposed of via the sanitary sewer if they are not hazardous due to the presence of other constituents (as subject to local regulations). Heavy metals may be precipitated from the liquid portion and disposed via the sanitary sewer (subject to local regulations).

Hazardous waste storage is limited to quantity and/or accumulation time and must comply with RCRA regulations as specified in the 40 CFR. These wastes should be packaged and separated according to the compatible groups (e.g. solvents, acids etc.).

Sample submitted to BRL for analysis are excluded from regulation as hazardous waste under 40 CFR Part 261.4(d) provided the samples are being transported to or from the laboratory, are being analyzed, are being held for analysis or are being maintained in custody for legal reasons. However, once a decision is made to dispose of the laboratory samples, the exclusion provisions of 40 CFR Part 261.4(d) no longer apply. Samples that have been identified as hazardous may be either: 1) returned to the generator; or, 2) disposed of according to applicable RCRA regulations. Samples which are determined to be non-hazardous may be subject to local environmental regulations. A sample collector shipping sample to a laboratory and a laboratory returning samples to a sample collector must comply with U.S. Department of Transportation (DOT), U.S. Postal Service (USPS), or any other applicable shipping requirements.

All sample and sample preparations must be disposed of in accordance with local, state and federal regulations. All remaining native samples must be separated by matrix (water, sediment/soil, and biota) and placed in the appropriate containers for disposal. All remaining sample preparation solutions must be separated by digestion type (acid, base, solvent extract) and place in the appropriate disposal containers. All disposal containers must be clearly labeled.

8.5.4 Clean Room Procedures

BRL currently has one clean room for ultra-trace mercury analysis and a "semi-clean room" for other trace metal analyses.

The mercury lab clean room is equipped with two laminar flow hoods providing incoming air to the lab. All incoming air is filtered through a particulate filter, then through a tri-iodated carbon mercury removal filter, and then through hepa-filters. Lab air is monitored on a weekly basis for atmospheric mercury levels to ensure that levels are suitably low for ultra-trace level mercury analysis. A warning level has been established at 15 ng/m^3 and a shutdown control level at 25 ng/m^3. The lab is arranged with the two laminar flow hood at one end of the lab (the cleanest area), with all air exiting the lab at the other end through either a fume hood or through the entrance to the lab. Positive pressure is maintained to ensure that there is no incoming air through other routes other than the laminar flow hoods. All persons entering the clean room are required to wear clean room shoe covers to minimize tracking in particles. In addition, clean room sticky mats are located at the entrance as an additional precautionary measure.

The "semi-clean room" is a new laboratory located in the north east corner of the 2nd floor, and is where BRL performs all metals analysis by Graphite Furnace AA, Flame AA, or Hydride Generation AA. This lab is contains one laminar flow hood with a hepa filter. All incoming air is prefiltered. The laminar flow hood is at one end of the room with the intake to the laminar flow hood at the other end of the room. In this way all room air is efficiently circulated through the hepa-filter. In addition, the entrance to this lab has an enclosed gowning area so that there is no direct flow of air from outside of the lab into the lab.

9.0 Calibration Procedures

9.1 Instrumentation List

Table 9.1 - Instrumentation List

Description	Manufacturer	Model #	Quantity
Analyzers			
CVAFS Hg Detector	BRL	III	3
Flame AAS	Perkin Elmer	703*	1
GF and Flame AAS	Perkin Elmer	5000	1
GF ZAAS	Perkin Elmer	4100ZL	1
Colorimetric Spectrophotometer	Sequoia-Turner	690	1
IR Spectrophotometer	Perkin Elmer	1420	1
Digestion Equipment			
Microwave digestor	CEM	MDS-81	1
Block digestor	Tecator	System 20 / 1015digestor	1
Hot Plates	various	various	4
pH Meter			
pH Meter	Orion Research	301	1
Balances			
Analytical (to 0.1 mg)	Mettler	H80	1
Analytical (to 0.1 mg)	Mettler	H10	1
Top loading (to 1 mg)	Sartorius	E1200	1
Top loading (to 1 mg)	American Scientific Products	Z-400-DR	1
Top loading (to 10 mg)	Mettler	BB2400	1
Geiger Counter			
Geiger Counter	Bicron	Surveyor 50	1
Ovens			
Scientific oven	American Scientific Products	Tempcon	1
Scientific oven	Precision Scientific	Thelco	1
Scientific oven	Fisher Scientific	630F	1

Refrigerators/Freezers			
Chest freezer	General Electric	CB22D	1
Chest Freezer	Sears	Coldspot	1
Upright freezer	Sears	Coldspot 31	1
Refrigerator/freezer	Kenmore	20	1
Refrigerator/freezer	Sears	Coldspot	1
Conductivity Meter			
Conductivity Meter	Cole Parmer	TDS Testr3	1

*modified with BRL manufactured parts for hydride generation AA analyses

9.2 Standard Receipt and Traceability

All stock standard solutions are received by the analytical laboratory and are documented in the appropriate standard solution lab notebook. Information to be documented in the standard solution notebooks includes: Source, type of standard, date of receipt, lot number (if applicable), expiration date and purity statement. Certifications for all stock standards are kept in the QA files by the Quality Assurance Manager.

All standard solutions are stored in a manner that is consistent with the manufacturers' recommendations.

Traceability of standards is achieved by documenting all standard solution information in the standard solution notebooks. In addition to the previously mentioned documentation for stock standards, documentation for intermediate standard solutions must include: identification of primary (stock) standard used, the preparation date, method of preparation (specifically dilution information), the preparer's name, the concentration prepared and the expiration date. Documentation for working standards must include: identification of the stock and intermediate standards used, the preparation date, method of preparation (specifically dilution information), the preparer's name, the concentration prepared and the expiration date.

9.3 Standard Sources and Preparation

All working standards are documented for traceability as discussed in section 9.2. All intermediate and working standards are made in accordance with the protocols of the specific procedure for which the standards shall be used.

Table 9.3 - Standard Sources and Preparation

Instrument Group	Standard Source(s)	How Received	Storage	Preparation from Source	Lab Stock Storage	Prep Frequency
Atomic Absorption	Johnson Mathey and High Purity Standards - all NIST traceable	Solutions of 1000 ppm	Room temp.	Intermediate standards are prepared from Stock standard	1% HNO_3 at room temp	annual
				Working standards are prepared from intermediate	1% HNO_3 at room temp	every 3 months
Atomic Fluorescence	Total Hg - High Purity Standards - NIST traceable	Solutions of 1000 ppm	Room temp.	Intermediate standards are prepared from Stock standard	5% BrCl at room temp	annual
				Working standards are prepared from intermediate	1% BrCl at room temp	monthly
	Methyl Hg - Strem Chemicals	raw chemical CH_3HgCl	Room temp.	Stock standard prepared from raw chemical	dissolved in isopropanol, stored at 4°C	annual
				Intermediate standards are prepared from Stock standard	in isopropanol at 4°C	annual
				Working standards are prepared from intermediate	1% HCl, dark at room temperature	every 3 weeks
pH Meters	Scientific Products	pH 4, 7, 10	Room temp.	NA	NA	NA
Conductivity Meters	Cole Parmer	1413 µmhos	Room temp.	NA	NA	NA

None of the above standards require standardization. MMHg standards (made from raw reagents at BRL) are checked against Certified Reference Materials but are not "standardized" due to a lack of certified MMHg standards.

All Quality Control Reference Materials are currently acquired from the National Resource Council of Canada (NRCC), the National Institute of Standards and Technology (NIST), the International Atomic Energy Agency (IAEA), Community Bureau of Reference (BCR), or Alpha Resources Inc. Most standard reference materials are used directly in audit studies and will reflect performance in both quality and usage.

9.4 Instrument Calibration

All calibration protocols and criteria are specified by the analytical methods, except as noted below. All instruments are calibrated daily prior to sample analysis. Full documentation for calibration is included in the sample data for the bath of samples analyzed on that day. In addition, each instrument has a log book in which summarized information is documented. This summarized documentation includes: date, analyst, batch #, calibration coefficient (or response factor), correlation coefficient (r), average blank level, and instrument noise level. In addition, any maintenance performed on an instrument is documented in the instrument log books.

9.4.1 CVAFS, HGAA, GFAA, and FLAA Instrument and Method Calibration

Instrument abbreviations are as follows: Cold Vapor Atomic Fluorescence Spectrophotometry (CVAFS), Hydride Generation Atomic Absorption (HGAA), Graphite Furnace Atomic Absorption (GFAA), and Flame Atomic Absorption (FLAA). Each instrument used to analyze samples must pass the calibration criteria established in the appropriate operating procedure. The instrument calibration consists of analyzing at least three standards covering at least one order of magnitude (one near the upper limits of linearity within the calibration curve, one in the middle and one at the low end) and a calibration blank. These standards should span the linear range of the instrument. The correlation coefficient of the initial calibration for CVAFS and HGAA must be $\geq$0.990 and for GFAA and FLAA must be $\geq$0.995. The initial calibration check, consisting of one standard at the mid-point of the calibration curve and one calibration blank, is performed immediately following this initial calibration, after every ten samples and at the end of each batch or sequence. After analysis of a batch (maximum 20 samples), the CVAFS and HGAA instruments must be recalibrated before use. Calibration criteria for instrument linearity and sensitivity must be met before samples are analyzed. Initial and continuing calibration checks are used to establish whether ongoing instrument calibration is acceptable. Initial and continuing calibration checks must be within 20% of the instrument calibration for CVAFS and HGAA and within 10% for GFAA and FLAA. QC check standards used for performing the initial and continuing calibration checks must be from a different source that the standards used for calibration and must meet the acceptance criteria of 80-120% recovery.

All standards used to prepare the calibration standard solution are obtained from chemical suppliers and are of high purity and concentration. The standards are routinely checked by the laboratory for traceability to National Research Council of Canada (NRCC) or National Institute of Standards and Technology (NIST) Standard Reference materials. These commercial standards

are used as stock standards. Working standards are made from the stock standards at appropriate concentrations to cover the linear range of the calibration curve as outlined in the individual procedures. The working standards used for initial calibration curves are independent of calibration check standards and spiking solutions. Preparation of all standards is recorded in a bound laboratory logbook (as described in SOP BR-0500). All solutions are labeled as follows: name of solution, concentration of solution, date prepared, analyst's initials. All laboratory analysis (including instrument calibration) is documented by the analysts on the lab bench sheets.

9.4.2 Periodic Calibration Procedures for other Laboratory Equipment

Periodic calibration checks are performed for associated equipment such as balances (daily), thermometers (quarterly), ovens (daily), and refrigerators (daily) that are required in analytical methods but that are not routinely calibrated as part of the analytical procedure. All the calibration measurements are recorded in a laboratory logbook (SOP BR-1200).

BALANCES

All balances are calibrated on an annual basis by a contracted, certified professional. Balances are checked routinely with Class S weights on a daily or as-used basis. Before each weighing session, the analyst is required to perform at least one calibration check in the range of the material to be weighed. The acceptance criteria for calibration checks are summarized in Table 7.1. In addition all balances undergo a four point calibration check monthly. All calibration checks are documented in a bound laboratory log book (SOP BR-1200).

Table 9.4.2
Criteria for Balance Calibration Checks

ANALYTICAL BALANCES

Class S Weight (g)	Warning Level (g)	Control Level (g)
0.0100	0.0098 - 0.0102	0.0097 - 0.0103
0.1000	0.098 - 0.102	0.097 - 0.103
1.000	0.995 - 1.005	0.990 - 1.010
10.000	9.995 - 10.005	9.990 - 10.010
50.00	49.98 - 50.02	49.94 - 50.05

TOP LOADING BALANCES

Class S Weight (g)	Warning Level (g)	Control Level (g)
1.00	0.95 - 1.05	0.90 - 1.10
10.0	9.9 - 10.1	9.8 - 10.2
50.0	49.7 - 50.3	49.5 - 50.5

OVENS, REFRIGERATORS AND FREEZERS

Temperatures are checked with thermometers and necessary adjustments to the temperatures are made as required. All refrigerators and freezers are checked on a daily basis, and all ovens are checked at least once during each use. Thermometers are checked annually against a NIST-grade thermometer. All temperatures are recorded in a bound laboratory log book (SOP BR-1200).

10.0 Preventative Maintenance

10.1 Routine Maintenance Measures

Table 10.1 - Preventative Maintenance

Instrument	Activity	Frequency
CVAFS	Change lamp	Monthly
	Change/clean quartz cell	Quarterly
	Check Electronics	Annually
AA - Flame	Clean burner head, check tubing, pump and lamps	Daily
	Clean spectrophotometer windows	Weekly
	Check O-rings	Monthly
	Clean nebulizer	Semi-Annually
	Fine tune wavelength; Check optics and electronics	Annually
AA - Furnace	Check graphite tubes; Flush autosampler tubing	Daily
	Clean furnace housing and injector tip	Daily
	Replace graphite contact cyclinders	Semi-Annually
	Check electronics	Annually
Colorimetric / Spectrophotometer	Clean sample compartment	Daily
	Windows cleaned	Monthly
	Check Electronics and lamp alignment	Annually
pH Meter	Clean; 2 pt. Calibration	After each use
Balances	Clean pans and compartment	After every use
	1 pt. Calibration check	Before every use
	4 pt. Calibration check	Monthly
	Certified Calibration	Annually
Geiger Counter	Certified Calibration	Annually*
Conductivity Meter	Check batteries and probe cables	Daily
Refrigerator / Freezers	Check temperature	Daily
	Clean interior	Monthly
	Check temperature against NIST certified thermometer	Annually

*or as required by project specific needs

10.2 Documentation

Instrument logbooks are maintained for all equipment. These logbooks contain a complete history of past performance and maintenance. For each CVAFS and AA instrument a log book is kept to document instrument usage, routine maintenance and non-routine repairs. In addition, a general lab instrument log book is kept to document all routine maintenance and non-routine repairs for all other instrumentation.

10.3 Contingency Plans

10.3.1 Major Equipment Failure

For major equipment failure of CVAFS instruments, the laboratory almost always has one or two instruments not in use. In addition BRL manufactures all of our mercury analyzers; therefore a large stock of replacement parts exists and expert service personnel are readily available.

For flame and furnace AA's, rental equipment is locally available in the case of major equipment failure while instrumentation is being repaired.

BRL currently has an excess of balances and refrigerators/freezers so that if any of this equipment fails backup equipment is immediately available. Other equipment (such as conductivity meter and pH meter are relatively inexpensive and will be purchased immediately if major equipment failure is determined.

10.3.2 Invalidation of work

Results for all samples analysis affected by equipment failure will be invalid, unless sample integrity is preserved and the sample can be reanalyzed using backup, rental or repaired equipment within the holding times specified by the project and/or in Table 6.4.

When QC criteria are not met during analysis, all instrumentation is thoroughly checked and appropriate maintenance is taken.

11.0 Quality Control Checks and Routines to Assess Precision, Accuracy and Calculation of Method Detection Limits

The laboratory uses quality control samples to assist in assessing the validity of the analytical results of field samples. Quality control samples only help to assess analytical accuracy and precision. However, errors made during sample collection can seriously effect the analytical results of field samples. Quality control samples are analyzed in the same manner as field samples at a frequency described either in the individual procedures or the contract with the client. If the quality control sample results fall within acceptable criteria (also detailed in the method), then field sample data is considered to be valid or acceptable as is. In other words, the validity, quality etc. of the field sample data is supported partially by the QC sample results. Field QC samples are the other necessary component for the validity of field sample results.

QC samples include method blanks, calibration checks, replicate, and spiked samples. The specific frequency and type of QC samples analyzed are described in the individual SOP or analytical method. A client will occasionally have specific QC requirements which are followed unless the method QA requirements are more stringent. In addition to these routine QC samples, EPA and USGS performance evaluation samples are analyzed periodically.

11.1 Quality Control Checks

11.1.1 Field QC Checks

BRL does not normally conduct field sampling. For most projects, the client is responsible for field sampling activities and therefore mandates the requirements for field QC checks.

11.1.2 Lab QC Checks

11.1.2.1 Method Reagent Blanks

A method blank is a sample of reagent water (ASTM Type I) that is treated as a sample in that it undergoes the same analytical process as the corresponding field samples. Method blanks are used to monitor laboratory performance, contamination introduced during the analytical procedure, and in some cases for determining method detection limits. A minimum of one method blank or 5% of the total number of samples (whichever is greater) is required per analytical batch. For any method where method blank levels are historically detectable, a minimum of two method blanks or 5% of the total number of samples are required per analytical batch.

11.1.2.2 Matrix Spikes

Matrix spikes are routinely included in the analysis for methylmercury due to the lack of standard or certified reference materials for many matrices and because there may be

matrix effects associated with particular samples which might interfere with the recovery. The methodology for analyzing methylmercury in water and sediment does not produce recoveries of 100% due to limitations with the preparation of the samples. Typical recoveries are 85% and this must be shown by analysis of spiked samples with every batch of samples analyzed for methylmercury.

For all Florida DER work, matrix spike must be analyzed at a minimum frequency of 5% for all samples with a similar matrices. If a batch contains samples of different matrices, matrix spikes must be prepared and analyzed for each matrix type. For all other clients, matrix spikes will be analyzed at a frequency specified by the client.

11.1.2.3 Reagent water or reagent matrix spikes

When reagent water or reagent matrix spikes are employed at the request of a client as an additional QC check to monitor the effectiveness of the method, a minimum frequency of 5% of the total number of samples or one per sample batch must be used.

11.1.2.4 Quality Control check samples

Quality Control check samples are analyzed as blind samples and must be analyzed semi-annually. BRL participates in both the EPA WP Performance Evaluation (PE) studies and the USGS Interlaboratory comparison tests. Both of these studies are conducted semi-annually and utilize samples that are blind to not only to the analyst but also to the entire laboratory until after results are submitted to the appropriate agency. All PE results are included in both internal and external QA reports.

11.1.2.5 Quality Control checks / calibration checks

Quality Control checks (QCC) or calibration checks are standards that are from a different source than the working standards. QCCs must be analyzed immediately after calibration (initial calibration check), after every twenty samples (continuing calibration check), and at the end of the analytical batch (closing calibration check). For each calibration check, one QCC must be at the mid-point of the calibration curve and one QCC must be at 1-2 times the Practical Quantitation Limit (PQL). The PQL is defined as 4 times the MDL.

11.1.2.6 Duplicate samples or matrix spike duplicates

Duplicate samples or matrix spike duplicates must be analyzed at a minimum frequency of 5% or once per analytical batch (whichever is greater). If a batch contains samples from more than one matrix, then duplicates should be analyzed for each matrix.

11.1.3 Lab QC Checks

11.1.3.1 Reagent purity checks

All reagents used in the preservation, preparation or analysis of samples must be checked for the appropriate parameters of concern prior to use on client samples. All reagent

testing results is documented in the reagent log book and the reagents are then labelled with the BRL assigned lot number, the date of testing and the measured concentrations of the analytes of concern. For acceptable analytical use, all reagents should have concentrations of analytes that would be less than the detection limit for the pertinent analytical method once adjusted for the volume of reagent used for a normal sample. For example, for aluminum (MDL = 7.8 µg/L) the HNO_3 must contain less than 3.9 mg/L assuming that 2 mL of HNO_3 is used for the preservation of a 1 Liter sample.

11.1.3.2 Internal standards

BRL does not possess GC/GCMS instrumentation and therefore does not use internal standards.

11.1.3.3 Surrogate spikes

Surrogate spikes are samples that are fortified with a compound of similar chemical characteristics to the compounds of interest. Known concentrations of these compounds are added to all samples in the batch before sample preparation. Surrogate spikes may be analyzed if specified by a particular method or if required by a particular client.

11.2 Routine Methods Used to Assess Precision and Accuracy

11.2.1 Accuracy and Precision

11.2.1.1 Precision

Precision from three or more replicates is expressed as % Relative Standard Deviation (or % RSD) and shall be calculated from the following formulae:

$$\%RSD = \frac{s}{\bar{x}}(100)$$

Where: $\bar{x}$ = Mean (average) of the data points, and
s = Standard deviation calculated as:

$$s = \sqrt{\frac{\sum_{i=1}^{n}\left(x-\bar{x}\right)^2}{n-1}}$$

Precision from duplicate samples is expressed as Relative Percent Difference (RPD) and is calculated as follows:

$$RPD = \frac{|a-b|}{\bar{x}}$$

Where: a = result a from native sample aliquot a
b = result b from native sample aliquot b

11.2.1.2 Accuracy from Spiked Samples

The accuracy of a measurement shall be determined by the recovery of a known amount of analyte in a real sample as:

$$\%\,R = \frac{Cs - Cu\,(100)}{S}$$

Where: Cs = concentration of spiked sample
Cu = concentration in unspiked sample
S = expected concentration
%R = percent recovery

11.2.1.3 Accuracy from Known Concentrations

The accuracy of a measurement based on known concentrations shall be calculated as:

$$\%\,R = \frac{\text{Sample concentration}\,(100)}{\text{Reported True Value}}$$

11.2.1.4 Upper and Lower Warning and Control Limits for Acceptance Criteria

Upper and Lower Warning Limits (WL) and Control Limits (CL) for determining acceptance criteria shall be calculated as follows:

$$CL = P_{av} \pm 3s$$

where: CL = Control Limit (upper and/or lower)
P_{av} = Mean of P (average percent recovery or average %RSD)
s = standard deviation

and

$$WL = P_{av} \pm 2s$$

where: WL = Warning Limit (upper and/or lower)

11.2.5 Quality Control Charts

Quality Control charts are used to determine acceptance criteria for the laboratory. Separate Quality Control charts should be established for each analytical method and for each parameter or analyte for both precision and accuracy. Control charts are updated and maintained by the Quality Assurance Manager. Control Charts are updated continuously with each set of analytical

data processed by the laboratory so that the Quality Assurance manager can quickly spot trends in performance. QC results from every batch are entered into the appropriate control charts as part of the final review process. Quality Control Charts are then distributed that state the last 20 points or annually, whichever comes first. Quality Control Charts are distributed to the laboratory personnel and used to update QA criteria information in Table 5.2 of this CompQAP.

Control charts are constructed and can be used to monitor laboratory performance and to evaluate out-of-control events. Control charts use both the mean and standard deviation in order to identify out-of-control events. Control charts are constructed for spike recoveries, SRMs/CRMs performance, and duplicate analysis.

Data is entered into a spreadsheet application program, where the mean and the standard deviation, warning and control limits are automatically calculated and updated. These statistical data are also plotted automatically in control chart format, giving the analyst immediate feedback on the performance of the method of the data set. The individual data points are plotted against the mean and the ± 2 and ± 3 standard deviations.

It takes a minimum of 7 data points from at least 3 non-consecutive calendar days of analysis to "define" a control chart. If a sample points falls in the interval delineated by ±3 standard deviations, then it is a valid analysis. The warning limits are defined as ±2 standard deviations and the control or action limits are defined as ±3 standard deviations from the mean. Those sample points that fall outside the control limits are termed out-of-control, and must be discussed in the cover letter or case narrative.

Examples of out-of-control data are:

- A single data point outside of the acceptance limits established by control charts
- Two consecutive data points that are between either the upper warning limit and the upper control limit, or that are between the lower warning limit and the lower control limits
- A series of seven consecutive points on the same side of the mean recovery value
- A series of five consecutive unidirectional data points deviating from the mean
- A series of consecutive points describing a periodic or recurrent trend

An outlier is an extreme value, high or low, that has questionable validity as a member of the measurement set with which it is associated. Outliers are **not** used in Quality Control Charts. Outliers may be rejected from the data set for the following reasons:

- A known experimental aberration occurred, such as instrument failure or inconsistency in the procedure or technique

- The T value for the data is larger than the tabulated values using the Grubbs test for outliers (Table 9.1). Outliers at BRL are determined with a 99% confidence level (or 1% risk of false rejection). The T value is calculated using the following equation:

$$T = \frac{|X_0 - \overline{X}|}{SD}$$

where: X_0 is the extreme value being measured
$\overline{X}$ is the mean of the measurement set for *n* observations including X_0
SD is the standard deviation associated with X including X_0

If a value is rejected, the mean and standard deviation are recalculated using the remaining data. This procedure can be reiterated using the next extreme value until no outliers remain.

Table 11.2.5
Grubbs Test for Outliers

Number of	Risk of False Rejection				
Data Points	0.1%	0.5%	**1%**	5%	10%
3	1.155	1.155	**1.155**	1.153	1.148
4	1.496	1.496	**1.492**	1.463	1.425
5	1.780	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**	2.110	1.977
10	2.606	2.482	**2.410**	2.176	2.036
15	2.997	2.806	**2.705**	2.409	2.247
20	3.230	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	**3.600**	3.207	3.017

Tabulated values obtained from Quality Assurance of Chemical Measurements by John Keenan Taylor, 1987.

11.3 Method Detection Limits and Practical Quantitation Limits

11.3.1 Method Detection Limit

The method detection limit (MDL) is the minimum concentration of an analyte of interest that can be measured and reported, with 95 percent confidence, that the value is above zero. MDLs are defined as three times the standard deviation of replicate analyses of a sample that is 1-5 times the estimated detection limit for the analyte of concern. The sample aliquots to be used may be a native sample or reagent water that has sufficient analyte (present or spiked) to make the concentration 1-5 times the estimated MDL. More specific information on the MDL procedure and the standard deviation calculation are found in the EPA in "Definition and Procedure for the Determination of the Method Detection Limit - Revision 1.11", 40 CFR 136, Appendix B.

A minimum of seven sample aliquots must be analyzed for determination of the MDL. The MDL is then calculated as the "students t" value (for the number of replicate analyses) times the standard deviation. When evaluating an MDL, the sample aliquots must be carried through the entire method under ideal conditions.

The Practical Quantitation Limit (PQL) is the concentration of an analyte of interest at which the relative confidence in the measured value is ± 30% at the 95% confidence level. PQLs are calculated as 12 times the standard deviation from the MDL determination or 4 times the MDL. If, for a particular method of analysis, the concentration in the sample aliquots is below the MDL, then they cannot be used to calculate the MDL or the QL. In such a case, a low level standard, slightly above the anticipated detection limit, will be carried through the entire method at least seven times. The standard deviation from these seven analyses will be used to determine the MDL and QL. MDLs and PQLs are determined for each method used at BRL prior to the analysis of client samples by that particular method. If any major changes in a procedure are made, then the MDL and PQL must be redetermined for the revised method.

MDLs and PQLs are verified on a continuous basis by the analysis of Quality Control check (QCC) samples that are 1-2 times the PQL (see section 11.1.2.4).

All MDL studies are documented and the documentation is kept on file by the Quality Assurance Manager. Documentation includes the date of the study, the name of the analyst conducting the study, the analytical method(s), the compounds studied, all preparation notes and all raw data from analysis.

11.4 General QC Requirement Statement

The QC requirements listed above are general minimum requirements only. Specific methods may have more stringent requirements in which case the method requirements must be followed.

12.0 Data Reduction, Validation and Reporting

Before the analytical results are released from the laboratory, both the sample and the associated quality control data are carefully reviewed.

12.1 Data Reduction

The analyst is responsible for reducing the raw data into data ready for reporting. Data reduction consists of performing any necessary calculations and determining the concentration of an analyte using a standard calibration curve. The following documentation must be present: preparation notes, Sample Processing Forms (SPF), lab bench sheets, and raw data or instrument printouts. All instrument printouts (chromatograms and/or strip chart recordings) must have the analytical batch recorded on them and the sample tracking and ID number for each instrument response. All calculations must be carried out as specified in each particular analytical procedure.

12.2 Initial Data Review

After the data has been acquired and any necessary calculations performed, the initial review is performed by an Analytical Technician other than the one who analyzed the samples. Items in the review include sample identity, instrument calibration, QC samples, detection limits, numerical computations, accuracy of transcriptions, sample preparation logs, instrument/analytical logs, internal chain of custody and compliance with the individual method.

12.3 Data Entry

The person performing data entry first ensures that all data is present and that the initial review has been completed and signed-off. All raw data is then entered into Excel® files and recalculated. All computer spreadsheet results are then compared to the analyst's calculations to ensure that they match. The Analyst who analyzed the samples is responsible for performing all data entry.

12.4 Final Data Review and Validation

Following the analyst's review, the QA director reviews all the raw data and calculations, as well as the analyst's chemical interpretation and any out-of-control conditions that may have been identified by the analyst. Additionally, the QA director examines the QC sample data and ensures that the analytical results are within laboratory-prescribed criteria for accuracy and precision.

Data validation is part of the review process whereby data are inspected and either accepted or rejected based on a set of criteria. Evaluation parameters that can be used for validation include, but are not limited to:

- Calibration data
- Specific checks unique to each measurement
- Statistical tests

After final data review and validation is complete, the QA director signs-off on the sample processing form.

12.5 Data Reporting

Data are reported using a format specified in the client contract. Generally, data are reported in tabular form with a case narrative or a cover letter attached. Reports for Florida State DEP must have all information required in 62-160.670, F.A.C. All of the data, including standard spike recoveries, control samples, triplicate analyses, and results from blank analyses, are reported along with the sample results. Any results lower than the method detection limit (<MDL) or less than the Quantitation Limit (<QL) are qualified. Footnotes are referenced to specific data if an explanation of reported values is required. All the reports are signed and transmitted by the laboratory supervisor. Data entry for the final report is performed by the lab manager and is verified by either the QA Manager or one of the Analysts. This verification process includes double-checking all final results against the original calculated results and checking the sample ID #'s in the final report against the ID #'s in the original COC. Final reports are then submitted to all required parties (project dependent). One additional copy of every report is made and kept on file for BRL's internal records (see 12.6 data storage). All laboratory report forms and reporting formats must be in compliance with the reporting requirements of the applicable project for which they are generated.

12.6 Data Storage

Hard copies of all data and documentation will be kept on file for a minimum of three years. Data and documentation to be stored includes: SPFs, preparation notes, lab bench sheets, lab notebooks used in reduction, instrument printouts, results spreadsheets (spreadsheets used to calculate values), a full copy of reports, sample receiving logs, and any sample information provided by the client including chain of custody forms, analysis request forms, airbills, etc. The QA Manager is responsible for maintaining files for all analytical work. All Client work is stored in the central filing cabinets and is sorted by project identification number, and then by tracking number. All MDL study documentation and other QA documentation is filed according to the method in the QA files.

Electronic summaries of data will be kept for a minimum of seven years. All computer files are stored both on the hard drive of the lab manager's computer and on backup disks. Computer files of reports are organized by sample tracking number, batch spreadsheets are organized by batch number, and all project information is organized by project identification numbers.

13.0 Corrective Action

13.1 Criteria Levels

Table 13.1 - Criteria Levels

Control Item	Method Reference	Analysis	Acceptance Criteria
Standard validation	All	All	95-105%[1]
Initial calibration curve - Correlation Coefficient (r)	All GFAA methods	*GFAA Metals	>0.995[2]
	BR-0011	MMHg	>0.990[2]
	BR-0020	As, Se	>0.990[2]
Initial calibration curve - Response Factor (RF)	All BRL SOPs, 1631, 1632	MMHg, Se, Hg, As	<20% RSD[2]
QC Check Standard	All	All	90-110%[3]
Continuing calibration	All GFAA methods	*GFAA Metals	90-110%[3]
	All BRL SOPs, 1631, 1632	MMHg, Se, Hg, As	80-120%[3]
Standard Reference Material	All	All	see Accuracy in Table 5.2[4]
Method/Reagent Blank	All GFAA methods	*GFAA Metals	<MDL[4]
	BR-0002, BR-0003	Hg (water)	<50 pg/blank[4]
		Hg (solids)	<500pg/blank[4]
	BR-0011	MMHg (water&sed)	<5 pg/blank[4]
		MMHg (biota)	<500pg/blank[4]
	1631	Hg	<25 pg/blank[4]
	1632	Inorganic As	<MDL[4]
Calibration blank	All Except CVAFS	All Except Hg	<MDL[2]
	BR-0002, BR-0003	Hg	ea. < 40 pg[2], ave. < 20 pg[2]
	1631	Hg	ea. < 50 pg[2], ave. < 25 pg[2]
QC Check Samples	All	All	see Accuracy in Table 5.2[4]

Table 13.1 - Criteria Levels (continued)

Control Item	Method Reference	Analysis	Acceptance Criteria
Matrix Spike	All	All	see Accuracy in Table 5.2[4]
Blank Spike	All	All	see Accuracy in Table 5.2[4]
Analytical Duplicate	All	All	see Precision in Table 5.2[4]
Method Duplicate	All	All	see Precision in Table 5.2[4]

*GFAA Metals include: Ag, Al, As, Ba, Be, Cd, Co, Cr, Cu, Fe, Mn, Ni, Pb, Sb, Se, Sn, Tl, Zn

[1] - purchase and/or make new standards
[2] - reanalyze and/or recalibrate
[3] - rerun standard, reprep/repour/rerun if ICV is out, if still out of control, then recalibrate, rerun samples from last continuing calibration check
[4] - reanalyze, if still out of control, determine cause, reprepare and analyze all samples if necessary

For spike recovery and duplicate precision criteria please refer to Table 5.2.

13.2 Corrective Action

The laboratory has a corrective action system so that for any situations which adversely affect data quality are identified, solved and documented. These situations include, but are not limited to:

- Results outside of quality control criteria
- Statistically out-of-control-events
- Deviations from normally expected results
- Suspect data
- Deviations from the method
- Special sample handling requirements

Corrective action may also be initiated as a result of other QA activities, such as performance or system audits. Anyone is able to initiate required corrective action.

Once a requirement for corrective action has been identified, the lab manager and/or the QA manager must be notified immediately. A verbal notification may be initially made; however, a non-compliance form is required (see following page). The QA Manager is responsible for evaluating the situation and determining the appropriate corrective action. Corrective action steps include, but are not limited to:

- Problem identification
- Investigation responsibility assignment
- Investigation to determine the cause of the condition
- Action to eliminate the problem
- Increased monitoring of the effectiveness of the corrective action
- Verification that the problem has been eliminated

Documentation of problems requiring corrective action is important to overall laboratory management. The QA Manager is responsible for verifying that initial action has taken place and appears effective and, after an appropriate time, for checking to see if the problem has been fully resolved. Examples of corrective action include, but are not limited to:

- Amending forms
- Reanalyzing samples if holding times permit
- Checking instrumentation to make sure that it is operating properly
- Recalibrating with fresh standards
- Replacing suspect reagents
- Examining calculations
- Additional training in sample preparation and analysis
- Evaluating and amending procedures
- Accepting the data and acknowledging the level of uncertainty or inaccuracy by flagging the data and providing an explanation for the qualification

Florida DEP recommended corrective action will be initiated (for projects in Florida) as a result of systems or performance audits, split samples or data validation review.

Non-Conformance/Resolution Form

Initiation of Form

Initiated by: _____ Date of Occurrence: _____

Sample Batch # Affected: _____ Matrix/Analyte: _____

Description of Non-Conformance

Symptom Observed: _____

Detailed Description of Problem: _____

_____

_____

_____

_____

_____

Communication and Discussion of Problem

Lab Manager Notified: ___ [Y/N] Meeting Held: ___ [Y/N]

Date: _____ Names of Attendees: _____ _____

_____ _____

Summary of Discussion and Action to be taken: _____

_____

_____

_____

_____

_____

_____

Action Taken

Name/Date/Description of Action and Findings: _____

_____

_____

_____

Preventative Measures: _____

_____

Resolution Approval

Initiator: _____ Lab Manager: _____

(signature/date) (signature/date)

14.0 Performance and System Audits

14.1 System Audits

14.1.1 Internal Systems Audits

General Laboratory Audits are performed at least annually. This audit process is used to ensure that:

- Approved procedures are in place and used
- Sample custody is properly maintained and documented
- Analytical methods are performed properly and documented
- Specific equipment is available, calibrated and in proper working order
- Analysts are properly trained and the training is documented
- Record keeping procedures are being followed and appropriate documentation is maintained

An Audit checklist is included at the end of Section 14 of this CQAP. After the audit, the QA manager prepares an audit summary and submits it to the lab director. The QA manager is responsible for tracking responses and follow-up corrective action.

In addition BRL conducts specific function audits on a monthly basis. The laboratory work is divided into twelve segments, one of which is thoroughly audited each month. The twelve segment are as follows:

1) Training and Training Records
2) Equipment Calibration and Maintenance
3) SOPs and CQAP Review and Revisions
4) Sample Receiving Practises
5) Sample Storage and Custody
6) Sample and Hazardous Waste Disposal
7) CVAFS Lab
8) HGAA Lab
9) Wet Chemistry Lab
10) GFAA/FLAA/CVAA Lab
11) Safety and Radiation and CHP
12) Data Handling/Record Keeping

Various intercalibration exercises with other laboratories also serve as a performance audit of laboratory analysis.

14.1.2 External Systems Audits

BRL has occasional audits from various clients and accrediting agencies. The principal organizations that conduct audits of BRL's facilities and operations are Lockheed Martin Energy Systems and the Washington State Department of Ecology. There are currently no regularly scheduled external systems audits of BRL. BRL views external audits as an excellent tool for evaluating our quality, and for finding areas for improvement. BRL always welcomes any client (current or potential) or government agency to conduct on-site audits.

14.2 Performance Audits

Internal Performance Audits must be conducted at least semiannually and may consist of blind samples, split samples with another laboratory (interlaboratory comparison study), QC samples (unknown to the analyst), performance evaluation samples, and/or blind spiked samples. BRL participates in the EPA WP PE study twice a year, and in the USGS PE study twice a year. In addition BRL frequently participates in assisting agencies to certify reference materials for use as blind interlaboratory samples. Any of the Analytical Technicians may analyze these performance audit samples. The Lab Manager is responsible for overseeing BRL's participation in each study, and all associated documentation, reporting and record keeping.

External Performance Audits are as follows:

Agency	Study Title	Frequency
US EPA	WP PE	Semiannual
USGS	Round Robin Evaluation	Semiannual

BRL Laboratory Audit Checksheet

Individual SOP's should be checked against the following lab practices to ensure agreement. Briefly explain any lab practice which disagrees with written procedures.

1. SAMPLE RECEIPT
   - __ Samples received and logged in sample receiving log book
   - __ Samples labeled correctly upon receipt
   - __ Samples preserved correctly

2. SAMPLE STORAGE
   - __ Samples stored in appropriate location
   - __ Samples stored for appropriate holding times
   - __ Samples disposed of after holding times expired
   - __ Sample containers cleaned and tested correctly

3. SAMPLE PREPARATION
   - __ Distillation of samples for methylmercury performed correctly and documented
   - __ Digestion of samples for methylmercury performed correctly and documented
   - __ Digestion of samples for total mercury performed correctly and documented
   - __ Filtrations are performed correctly and analysis of blank filters is documented
   - __ Total Suspended Solids and Dry weights are performed and documented correctly

4. SAMPLE ANALYSIS
   - __ Samples analyzed according to written procedures
   - __ Triplicates run at correct frequency
   - __ Blanks run at correct frequency
   - __ Spiked samples or Certified Reference Materials run at correct frequency

5. STANDARDS AND REAGENTS
   - __ Standards and reagents are labeled correctly
   - __ Use of standards and reagents is documented correctly
   - __ Standards and reagents are stored correctly
   - __ Standards and reagents are traceable to original lot numbers of all components
   - __ Expired standards and reagents are disposed of properly
   - __ New standards and reagents are analyzed and documented

6. EQUIPMENT MAINTENANCE AND CALIBRATION
   - __ Pipettes are calibrated and documented routinely
   - __ Balances are maintained, checked and documented appropriately
   - __ Temperatures (ovens, refrigerators, freezers) are checked and documented.

7. INSTRUMENT CALIBRATION
   - __ Instrument log books are filled out appropriately
   - __ MDL's are calculated and documented appropriately
   - __ Calibration curves are established each analytical day
   - __ Calibration checks are performed accordingly
   - __ Corrective action is taken and documented when calibration curves are not linear or calibration checks are not acceptable.

8. DATA HANDLING/RECORD KEEPING
   - __ All appropriate documents are present with a given set of samples
   - __ All data checks are performed and documented
   - __ All records of results are kept for appropriate length of time

GENERAL or MISCELLANEOUS COMMENTS

REVIEWER/DATE _____

REVIEWER/DATE _____

15.0 Quality Assurance Reports

The Quality Assurance Manager is responsible for the preparation and submittal of all Quality Assurance Reports. All QA Reports are kept on files at BRL.

15.1 Internal QA Reports

Internal Quality Assurance Reports are submitted every month to the President of Brooks Rand, Ltd. Monthly reports must be complete and submitted within the first two weeks of the following month. These Internal QA Reports discuss each topic as listed below:

Personnel Changes
- List new hires, exiting employees, and changes in position of current employees

SOPs/QA Manual
- List SOP numbers and dates of review, revision, new accepted for current month
- List copies of SOPs/QAM distributed to clients
- Discuss any QA Manual issues, revisions

Training
- List training of all personnel on new tasks (from training records)

Unacceptable Analytical Results
- High blanks, bad duplicates or replicates, bad SRMs, bad spikes, bad standards....
- Describe problem and corrective action

Method Development
- Summarize progress

Performance Evaluations
- EPA (water)
- USGS (water)
- Health and Welfare, Canada (hair)
- Department of Fisheries and Oceans, Canada (fish)

Certifications
- WA accreditation
- Radioactive materials license

Instrument and Facility Changes
- Set up of new equipment/ equipment changes
- Problems with equipment/equipment maintenance

Use of new facilities (new lab)

Bottle Shipping/Sample Receiving Issues

Database Issues

Audits (Internal and External)
When they will occur
Audit findings
Resolution of findings

Lab Supply Issues
shortages of supplies, gasses etc.
bad supplies (contaminated acids etc.)

Project Specific Issues
New projects, required QAPPs, DQOs etc.
Existing Project QA Issues

Miscellaneous Issues

15.2 QA Reports for Florida DEP

QA Reports must be prepared and submitted to the Florida Department of Environmental Protection. These QA Reports must contain the following topics:

Assessment of measurement data accuracy, precision and method detection limits
Results of performance and system audits
Significant QA/QC problems and recommended solutions
Outcome of any corrective action

In addition, BRL will provide QA report to Florida DEP for all Category IV work (QAPPs). If no project audits are performed and no significant QA/QC problems occur for a specific project in the state of Florida, a letter stating these facts will be submitted to the Florida DEP in lieu of a QA report. All QA reports submitted to the Florida DEP must be submitted at the frequency required and must include all criteria as outlined in Appendix D of Florida DEP Document # QA-001/90.

APPENDIX A

for

COMPREHENSIVE QUALITY ASSURANCE PLAN

of

Brooks Rand, Ltd.

BROOKS RAND, LTD. (BRL)

Revised and Resubmitted 11/13/97

Method Validation Package (limited-use) for:

Total Mercury in Water by CVAFS: EPA Draft Method 1631, BRL Modification #1, *Determination of Total and "Acid-Labile" Mercury in Aqueous Samples by Cold Vapor Atomic Fluorescence Spectrophotometry (CVAFS).*

Contents include:

BRL Modification #1 to 1631

Summary of MDL, Precision, and Accuracy Data from Batches analyzed from 2/28/97 through 11/7/97

Public Document (not required for submittal):

EPA Draft Method 1631 (7/96)

Published Papers/References: EPA Method 1631, Draft, April 1995
Liang and Bloom, 1993

Comments:

This method is functionally the same as the previously approved method BR-0003. For purposes of clarity and consistency we perform CVAFS analysis of total mercury in water using EPA method 1631 with our modification #1. Modifications are allowed under this performance based EPA method.

Method 1631 (Draft, July 1996)
Brook Rand, Ltd. (BRL) Modification #1

Effective 2/27/97 to date

Analysts: Rebecca Els, Patrick Moore, Rick Manson, Steve Senter

QC Officers: Colin Davies, Paul Swift

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Analytical Services Division
3950 6th Ave. N.W.
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Telephone #: 206-632-6206

Narrative: BRL has been performing mercury analysis by atomic fluorescence since 1989, and during this time have identified a number of ways to improve both the quality of the data and the efficiency of the analytical process. Our current improvements that are not included in Method 1631 are as follows:

1) Section 3.1 - For oxidizing samples BRL uses the additional step of UV photo-oxidation to ensure recovery of all Hg.
2) Section 6.4.3 - BRL uses commercially available 6-12 mesh soda lime for the acid fume pretrap as opposed to the 8-14 mesh size that is listed in Method 1631 but is not commercially available. BRL has seen no difference in performance for 6-12 mesh and in-lab generated 8-14 mesh. In addition BRL cleans the pretraps for 15 minutes, which is sufficient, instead of one hour. Bubbler blanks are always analyzed first and therefore any indication of not thoroughly cleaned pretraps would be immediately demonstrated.
3) Section 6.5 - BRL uses a single trap preconcentration system instead of dual trap. This allows for faster analytical times, without masking of potential problems on the sample collection trap. No disadvantages in terms of data quality have been observed.
4) Section 6.6 - BRL uses direct electronic data acquisition with the BRL Guru integration software instead of a chart recorder or integrator. This is faster, eliminates the expense of chart recorders and/or integrators and eliminates possible transcription errors.
5) Section 7.9 - BRL uses 2 working standards: one at 10 ng/mL and one at 1.0 ng/mL. This allows for more accurate delivery of the low level standards. As written, method 1631 calls for only a 10 ng/mL standard which means that for the 50 and 100 picogram standards 5 and 10 µL respectively would need to be pipetted. This change from 1631 results in improved calibration.
6) Section 9.4.1 - 1631 states that "The mean bubbler blank for an analytical batch, if within acceptance criteria, is subtracted from all raw data for that batch prior to the calculation of results." This does not allow for the continuous determination of whether QA results are in control, thereby forcing the analyst to analyze all samples prior to determining if all QA criteria are met. This point was raised with DynCorp (contracted to facilitate method approval) on 2/27/97. BRL's suggestion of using the mean of the first 3 bubbler blanks to correct with, will hopefully be adopted. Criteria for subsequent bubbler blank checks is tentatively set at ± 20 pg from the mean.
7) Section 11.2 - BRL purges samples and standards for 15 minutes at 350 mL/min instead of 20 minutes at 300-400 mL/min. High level standard recoveries and subsequent bubbler blanks indicate that 15 minutes is adequate purge time to volatilize and collect all of the mercury. Longer purge times may, however, be necessary if the lab and/or sample temperature is <16° C.
8) Section 11.2.2 - BRL uses a minimum of 0.1 mL of $NH_2OH{\cdot}HCl$ per 0.5 mL of BrCl instead of 0.2 mL.

Brooks Rand Ltd.

Hg by Method 1631 (BRL modification #1)
Quality Control Chart Summary
11/20/97

Matrix Spike/Matrix Spike Duplicate (MS/MSD)

Dates		Average		Range		
From	*To*	*Recovery*	*St. Dev.*	*From*	*To*	*n*
2/28/97	11/7/97	98.1%	6.2%	85.6%	111%	151
			Method Criteria:	*75%*	*125%*	

Initial and Ongoing Precision and Recovery (IPR/OPR)

Dates		Average		Range		
From	*To*	*Recovery*	*St. Dev.*	*From*	*To*	*n*
2/27/97	11/7/97	98.9%	5.1%	88.8%	109%	108
			Method Criteria:	*77%*	*123%*	

Quality Control Sample (QCS)

Dates		Average		Range		
From	*To*	*Recovery*	*St. Dev.*	*From*	*To*	*n*
2/28/97	11/7/97	95.5%	6.5%	82.4%	109%	44
			Method Criteria:	*77%*	*123%*	

QCS Sample used is 1641c diluted to 7.35ng/L

Method Detection Limit (MDL)

	MDL	*ML*
Date	*(in ng/L)*	*(in ng/L)*
2/27/97	0.11	0.5
3/19/97	0.11	0.5
Method Criteria:	*0.2*	*0.5*

BROOKS RAND, LTD. (BRL)

Submitted 1/20/97

Method Validation Package (limited-use) for:

Total Mercury in Solids (sediments and biota) by CVAFS: SOP #BR-0002, revision 003, *Determination of Total Mercury in Solids by Cold Vapor Atomic Fluorescence Spectrophotometry (CVAFS).*

Previously submitted:

Summary of MDL, Precision, and Accuracy
SOP #BR-0002, revision 003
Data from Batches: 96-190
96-194
96-199
96-210
Published Papers/References: Liang and Bloom, 1993, (See Total Mercury in Water package)
Bloom, 1992, (See Methyl Mercury in Biota package)

BROOKS RAND, LTD. (BRL)

Submitted 1/20/97

Method Validation Package (limited-use) for:

Methylmercury in Water by CVAFS: SOP #BR-0011, revision 003, *Determination of Methylmercury by Aqueous Phase Ethylation, Trapping Pre-Collection, Isothermal GC Separation, and CVAFS Detection.*

Previously submitted:

Summary of MDL, Precision, and Accuracy
SOP #BR-0011, revision 003
Data from Batches: 96-184
96-215
96-218
96-280
Published Papers/References: Bloom, 1989
Horvat, Liang, and Bloom, 1993

BROOKS RAND, LTD. (BRL)

Submitted 1/20/97

Method Validation Package (limited-use) for:

Methylmercury in Sediment by CVAFS: SOP #BR-0011, revision 003, *Determination of Methylmercury by Aqueous Phase Ethylation, Trapping Pre-Collection, Isothermal GC Separation, and CVAFS Detection.*

Previously submitted:

Summary of MDL, Precision, and Accuracy
SOP #BR-0011, revision 003 (see Methylmercury in Water package)
Data from Batches: 96-202
96-214
96-223
96-233
Published Papers/References: Horvat, Bloom, Liang, 1993

BROOKS RAND, LTD. (BRL)

Submitted 1/20/97

Method Validation Package (limited-use) for:

Methylmercury in Biota by CVAFS: SOP #BR-0011, revision 003, *Determination of Methylmercury by Aqueous Phase Ethylation, Trapping Pre-Collection, Isothermal GC Separation, and CVAFS Detection.*

Previously submitted:

Summary of MDL, Precision, and Accuracy
SOP #BR-0011, revision 003 (see Methylmercury in Water package)
Data from Batches: 96-253
96-258
96-271
96-276
Published Papers/References: Bloom, 1992
Liang, Horvat, Bloom, 1994

Brooks Rand Ltd (BRL)

Arsenic & Arsenic Speciation Methodology Information

Method Numbers and Titles

BR-0020 Determination of Selenium and Arsenic in Environmental Samples by Hydride Generation-Atomic Absorption with Cryogenic Trap Preconcentration

BR-0021 Determination of Arsenic Species in Environmental Samples by Extraction and Hydride Generation-Atomic Absorption with Cryogenic Trap Preconcentration

Method Summaries

BR-0020 / Total As

Water samples are oxidized with the addition of $KBrO_3$/KBr/HCl and UV irradiation. Solid samples are digested with a 80:20 HNO_3:$HClO_4$ acid mixture in a sand bath. The samples were brought to volume with 15 mL of HCl and diluted to 50 mL with DI water. All samples are then reduced with NH_2 OH HCl and microwave heating. Samples are analyzed by hydride generation with $NaBH_4$, cryogenic trap precollection, H2/Air flame quartz furnace decomposition and Atomic Absorption detection (HGAAS).

BR-0021 / $As^{(III)}$, $As^{(V)}$, Monomethyl As, Dimethyl As

Water samples are adjusted to a pH of 6 with the addition of tris buffer solution. Samples are analyzed by hydride generation with $NaBH_4$, cryogenic trap precollection, H2/Air flame quartz furnace decomposition and Atomic Absorption detection (HGAAS). After water samples are analyzed for $As^{(III)}$ the same samples are then analyzed for $As^{(V)}$, monomethyl As and dimethyl As. HCl is added followed by the addition of a sodium borohydride solution which reduces the species to the corresponding arsines. The arsines are analyzed by hydride generation with $NaBH_4$, cryogenic trap precollection, H2/Air flame quartz furnace decomposition and Atomic Absorption detection (HGAAS).

Sediments/soil samples are sequentially extracted with a sodium acetate solution followed by HCl extraction and NaOH extraction. The combined extracts are then extracted with a benzene-potassium iodide-hydrochloric acid system. After extraction and back extraction, the arsenic species are separated from the sample matrices, and are then analyzed by hydride generation with $NaBH_4$, cryogenic trap precollection, H2/Air flame quartz furnace decomposition and Atomic Absorption detection (HGAAS). Biological Material is pretreated with KOH and then treated as sediment/soil samples (see above).

References

BR-0020

American Public Health Association (1989). "Standard Methods for the Examination of Water and Wastewater", 17th Edition.

Cutter, A. G. (1978). "Species Determination of Selenium in Natural Waters." *Analytical Chimica Acta,* 98:59-66.

Electric Power Research Institute. (1986). Volumes 1 and 2, "Speciation of Selenium and Arsenic in Natural Waters and Sediments", Research Projects 2020-1 and 2020-2, Final Report, EA-4641.

EPA. (1996). "Inorganic Arsenic in Water by Hydride Generation Quartz Furnace AA", EPA Draft Method 1632.

Liang, L., Danilchik, P., and Huang, Z.R. (1994). "Elimination of Dependence on Experimental Conditions in the Determination of Se in Water, Sediment, Coal and Biological Samples by Hydride Generation." *Atomic Spectroscopy,* July/August:151-155.

BR- 0021

Andrea, M.O. (1977). "Determination of arsenic species in natural waters." *Anal. Chem.* 49/6:820-823.

Braman, R.S., Johnson, D.L., Foreback, C.C., Ammons, J.M., and Bricker, J.L. (1977). "Separation and determination of nanogram amounts of inorganic arsenic and methylarsenic compounds." *Anal. Chem.* 49/4:621-625.

Electric Power Research Institute. (1986). "Speciation of Selenium and Arsenic in Natural Waters and Sediments, Volume 2, Arsenic Speciation", Research Projects 2020-1 and 2020-2, Final Report, EA-4641.

EPA. (1996). "Inorganic Arsenic in Water by Hydride Generation Quartz Furnace AA", EPA Draft Method 1632.

EMAX LABORATORIES, INC.

QUALITY ASSURANCE MANUAL
JANUARY 1998
REVISION 9

Approved By:

2/18/98

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TABLE OF CONTENTS

1. COMPANY BACKGROUND

EMAX Laboratories Incorporated is a full-service environmental analytical laboratory located in Torrance, California. Our services have been utilized by government and private sector clients since 1987. The company is a woman-owned, small business enterprise and is certified as such with numerous state and local governmental agencies.

The firm currently employs a full-time staff of over forty professionally qualified chemists and environmental scientists. Over twenty percent of our technical personnel possess advanced academic degrees. Management personnel have extensive experience in providing analytical services for environmentally-related projects. All personnel participate in a thorough training period upon the commencement of their employment and are proficient in following a variety of methodologies, including 40 CFR Part 136; US EPA SW-846; California Title 22, Division 4; California AB 1803 and 3374; US EPA Contract Laboratory Program and all NPDES requirements. Employees are also encouraged to attend educational seminars to keep abreast of the latest technologies. Analytical personnel are certified as proficient in specific methods before being allowed to perform analyses, and annual re-certification assures continued proficiency. EMAX management, laboratory client services, and other employees are available to assist clients at every stage of a project and will answer questions concerning data interpretation after completion of a project.

Our professionals are supported by the state-of-the-art analytical instruments (Appendix 1) and information processing system (Appendix 2). The laboratory is certified and evaluated by many states, government agencies and special programs to perform environmental services (Appendix 3). The laboratory is fully equipped to quantitatively identify environmental contaminants in soil, groundwater, waste waster, hazardous waste, sludge, oil and air. Analyses are performed using Gas Chromatography (GC) with a variety of detectors, Gas Chromatography/Mass Spectrometry (GC/MS), High Performance Liquid Chromatography (HPLC), Atomic Absorption Spectrophotometry (flame and furnace), Inductively Coupled Argon Plasma (ICP) Emission Spectrophotometry, Infrared Spectrophotometry (IR) and other instruments.

All the stationary equipment is housed in a 15,000 sq.ft. facility which is part of a larger business park. The floor plan of the laboratory appears in Appendix 4. There are also two mobile laboratories which are equipped to provide certain analytical services on site in the field.

2. QUALITY ASSURANCE MANAGEMENT

2.1. Scope of Quality Assurance Program

In accordance with the applicable guidelines from the California State Department of Health Services and U.S. Environmental Protection Agency (EPA)[1], EMAX has developed this QA Manual to define the authority and responsibility of involved individuals and establish the requirements by which the desired quality of equipment and services shall be achieved and verified. EMAX's policy is to assure that all data collected and processed through EMAX are scientifically valid, defensible and of known precision and accuracy. In support to this policy, EMAX has established a QA program which includes the following essential elements:

- To establish and maintain the line of authority and responsibility.
- To establish and maintain standard operating procedures which cover the entire laboratory operations from sample receiving to reporting.
- To establish and maintain an audit system which identifies problems at the earlier stages and provides direction for corrective action.
- To ensure all QC requirements are implemented by employees through extensive technical and procedural training.

The scope of EMAX's QA program describes the procedures involved in implementing these elements in the following areas:

- Organization and Responsibility
- Personnel Training
- Quality Assurance Objectives
- Sample Handling
- Chain of Custody
- Analytical Procedures
- Data Reduction, Validation and Reporting
- Quality Control Check
- Performance and System Audit
- Instrument Maintenance
- Corrective Action
- Material Procurement and Control

[1] Calif. Dept. of Health Services, Environmental Laboratory Accreditation Program, "Information Appendices" (1991) (as updated); QA/R-2, "EPA Requirements for Quality Management Plans" (Aug. 1994).

- Facilities and Equipment
- Security
- Software Quality Assurance
- Special Program/Project Requirements

2.2. Assessment of Quality Assurance Program

Assessment of QA Program shall be performed using the following guidelines:

- Effectiveness of the system as described by this manual.
- Adequacy of resources and personnel provided to achieve the quality objectives set forth by this manual.
- Review of existing system and identify opportunities for quality improvements based from QA reports to management, internal and external audits, performance evaluations and other functions related to quality assurance evaluation.

2.3. Management of Controlled Documents

It is the policy of EMAX to promote operation standardization; accuracy and precision in implementing the standards; the establishment of operation controls; and the systematic archiving of controlled documents when the same have been rendered obsolete. All controlled documents shall be processed as described in SOP GP-0013. Controlled documents shall include but not limited to, QA Manuals, SOPs, Laboratory Logbooks, MDLs, In-house QC Criteria, and other related documents that have similar importance.

2.4. Corporate Ethics Policy on Waste, Fraud and Abuse

It is the policy of EMAX Laboratories, Inc., to promote and maintain the integrity of all data generated in the conduct of laboratory business as well as to prevent any malpractice on behalf of its employees. Malpractice, as used in this corporate ethics policy, encompasses the improper manipulation or falsification of data, destruction or removal of data, or other staff misconduct which does or may cause waste, fraud, or the abuse of corporate assets. This includes the goodwill of the corporation's clientele as well as EMAX's physical assets.

If malpractice is suspected of having occurred, EMAX will conduct a thorough investigation, including gathering and securing all pertinent records related to the incident. A written incident report shall be prepared at the conclusion of the investigation, detailing the reason for suspicion, the conclusion of the investigators as to the existence or non-existence of malpractice, and if malpractice occurred, the cause, process and effect of the incident. All individual staff members responsible for malpractice incident shall be identified and their

respective roles shall be stated in the incident report thoroughly. The appropriate actions shall be taken by EMAX to uphold this policy under the circumstances of the particular case.

2.5. Discipline Measures For Employees and Other Agents Who Violate This Policy

Any employee or agent found to have engaged in malpractice is subject to disciplinary action up to and including termination of employment. For example, if an employee is found to have willfully and knowingly manipulated or falsified data to mislead EMAX management or its client(s), his or her association with EMAX may be summarily terminated. Lesser consequences may be imposed, depending on how critical the offense is to EMAX's business interests. In addition to internal disciplinary action, in appropriate cases, EMAX may bring civil or criminal charges against the employee or agent in the courts.

3. LABORATORY ORGANIZATION

3.1. Overview

Well defined personnel responsibility and accountability is necessary to ensure the highest quality of service. Within EMAX, each employee is personally responsible and accountable for the work he or she performs. However, the QA Manager had overall quality assurance responsibility concerning the laboratory operations and reports directly to the Laboratory Director.

The organization of the laboratory function is shown in Appendix 5-A and 5-B respectively. Professional qualifications and experience for the individuals filling these positions is maintained in the personnel and training records. The specific duties and responsibilities of the various managers and support personnel are summarized in the following sections.

3.2. Summary of Personnel Responsibility and Qualifications

3.2.1 Laboratory Director

Responsibility

- To establish the quality policy for all laboratories and perform assessment of the QA program.
- To establish and support a positive attitude and professionalism.
- To oversee and monitor the QA Manager in the execution and implementation of the QA programs.
- To assign, oversee and monitor the Project Manager(s) in execution of the program/project specific requirement.

Qualifications

- Education: Minimum of Bachelor's degree in chemistry of any science/engineering discipline.
- Experience: Minimum of five years of laboratory experience directly related to environmental testing, including at least three years of supervisory experience.

3.2.2 Laboratory Operations Manager

Responsibility

- To oversee the department supervisors in accomplishing the goals towards timely and cost effective production of quality data.
- To ascertain the reliability of laboratory data.

- To ensure that all laboratory personnel are qualified to perform their functions.
- To assess the load of the laboratory, its personnel and instrumentations, and ascertain that all samples received can be acted upon accordingly.
- To assist department supervisors in reviewing data packages whenever necessary.
- To take charge in the absence of the Laboratory Director.
- To attend to the functions of the Project Manager in his/her absence with respect to client coordination.

Qualifications

- Education: Minimum of Bachelor's degree in chemistry of any science/engineering discipline.
- Experience: Minimum of five years of laboratory experience in analytical field, including at least three years of supervisory experience.

3.2.3 QA Manager

Responsibility

- To establish and maintain QA and QC system to accomplish the stated quality assurance objectives.
- To monitor the state of quality assurance in the laboratory operations and the results of any corrective actions and recommendations of future improvement.
- To perform system audits to monitor completeness and effectiveness of QC system.
- To stop work in the case of a severe QA/QC problem.
- To monitor the training and technical review program in accordance with applicable Standard Operating Procedures (SOPs).
- To interact with Laboratory Operations Manager on matters pertaining to data integrity and quality.
- To support the laboratory operations manager, supervisors, group leaders, analysts, and technicians in addressing quality and/or training in their areas of operation.
- To authorize all issues dealing with data quality.
- To conduct and evaluate results from system and compliance audits.
- To establish and review standard operating procedures.
- To assess QC data to insure that the analytical systems are operating in a state of statistical control.
- To report the implementation of QA Program to the Company President on a quarterly basis.

Qualifications

- Education: Minimum of Bachelor's degree in chemistry of any science/engineering discipline.
- Experience: Minimum of three years of laboratory experience, including at least one year of applied experience with QA principles and practices in an analytical laboratory.

3.2.4 Project Manager

Responsibility

- To review the project specific QA Plan and other pertinent records related to the project.
- To disseminate project specific requirements to the laboratory during the course of the project.
- To maintain a line of communication and maintain documentation of all transactions with the client.
- To initiate request for variance whenever necessary.
- To report project status to the Laboratory Director and the Client's representative.
- To ensure that analytical process and its results conforms with the project specific requirements.

Qualifications

- Education: Minimum of Bachelor's degree in Chemistry or any science/engineering discipline.
- Experience: Minimum of three years of analytical experience including sample analysis, data validation, and QA activitities.

3.2.5 Department Supervisors

Responsibility

- To direct the operations of their respective Departments and accomplish the goal of timely and cost effective production of quality data.
- To support the QA Manager in the execution of all the procedures in their respective Departments.
- To monitor compliance to the project specific QA Plan and SOPs by the analysts and technicians through supervisor review and have the authority to reject data based on well defined QC guidelines.
- To train new personnel and make recommendation for continuing education as detailed in EMAX SOP GP-0009.

- To review the data for accuracy, precision, and completeness in accordance with standard operating procedures.

- To ensure all analyses are done on time.

Qualifications

- Education: Minimum of Bachelor's degree in chemistry or any science/engineering discipline.

- Experience: Minimum of three years of analytical laboratory experience , including at least one year of supervisory experience.

3.2.6 Analysts/Technicians

Responsibility

- To understand and operate in compliance with QC Program and standard operating procedures, utilizing good laboratory procedures.

- To identify problems that have a potential impact on data quality and to inform the appropriate individual(s) of such problems in accordance with established procedures.

- To submit all analytical data on time.

Qualifications for Analysts

- Education: Minimum of Bachelor's degree in chemistry or any science/engineering discipline or in lieu of minimum education requirement, two years experience in operating and maintenance in the related field of service.

Experience: Minimum of two years experience in related field of service, such as GC/MS, GC, ICP, etc.

Qualifications for Technicians

- Education: Minimum of high school diploma and a college level course in general chemistry.

- Experience: Minimum of one year experience of laboratory works.

3.2.7 Laboratory Information Manager

Responsibility

- To ensure that computing resources and information systems are current and reliable to support laboratory operation.

- To define the personnel requirements and responsibilities for the management, operation and maintenance of the Laboratory Information Management System.

Qualifications

- Education: Minimum of Bachelor's degree with advanced training in programming, information management, database management systems, or systems requirement analysis.

- Experience: Minimum of three years experience in data or systems management or programming including one year experience in laboratory information management system.

3.2.8 Data Processing Supervisor - QA Responsibility

Responsibility

- To ensure that all analytical data are in a format that is in compliance with clients' requirements.

- To oversee the data reporting process.

- To archive all documents pertaining to data processing.

- To ensure all reports are done on time.

Qualifications

- Education: Minimum of Bachelor's degree in management with advanced training in computer software applications.

- Experience: Minimum of three years experience in data processing including one year of supervisory experience.

3.2.9 Electronic Data Deliverables Supervisor

Responsibility

- To ensure that all samples received are properly logged in.

- To ensure that all EDDs are written in accordance to project specific requirements.

- To ensure that electronic data deliverables (EDD) are error-free prior to submittal.

Qualifications

- Education: Minimum of Bachelor's degree with advanced training in programming, information management, information systems, database management systems or systems requirements analysis.

- Experience: Minimum of two years experience in systems or applications programming including one year experience of data management and EDD generation.

3.2.10 Sample Management Manager

Responsibility

- To ensure all incoming samples are properly documented and stored.

- To supervise the creation and distribution of analytical folders in a timely manner.
- To keep track of the internal sample management.
- To inform project manager/customer liaison of any discrepancy or anomaly in samples and to follow up with any corrective action.
- To supervise the sample preparation section and implement the quality assurance program pertaining to this section.
- To dispose of samples according to SOP GP-0010.

Qualifications

- Education: Minimum of Bachelor's degree in chemistry or any science/engineering discipline.
- Experience: Minimum of two years of laboratory experience in sample management and sample preparation including at least one year of supervisory experience.

3.2.11 Health and Safety Manager

Responsibility

- To establish the policy on health and safety issues.
- To ensure all health and safety regulations are observed and reported to the Laboratory Director on issues of concern.
- To train new employees on safety procedures.

Qualifications

- Education: Minimum of Bachelor's degree in chemistry or any science/engineering discipline, with 40-hour training on Hazardous Waste Management.
- Experience: Minimum of one year experience in administering health and safety regulations.

3.3. Delegations in the Absence of Key Personnel

Planned absences shall be preceded by a written delegation of authority from the person whose function is described in Section 3.2 above, to another staff member. Delegations may be given to subordinate staff members whose qualifications and experience are similar. If no such staff member is available, the responsibilities and duties of the person will be delegated to the person's organizational superior as indicated in Appendix 5-B. In the case of unplanned absences, the organizational superior shall either assume the responsibilities and duties or issue a written delegation of these authorities to an appropriately qualified member. Any staff member carrying out another person's function pursuant to a delegation of authority shall attach a copy of the written delegation to any document that staff member approves on behalf of the absent person.

3.4. Laboratory Personnel Qualification and Training

All personnel joining EMAX organization shall undergo Orientation and Training as described in SOP GP-0009. During this period the new personnel shall be introduced to the organization, their roles, responsibilities and authorities as well as the policies and procedures of the company. They shall also undergo on the job training and initial demonstration of proficiency prior to performing their work assignments on their own. To ensure a sustained level of quality performance among EMAX staff members, continuing demonstration of proficiency shall be performed at least once a year by successfully passing an external PE sample or internal PE sample. Personnel QA record, shall include, resume; signed Corporate Ethics Policy on Waste, Fraud, and Abuse; Safety & Health Orientation, Training Records; Demonstration of Proficiency; as well as training performed outside EMAX. These records are filed in individual binders and are maintained by the QA Manager.

3.5. Subcontractors and Contract Labor Personnel

EMAX by all means, prefers to perform all analytical work in its own facility with its own staff members. However, service demands sometimes exceeds EMAX's capacity or are beyond EMAX's analytical capability. In such instances, EMAX may add contract labor personnel to its work force or seek out other laboratories to perform subcontracted work.

As part of EMAX's policy, Subcontractors shall only be awarded work upon client's written approval. In selecting a proposed subcontractor, EMAX shall determine that capability to do the work exists, the same certifications, approvals, and evaluations required of EMAX are available at the proposed subcontractor, and an adequate QA Manual governs the proposed subcontractor's laboratory operations. Upon request, EMAX shall provide its client copies of the proposed subcontractor's applicable certifications, approvals, and evaluations and its QA Manual. EMAX's QA Manager shall maintain such documents on file for each subcontractor approved by an EMAX client and shall provide the name of the subcontractor's point of contact, phone number, fax number, and the subcontractor's address to interested EMAX and client staff member upon request.

Utilization for subcontract laboratories policies and procedures are detailed in SOP GP-0005.

Contract labor personnel are required to meet the same qualifications criteria as EMAX's own staff engaged in the same work and are required to undergo the same training as EMAX's own staff.

4. QUALITY ASSURANCE OBJECTIVES

The purpose of a quality assurance program is to produce data of known quality which satisfy the project objectives and meet or exceed the standard requirements for the analytical methods that generated the data. Data whose known quality is substandard and/or does not satisfy project objectives will not be relied upon by EMAX for any purpose.

As reflected by controlled document work instructions issued to EMAX personnel, the QA program: (1) provides measures of data quality in terms of precision, accuracy, representativeness, completeness and comparability and (2) provides a mechanism for ongoing control and evaluation of data quality.

4.1. Precision

Precision measures the reproduction of repetitive measurements. It is strictly defined as the degree of mutual agreement among independent measurements. Results from sample duplicate analyses provide a measure of precision. Precision can be expressed in terms of relative percent difference (RPD) using the following formula where D2 is the concentration of one of the replicates and D1 is the concentration of the remaining replicates:

$$RPD = \frac{D2 - D1}{(D2 + D1)^{1/2}} \times 100$$

Duplicate samples will be prepared at a minimum of one per analytical batch. A batch is defined as a group of samples of similar matrix (i.e., a matrix which behaves similarly with respect to the procedures to be employed in analysis). They are processed together with the same method sequence, same lots of reagents and subject to manipulations common to each sample within the same time period or in a single continuous time sequence without interruption. The maximum number of environmental samples in one batch is 20.

The laboratory precision objectives of each method and matrix are presented in individual SOPs concerning the method.

4.2. Accuracy

Accuracy is a statistical measurement of correctness and includes components of random error (variability due to imprecision) and systematic error. It therefore reflects the total error associated with a measurement. Analytical accuracy is expressed as the percentage recovery (%R) of an analyte which has been used to fortify a field sample or blank at a known concentration prior to analysis, an is expressed by the following formula:

$$\%R = \frac{Amount\ Recovered}{Amount\ Spike} \times 100$$

One (1) sample out of twenty (20) or one in every analytical batch of less than 20 samples is spiked with appropriate concentrations of analytes in order to determine the matrix effects

and recoveries. The laboratory accuracy objectives of each method and matrix are presented in the SOP concerning the individual method.

4.3. Representativeness

Representativeness of data with respect to the site conditions is significantly dependent upon field activities that are outside the control of the laboratory. However, EMAX endeavors to assure the representativeness of data with respect to the sampling and analysis tasks that are within the laboratory's control. Objectives for this representativeness will be defined in the SAP as a result of DQO development and will reflect the investigative objectives for each particular task. In all instances other than VOA analyses, samples will be thoroughly mixed before preparation to ensure the representativeness of a subset. Furthermore, the participation of all technical disciplines involved in producing and using sampling and analysis data is mandatory at the planning and DQO development stages of work to ensure that appropriate objectives are selected for inclusion in the SAP.

4.4. Completeness

Completeness is defined as the percentage of measurements made which are judged to be valid. Completeness is calculated for the aggregation all methods of any particular sampling event or other defined set of samples. The number of valid, unqualified results. divided by the number of possible individual analyte results, expressed as a percentage, determined the completeness of the data set. The objective for completeness if 90 percent for most projects unless specified otherwise for specific project needs.

$$Completness = \frac{Number\ of\ valid\ results}{Number\ of\ possible\ individual\ results}$$

4.5. Comparability

Comparability is a qualitative parameter expressing the confidence with which one data set can be compared with another. It depends significantly on the on-site sampling plan which is generated and executed outside the control of the laboratory. However, EMAX endeavors to assure the greatest degree of comparability possible for data sets generated under the control of the laboratory. In this regard, EMAX employs established methods for sampling and analysis, reporting data in standard units and comprehensive reporting format, and using reagent and standards of the highest quality available. Reference standard solutions used in analytical processes will always be traceable to the National Institute of Standards and Technology (NIST).

4.6. Control of Data Quality

EMAX personnel shall not employ non-standard or significantly modified methods [e.g., work performed under instructions not sanctioned by EMAX controlled documents] unless the client approved the methods in writing after full disclosure of the non-standard or modified aspects of the method(s). The project manager shall not request the client's approval unless the QA Manager has first determined that the non-standard or modified method: (1) is supported by in house data or literature, (2) is within the scope of governing regulations (if any) of appropriate agencies. All this information shall be provided to clients as part of the disclosure accompanying a request for approval. A copy of the client approval shall be kept in the project file.

The company uses SOPs with Method Detection Limits (MDLs), Reporting Limits, and In-house QC Schedule and Criteria (IQCs) to communicate the implementation of data quality objectives to the analytical staff (Refer to Appendix 6 for SOP Index). These are controlled documents issued by the QA Manager to related individuals and recalled from such persons when revisions make the documents obsolete. (Refer to SOP GP-0013 for detailed information concerning the control of documents).

5. SAMPLING

Sample testing methods and holding times must be strictly observed. Recognizing this imperative need, EMAX has adopted policies and procedures governing all aspects of sample handling, processing, and disposal and requires its personnel to comply with such policies and procedures. Additional policies and procedures govern internal EMAX systems for monitoring and reporting on adherence to sample handling, processing, and disposal requirements. The QA Manager shall provide assistance upon request in identifying pertinent controlled documents which establish work instructions on these topics.

5.1. Collection and Preservation

EMAX prepares properly cleaned and preserved bottle containers for use in sample collection according to the published EPA guideline. Appropriate containers, sample volume and preservatives for routine organic, metal and wet chemistry parameters are summarized in Appendix 1 of GP-0001, Required Sample Containers and Preservatives.

5.2. Sample Receipt

5.2.1 Sample Receipt is the responsibility of the Sample Custodian (SC). Date and time of sample receipt is acknowledged by the SC on the chain-of-custody form (Appendix 8). The SC may designate an alternate person to fulfill his duties when he is absent.

5.2.2 Each sample received will be inspected by the sample custodian. Any anomaly will be recorded. The inspection will include:

- If containers are intact;
- If there was no headspace in VOA vials;
- If the sample holding time has not exceeded;
- If the sample was received within 4°C + 2 °C;
- If the custody seals or tapes on the shipping containers are present and intact;
- If preservatives are present;
- If there was any discrepancy between Chain-of-Custody and sample received.

The sample custodian will fill out sample receipt and description form (Appendix 9) and will notify client services of any problem. In turn, client services will notify the client of any violations in holding time, preservatives or storage requirements. Such violations, even approved by the client, will be noted in the analytical report.

5.2.3 All samples are logged into the Sample Receipt Record when they are received. A unique EMAX accession number is assigned to the analytical batch, and a sample ID number is assigned to each sample within the analytical batch. The storage code is assigned. The EMAX reporting batch accession number and sample number are designated as follows:

YYMSSSNNQQ

YY = Last two digits of the current year, i.e. 96- 1996

M = Calendar month in alphabetical order, i.e. A = Jan,

B = Feb, etc.

SSS = Reporting batch number, assigned in ascending order

from 001 each month

NN = Individual sample numbers assigned in ascending order

within each analytical batch, from 01 through 99

QQ = QC designated by client

The client's name, sample ID, matrix, date received, analyses required, and storage locations are also listed in the receipt record. A file is created for all paperwork.

5.2.4 Work-order-forms of each analysis will be distributed to the management and the appropriate analyst. These forms provide the necessary information needed to perform the analysis. Unless QC sample is designated, it is the responsibility of a chemist/extraction supervisor to assign QC samples for the analytical batch in accordance with project specific requirements.

5.2.5 Laboratory Information Management System

EMAX operates an in-house Laboratory Information Management System (LIMS) which serves as a central point to manage all data related to sample status and report deliverables and disseminate these information to responsible parties. Key components of the EMAX LIMS are LABWORKS, a PC network-based commercial LIMS package, and SAMPLETARCK, an in-house sample tracking system which interfaces with and complements LABWORKS.

5.2.5.1 Sample Log-In

Sample log-in is performed on the LABWORKS system by a designated data processing personnel. The process involves checking a sample or group of samples into the system, recording field data, and specifying analyses and reporting details. Specific information logged in include:

- client sample ID and corresponding control number
- sample collection and receiving date/time
- sample matrix and analysis requested

- holding time and internal due date
- QC sample information

LABWORKS has login and password control, and it has different levels of module privileges to achieve system security and data integrity. Different laboratory personnel are given different access privileges to control operations that can be performed on a given sample.

5.2.5.2 Sample Tracking

SAMPLETRACK provides the environment and the operations to monitor the status of samples as soon as they are received, as they progress from one stage of laboratory operation to another, until they are reported. Sample preparation technicians and analysts receive their work orders, update the relevant information as their assignment is finished, and browse these status information in SAMPLETRACK. Data entry personnel will prepare the final reports according to their due dates and update the system when the report is complete.

The LIMS is an essential management tool for the laboratory operations manager and supervisors to have up to date information regarding the status of each project. Holding time information is stored in the LIMS for the different methods. The user is alerted of holding time requirements by looking at the SAMPLETRACK. For any violation of holding time, the client will be notified promptly and follow up action will be noted in the analytical report.

5.3. Sample Storage

Samples will generally be stored in refrigerators maintained at 4°C (± 2 °C) to reduce the possibility of sample contamination and degradation. Liquid and soil samples designated for volatile analyses are stored separately. Aqueous samples for the determination of metals shall only be stored at 4°C (± 2 °C) at client's request.

5.4. Internal Sample and Extract Custody

When samples are taken out to be prepared or analyzed, the Sample Custodian will transfer custody to the responsible party. The transfer of custody is recorded in the internal COC log and verified by the signature of both the Sample Custodian and the analyst. When sample preparation or analysis is finished it is returned to the sample storage, the analyst must transfer custody back to the Sample Custodian who will return the sample to the refrigerator. This transfer is also indicated on the internal chain-of-custody record logbook by the signature of the analyst and the Sample Custodian. The date and time of each transfer must also be shown on the internal chain-of-custody.

When sample extracts are ready for analysis, the custody of extract is transferred to the analyst, and the recipient initials the extraction log. The analyst will keep the custody of the extract until endorsed for disposal.

5.5. Sample Disposal

After completion of the analysis, the sample will be returned to its original storage place and kept for 30 days. Before actual disposal, the client will be notified of EMAX's intention to dispose of the sample. Upon the client's request and with the approval of the laboratory management, the sample may be stored for a longer period of time either at no charge or for a fee. The laboratory is registered with the EPA as a Hazardous Waste generator. Waste disposal for the laboratory is contracted to a qualified waste disposal company. At the time of disposal, the laboratory will separate samples according to hazardous material classification code and dispose of them according to SOP GP-0010 and in compliance with applicable federal, state, and local hazardous waste regulations. In the event that the project has a different sample disposal/handling specification, the project specification shall prevail.

6. MATERIAL PROCUREMENT AND CONTROL

Material procurement and control procedures ensure quality control of chemicals and any related materials essential to the laboratory operation. Purchase orders will specify the quality of material needed. Such specifications may include purity, requirements for traceability or certifications, lot number or any other pertinent information.

6.1. Procurement and Quality Control

6.1.1 All reagents/chemicals and related materials needed for analyses are pre-analyzed prior to placing any order. A requisition for sample(s) is placed with the supplier to pre-qualify the chemicals.

6.1.2 All reagents/chemicals and related materials purchased are logged as they are received. A reagent log shall be maintained to contain the following:

- RL Number
- Date received
- Description
- Lot Number
- Comments

6.1.3 All reagents/chemicals and related materials shall be analyzed per lot prior to use to ensure that no interference exists as required by the analyses. The detailed procedure and acceptance criteria are based on the intended use of reagents and is described in SOP GP-0025. Documentation of such analyses shall be maintained by QA department.

6.1.4 Prior to use, the lot number of reagents/chemicals and other related materials shall be verified as corresponding to the latest lot analyzed. All reagents/chemicals and related materials shall be dated when first opened.

6.1.5 All reagents/chemicals that are expired as specified by the label and/or chemicals that did not pass the QC requirements shall be disposed properly as described by the waste disposal procedures.

6.2. Chemical and Standard Inventory

6.2.1 Chemical Inventory

The supplies (reagents/chemicals/general laboratory supplies) requisitions and inventory system is maintained by the Purchasing Officer with the cooperation of the end users.

6.2.2 Standard Inventory

The chemical standards are controlled by each department. All standards are purchased from vendors of known integrity. All standards purchased are either accompanied with certificate of analysis or traceable to a documented source. To ensure availability of the standards used in the Organic Department, a purchase order is placed prior to the release of the last container of the standard in inventory for consumption in the laboratory. For the Inorganic Department, a purchase order is placed for the projected requirements at least a month prior to its expected consumption. The detailed description of tracking and maintaining quality control of standards can be found in SOP GP-0020 and GP-0023.

6.3. Storage Control

Chemical storage is strictly governed by compatibility considerations and by concerns for maintaining the integrity of the stored chemicals.

6.3.1 All solvents shall be stored in flammable liquid safety cabinets in a well ventilated area.

6.3.2 All acids shall be stored in a non-metallic cabinet.

6.3.3 All reagents shall be stored in appropriate cabinets, systematically observing chemical compatibility criteria.

6.4. Laboratory Waste Disposal

6.4.1 Chemical Wastes

Chemical wastes are properly identified and are temporarily stored in drums seated on spill containment pallets, awaiting for disposal.

6.4.2 Organic Waste Waters

Organic waste waters generated from samples that are extracted are boiled under fume hoods for at least two hours or purged with nitrogen gas prior to its disposal as non-hazardous waste.

6.4.3 Inorganic Waste Water

Inorganic waste waters generated by digestate and/or distillates are neutralized to pH 7 prior to its disposal as non-hazardous waste.

6.4.4 Organic Waste Solids

Wastes soils generated after solvent extraction are placed in fume hoods for at least one week prior to its disposal as non-hazardous waste.

6.4.5 Sample Disposal

Samples are generally stored for at least 30 days from date of receipt unless otherwise specified by the client and/or by the project

Samples are classified and a profile is generated by the laboratory to identify the waste stream.

6.4.6 Hazardous Waste Disposal

All wastes generated by the laboratory and identified as hazardous are so labeled and packaged appropriately for pick up by the contract waste disposal company for proper disposal.

6.4.7 Effluence Discharge

Wastewater effluent from EMAX's Torrance laboratory is discharged under the jurisdiction of the Los Angeles County Sanitation District. File correspondence with the District states that no additional permitting or monitoring of the EMAX discharge is required.

EMAX's Internal Hazardous Waste Management Plans specified procedures to minimize the potential for hazardous materials entering the sanitary sewer collection system.

7. ANALYSES

7.1. Analytical Procedures

The methodologies and procedures for analysis set forth in various documents are used by EMAX in the analysis of soil, water, and air samples. These documents are issued by various government regulatory agencies for regulatory concerns. Unless otherwise informed, EMAX will use the most recently promulgated version of the method. When applicable, EMAX will discuss the availability of different versions with the client to ascertain their need, and this information will be included in the project-specific notes. The source documents are:

- "Test Methods for Evaluating Solid Waste, Physical/Chemical Methods", USEPA SW846, Third Edition, 1986 (as updated)
- "Methods for Organic Chemical Analysis of Municipal and Industrial Wastewater" (EPA-600/4-85-054) (as updated)
- "Methods for Analysis of Water and Wastes", EPA 600/4-79/020 (as updated)
- "Method for Determination of Organic Compounds in Drinking Water" (EPA-600/4-88/039) (as updated)
- "Statement of Work for Organic Analysis", USEPA Contract Laboratory Program OLM01.8, Aug. 1991 (as updated)
- "Statement of Work for Inorganic Analysis", USEPA Contract Laboratory Program, ILM02.1, Sept. 1992 (as updated)
- Title 22, Article 11 of the California Administrative Code, "Criteria for Identification of Hazardous and Extremely Hazardous Wastes"
- Guidelines Establishing Test Procedures for the Analysis of Pollutants Under the Clean Water Act", 40 CFR, Part 136 (Federal Register 49 (209), 26 October 1984) (as updated)
- "Annual Book of ASTM Standards", Volume 4.08 (as updated)
- "Methods for Determination of Organic Compounds in Drinking Water (500 Series)", PB-89-220461/AS (as updated)
- "The LUFT Field Manual", October 1989, California (as updated)
- Alaska Methods & Alaska Regulations 18 AAC 7.800 (b)
- "EPA Compendium of Methods for the Determination of Toxic Organic Compounds in Ambient Air" EPA/600/4-89/017 (as updated)
- "Methods for Determining Emissions of Toxic Air Contaminants from Stationary Sources". State of California Air Resources Board, 1991.

A list of analytical methods performed by EMAX is summarized in Appendix 7.

The choice of method depends on the data quality objectives, regulatory program and sample matrix. The operation of each method is detailed in an SOP which provides work instructions concerning the sample preparation, instrumentation, analysis, performance criteria and corrective action. Adherence to the SOP is essential in producing consistent data of known quality. Deviation from standard operation procedures due to the nature and composition of the sample must be documented. The SOPs are reviewed annually and updated, if necessary, to reflect the current practice. Controlled copies of the SOPs are issued for easy access by related personnel. An index of SOPs issued before the revision date of this manual is provided in Appendix 6-A, 6-B, 6-C.

EMAX analytical personnel has to demonstrate proficiency and are certified for every analysis that they perform as described in GP-0009. Under GP-0013, a control system is established to ensure that analytical personnel do not retain obsolete versions of methods documentation and do have the most current revision of such documentation.

7.2. Detection Limits

EMAX's policy and practices for defining instrument detection and method detection limits, reporting limits, and quantitation limits, as well as the method(s) and algorithm(s) used to determine such limits, are set forth in SOP GP-0026.

7.2.1. Instrument Detection Limit (IDL)

The operation definition and procedure of IDL are taken from USEPA Statement of Work for Inorganic Analysis ILM02.0 (as updated) and applied only to instruments for metal analysis. It is determined by taking 7 measurements per day for three non-consecutive days at a concentration of 3 to 5 times the manufacturers recommended instrument sensitivity. IDL is calculated by multiplying 3 to the standard deviations obtained from these measurements. The IDL is updated quarterly if CLP samples are analyzed and after major instrument repairs.

7.2.2. Method Detection Limit (MDL)

The method detection limit (MDL) is defined as the minimum concentration of a substance that can be measured and reported with 99 percent confidence that the analyte concentration is greater than zero. The laboratory performs MDL studies annually to demonstrate that it can meet or exceed the method recommended MDLs and project requirements.

EMAX procedure of performing MDL is adopted from the "U.S. EPA Procedure Used for Establishing MDLs and Procedure for the Determination of the Method Detection Limit - Revision 1.1", 40 CFR 136, 1984 (as updated). This procedure consists of analyzing seven aliquots of a standard spiked at three or five times the expected MDL. Then, the aliquots are taken through the entire sample processing steps of the analytical method. The MDL is established at three times the standard deviation of the mean value for the seven analyses. Results for similar methods performed for each matrix will be applied to all methods. MDL(s) for analytical methods listed in Appendix 6 is available upon request.

7.2.3. Reporting Limit (RL)

Reporting limit (RL) is used for reporting purpose. It is set at a level where the precision and accuracy are assumed to be defined and it must be greater than or equal to the empirically derived MDL. The QA Manager determines the validity of laboratory method reporting limits. Reporting limit is project specific.

7.3. Standards

7.3.1 Standard Storage

Standards are stored according to the EPA or manufacturer recommended temperature in either subzero, 4 °C (± 2°C) or room temperature (22 ± 2°C). Standard storage is organized so that possible standard cross contamination is avoided. Pure or high concentrated standards are stored away from the low concentration dilution standards. Maximum shelf life of standard is less than or equal to manufacturer's expiration date for neat compounds or pre-mix solutions. On occasions, the expiration date of standard can be extended upon validation with certified source. Expiration of secondary standards must meet or exceed the proposed standard expiration criteria for the specific method of analysis. Diluted standards will be discarded when analysis indicated decomposition.

7.3.2 Standard Verification

Before using a new standard, its acceptable condition must be verified through analysis which compares the new standard to a standard known to be in acceptable condition. The acceptance criteria of standard for each method is listed in method specific QC schedule in the SOP. Once the standards is verified, it may be used for routine analysis.

7.3.3 Standard Logbook

All standards used for analytical processes by EMAX will be logged in the standard logbook according to SOP GP-0023 with the following information:

- Source of Standard (Manufacturer's Name)
- Expiration date of source standard
- Date of standard arrival, date when standard first opened
- Lot number or analytical batch number of the standard
- Standard name and original concentration
- Date of dilution
- Solvent used for dilution
- Concentration value after dilution
- Where standard is stored
- Identification code for diluted standard solution

- Any unusual observable characteristics

7.3.4 After log-in, an ID associated with the standard is generated. The standard ID is used in all analytical documentation to provide traceability and ensure that the process being documented employed a standard.

7.4. Method Development

When methods are revised, updated or modified, a new procedure shall be developed as a standard practice in the laboratory.

If the method contains definitive criteria, the specified criteria shall be used without modification. Clarification from the method originator may be required on areas where the criteria is subject to interpretation.

If the method contains only guidelines, the criteria shall be defined using the guidelines provided, the result of the method development and the objective of the method.

If the method lacks criteria, the criteria shall be defined using the similar in-house method as advisory control limits, until enough data points are accumulated. Refer to SOP GP-0030 for generating control limits.

8. DATA MANAGEMENT AND LABORATORY RECORD

The data reduction, review, validation and reporting procedures described in this section will ensure the data validity, that complete documentation is maintained, that transcription and data reduction errors are minimized, the data are reviewed and documented, and results are properly qualified. If data is generated by an EMAX subcontractor, it must be verified and reviewed to the same extent as internal data before EMAX accepts the data. A flow diagram of data management is presented in Appendix 10, and the procedures are presented in SOP-GP-0009.

All analytical data produced within EMAX or by EMAX subcontractors are extensively reviewed prior to report generation to ensure the validity of the reported data.

8.1. Data Generation

Analytical data are generated either manually or electronically from instruments. Manual data are recorded into bound laboratory notebook issued by the QA Department. Notebook pages contain the following information, as applicable: analytical method, analyst, date, sequential page number, reagent concentrations, instrument settings and raw data. Entries for instrument logs are in chronological order. Notebooks are maintained in accordance with SOP GP-0013. The notebook is reviewed periodically by the QA Manager/Supervisor. For data generated electronically, the data and a copy of the run-log (with method name, instrument ID, date, data file ID with associated sample control number and other pertinent information) are kept on file. Instrument outputs (quantitation reports, chromatograms, etc.) are also kept on file. The filing of instrument data is based on the method and EMAX control number.

8.2. Data Reduction

8.2.1 Data reduction is performed by the individual analysts and consists of calculating concentrations in samples from instrument output. The complexity of data reduction efforts depends on the specific analytical method and the number of discrete operations (e.g., extractions, dilutions, and concentrations) involved in obtaining a sample that can be measured. The analyst will reduce all raw data into the final reportable value with the appropriate data qualifiers. Analysts apply their professional judgment in manual integration.

8.2.2 Manual integration shall be initialed, dated, and properly explained. The original hard copy and the integrated result shall be retained and archived with the raw data.

8.3. Data Review

Every data package generated shall undergo levels 1, 2, and 3 data review.

8.3.1 Level 1

8.3.2 The analyst, who generated the results, performs the first level of review and is responsible to report the results accurately and completely. It is also the analyst's prime responsibility to generate and reduce the data in accordance to the specified protocols set forth by the laboratory SOPs. The Level 1 review guidelines includes but not limited to the following:

- Completeness of information from sample preparation to analysis
- Correctness on execution of SOP
- Correctness of quantitation of analytical results
- Completeness of analytical results, to include case narrative, method blanks, LCS, MS/MSD and/or duplicate samples, sample results accounted from the COC, calibration results, raw data, NCR (if any) and any other pertinent record(s) that in the analyst's professional judgment deemed necessary to enclose be included as part of the record.
- Correctness on implementation of control limits
- Correctness on project specific requirements
- Completion of analytical checklist to include the analyst's signature on Level 1 review of the checklist. The analyst shall then compile the analytical report into an analytical folder and submit the folder to the supervisor for technical review.

8.3.3 Level 2

The supervisor or his/her designee, performs the 100% second level of review. The primary function of this review is to ensure that results are scientifically sound and accurate. It shall also counter check whether or not the analytical checklist conforms with the contents of the analytical folder. Level 2 review guideline includes, but not limited to, the following:

- Calibration data are scientifically sound, appropriate for the method, and completely documented
- QC samples are within the established guideline
- Qualitative identification of sample component is correct
- Quantitative results are accurate
- Completeness of the analytical folder

During secondary level of review, it is essential to make immediate action to correct any errors, and at the same time, to investigate the root cause of the error. Once the deficiency is identified, a corrective action plan shall be implemented to prevent recurrence of similar incidence. Errors that potentially may have a great impact on the project will be reported to QA Management/project manager immediately. The project manager, after investigation, informs the client and recommends remedial action. The final resolution will be documented in the project file.

This review process is documented by the signature of the reviewer and the date of review in the analysis review form.

After the secondary review, the data are compiled into a final report by the Data Processing Department and the department supervisor reviews for transcription error as well as the completeness of the data package.

8.3.4 Level 3

The Project Manager (PM) performs the third level of review. The primary function of this review is to ensure that the analytical process and its results conform with the project specific requirements. In addition, the PM shall also review the overall quality of the data package. The Level 3 review guideline includes, but not limited, to the following:

- Analytical method used conforms with the project specific requirements
- Detection Limits conforms with the project specific requirements
- Data packaging conforms with the project specific requirements
- Completeness on all of the deliverables

After satisfactorily meeting all of the above requirements, the PM shall initial the draft and returns the package to the data processing department for final pagination.

Subsequently, the data package goes to the Laboratory Director. The Laboratory Director reviews the case narrative of each method before signing off.

The data processing department reviews all generated copies of the data package to ensure readability and completeness on the data package replicates.

8.3.5 QA Review

The QA Manager or his/her designee randomly reviews at least 10% of the generated data packages to ensure that the data generated meets the laboratory QA objectives. The examiner shall perform all of the three levels of reviews mentioned above. The QA Manager shall document the review accordingly.

The purpose of QA review is to ensure that all quality control procedures are properly implemented and project specific requirements are met; it also provide an opportunity to evaluate the effectiveness if the QA program and make recommendation for improvement.

8.4. Data Reporting

A variety of reporting formats ranging from computerized data tables, to complex reports discussing regulatory issues, to a CLP-deliverable package, are available. In general, a standard report consists of a transmittal letter with the following information:

8.4.1 Case Narrative

This form contains the description of sample types, tests performed, the reference for the analytical method, and any technical problems encountered during analysis.

8.4.2 Sample Result

This form contains results and reporting limits of samples with client's ID and EMAX's control number in a format required by the project. Results and reporting limits are calculated base on dilution factor and/or dry weight. Additional pertinent information, to include dates sampled, received, prepared, extracted and/or analyzed is also provided on this form.

8.4.3 Laboratory Performance QC Information

The result of a method blank, the precision and accuracy of laboratory control sample and laboratory control sample duplicate are summarized in this section together with project acceptance limits.

8.4.4 Matrix-Specific QC Information

This form contains the results of sample duplicates, matrix spikes, matrix spike duplicates or other project-specific QC required by the contract. The results include supporting information such as amount spiked, and percent recovery or relative percent difference.

8.4.5 Other Deliverables

Other deliverables required by the client which may include electronic disk deliverables, sample raw data packages, and custom report formats shall also be provided as required by the contract.

8.5. Record Keeping

Each analyst or technician is responsible for maintaining accurate, legible records and logs in accordance with the SOPs outline in SOP No. GP-0013. The supervisors are responsible for insuring adherence to the SOP. The QA Manager is responsible for routinely auditing all records and logs and reporting deficiencies to the appropriate supervisor for corrective action.

The extraction sections utilized method-specific bound books to record all data associated with sample extraction and preparation. A copy of the extraction log is transferred to the respective analytical area to process the extracted sample.

Analysis-specific laboratory notebooks are issued to the various groups as needed. The notebooks are sequentially numbered and the inclusive dates recorded on the cover. They are maintained within the group until they have been completed. Competed laboratory notebooks are returned to the QA department for appropriate archiving.

EMAX maintains specific analytical batch folders for all data (including computer print out, quantification reports and chromatographs, extraction log, and analytical log). All analytical folders are archived chronologically by the data processing department in a secure area. All analytical records are kept in a secured storage for a minimum of three years. Upon client

request and subject to managerial approval, analytical records can be stored for a longer period of time.

8.6. Electronic Media

Back up and archival methods are defined for all data files. Backup data are used to recover data when files have been destroyed. All analytical files are periodically copied on tape. Archived data shall be kept permanently for future reference. EMAX normally keeps all electronic files for three years, unless specified in the contract or pre-arranged by the client. The details of storing electronic data is described in SOP SP-5010.

9. QUALITY CONTROL CHECKS

This manual defines a batch as a group of samples of similar matrix, not to exceed 20 original field samples, analyzed for a specific method. Each batch shall include the analysis of a method blank and a laboratory control sample. For organic analyses, it shall also include a matrix spike and a matrix spike duplicate. For inorganic analyses, it shall also include a matrix spike and a duplicate sample.

In order to asses the validity of an analytical result, quality control elements are introduced into the entire analytical process. The following subsections describe the quality control parameters used by EMAX. By examining the completeness and quality of all QC parameters, method performance can be assessed and the validity of analytical data can be evaluated. The applicable QC procedure(s), the frequency of each procedure(s), the acceptance criteria, and the corrective action are summarized for each analytical method in a QC table of each SOP as a quick reference source for laboratory personnel. The QC acceptance criteria for each methods are either derived from the reference or established based on statistical consideration and performance requirements. QC tables for specific analysis is available upon request.

9.1. Calibration

Calibration defines the quantitative response, linearity and dynamic range of the instrument for each analyte. Calibrations that are used in analyses can be classified into two categories: Initial and Continuing Calibration. Initial calibration establishes the calibration range of the instrument and determines the response over that range. Continuing calibration provides a measure of the instrument stability over a period of time by comparing to initial calibration. All analytical instruments shall be calibrated in accordance with requirements which are specific to the instrumentation and procedures employed.

9.1.1 Organics by Chromatography

Chromatography involves a variety of instrumentations and detectors. The commonly used systems includes, GC/MS (Gas Chromatography-Mass Spectrometry), GC (Gas Chromatography), and HPLC (High Performance Liquid Chromatography). Though the sensitivity of each system differs, they all aim at identifying target analytes with specific means of separation and detection. An initial calibration curve of target analytes using known standards defines the working range. Concentration levels and acceptance criteria are determined in each method.

The continuous calibration is evaluated based on the method specific criteria at designated intervals in predetermined concentration to ensure the system is within control. If the continuing calibration does not meet the established criteria, corrective action shall be initiated. This may involve examination of instrument performance, verification of standard, recalibration or re-analysis of affected samples.

9.1.2 Metals by ICP, AA

Metals are analyzed by two types of instruments: Inductively Coupled Argon Plasma Emission Spectroscopy (ICP) or Atomic Absorption Spectroscopy (AA). Each instrument is calibrated prior to any analyses. A calibration curve is prepared daily

using criteria prescribed in individual method. The calibration is then verified using a standard prepared from an independent source (Initial Calibration Verification (ICV)). The calibration is monitored through the analytical process by analyzing a continuous calibration verification standard at designated intervals. If the verification does not meet the QC criteria, corrective action is initiated. This may include examination of instrument performance, standard preparation, analytical procedures, recalibration or reanalysis of sample back to the previously acceptable calibration check.

9.1.3 Wet Chemistry

The field of classical non-metal inorganic analysis involves a variety of instrumentation and technique such as spectrophotometric, chromatographic, electrometric, gravimetric, titrametric. While calibration or standardization procedures depend on the type of system required for a specific method, the general principals involve defining the working range by standards. The calibration is checked on a predetermined interval to ensure that the system is remained in control and all measurements are derived from consistent source. If the on-going calibration check does not meet QC criteria, corrective action may follow. The corrective action procedures include examination of instrument performance, external calibration check, recalibration or reanalysis.

9.2. Blanks

Blank is an artificial sample designed to monitor the introduction of contamination into the process. For aqueous samples, reagent water is used as a blank matrix; however, a universal blank matrix does not exist for solid samples, therefore, no matrix is used.

The blank is taken through the appropriate steps of the process.

9.2.1 Trip Blanks

Trip blanks are samples containing reagent water appropriately prepared for the project. The trip blanks are transported along with the other sample containers to the sampling site and back to the laboratory. The trip blank is never opened until it is ready for analysis. The trip blank will provide information concerning any possible contamination during the transportation of the samples. One trip blank should accompany each cooler containing VOAs.

9.2.2 Equipment Blanks

Equipment blanks are collected in the field. Reagent water is poured over or through the sample collection device, and collected in a sample container which is then sealed with a teflon lined cap and returned to the laboratory as a sample. The equipment blank provides information concerning possible contamination during the sampling process. Equipment blanks are collected daily during a sampling event.

9.2.3 Method Blanks

Method blanks are samples containing reagent water that are taken through the entire analytical procedure and analyzed on the instrument. The method blanks

provide information concerning possible contamination during the sample preparation and analysis. One method blank per analytical batch is required.

9.2.4 System Blanks

System blanks are either reagent water or solvent that has not gone through the sample work-up. These blanks are analyzed by the instrument on the same condition that samples are analyzed. The system blanks provide information for possible presence of contamination in the instrument being used. One instrument/system blank is analyzed per day.

9.2.5 Field Blanks

Field blanks are reagent water samples exposed in the field, during sampling, to monitor contamination from the field operation. The frequency of field blank analysis is described in the sampling and analysis plan.

9.3. Laboratory Control Sample (LCS)/Laboratory Control Sample Duplicate (LCSD)

The LCS/LCSD samples are method blanks spiked with known concentration of analyte(s) or surrogate(s) and subjected to the entire preparation and analytical processed along with the environmental samples. The LCS checks the laboratory performance on the sample preparation and analyses procedures without the matrix interference. Relative percentage difference (RPD) of LCS/LCSD checks the precision of the laboratory performance without matrix effect.

One LCS is prepared for each analytical batch of up to 20 samples. One LCSD is prepared with each LCS in the absence of matrix spike/matrix spike duplicate sample in the analytical batch to provide data on precision.

9.4. Matrix QC Parameters

Matrix spike, matrix spike duplicate, sample duplicate and surrogate recoveries results provide indication of the effect of the matrix on accuracy and precision. These samples are analyzed concurrently with other environmental samples. To prepare a matrix spike sample, a known concentration of analyte/surrogate is added to sample prior to preparation and extraction. Laboratory duplicate samples are prepared from the same field samples. It is a standard practice in established methods for organic analyses to prepare and analyze one matrix spike/matrix spike duplicate for each analytical batch. Surrogate is added in every sample, method blank, LCS/LCSD, MS/MSD. For inorganic analyses, one matrix spike/one duplicate is included in every batch of samples. EMAX sets control limits for precision and accuracy of each method. The limits are either based on the laboratory's past performance, adapted from specific programs or established as internal goal. The specific acceptance criteria appears on the QC Schedule of each SOP. They are reviewed annually based on results from control charts (Section 9.13).

9.5. ICP Interference Check Sample

The ICP Interference Check Sample is used to verify the ICP inter-element and background correction factors remain constant in the analytical process. It is analyzed at the beginning and end of each analytical run, prior to the last CCV and CCB at a predetermined time interval.

9.6. GC/MS Performance Check

Mass spectrometer performance is monitored every 12 hours of operation or method specific time period by measuring the mass/ion distribution of BFB (volatiles) of DFTPP (semivolatiles). The mass/ion distribution of these compounds has to fulfill the method project specific requirements before analysis can start. Furthermore, mass assignments is checked periodically by using perfluorobutylamine to ensure that mass number is properly assigned.

9.7. Pesticide Performance Evaluation

Pesticide analysis is subjected to the following additional QC to ensure the quality of data.

9.7.1 Resolution Check

A mixture of known pesticides is analyzed at the beginning of the analytical sequence. The two adjacent peaks have to be resolved to a method specific criteria to be acceptable.

9.7.2 Performance Evaluation

A mixture of known pesticides, to include DDT and Endrin is analyzed at the beginning of analysis and at a certain interval. Individual breakdown and the combined breakdown of Endrin and DDT should be less than a predetermined criteria for the system to be acceptable.

9.8. Internal Standard

An internal standard, which is a non-target analyte having similar properties to target analytes, is added to every standard, blank, laboratory control sample, matrix spike, matrix spike duplicate (for volatiles), and sample extract (for semivolatiles) prior to analysis to compensate for the local variation during analysis. Internal standards are used as the basis for quantitation of the target compounds.

9.9. Method of Standard Addition

Method of standard addition (MSA) is performed on samples found to contain matrix interferences when analyzed for metals by GFAA. This procedure assumes the constant effect of matrix interference and allows quantitation of analyte based on the linearity.

9.10. Post Digestive Spike

When the matrix spike recovery falls outside QC limits and the unspiked sample does not exceed four times the amount spike, a spiked sample at a predetermined concentration will be analyzed and the resulting data compared to the data for the corresponding unspiked sample. This analysis allows any operational error in metal or cyanide analyses to be distinguished from matrix effect.

9.11. Serial Dilution

For metals, a designated dilution (4X or 5X, depending on the protocol) of an investigative sample is analyzed to check for possible physical and/or chemical interference. One serial dilution per batch is analyzed. The diluted value should agree within 10% of the original analysis to demonstrate no interference. Exceptions are described in the SOP for each individual method.

9.12. Laboratory Operations QC Checks

9.12.1 Sample Containers

All sample containers are purchased pre-cleaned. All glasswares used in this laboratory shall be subjected to decontamination treatment prior to its use. Procedures are detailed in SOP GP-0006.

9.12.2 Thermometer, Balance and Refrigerator

Thermometers are calibrated against National Bureau of Standards (NBS) on an annual basis in accordance to SOP GP-0016. In turn, these calibrated thermometers are used to monitor furnace, oven, and refrigerators. Balances are services and certified by a service representative annually. The accuracy of balances as well as refrigerator temperatures are monitored daily and permanent records are kept. The tolerance limits and plan of action is written in the daily logs.

9.12.3 Stationary Blank

Stationary blanks are samples containing reagent water that are stored in the sample storage refrigerator. These blanks are analyzed weekly to monitor the storage refrigerator for volatile water samples. The stationary blanks provide information concerning possible contamination in the sample storage refrigerator. Analytical results are maintained by the QA Department.

9.12.4 Standard Verification

Before using a new standard, it must be verified that it is in acceptable condition through analysis. This is accomplished by analyzing the new standard against a second standard known to be in acceptable condition. Once the standard is verified acceptable, it may be used for routine analysis.

9.12.5 Reagent Water

Reagent water is available for routine laboratory use. Reagent water is defined as water in which no interference is observed at the MDL level of each parameter of interest. For organic analysis, tap water is filtered through a charcoal trap and analyzed for trace level of the analyte of interest before use. For inorganic analysis, Type I water is required. Criteria for Type I water are: Minimum electrical resistivity of 16.67; maximum electrical conductivity of 0.1 mmho/cm and maximum total matter of 0.1 mg/L. Reagent water (Type I) is generated by the laboratory employing a Barnistead's Nanopure system that utilizes a combination of macroreticular resin, carbon, anioncation resin and membrane filtration. The conductivity of this water supply is monitored and recorded daily in a logbook containing the date, person recording the data and conductivity. The water source will be removed from use until the problem is corrected when an out-of-control event occurs.

9.12.6 Reagent QC

All chemicals used in the laboratory are reagent grade or better. Whenever a new lot of reagent is purchased, it will be subjected to a QC check to ensure that the chemical is free of any contamination that will interfere with analysis. A detailed description of reagent QC appears in Section 6.1. A record is kept in each department until the reagent has been used up in the course of analysis.

9.13. Control Chart

Control charts provide a graphic display of parameters over a period of time. They are a useful tool in assessing the quality control effort. Before the assembly of control chart, the analytical procedures and quality control criteria for respective analytical method should be verified. A control chart compiles data from laboratory control sample, matrix spike recovery and RPD for soil and water, and surrogate recovery from each analytical method. Each control chart shall consist of a center line, two warning limits, and two control limits. The control chart parameters should be calculated according to the equations below. A minimum of 20 points per control chart parameter shall be obtained prior to the initial attempt to establish the control chart parameter. If the laboratory does not have 20 points to use in setting control limits, the recommended EPA recoveries for the method will be used until such time as 20 points are obtained.

$$\bar{x} = \frac{x_1 + x_2 + \ldots x_n}{n} \qquad SD = \sqrt{\frac{\sum_{i=1}^{n} (x_i - \bar{x})^2}{n-1}}$$

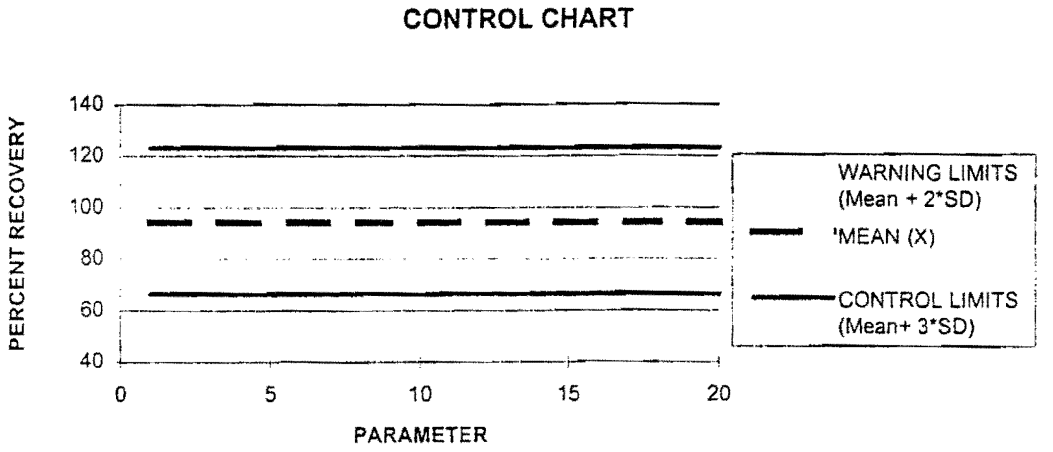
where:

$\bar{x}$ – mean

SD – standard deviation

n – number of data points

Control Chart of Parameter Estimation



On a real time basis, the analyst shall monitor one representative parameter for trends and shifts (refer to SOP GP-0030 for details). A control chart monitors laboratory performance on a long term basis. The QA Manager updates the control chart annually. When updating the control chart, the data for the most recent 12 month period are kept while the rest of the historical data are dropped off from the chart. In this manner, a 12 month period chart is generated. Once a year, the QA Manager compiles the control chart parameters (upper control limit and lower control limit) and compare the relevant parameter to the established method control limit in the QC schedule. If the percent difference between the established method control limit and the control chart parameter is greater than 10%, the new control limit will replace the established one.

Only control charts from the laboratory control sample will be monitored for trend purposes since they represent the laboratory performance of a method without the problem from matrix effect and sample non-homogeneity. A laboratory process for a particular analyte is considered out-of-statistical control whenever one of the following conditions, as demonstrated by a control chart monitoring that analyte, exists:

- Any one point is outside of control limit.
- Any three consecutive points are outside the warning limits.
- Any obvious cyclic pattern is seen in the points.

If a control chart reveals any of these problems, the QA Manager will generate a non-conformance report detailing the problem and initiate an investigation. The investigation should include review of data generated during the period, history of the instruments, quality

of supplies (reagents, standards, gases, etc.), and other relevant information. The purpose of the investigation is to isolate the cause of the out of control event(s) and the possible corrective action that may be required to bring the system back in control. The maintenance of control chart is described in SOP GP-0030.

9.14. QA Manual Review, Revision, and Control

The QA Manager is responsible for the contents of the QA Manual. As clients modify or issue new guidance documents affecting the operations of laboratories, the QA Manager will review the manual annually and revise it when appropriate. Similarly, when corrective action affecting the manual is identified as necessary during normal business operations, the QA Manager will take the appropriate action.

The QA Manual is a controlled document as described in SOP GP-0013. The master document is retained as a historical document by the QA Manager and copies are issued pursuant to the control processes defined in that SOP.

10. SYSTEM AND PERFORMANCE AUDITS

System audits, performance audits, and data quality audits are independent assessments of sample collection and analysis procedures. Audit results are used to evaluate the ability of the system to produce data that fulfill the objectives established for the program, satisfy the QC criteria and identify any area requiring corrective action.

A system audit is a qualitative review of the overall sampling or measurement system while performance and data quality audit are quantitative assessment of a measurement system.

10.1. System Audit

All records, logs, and data files are audited for completeness, accuracy, and adherence to SOPs on a semiannual basis by the QA Manager, and several random project files are also pulled and audited for compliance to procedure throughout the analytical process (i.e. from sample receipt through the final report) during the system audit.

10.2. Performance Audit

10.2.1 Internal Performance Evaluation Samples

10.2.2 The QA Manager may, at any time, prepare blind QC samples for routine analysis. The QC sample will be logged in under a fictitious client name so that they would be treated as a normal client sample. If the results of the internal blind QC Performance Evaluation (PE) samples are not acceptable, the QC Officer will investigate and determine the cause of error. Once the problem has been identified, corrective actions will be taken. A written report is kept on file. If the problem continues to exist, the Laboratory Director will be informed and further actions taken.

In the absence of an external PE sample for a period of 6 months, covering at least 90% of EMAX full suite of analyses, the QA Manager shall administer an internal PE sample, either commercially prepared for double blind PE samples or internally prepared for single blind PE samples. Acceptance criteria for commercially prepared PE samples shall follow the acceptance criteria established by the manufacturer. The acceptance criteria for the internally prepared PE samples shall be based on the characteristic of the parameter as observed in the LCS control limits.

10.2.3 Internal Audit

The QA Manager regularly conducts data review, which serves to check if holding time has been met, calibration are adequate, data calculations are correct, QC results are correct and accurate and documentation is complete. Any problems identified shall require corrective action. Furthermore, the QA Manager will audit the following areas on a regular basis:

- adherence to specific project QC protocol

- review of control chart
- result of external performance evaluation sample
- result of internal performance evaluation sample
- corrective action step taken for specific out of control event
- follow up on recommendation of external audit report

The result of these audits will be discussed with the Laboratory Director to decide cause of action. In cases, where a serious problem was identified, the QA Manager shall isolate and suspend that part of the operation and consult the President of the company for direction.

10.2.4 External Audit

EMAX is also audited as required by various regulatory agencies to maintain laboratory certifications, and by various commercial clients with laboratory auditing programs.

- USEPA Performance Evaluation Studies: EMAX participates in the EPE semi-annual drinking water (WS Series) and semi-annual wastewater (WP Series) performance evaluation studies (4 studies per year).
- EMAX participates in various client sponsored performance evaluations by analyzing QC samples and submitted by commercial clients in conjunction with their own QA programs.
- Several governmental proficiency samples are analyzed annually to maintain various laboratory certifications such as: HAZWRAP, US Army Corp. of Engineers, etc.

The findings of all audits are released to the Laboratory Operations Manager and the supervisors. Corrective action plans are formulated and implemented by the supervisors working in conjunction with the QA Manager.

The effectiveness of all corrective actions, whether issued in response to an audit deficiency or an out-of-control event, is determined through the use of follow-up blind QC samples or spot audits, depending upon the specific situations.

11. INSTRUMENT MAINTENANCE

EMAX's instrument maintenance program, an integral part of the QA program, consists of three elements: evaluation of instrument performance, good record keeping and a comprehensive preventive maintenance program. The laboratory operations manager and supervisors are responsible for providing technical leadership to all staff involved with chemical analysis. The leadership role includes serving as technical resource to help solve equipment and method problems and coordinate instrument repair and maintenance. The department supervisors are further responsible for developing procedures and schedules for maintenance of each major instrument readily respond to out of compliance monitoring measurements and corrective actions, and for delegating specific maintenance responsibilities to department staff. Critical parts inventory is maintained for each instrument in every department.

11.1. System Performance Check

The System Performance Check will be performed on each new instrument after installation and on existing instruments after major modifications have been made. In addition, portions of the System Performance Check may be used to troubleshoot the instrument whenever analytical problems arise during its use. The purpose of the System Performance Check is to establish the general instrument and specific method capability limits. This information will establish instrument control limits so analysts will know whether or not the Method Detection Limit (MDL) of the analysis they are performing is within the performance capabilities of the instrument and method.

The System Performance Check includes the following tasks:

- Establish Stability and Sensitivity of the instrument based on manufacturer's recommendation.
- Establish a Method Detection Limit (MDL) for each parameter of interest.
- Establish a dynamic range (linear range) of the instrument for each parameter of interest.

11.2. Instrument Maintenance

Each analyst is responsible for the daily instrument maintenance activities. For more extensive repair or preventive maintenance, EMAX maintains a service contract on all major instruments and expects quick response (24 to 48 hours) from the vendor. Each instrument is serviced for preventive maintenance according to the schedules specified by the manufacturer. Preventive maintenance services help reduce instrument downtime and allow instruments to perform at their peak level. A list of instruments in use as of the revision date of this manual is provided in Appendix 1.

In addition, an inventory of spare parts will be kept in case of an unexpected need to replace damaged or defective instrument parts. The section supervisors ensures that there are adequate supplies of routine spare parts (liners, lamp, EM tube, etc.).

11.3. Instrument Run Logbook

The Instrument Run Logbook is an on-going record of all analytical runs performed on the instrument. Each run (data file) appears in the logbook in chronological order indicating the sequence of runs associated with each complete analysis. In addition to providing evidence of whether or not a sample was run on a particular day, the logbook can serve as a diagnostic tool used for additional checks against possible carry-over contamination problems. On occasions, the instrument run log may serve as an official document used in verifying the validity of analysis in a courtroom situation. The Instrument Run Logbook will include the following information:

- Data file name, Laboratory sample ID, analysis batch
- Sample matrix
- Analytical method
- Date of analysis and dilution factor
- Standard ID
- Instrument ID
- Name of analyst

11.4. Instrument Maintenance Logbook

The instrument maintenance logbook contains documentation of all servicing and maintenance performed on the instrument. In addition, any instrument problems, including the symptoms, are noted in the logbook.

The instrument maintenance logbook will serve as evidence that proper care was taken to keep the instrument at its peak condition. The following information will be included in the instrument maintenance logbook:

- Date and description of instrument problems including the symptoms.
- Date and description of corrective actions taken.
- Date and description of routine maintenance.
- Initial and comments of individual performing the service.
- Any recommendations for servicing that will be required in the future.
- Documentation of return to control after repair.

12. CORRECTIVE ACTION

The objective of a QA/QC program is to ensure the quality of data as well as the service. If unsatisfactory performance is revealed, corrective actions shall be implemented immediately. An SOP (GP-0027) is developed to address the proper course of action if out of control event is observed in laboratory operation. Each analytical process is equipped with a self-correcting QC procedures as an integral part of every analytical SOP. The QA Manager and laboratory management will monitor the implementation of corrective action and to ensure that the root cause has been addressed. Of several possible out of control occurrences, four well-recognized events and their associated plans of corrective action and documentation are set forth below:

12.1. Corrective Action At The Receiving Level

The Sample Control Officer will document and notify client services of any discrepancies in samples received. Client service will then notify the customer for the proper course of action. Instructions on discrepancies will be documented in a sample receipt form and relevant information on that sample will be disseminated to the analyst involved in the project through the project alert form.

12.2. Corrective Action At The Bench Level

Laboratory personnel shall follow each method specific QC schedule and apply corrective action, when necessary. For example, if the calibration of an instrument is not linear, the analyst shall correct it prior to continuing to sample analysis. The reason for recalibration is noted on run log. Similarly, if a balance is out of calibration, service call shall be placed by supervisor and the instrument shall be removed from the operation until the problem is fixed and proper calibration is restored and documented.

12.3. Corrective Action At Review Level

Supervisors shall review all calculations, raw data and QA/QC criteria for possible out of control event. Results that are out of control due to matrix effect shall be documented as such with supporting evidence; no corrective action is necessary in such circumstances. However, out of control events that are not due to matrix interference shall be investigated for causes and corrective actions must be implemented. Corrective action can consist of reanalysis or resampling, depending on holding time and applicable project specification.

12.4. Corrective Action Required By Audit Issues, PE Samples, Control Charts And Other Related Circumstances.

External and/or internal audit, control chart evaluation, performance evaluation sample result and other circumstances shall be resolved by the closed loop corrective action process described below:

- Define the problem.
- Assign responsibilities for problem investigation.
- Investigate and determine the cause of the problem.
- Determine the corrective action(s) necessary to eliminate the problem.
- Assign and accept responsibilities for implementing the corrective action.
- Establish the effectiveness of the corrective action and implement the correction.
- Verify and document that the corrective action has eliminated the problem and fill out an out-of-control discrepancy report.

13. SECURITY

The security of samples, information, and data is a necessary component of analytical laboratory procedures. To address client security concerns, EMAX has instituted multiple levels of security so that disruptions of protocols will be minimized and data security will b maximized. These levels of security may be described as follows:

13.1. General Building Security

The physical facilities of EMAX Laboratories, Inc. are located in metropolitan Los Angeles, at 630 Maple Avenue in the City of Torrance. The laboratory facility itself is located in a very low-crime area, directly opposite from the Regional Courthouse for Los Angeles County and the Municipal Offices of the City of Torrance, including the Police Department.

EMAX shares no common areas in its physical location with other commercial tenants of the building complex. All external doorways are keyed with tumbler and deadbolt locks. Key holders are logged and recorded. Return of keys is an identified transfer;/termination procedure. In addition to the keying of external doors, EMAX employs an electronic alarm system that notifies a commercial security firm, in the event of an unauthorized entry. Electronic code entry is required after regular business hours and on company holidays as a second level of security once key entry is made. Each employee who is given key access is also given a discreet identification code for entry into the electronic data system. Security codes are changed on an intermittent and random basis.

During working hours, all persons visiting or working in an EMAX area must pass through either Sample Receiving or General Reception where designated staff control access to the facilities. Visitors are required to log into and out of the facility and are identified by either Visitor or Vendor badge while at the facility.

13.2. Storage and Access to Samples

All samples received by EMAX are stored in a discrete, sample storage area. Access to this area is secured by a third level of punch-key combination locks. During the course of a staff work day, access to the sample storage area is available only through direct contact with the Sample Custodian or the Extraction Supervisor. The Sample Storage Area is locked at all times when the Sample Custodian is away from the Area.

Sample extracts are kept in individual refrigerators in the cognizant organic/inorganic laboratories to minimize traffic and access into the Sample Storage Area. Each laboratory is locked during off-hours. Access is restricted to personnel working in that department.

13.3. Access to Data

As data is produced and prior to final review by supervisory staff, it is maintained in the custody of individual analysts during the work day. During after-hours periods, data in production is secured by the two levels of building access security. In addition, analyst work areas are locked after hours such that only personnel assigned to that area can access it

(e.g., organic personnel cannot access inorganic areas, etc.). During the data review process, data is filed in job folders either as paper documentation or electronic media and the resulting files are maintained in storage accessible only to laboratory management. Such files are secured in a locked room during non-business hours. When the client project is completed and on-site storage is no longer required, the files are transferred to off-site locked storage facilities for the remaining retention period required by the contract or pertinent regulations and/or statuses.

14. SOFTWARE QUALITY ASSURANCE

Software quality assurance functions are detailed in a separate EMAX manual entitled Software Quality Assurance Plan (SQAP). The SQAP addresses the requirements and responsibilities for the Laboratory Information Management system (LIMS), local software (e.g., spreadsheets and other data manipulating software), instrument software provided by instrument manufacturers, and other systems employed to generate, compile, reduce, or report analytical data or supporting information. When deemed necessary to support the SQAP, EMAX issues controlled documents such as SOPs i.e., policies and procedures for electronic data security including documented authorization for access to such data files; policies and procedures for validation and verification of purchased and internally developed or modified software, etc.

The Software Quality Assurance Plan is kept by the QA Department and is available upon request.

15. SPECIAL PROGRAM/PROJECT REQUIREMENTS

15.1 Other QA requirements specific to a program and/or project shall be addressed in a form of an addendum. The addendum shall include the following:

- Reference #
- Program/Project
- Changes and/or Additions
- Prepared by
- Approved by

16. REFERENCES

16.1 Environmental Laboratory Accreditation Program, California Department of Health Services (1991) (as updated).

16.2 Data Quality Objectives for Remedial Response Activities", EPA 540-G87-004 (as updated).

16.3 Standard Methods for the Examination of Water and Wastewater, 18th Edition, 1992 (as updated).

16.4 Methods for Chemical Analysis of Water and Wastes EPA/600/4-79/020 (as updated).

16.5 Methods for Determination of Organic Compounds in Drinking Water (500 Series) (as PB-89-220461/AS (as updated).

16.6 Test Methods for Evaluating Solid Waste Physical/Chemical Methods S.W. 846 USEPA SW846, 3rd Edition, 1986 (as updated).

16.7 EPA 40 CFR Part 136 for 600 Series Methods (as updated).

16.8 USEPA Contract Laboratory Program, Statement of Work for Organic anlysis (OLM01.8, August 1991) (as updated).

16.9 USEPA Contract Laboratory Program, Statement of Work for Inorganic Analysis (ILM02.1, September 1992) (as updated).

16.10 USEPA Compendium of Methods for the Determination of Toxic Organic Compounds in Ambient Air. EPA/600/4-89/017 (as updated).

16.11 Methods for Determining Emission of Toxic Contaminants from Stationary Sources. State if California Air Resources Board, 1991.

APPENDIX 1 LIST OF MAJOR ANALYTICAL INSTRUMENTS

Units	Instrument	Manufacturer	Model	Detector
4	GC/MS	Hewlett Packard (Volatiles)	5890/5970	MS
3	GC/MS	Hewlett Packard (Semivolatiles)	5890/5970	MS
2	GC	Hewlett Packard	5890	FPD
3	GC	Hewlett Packard (Dual Detectors)	5890	ECD
1	GC	Varian (Dual Detectors)	3400	ECD
2	GC	Hewlett Packard	5890	FID
2	GC	Varian (Dual Detectors)	3400	FID
2	GC	Varian	3400	FID/PID/HALL
2	GC	Hewlett Packard	5870	FID/PID/HALL
1	HPLC	Hewlett Packard	1050	UV/Fluorescence
1	HPLC	Waters	LC Module 1 Plus	UV/Fluorescence
2	IC	Dionex	100	Conductivity
1	TOC Analyzer	Shimadzu	TOC-500	Fluorescence
1	Mercury Analyzer	Leeman	PS200	UV
1	Mercury Analyzer	Bacharach	Coleman 50B	UV
1	GPC	Zymark	Benchmate WorkStation	UV
1	GPC	ABC	GP-1000	UV
1	GPC	Waters	717 Plus/486	UV
2	ICP-AES	Thermo Jarell Ash	61E	ICP
1	ICP-AES	Themo Jarell Ash	Tracer	ICP
1	ICP-AES	Perkin Elmer	P40	ICP
2	GFAA	Varian	Spec400	GFAA

APPENDIX 2 INFORMATION PROCESSING EQUIPMENT

18.1 Network

18.1.1 Networking Hardware

- DELL PowerEdge 2100/200 Pentium Pro 200MHz File Server with 64MB RAM, 8GB SCSI Hard Drive, 4/8GB Tape Backup, 8x SCSI CD-ROM Drive, and 1.44MB Floppy Drive.
- PCI SCSI 2940 Controller (2)
- 3COM PCI Combo Ethernet Adapter (2)
- PCI and Enet-16 Adapters (25)

18.1.2 Peripherals on the Networking System

- Pentium and 486 PCs connected to the network (25)
- HP Laser Jet 4000 TN network printers (2)
- HP Laser Jet II/III/4/4L/4P/4V/5 workgroup printers (9)
- Fax/Modem, 56K X2 (1)
- Fax/Modem, 14.4K baud (1)
- Internal Tape Drive, ioMega Ditto Travan TR-3 3.2GB (1)
- External Tape Drive, Colorado Trakker 250 MB (2)
- External ZIP DISK Drive, iomega 100 MB (5)
- SmartUPS 1000VA Power Back-up System

18.1.3 Software on the Networking System

- Novell Netware Version 3.12
- DOS Operating System
- Windows 3.X
- Windows 95
- IBM AntiVirus for Netware Version 2.5.2
- Seagate Backup Exec for Netware Version 7.11
- APC PowerChute Plus for Netware
- Labworks 300 Laboratory Information Management System

- Installation Restoration Program Information Management System (IRPTools PC Version 1.2)
- Installation Restoration Data Management Information System (IRDMIS Version 5.2, 5.3)
- Jacobs Environmental Management System (JEMS) QC Tool Version 1.04
- Corps of Engineers Loading Tool (COELT Version 1.2a)
- Corps of Engineers Electronic Data Consistency Checker (EDCC Version 1.2a)
- Bechtel EDD Checker
- Ward Scientific Environmental Data Reporting/CLP Report Generator Software
- USEPA Inorganic Contract Compliance Screening Software
- Programming Languages (QuickBasic 4.5, Pascal 6.0, Foxpro 2.5)
- Utilities (Norton, PC Tools, Trakker)
- Office Suite (Microsoft Office 95 Professional)
- Database Management (MS Access, Foxpro 2.5)
- Spreadsheets (MS Excel, Lotus 1-2-3)Word Processing (MS Word, Wordperfect, Lotus AMI Pro)
- Graphics (DrawPerfect, Visio, MS Powerpoint)

18.2. Computer Systems and Printers

18.2.1 Windows 95/Windows 3.x/DOS Workstations

18.2.2 DELL Pentium Pro 200 MHz PC (1 system) equipped with 64MB RAM, 8GB hard disk, 4/8GB Tape Backup, 8x CD-ROM, and 3-1/2 floppy disk. Network file server.

18.2.3 Pentium 200/166/133/75 PCs (15 systems). Brand-names and clones; equipped with 32, 16, or 8 MB RAM, color monitor, 2.4, 1.2 or 540 MB hard disk, and 3-1/2 floppy disk. Windows 95 or Windows 3.11. Networked and stand-alone.

18.2.4 Intel 486 computers (20 systems). Brands: Brand-names and clones; equipped with 16, 8 4 or 2 MB RAM, color monitor, 210 MB to 1.2 GB hard disk, and 5-1/4 and/or 3-1/2 floppy disk. Networked and stand-alone.

18.2.5 Intel 386 computers (4 systems). Brand-name and clones; equipped with 2MB-4MB RAM, color monitor, 80-500MB hard disk, and 5-1/4 and/or 3-1/2 floppy disk. Stand-alone.

18.2.6 Intel 286 computers (2 systems). Brand-names and clones; equipped with 640K-2MB RAM, color monitor, 20-96MB hard disk, and 5-1/4 and/or 3-1/2 floppy disk. Stand-alone.

18.3 HP Workstations

18.3.1 HP 1000 A-Series instrument computers (3 systems) each with HP tape backup systems.

18.3.2 HP terminals serving the HP A-Series computers (10 terminals).

18.4 Printers

18.4.1 HP Laser Jet 4000 Series network printers (2 units)

18.4.2 HP Laser Jet 5 Series (3 units)

18.4.3 HP Laser Jet 4 Series (4 units)

18.4.4 HP Laser Jet 2/3 Series (3 units)

18.4.5 HP Desk Jet (2 units)

18.4.6 HP RuggedWriter (5 units)

18.4.7 Panasonic, Epson, Okidata, NEC, Citizen Dot Matrix Printers (14 units).

18.5. Data Communications Equipment

18.5.1 Internet PC. One(1) 586 computer system with 16MB RAM, color monitor, 6x CD-ROM, 1.2GB hard disk, and 3-1/2 floppy disk. US Robotics fax/modem.

18.5.2 Communications PC. One(1) 486 computer system with 8MB RAM, color monitor, 530MB hard disk, and 5-1/4 and/or 3-1/2 floppy disk. Hayes fax/modem. In Accounting and Finance.

18.5.3 Communications PC. One(1) 486 computer system with 8MB RAM, color monitor, 530MB hard disk, and 5-1/4 and/or 3-1/2 floppy disk. Hayes-compatible internal 9600 fax/modem. Stand-alone. In Purchasing.

18.5.4 Modem. One(1) US Robotics external fax/modem, 56K X2

18.5.5 Modem. One(1) Hayes external fax/modem, 14.4 K

18.5.6 Modem. One(1) Hayes-compatible internal modem, 9600 K.

18.5.7 Communications Software.

18.5.8 One(1) CrossTalk XVI

18.5.9 One(1) pcAnywhere software

18.5.10 One(1) Netscape Internet Browser v3.0

18.5.11 One(1) Netcom NetComplete Internet Browser

18.6 Data Backup Equipment

18.6.1 Uninterruptible Power Supply. One(1) UPS power back-up system, 1000VA.

18.6.2 Tape Drive.

18.6.3 One(1) File Server tape drive, 4/8 GB.

18.6.4 One(2) Trakker external tape drive, 250 MB.

18.6.5 One(1) ioMega Ditto Travan TR-3 3.2GBinternal tape drive.

18.6.6 Five(1) ioMega ZIP external drive, 100MB

APPENDIX 3 LIST OF CERTIFICATIONS

Federal Government Validations

- Department of Army, USATHAMA
- Army Corps of Engineers, HTRW Center of Expertise
- Department of Energy, HAZWRAP
- Department of Food and Agriculture, Permit to Import Soils
- Department of Navy, NFESC
- United States Air Force, AFCEE
- United States Environmental Protection Agency, CLP

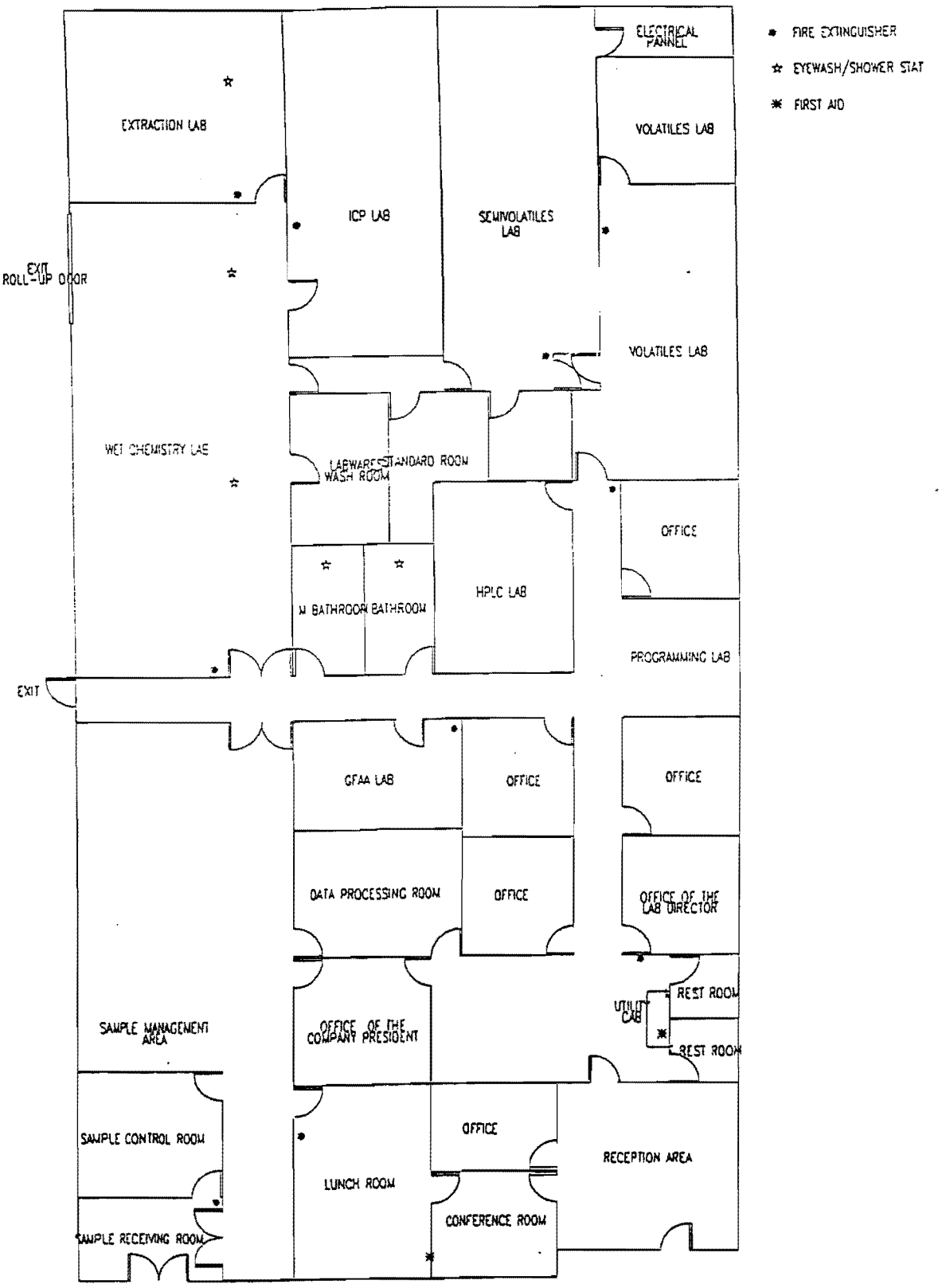
State Certifications

- Alaska
- Arizona
- California
- Florida
- Massachusetts
- North Carolina
- Kansas
- Oklahoma
- South Carolina
- Tennessee
- Washington
- Utah

Inter-Laboratory Quality Control Programs

- EPA-Water Supply Laboratory Performance Evaluation
- EPA-Water Pollution Laboratory Performance Evaluation

APPENDIX 4 DESCRIPTION OF FACILITY

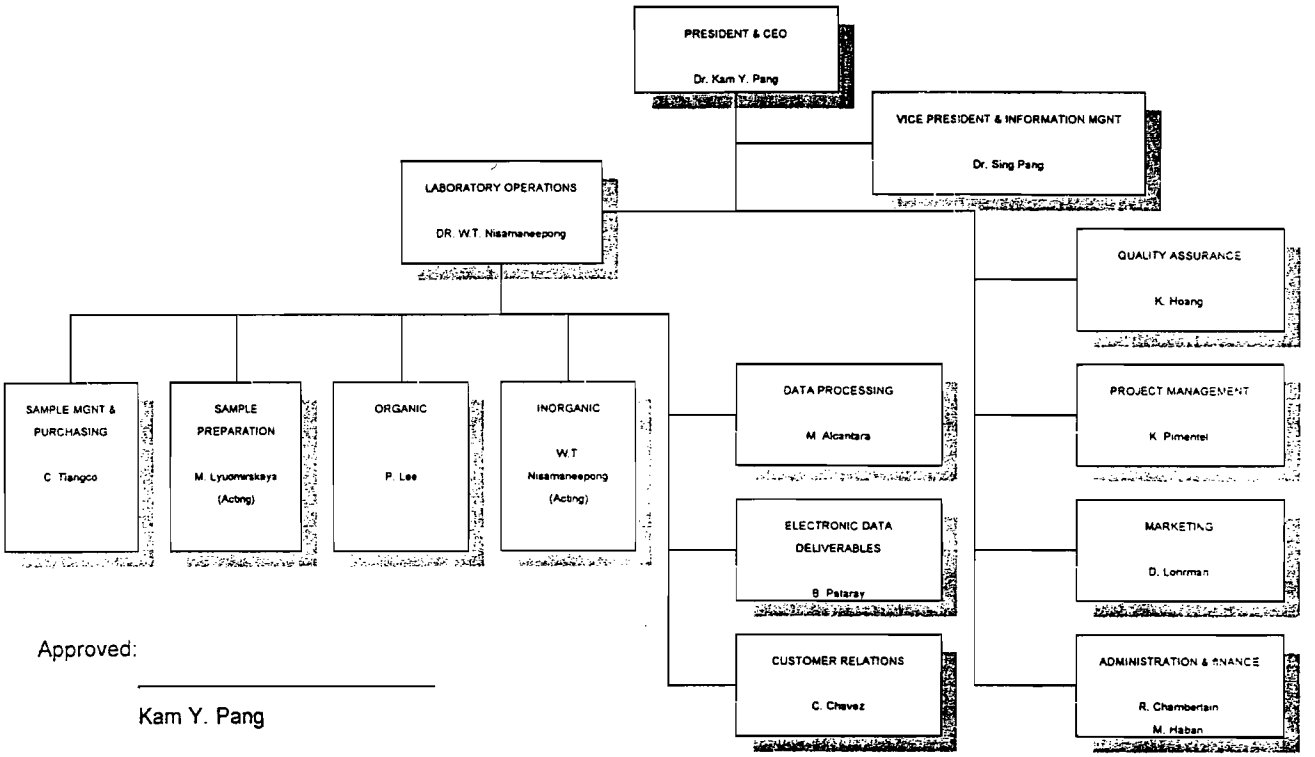


APPENDIX 5 EMAX ORGANIZATIONAL CHART

5-A LABORATORY ORGANIZATIONAL CHART

EMAX LABORATORIES, Inc.

JANUARY 1998



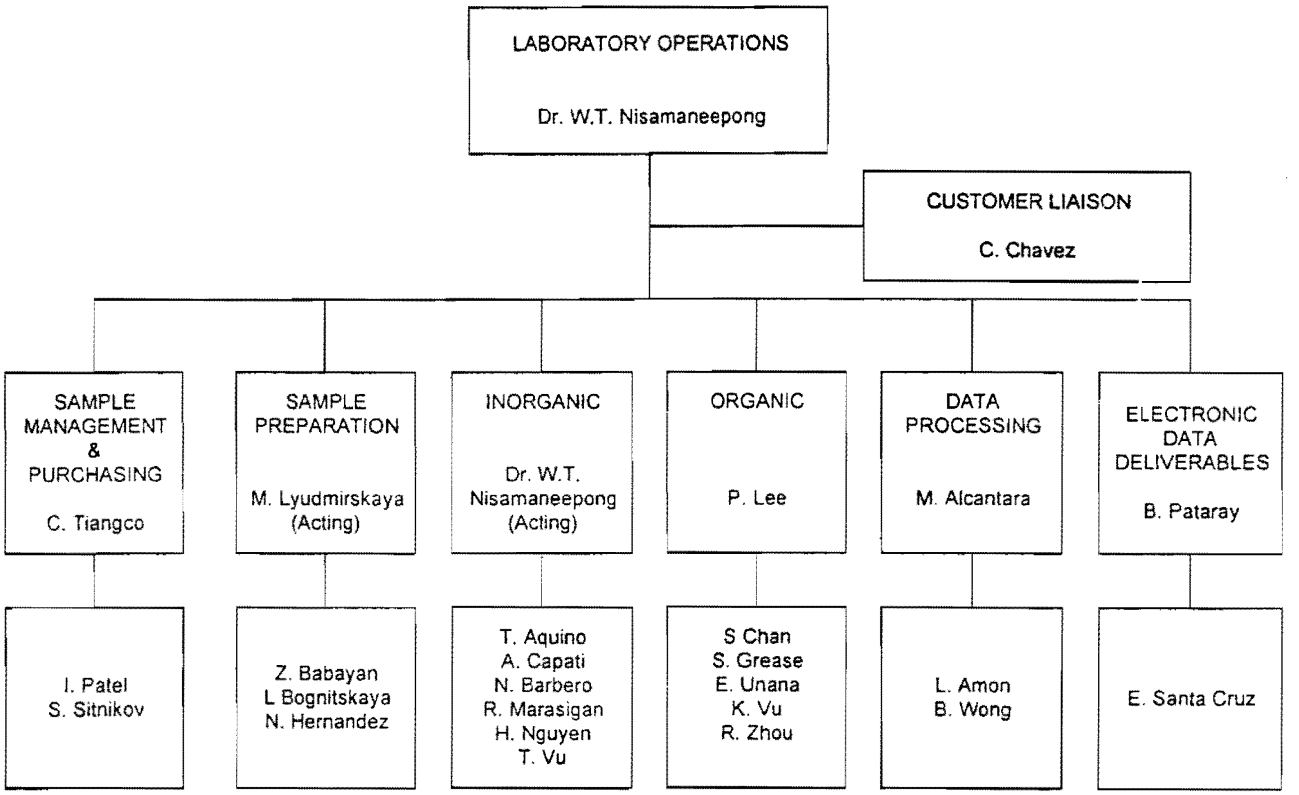
Approved:

Kam Y. Pang

5-B LABORATORY ORGANIZATIONAL CHART

EMAX LABORATORIES, Inc.

JANUARY 1998



Approved:

Dr. T.W. Nisamaneepong

APPENDIX 6 STANDARD OPERATING PROCEDURES INDEX

6-A GENERAL PROCEDURES

SOP NO.	TITLE
GP-0000	Writing SOP
GP-0001	Sample Receiving
GP-0002	Sample Management
GP-0003	Security
GP-0004	Refrigerator Control
GP-0005	Utilization of Subcontract Laboratories
GP-0006	Glassware Cleaning
GP-0007	Balance Calibration
GP-0008	Data Flow and Review
GP-0009	Training
GP-0010	Waste Disposal
GP-0011	Mobilization
GP-0013	Controlled Document
GP-0014	Assembly of Complete CLP Data Package
GP-0015	Calibration of Micropipets
GP-0016	Calibration of Thermometer
GP-0017	Project Management
GP-0018	System Audits
GP-0020	Analytical Standard Preparation
GP-0021	Document Archival
GP-0023	Analytical and QC Sample Labeling
GP-0024	Monitoring Instrument Clock Time
GP-0025	Quality Control for Chemicals
GP-0026	Generation of IDL, MDL and RL
GP-0027	Corrective Action
GP-0028	Trip Blank Preparation
GP-0029	Sample Containers, Handling, and Shipping
GP-0029A	Preserving and Shipping Sample Containers
GP-0030	Control Chart

6-B ORGANICS ANALYTICAL PROCEDURES

SOP No.	ORGANICS ANALYTICAL PROCEDURES	Adaptation
AP-2001	Cleanup	3600
AP-2003	Aromatic Volatile Organics	8021
AP-2004	Aromatic Volatile Organics (Mobile Lab)	8021
AP-2005	Halogenated Volatile Organics	8010B
AP-2006	Halogenated Volatile Organics (Mobile Lab)	8010B
AP-2007	Total Petroleum Hydrocarbons by Extraction	M8015
AP-2007A	Total Petroleum Hydrocarbons by Extraction	M8015
AP-2009	Total Volatile Petroleum Hydrocarbons (TPH)	5030/M8015
AP-2010	Extraction of Organic Compounds by Continuous Liquid/Liquid Extraction	3520
AP-2011	Extraction of Organic Compounds From Solid Samples by Pulse Sonication	3550
AP-2013	Total Recoverable Petroleum Hydrocarbons	418.1
AP-2015	Nitroaromatics and Nitramines by HPLC	8330
AP-2016	Volatiles Organics by GC/MS	CLP
AP-2017	Semivolatile Organics by GC/MS	8270B
AP-2019	Chlorinated Pesticides and PCBs	CLP
AP-2020	Nitrogenous and Phosphorous Pesticides in Water by GC	507
AP-2021	TCLP	1311
AP-2022	Organophosphorous Pesticides by GC	8140
AP-2023	Carbamate and Urea Pesticides by HPLC	632
AP-2024	Organochlorine Pesticides and PCBs	8081
AP-2025	Semivolatile Organics by GC/MS	CLP
AP-2026	Volatile Organics	8240B
AP-2028	Ethylene Glycol	M8015
AP-2029	Organic Compounds in Water	524.2
AP-2030	Chlorinated Herbicides by GC	8150
AP-2031	Organic Compounds in Water	502.2
AP-2032	Volatile Organics by GC/MS	8260A
AP-2033	Volatile Aromatic and Unsaturated Organic Compounds in Water	503.1
AP-2034	Semivolatile Organics by GC/MS	525
AP-2035	EDB & DBCP in Water by Microextraction	504
AP-2036	Volatile Organics by GC/MS	624
AP-2038	GC/MS Data Reduction	

AP-2039	Nitrogen and Phosphorous Pesticides in Soil by GC	8000
AP-2040	Fuel Class Hydrocarbons by GC	
AP-2041	Halogenated Volatile Organics by GC PID/Hall in Series	8021
AP-2042	Toxicity Characteristics Leaching Procedure (TCLP) for Volatile Organics	1311
AP-2043	Polynuclear Aromatic Hydrocarbons by HPLC	8310
AP-2044	Total Petroleum Hydrocarbon	
AP-2046	Purge and Trap	5030
AP-2047	Semivolatile Organics by GC/MS	8270 SIM
AP-2050	Dissolved Methane, Ethene and Ethane in Water by a GC Headspace Equilibration Technique	

6-C INORGANICS ANALYTICAL PROCEDURES

SOP No.	INORGANICS ANALYTICAL PROCEDURES	Adaptation
AP-3001	Ion Chromatography Analysis	300
AP-3002	Acid Digestion of Soil, Sludge, and Sediment Samples for Furnace AA, Flame AA, and ICP Analyses of TAL Metals	CLP
AP-3003	ICP Emission Spectrometric Method for Trace Metal Analyses of Soil, Water, and Wastes	CLP
AP-3004	Trace Element Analysis of Water, Soil, Aqueous, Sludge, Sediment, and Water Samples by GFAA	7000
AP-3005	Percent Solids Determination	CLP
AP-3006	Alkalinity	310.1
AP-3007	Specific Conductance	120.1
AP-3008	Mercury Analysis of Water, Extract, Soil, and Sediment by Cold Vapor AA	7470/7471A
AP-3009	ICP Emission Spectrometric Method for Trace Metal Analyses	6010A
AP-3010	Ion Chromatography Analysis	9056
AP-3011	Acid Digestion of Aqueous Samples for Total Metals by GFAA	3020A
AP-3015	Total Cyanide (Spectrophotometric Determination)	CLP
AP-3016	Total & Amenable Cyanide	9010A
AP-3017	pH Measurement	9045
AP-3017A	pH Measurement	9040/151.1
AP-3018	Total Filterable Residue (Total Dissolved Solids)	160.1
AP-3019	Total Suspended Solids (Non-Filterable Residue)	160.2
AP-3020	Ammonia-Nitrogen (Ion Selective Electrode)	350.3
AP-3021	Total Kjeldahl Nitrogen (Potentiometric, Ion Selective Electrode)	351.3
AP-3022	Total Phenolics (Spectrophotometric)	420.1
AP-3025	Chemical Oxygen Demand (Colorimetric Manual)	410.4
AP-3026	Waste Extraction Test (WET)	Title 22

AP-3027	Hardness in Water (Total, Titrimetric, EDTA)	130.1
AP-3027A	Hardness in Water (Total, Titrimetric, EDTA) by Calculation	130.1
AP-3028	Fluoride (Ion Selective Electrode)	340.2
AP-3029	Chloride (Titrimetric Mercuric Nitrate)	325.3
AP-3034	Acid Digestion of Soil, Sludge, and Sediment Samples for Furnace AA, Flame AA, and ICP Analyses of Metals	3050A
AP-3035	Acid Digestion of Aqueous Samples for Total Recoverable and Dissolved Metals by ICP	3005A
AP-3036	Acid Digestion of Aqueous Samples for Total Metals by ICP	3010A
AP-3037	Mercury Analysis of Water and Soil Samples by Cold Vapor AA	CLP
AP-3039	Acid Digestion of Water and Aqueous Samples for Furnace AA, Flame AA, and ICP Analyses of TAL Metals	CLP, Water
AP-3040	Extractable Procedure Toxicity Test (EPTOX)	1310
AP-3044	Volatile Residue (Gravimetric)	160.4
AP-3049	Sulfate (Gravimetric)	375.3
AP-3050	Color (Colorimetric-Platinum-Cobalt)	110.2
AP-3053	Methylene Blue Active Substances (MBAS)	425.1
AP-3054	Turbidity (Nephelometric)	180.1
AP-3055	Nitrogen, Nitrite (Spectrometric)	354.1
AP-3056	Phosphorous (Colorimetric, Ascorbic Acid, Single Reagent)	365.2
AP-3057	Orthophosphate (Colorimetric, Ascorbic Acid, Single Reagent)	365.2
AP-3058	Nitrogen-Nitrate/Nitrite (Spectrometric, Cadmium Reduction)	
AP-3059	Determination of Solid Content on Filter	
AP-3060	Organic Lead (Atomic Absorption)	
AP-3061	Sulfate (Turbidimetric)	375.2
AP-3063	Sulfide (Trimetric, Iodine)	376.1
AP-3064	Chlorine, Total Residue (Titrimetric, Iodometric)	330.3

AP-3065	TJA ICAP 611; Start-Up Procedure	
AP-3066	Varian Zeeman 400 Start-Up Procedure	
AP-3067	Metals Reporting Using Telecation Software	
AP-3068	Metals Reporting by Ward Software	
AP-3070	Acid Volatile Sulfide and Simultaneously Extractable Metals	
AP-3071	pH Measurement	
AP-3072	Biochemical Oxygen Demand	405.1
AP-3073	Dissolved Oxygen (Azide Modification of Thelodometric Method)	
AP-3074	Reactivity Cyanide and Sulfide	
AP-3075	Hexavalent Chromium (Colorimetric)	7196
AP-3075A	Hexavalent Chromium by Coprecipitation	7195
AP-3076	Paint Filter Liquids Test	9095
AP-3077	Oil and Grease, Gravimetric Method	413.2
AP-3078	Specific Gravity, Erlenmeyer Flask Method	
AP-3078A	Specific Gravity	D-845
AP-3079	Acidity	305.1
AP-3080	ICP Trace Metal Analysis in Paint Chips	3050A
AP-3084	Total Organic Carbon Analysis	415.1/9060
AP-3085	Settleable Matter	160.5
AP-3086	Total Organic Compound by Walkley-Black Method	

APPENDIX 7 ANALYTICAL METHODS

EMAX Laboratories, Incorporated is currently capable of performing the following analyses:

ORGANIC

EPA 8010/601	-	Halogenated Volatile Organics
EPA 8015	-	Non-Halogenated Volatile Organics
EPA M 8015	-	Total Petroleum Hydrocarbons
EPA 8020/602	-	Aromatic Volatile Organics
EPA 8030/603	-	Acrolein, Acrylonitrile, Acetonitrile
EPA 8040/604	-	Phenols
EPA 8080/608	-	Organochlorine Pesticides and PCBs
EPA 8100	-	Polynuclear Aromatic Hydrocarbons
EPA 8120/612	-	Chlorinated Hydrocarbons
EPA 8140/614	-	Organosphosphorus Pesticides
EPA 8150	-	Chlorinated Herbicides
EPA 8240/624	-	Volatile Organics by GC/MS
EPA 8270/625	-	Semivolatile Organics by GC/MS
EPA 8310	-	Polynuclear Aromatic Hydrocarbons by HPLC
EPA 8330	-	Explosives
EPA 502.2	-	Volatile Organic Compound In Water
EPA 504	-	EDB and DBCP in Water
EPA 508	-	Chlorinated Pesticides in Water
EPA 515	-	Chlorinated Acids in Water
EPA 524.2	-	Purgeable Organic Compounds in Water
EPA 525	-	Organic Compounds in Water
EPA 531.1	-	Carbamates in Water by HPLC
VOA by GC/MS	-	USEPA CLP SOW OLM03.0
Semivolatiles by GC/MS	-	USEPA CLP SOW OLM03.0
Pesticides by GC/ECD	-	USEPA CLP SOW OLM03.0
TO3	-	Total Petroleum Hydrocarbons
TO14	-	VOA in Ambient Air

* Special inhouse developed methods for dissolved gases, degradation products, ethylene glycol, and tetrabutyl tin.

INORGANIC

EPA 7000	-	Metals by Graphite and Cold Vapor Atomic Absorption
EPA 6010	-	Metals by ICP
EPA 300	-	Anions
EPA 310.1	-	Alkalinity
EPA 325.3	-	Chloride
EPA 335.2	-	Cyanide
EPA 340.2	-	Fluoride
EPA 130.2	-	Total Hardness
EPA 353.3	-	Nitrate
EPA 354.3	-	Nitrite
EPA 413.2	-	Oil & Grease
EPA 418.1	-	Petroleum Hydrocarbons (IR)
EPA 150.1	-	pH
EPA 415.1	-	Total Organic Compound
EPA 365.3	-	Orthophosphate
EPA 160.1	-	Total Dissolved Solid
EPA 120.1	-	Specific Conductance
EPA 375.3	-	Sulfate
EPA 376.1	-	Sulfide
EPA 410.1	-	Chemical Oxygen Demand
EPA 1010	-	Flashpoint
TAL	-	USEPA CLP SOW ILM04.1 (low and high concentration)

WASTE EXTRACTION

EPA 1311	-	Toxicity Characteristic Leaching Procedure
EPA 1312	-	
WET	-	Waste Extraction Test (Calif.)

APPENDIX 8 CHAIN OF CUSTODY

EMAX Laboratories, Inc.

630 Maple Ave., Torrance, CA 90503
Tel # 310-618-8889 Fax # 310-618-0818
Email emaxlabs@ix.netcom.com

CHAIN OF CUSTODY RECORD

Sample Storage | **Lab Batch Control #**

		Matrix Codes		Preservative Codes	Analysis Required	TAT
CLIENT						
PROJECT	TEL	DW = Drinking Water	SS = Soil/Sediments	IC = Ice		☐ RUSH ____hrs
COORDINATOR	FAX	GW = Ground Water	SD = Solid Waste	HC = HCl		☐ RUSH ____days
SEND REPORT TO		WW = Waste Water	SL = Sludge	HS = H_2SO_4		☐ 7 days
EMAIL		AR = Air	PP = Pure Products	HN = HNO_3		☐ 14 days
COMPANY	TEL	WP = Wipes	OT = Others	SH = NaOH		☐ 21 days
ADDRESS	FAX			ST = $Na_2S_2O_3$		☐ 30 days
				ZA = ZnAc		☐
EMAX PM						☐

Sample ID		Sampling		Container			Matrix	QC	Preservative Codes	Comments
Lab	Client	Date	Time	No.	Size	Type	Code			
1										
2										
3										
6										
7										
8										
9										
0										

Instructions

Sampler	No. of Coolers		Courier/Airbill	Cooler Temp.(°C)
Relinquished By	Date	Time	Received By	(Notes for samples sent out)

NOTICE: Turn-Around-Time (TAT) for samples shall not begin until all discrepancies have been resolved. For samples received and discrepancies resolved after 1500 hrs, TAT shall start at 0800 hrs the next business day. The client is responsible for all costs associated with sample disposal. Samples shall be disposed of as soon as practical (but not prior to fifteen (15) calendar days) after issuance of analytical report unless a different sample disposal schedule is pre-arranged with EMAX. Disposal fee for samples defined by CA Title 22 as non-hazardous shall be $5.00 per sample. EMAX will return hazardous samples to the client at the client's expense unless directed in writing otherwise.

APPENDIX 9 SAMPLE RECEIPT FORM

BCN:		Date		Reviews	
Client		Time		Sample Labeling	
Project		Recipient		SRF	
				CRO	

Type of Sample Delivery to EMAX		
☐ EMAX Courier	☐ Client Delivery	☐ Third Party/Airbill No.
By :		
Date :		
Time :		
Comments:		

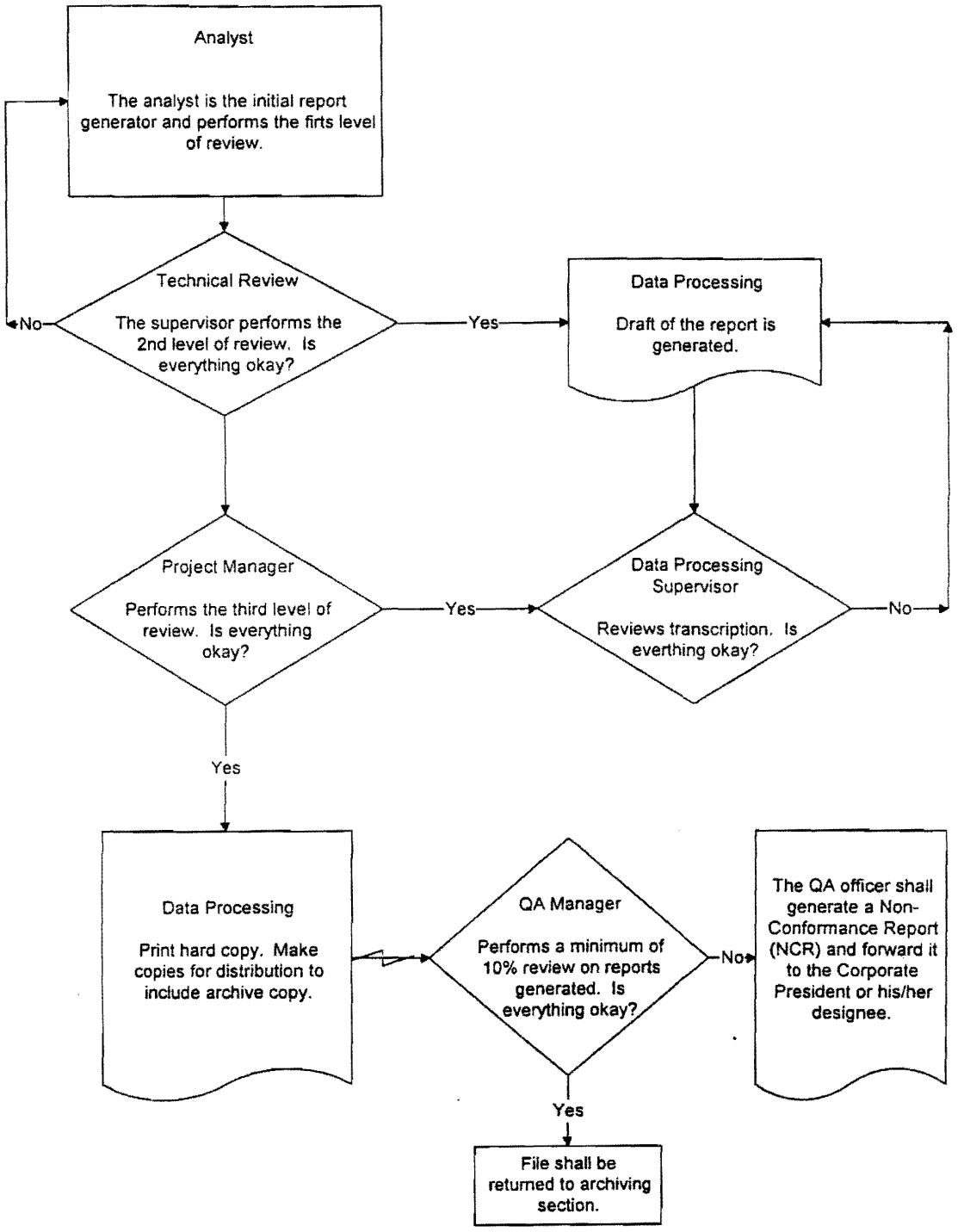
COC Inspection (Check for presence)		
☐ Client Name	☐ Sampler Name/Signature	☐ Sampling Date/Time
☐ Address	☐ Courier Signature w/ Date & Time	☐ Analysis Required
☐ Tel # / FAX #	☐ TAT	☐ Sample Container
☐ Prj Name/Contact Person	☐ Sample ID	☐ Matrix
Safety Issue(s) ☐ None ☐	☐ High concentrations expected	☐ Superfund Site Samples
Comments:		

Packaging Inspection				
Container :	☐ Cooler	☐ Box	☐	
Condition :	☐ Custody Seal	☐ Intact	☐ Damaged	☐
Packaging:	☐ Bubble Pack	☐ Styro Foam	☐	☐ Sufficient
Temperature(s)	☐	☐	☐	☐
Comments:	☐ RAD Screened	☐		

Sample Inspection				
Container:	☐ Custody Seal	☐ Intact	☐ Damaged	☐ Appropriate
Identity:	☐ Client Sample ID	☐ Sampling Date/Time	☐ Sampler Initial	☐ Analysis
Preservation:	☐ NaOH [pH≥12]	☐ HNO_3 [pH≤2]	☐ H_2SO_4 [pH≤2]	☐ Holding Time OK
Sample:	☐ Sufficient	☐ Not enough(see comment)	☐ Appropriate	
	☐ No head space on VOA water samples		☐ RAD Screened	
Comment:				

Sample Control #	Client ID	Discrepancy	Corrective Action

APPENDIX 10 DATA FLOW MANAGEMENT



APPENDIX 11 NON-CONFORMANCE REPORT (NCR)

Instrument No.	
Project Name	
Analysis	

Analysis Date	
Batches Affected	
Lab Nos. Affected	

OUT OF CONTROL EVENT(S)

Initiator _____ Date _____

RECOMMENDED CORRECTIVE ACTION

Supervisor Date _____

ACTION TAKEN

Analyst _____ Date _____

Reviewer Date _____

QA/QC COMMENTS

QA/QC Officer Date _____

APPENDIX 12 ADDENDUM FORM

	ADDENDUM TO	
	Document	
	Revision Number	
	Section	
	Date	
	Reference Number	

PREPARED BY: _____ Date: _____

Name _____

Title Project Manager

REVIEWED BY: _____ Date: _____

Name _____

Title QA Manager

APPROVED BY: _____ Date: _____

Name Kam Pang

Title Laboratory Director

MEMO

==========

February 26, 1998

To : Distribution

From : Kenette Pimentel

RE : **HOLDING TIME FOR DISSOLVED GASES**

It has bee noticed that there is a misprint in holding time specified in Section 5.0 of SOP AP-2050, Dissolved Methane, Ethene, and Ethane by GC Headspace Equilibration Technique, Revision No. 0.

The correct Holding Time should be 14 days from the time of collection.

Please take note of the correction. If you have received an issue of the SOP, please attached this memo to it.

Thank you for your cooperation.

STANDARD OPERATING PROCEDURE

DISSOLVED METHANE, ETHENE, AND ETHANE IN WATER BY A GC HEADSPACE EQUILIBRATION TECHNIQUE

SOP No.:	AP-2050	Revision No. 0	Date: 4-Apr-97
Prepared By:	Phillip Lee		Date: 4/4/97
Approved By:	Kevin Hoang QA Manager		Date: 4/4/97
Approved By:	Kam Pang Laboratory Director		Date: 6/4/97

Control Number: 2050-00-

1.0 SCOPE AND APPLICATION

1.1 This method is used to determine the concentration of dissolved methane, ethene and ethane in water. It is an adaptation of the procedure published in International J. Environmental Analytical Chem. Vol. 36, p. 249, by Kampbell et al (1989).

2.0 SUMMARY OF METHOD

2.1 This method provides a sample preparation method to generate headspace within sample container and then describes gas chromatographic conditions for the detection of methane, ethene and ethane in water by analyzing gas sample taken from the head space.

3.0 METHOD DETECTION LIMITS AND REPORTING LIMITS

3.1 Method detection limits and reporting limits are presented in Appendix 1.

4.0 DYNAMIC RANGE

4.1 Methane: 0.7 to 34 µg/L

4.2 Ethene: 1.2 to 59 µg/L

4.3 Ethane: 1.3 to 63 µg/L

5.0 SAMPLE COLLECTION, PRESERVATION, AND HOLDING TIME

5.1 Water samples should be stored in 40 ml vial with teflon-lined septa preserved with 0.10 ml of 6 N HCl at 4°C. The holding time is 14 days from the time of collection. For unpreserved water samples, the holding time is 7 days. Samples should be collected by adding water to the vial down the side to prevent agitation. Also, care must be taken to make sure that no air bubbles are entrapped in the sample.

6.0 ASSOCIATED SOPs

GP-0020	Standard Preparation
GP-2023	Analytical and QC Sample Labeling

STANDARD OPERATING PROCEDURE

DISSOLVED METHANE, ETHENE, AND ETHANE IN WATER BY A GC HEADSPACE EQUILIBRATION TECHNIQUE

SOP No.: AP-2050 Revision No. 0 Date: 4-Apr-97

7.0 SAFETY

7.1 All reagents, standards, and samples shall be treated as potential hazards. Observe the standard laboratory safety procedures. Protective gear, i.e., lab coat, safety glasses, gloves, shall be worn at all times when performing this procedure.

7.2 All wastes generated during analytical process shall be placed in the wastes containers. These wastes shall be endorsed to waste disposal section for proper disposal.

7.3 Water samples shall be neutralized to pH 7 ± 2 prior to disposal.

7.4 If for any reason, solvent and/or other reagents gets in contact with your skin or any other part of your body, rinse the affected body part thoroughly with tap water. If irritations persist inform your supervisor immediately so that proper action can be taken.

8.0 INSTRUMENTS, CHEMICALS, AND SUPPLIES

8.1 Instruments and Supplies

8.1.1 Gas Chromatography: Varian 3400 with FID

8.1.2 Column: SPB-1, 60 m x 0.53 mm ID, 5 µm thickness

8.1.3 Gas: ultra-high purity helium; ultra-high purity hydrogen

8.1.4 Syringes: 5, 25 and 50 ml Luerlok hypodermic gas-tight with shut-off valve

8.1.5 Microsyringes: 25, 100, and 250 µl with a 0.006 mm ID needle (Hamilton 702N or equivalent) for dilution purposes

8.1.6 Data System: P-E Nelson

8.2 Chemicals and Reagents

8.2.1 Solvent: Organic-free water

9.0 STANDARDS

9.1 Stock standards are purchased as certified gas mixture at 1% (v/v).

9.2 Working Standard

9.2.1 Take 5 ml of 1% standard and mix with 95 ml Helium in a tedlar bag. This will create a standard at 500 µL/L.

9.2.2 Take 1 ml of 1% standard and mix with 99.0 ml of Helium. This will create a standard at 100 µL/L.

9.2.3 Take 10 ml from 9.2.2 and mix with 90 ml of Helium. This will create a standard at 10 µL/L.

STANDARD OPERATING PROCEDURE

DISSOLVED METHANE, ETHENE, AND ETHANE IN WATER BY A GC HEADSPACE EQUILIBRATION TECHNIQUE

SOP No.: AP-2050 Revision No. 0 Date: 4-Apr-97

10.0 SAMPLE PREPARATION

10.1 Water sample is collected in the field in a 40 ml vial with a teflon septum. The exact volume of the bottle is measured by refilling with water.

10.2 Place the sample vial upside down in a three finger clamp. A 20 gauge needle on a 10 ml Luerlok glass syringe, set for dead volume, is inserted into the sample by penetrating the septum about one centimeter.

10.3 An 8 cm 22 gauge needle attached to Teflon lubing via a mininert syringe valve is then inserted through the septa to the top of the water.

10.4 A flow of 4 ml per minute of high purity helium is passes through the syringe valve. After 4 ml of water is forced from the bottle into the syringe, remove both needles. This procedure creates a headspace of 4 cc in the vial.

10.5 Shake the vial for five minutes by quick wrist motion to allow gas to equilibrate between the liquid and gas phases.

10.6 Samples are ready for analysis. It should be analyzed immediately.

11.0 INSTRUMENT PARAMETERS

11.1 Gas Chromatography Condition:

11.1.1 Column: 40°C, isothermal

11.1.2 Injector: 200°C

11.1.3 Detector: 220°C

11.1.4 Injection Volume: 100 µl

11.1.5 Helium Pressure: 10 PSI

11.1.6 Carrier gas flow: 9 mL/min

11.1.7 Make up gas flow: 20 mL/min

12.0 CALIBRATION

12.1 Calibration Standard

12.1.1 Following Equation 12.1, convert the calibration standards in µL/L into µg/L of dissolved gas in water.

$$C = \frac{V_H}{V_W} \times Cv \left(\frac{273}{273 + Tc}\right) \times \frac{1}{22.4} Mwt \qquad \text{Eq.-12.1}$$

where:

C - concentration of gas in water in ug / L

V_H - Head space volume (4 ml)

V_W - Total volume of water (39 ml)

Cv - concentration of gas in uL / L

T - room temperature ($23^{\circ}C \pm 2^{\circ}C$)

M - mol. wt. of gases

CH_4 - 16 (ug / umol)

C_2H_4 - 28 (ug / umol)

C_2H_6 - 30 (ug / umol)

12.1.2 Concentration (µg/L) of Calibration Standard in Water

	Std 1 (10 µL/L)	Std 2 (100 µL/L)	Std 3 (500 µL/L)
CH_4	.676 µg/L	.676 µg/L	33.8 µg/L
C_2H_2	1.182 µg/L	11.82 µg/L	59.1µg/L
C_2H_4	1.267 µg/L	12.67 µg/L	63.35 µg/L

12.2 Initial Calibration

12.2.1 3 initial calibration solutions are prepared as described in Section 9.3 and analyzed as described in Section 13.0.

12.2.2 Results are calculated as follows:

Calibration Factor	Equation 14.1.1
Average Calibration Factor	Equation 14.1.2
Standard Deviation	Equation 14.1.3
% RSD	Equation 14.1.4

12.2.3 Acceptance criteria are specified in Appendix 2 or check project specific requirements.

12.3 Continuing Calibration

12.3.1 Use Standard 2 to check the calibration factor in ten sample interval.

12.3.2 Results are calculated as follows:

Calibration Factor	Equation 14.1.1
% Difference (%D)	Equation 14.2

STANDARD OPERATING PROCEDURE

DISSOLVED METHANE, ETHENE, AND ETHANE IN WATER BY A GC HEADSPACE EQUILIBRATION TECHNIQUE

SOP No.: AP-2050 Revision No. 0 Date: 4-Apr-97

13.0 ANALYSIS

13.1 Set GC parameters as described in Section 11.

13.2 Analyze an instrument blank by injecting 100 µl of Helium to ensure the system is free of contamination.

13.3 Analyze an initial calibration curve.

13.3.1 Carefully fill a 40 ml VOA vial with reagent water. Care should be taken to avoid any trap bubbles.

13.3.2 Follow Sections from 10.2 to 10.5 to create 4 ml of head space with Standard 1 gas mixture (Section 9.2.2).

13.3.3 Immediately withdraw 100 µl gas from the head space and analyzed by GC.

13.3.4 Repeat Standard #2 and #3 with the same procedures.

13.3.5 Establish the ACF from initial calibration curves.

13.4 Prepare analytical sequence and analyze sample if ICAL meets the QC requirements. Sample is prepared individually by procedures described in Section 9.0 and analyzed immediately.

Analytical Sequence	Sample
1	Blank
2	LCS
3	Sample 1
4	Sample 2
5	Sample 3
6	Sample 4
7	Sample 5
8	Sample 6
9	Sample 7
10	Sample 8
11	Sample 9
12	Sample 10
13	DCC1
14	Sample 11
15	Sample 11/MS
16	Sample 11/MSD
17	Sample 12
18	Sample 13
19	Sample 14
20	Sample 15
21	Sample 16
22	Sample 17
23	Sample 18

24	DCC2
25	Blank
26	LCS
27-37	10 samples
38	DCC

The analytical sequence should be recorded in GC Run Log Book..

13.5 Identification

13.5.1 When a chromatographic peak falls in the retention time window (Section 13.8) of an analyte, which is considered as tentatively identified. Its concentration could be calculated according to Section 14.3 .

13.6 Retention Time Windows

Make three analyses of all analytes through a course of a 72-hour period. Calculate the standard deviation of the three absolute retention times for each analyte (Equation 14.1.3). Plus or minus three times the SD will be used to define the absolute retention time window. The daily retention time window derives from the average retention time of the analytes on ICAL plus and minus 3 * SD.

14.0 CALCULATIONS

14.1 Initial Calibration

14.1.1 Calculate for Calibration Factor (CF).

$$CF = \frac{R_a}{C_a}$$ Eq. - 14.1.1

where:

R_a – Response for analyte measured in peak area

C_a – Concentrationof analyte to be measure (ug / L)

14.1.2 Calculate for Average Calibration Factor (ACF)

$$ACF = \frac{\sum CF_a}{n}$$ Eq. 14.1.2

where:

ACF - average response factor

$\sum CF_a$ - sum of calibration factors

n - number of calibration points

14.1.3 Calculate for Standard Deviation

Eq.-14.1.3

$$SD = \sqrt{\frac{\sum_{i=1}^{N}(x_i - \bar{x})^2}{N-1}}$$

where:

SD = Standard Deviation

x_i = result at i^{th} measurement

$\bar{x}$ = mean

N = number of measurements

14.1.4 Calculate for % relative standard deviation (%RSD).

$$\%RSD = \frac{SD}{ACF} * 100\% \qquad \text{Eq.-14.1.4}$$

where:

SD - standard deviation

ACF - average response factor

14.2 Calibration Check/Continuing Calibration

$$\%D = \frac{ACF - CF}{ACF} * 100\% \qquad \text{Eq.-14.2}$$

where:

ACF - average response factor

CF - calibration factor at calibration check standard

14.4 Sample Results

14.4.1 Water Sample

$$C = \frac{R_a}{AFC} * D \qquad \text{Eq.-14.4.1}$$

where:

C - Concentrationof analyte to be measure (ug / L)

R_a - response for analyte measured in peak area

AFC - average response for an analyte

D - sample dilution factor

14.5 Accuracy and Precision

14.5.1 Percent Recovery

$$\%R = \frac{C_f - C}{C_s} * 100 \qquad \text{Eq.-14.5.1}$$

where:

$\%R$ - percent recovery

C_f - concentration found

C - sample concentration

C_s - concentration of spike

14.5.2 Relative Percent Difference

$$\%RPD = \left[\frac{R_1 - R_2}{R_{ave}} * 100\% \right] \qquad \text{Eq.-14.5.2}$$

where:

R_1 - % recovery of first measurement

R_2 - % recovery of first measurement

R_{ave} - average of % recoveries

14.6 Method Detection Limit

MDL = 3.14 * SD Eq.-14.6

where:

MDL - method detection limit

SD - standard deviation (see Eq-14.1.3) from 7 measurements.

15.0 QUALITY CONTROL

15.1 Sample Preparation

15.1.1 The maximum number of original field samples in an analytical batch shall be 20 unless otherwise specified by the project.

15.1.2 All labware used in the sample preparation shall be properly treated as specified in GP-0002.

15.2 Analytical Batch

15.2.1 An instrument/solvent blank shall be performed prior to the first daily continuing calibration. The acceptance criteria shall be the same as the method blank. If the blank fails to meet the acceptance criteria, identify and correct the problem before proceeding to the analytical process.

15.2.2 Samples shall be analyzed at an acceptable initial calibration. Acceptance criteria is specified at Appendix 2.

If the initial calibration %RSD is outside the acceptance limits. Prepare a new initial calibration. If the problem persists, identify whether the deviation is due to a high standard or a low standard. Once identified, adjust the standard so that it falls within the acceptable range. In this process, the range of concentration is narrowed.

15.2.3 A continuing calibration shall be performed as specified in the QC schedule.

If the continuing calibration %D exceeds the allowed limits. The analyst may repeat the continuing calibration with a freshly prepared standard.

If the newly prepared standard does not meet acceptance criteria, a new initial calibration shall be performed.

All samples analyzed after the last acceptable continuing calibration must be re-analyzed.

15.2.4 A method blank, LCS, and MS/MSD shall be prepared in every analytical batch, unless otherwise specified by the project. They shall be subjected to the same analytical process as that of the field samples (Section 10.0).

15.2.5 Organic free water shall be used for method blank and LCS.

15.2.6 A Laboratory Control Sample at 100 µl/L (LCS or method blank spike) shall be analyzed for each analytical batch and all recoveries shall be within the acceptable limits (Appendix 2) prior to sample analysis.

15.2.7 Matrix spike samples shall be taken from a specified field sample within the analytical batch. In the absence of any designation, the laboratory may choose any sample to spike. The spike concentration shall be 100 µl/L.

15.2.8 In the event that there is insufficient amount of sample to be used for MS/MSD, LCS duplicate shall be prepared to demonstrate precision.

15.2.9 If MS/MSD recoveries were not within the acceptable range, and the concentration of the unspiked sample is $\geq$ 4 x the unknown matrix spike concentration, flag the sample result as "J" (estimated), due to precision uncertainty associated with matrix effect. However if the concentration of unspiked sample is < 4 x, flag all the associated samples as "J", due to precision uncertainty possibly caused by matrix effect.

If MS/MSD recoveries were within the acceptable, but the RPD is not within the acceptable range, flag the associated data as "J" (estimated) due to precision uncertainty.

However, if the project specifies other than as stated above, the project requirement shall prevail.

15.3 Method QC

15.3.1 All analyses must be performed on a valid initial calibration curve. The initial calibration procedure is discussed in Section 12.1 and acceptance criteria is described in Appendix 2.

15.3.2 Determine the method detection limit (MDL) at least once a year.

15.3.3 Reporting limit shall be 2 to 10 times of the MDL.

15.3.4 Update the in-house quality control limits (IQC) at least once a year.

15.3.5 Update the Retention Time Window at least once a year or when a major instrument repair is done.

16.0 PREVENTIVE MAINTENANCE

16.1 Clean the purge needles thoroughly with high purity helium, after every use to ensure that there are no particulates adhering to it.

16.2 Check the gas flow from time to time to ensure that ideal gas flow is maintained accordingly.

16.3 Maintain an inventory of instrument parts and supplies for routinary maintenance. See Appendix 4 for Minimum Instrument Parts & Supplies Inventory List.

17.0 CORRECTIVE ACTION

17.1 If a QC parameter proves to be unsatisfactory, corrective action shall be implemented. Sample analysis should be stopped immediately. The Department Group Leader or the Department Supervisor, and sometimes the QA officers, may be involved in the corrective action. Corrective Action Form must be filed and circulated to the appropriate staff.

If previously reported data are affected by a situation that requires correction, or if the corrective action impacts a project budget or schedule, the action will directly involve the project manager.

17.2 For immediate or long-term corrective actions, steps comprising a closed-loop corrective action system are as follows:

- Define the problem.
- Assign responsibilities for problem investigation.
- Investigate and determine the cause of the problem.
- check all calculations
- verify the integrity of the spiking solution, LCS, or DCC
- check instrument and operating conditions to preclude the possibility of malfunctions or operator error.
- Determine the corrective action(s) necessary to eliminate the problem.
- Assign and accept responsibilities for implementing the corrective action.
- Establish the effectiveness of the corrective action and implement the correction.
- Verify and document that the corrective action has eliminated the problem.

18.0 SUPPLEMENTARY NOTES

None.

19.0 REFERENCES

19.1 DH Kampbell, JJ Wilson and S.A. Vandegrift. "Dissolved Oxygen and Methane in Water by a GC Head space Equilibration Technique". Internation Journal Environ Analytical Chem., Vol 30, pp. 249-257, 1989

19.2 Corporate QA/QC Manual, as updated.

20.0 APPENDICES AND TABLES

Appendix 1 Analyte list with RL

Appendix 2 In-house QC Schedule

SCHEDULED QC
DISSOLVED METHANE, ETHENE, AND ETHANE IN WATER

QC Procedure	Frequency	Acceptance Criteria	Corrective Action
Instrument Blank	At the beginning of an analytical sequence	Free of any target analyte above R.L.	Clean the system before any analysis.
3 point Initial Calibration	Daily or if needed	RSD < 20%	Repeat before sample analysis.
Continuing Calibration (Calibration Check)	Every 10 samples and at the end of a sequence	%D < 15	Repeat before sample analysis.
Method Blank	Every batch < 20	Free of any target analyte above R.L.	Repeat until the system is free of contamination.
LabControl Sample (Blank Spike)	Each batch < 20	60-130% of true value	Reanalyze LCS and all associated samples.
Matrix Spike/Matrix Spike Duplicate	Each batch < 20	60-130% RSD = 30%	No action based on MS/MSD alone

EMAX LABORATORIES, INC., 630 Maple Ave., Torrance, CA 90503 TEL: (310) 618-8889 FAX: (310) 618-0818

METHOD DETECTION LIMIT STUDY
DISSOLVED METHANE, ETHENE AND ETHANE

Control Number	97E -
Matrix	Water
Extraction SOP	AP-2050
Analysis SOP	AP-2050
Adaptation (SW846)	

Standard	Standard Source	Lab Standard ID
Initial Calibration	Scott	S14A01-01-01
		S14B01-01-01
Spike	Scott	S14B01-01-01

Initial Cal. Date	4/4/97
Extraction Date	4/4/97
Analysis Date	4/4/97
Effectivity Date	Apr-97 Apr-98
Analyst	P. Lee

COMPOUND	True Value	1		2		3		4		5		6		7		Ave. Conc.	Ave.	SD	MDL (3.14XSD)	PQL
(Water)	ug/L	ug/L	%R	ug/L	%R	ug/L	%R	ug/L	%R	ug/L	%R	ug/L	%R	ug/L	%R	ug/L	%R		ug/L	ug/L
Methane	1.234	1.256	1.018	1.203	0.975	1.34	1.086	0.946	0.767	1.025	0.831	1.189	0.964	1.07	0.867	1.15	0.93	0.139	0.435	1
Ethene	1.182	1.383	1.170	1.401	1.185	1.334	1.129	1.282	1.085	1.204	1.019	1.309	1.107	1.2	1.016	1.30	1.10	0.079	0.249	1.2
Ethane	1.267	1.340	1.058	1.264	0.998	1.178	0.930	1.304	1.029	1.204	0.950	1.256	0.991	1.108	0.875	1.24	0.98	0.079	0.248	1.3

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Committed To *Your* Success

Severn Trent Laboratories
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to reflect the change of ownership to

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Monroe CT 06468

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IEA-CT Laboratory Quality Assurance Program

Prepared by:

Marsha K. Culik
QUALITY ASSURANCE MANAGER

for

IEA - Monroe, Connecticut

This document has been prepared by IEA Corporation and will be updated annually. The material contained herein is not to be disclosed to, or made available to any third party without the prior expressed written approval of the Corporate Quality Assurance Director.

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APPENDIX

1.0 QUALITY ASSURANCE PROGRAM-IDENTIFICATION FORM

Document Title: **IEA-CONNECTICUT QUALITY ASSURANCE PROGRAM PLAN**

Corporate Address: IEA Connecticut
200 Monroe Turnpike
Monroe, Connecticut 06468

Company Official: Mr. Michael V. Bonomo
Title: Vice President of Connecticut Operations
Telephone: (203) 261-4458

Company Official: Mr. Jeffrey C. Curran
Title: Laboratory Manager

Company Official: Ms. Marsha K. Culik
Title: Quality Assurance Manager

Plan Coverage: IEA-Connecticut Laboratory including the following functions:

- Administration
- Sample Receipt
- GC Laboratories
- Quality Assurance
- Data Entry
- Report Production
- Computer Systems
- Inorganics Laboratories
- GC/MS Laboratories
- Facilities and Safety
- Sample Preparation Laboratories

Concurrences:

Name: Mr. Michael Bonomo
Title: Vice President of Connecticut Operations
Signature: Michael V. Bonomo
Date: 2/20/97

Name: Mr. Jeffrey Curran
Title: Laboratory Manager
Signature: Jeffrey C. Curran
Date: 02/20/97

Name: Ms. Marsha Culik
Title: Quality Assurance Manager
Signature: Marsha K. Culik
Date: 2/20/97

Name: Mr. David Houle
Title: President - IEA
Location: Cary, North Carolina
Signature: David E. Houle
Date: 2/17/97

2.0 INTRODUCTION

2.1 Background

Industrial & Environmental Analyst's, Inc. (IEA) is a full-service environmental organization specializing in laboratory analytical services and field support services.

The IEA organization is a network of seven (6) integrated environmental laboratories located throughout the Eastern United States with over 300 employees, making it one of the top ten environmental testing companies in the United States. The corporation serves a broad range of industries including environmental consulting and engineering firms, state and federal agencies, pharmaceutical, petroleum, and electronic component manufacturers. In support of these activities the corporation presently maintains environmental laboratory certifications in over twenty five state programs. IEA Corporate headquarters are located in Cary, North Carolina.

IEA is a wholly-owned subsidiary of the AQUARION Company, headquartered in Bridgeport, Connecticut. AQUARION is listed on the New York Stock Exchange and has annual revenues exceeding 100 million. It is also the largest investor-owned water utility in the country.

The IEA laboratories are located as follows:

IEA/Connecticut	Monroe
IEA/Illinois	Schaumburg
IEA/North Carolina	Cary
IEA/NC-Radiological	Morrisville
IEA/Massachusetts	N. Billerica
IEA/New Jersey	Whippany

Detailed information such as mailing addresses and telephone numbers for each of the laboratories is presented in Table 2.2.1.

HISTORY OF IEA

IEA was founded in 1977, in Burlington, Vermont, as a water resources testing facility in support of IBM's facility in Essex Junction, Vermont. IEA served the IBM site exclusively for three years performing ultrapure water analysis, wastewater treatment and pollution control. In 1982, IEA opened a second facility in Research Triangle Park (RTP), North Carolina in order to provide desired services from the IBM facility in RTP. In 1984 IEA expanded its market and began serving the developing environmental testing market. By 1985 IEA had expanded to a full service laboratory offering complete soil and water analysis, field sampling, groundwater analysis and evaluation of hazardous waste. The North Carolina laboratory, which serves as IEA's corporate headquarters, is located in Cary, North Carolina.

In the fall of 1988, IEA positioned itself as one of the leading laboratories in the country by qualifying for the USEPA Contract Laboratory Program (CLP). This development created a favorable position for winning major consulting engineering contracts. As such, IEA grew rapidly and expanded its commercial client base considerably. Due to the rapid increase in demand for environmental services IEA sought potential buyers in 1989 in order to provide resources for future expansion. As a result, IEA was purchased by The Aquarion Corporation, based in Bridgeport, Connecticut in 1989. Aquarion is a New York Stock Exchange-listed corporation that traces its roots to 1857. It has the distinction of being the largest investor-owned water utility in the nation. Annual revenues of Aquarion exceed 100 million.

Since the initial purchase, IEA has acquired several existing environmental laboratories which were operated in strategic locations along the Eastern United States. As a result, IEA now offers very comprehensive environmental testing services including mixed waste radiological testing and a full range of chemical testing performed in support of DOD, DOE, RCRA, CERCLA, NPDES, TSCA and SDWA regulations.

This plan is intended to describe the quality assurance program of the IEA-Connecticut facility located at 200 Monroe Turnpike, Monroe, Connecticut. IEA operates a corporate wide quality assurance program (Doc.# QAQ00102.NET) and this facility QA program complies with the requirements set forth in the corporate program. In some cases, the requirements in the facility QA program may be more stringent than the corporate program, but in no case can they be less stringent.

TABLE 2.1.1 IEA NETWORK LOCATIONS

North Carolina
Corporate Headquarters
3000 Weston Parkway
Cary, NC 27513
(919) 677-0090
(919) 677-0427 (Fax)
(800) 444-9919

Connecticut
200 Monroe Turnpike
Monroe, CT 06468
(203) 261-4458
(203) 268-5346 (Fax)

North Carolina
Radiological Laboratory
120 South Center Court
Suite 300
Morrisville, NC 27560
(919) 460-8505
(919) 469-2646 (Fax)

New Jersey
628 Route 10
Whippany, NJ 07981
(201) 428-8181
(201) 428-5222 (Fax)

Massachusetts
149 Rangeway Road
N. Billerica, MA 01862
(617) 272-5212
(508) 667-7871 (Fax)
(800) 950-5212

Illinois
126 West Center Court
Schaumburg, IL 60195
(708) 705-0740
(708) 705-1567 (Fax)
(800) 933-2580

2.2 Definition of Terms

A number of terms are used within this document to describe the corporate QA program in effect at IEA laboratories. To ensure effective communication, the following terms are being defined:

Accuracy	- the degree of agreement of a measurement with an accepted reference or true value. Accuracy is usually expressed as the difference between the measurement and the true value. It is a measurement of the bias in a system.
Analytical Report Turnaround Time	- in order to ensure proper communication is maintained, IEA has defined analytical report turnaround times to be always based upon calendar days, not business days. Analytical holding times are also based on calendar days.
Audit	- a systematic check to determine the quality of some function or activity. Audits may be of two basic types, performance audits or system audits. Performance audits involve a quantitative comparison of the labs results to that of a proficiency sample containing known concentrations of analytes. A system audit is a qualitative evaluation that normally consists of an on-site review of a laboratory's quality assurance system and physical facilities.
Batch	- the basic unit for analytical quality control. It is defined as a group of samples which are analyzed together with the same method sequence and the same lots of reagents and with the manipulations common to each sample within the same time period or in continuous sequential time periods. Samples in each batch should be of similar composition (matrix). At IEA laboratories, the maximum batch size has been set at 20 samples. At IEA's smaller laboratories where the number of samples received daily may be low, samples received in a given week may be combined into one analytical batch. Due to holding time constraints, individual samples may be extracted on different days as compared to other samples in the batch. If this is the case, a method blank must be performed daily with every sample extraction. The other QC samples such as MS and MSD are only performed for the total analytical batch.
Comparability	- a measure of the confidence with which one data set can be compared to another.
Completeness	- a measure of the amount of valid data obtained from a measurement system compared to the amount that was expected to be obtained under routine operating conditions.
Data Quality Objectives	- during the planning phase of a project requiring laboratory support, the data user must establish the quality of data required from the investigation. Such statements of data quality are known as data quality objectives (DQOs). The DQOs are qualitative and quantitative statements of the quality of data required to support specific decisions or regulatory actions.
Data Validation	- a systematic effort to review data to identify any outliers or errors and thereby cause deletion or flagging of suspect values to assure the validity of the data to the user. This process may be done by manual or computer methods.
Field Blank	- contaminant free water, or appropriate matrix, used during sampling activities to determine if there is any potential for sample contamination associated with the field sampling or equipment.
Library Search	- a technique used by which a mass spectrum of an unknown compound is compared to the mass spectrum of compounds contained in a computer library in an effort to identify

unknown compounds. Compounds identified in this manner are referred to as "tentatively identified compounds" (TICs).

Matrix Spike - the process of adding a known amount of analyte to a sample and analyzing the sample. The amount of analyte recovered is calculated as a percent recovery. This technique is used to assess accuracy of analysis.

Matrix Spike Duplicate - a second matrix spike is compared to the results of the matrix spike to assess precision of the analysis.

Method Blank - contaminant free water, or appropriate matrix, taken through the entire analytical process to determine if there is any contamination associated with the analytical procedures.

Method Detection Limit (MDL) - the minimum concentration of a substance that can be measured and reported with 99% confidence that the analyte concentration is greater than zero.

Practical Quantitation Limit (PQL) - is the lowest level that can be reliably achieved within specified limits of precision and accuracy during routine operating conditions.

Precision - a measure of mutual agreement among individual measurements of the same property, usually under prescribed similar conditions. Precision is usually expressed in terms of standard deviation.

Quality Assurance (QA) - the total integrated program put in place to assure the reliability of data generated in the laboratory.

Quality Control (QC) - the routine application of specific, well-defined procedures which ensure the generation of data which fulfill the objectives of the QA program.

Quality Assurance Program Plan (QAPP) - a written assembly of management policies, objectives, principles and general procedures which outline how the laboratory intends to generate data of known and accepted quality.

Quality Assurance Project Plan (QAPjP) - a written document, which presents, in specific terms, the policies, organization, objectives, functional activities and specific QA/QC activities designed to achieve the data quality objectives of a specific project. There are 16 essential elements which EPA has mandated to be addressed in a project plan.

Relative Percent Difference (RPD) - relative percent difference (RPD) is used as the measure of precision between sample duplicates. The formula utilized to calculate RPD is as follows:

Relative Percent Difference (RPD)

$$RPD = \frac{\text{(Sample Result - Duplicate Result)}}{\text{Mean of Sample and Duplicate Results}} \times 100$$

Note: RPD is expressed as the absolute value obtained from the above formula.

Representativeness	- the degree to which data accurately and precisely represents a characteristic of a population, parameter variations at a sampling point, or an environmental condition.
Standard Operating Procedure (SOP)	- a detailed, written description of how a laboratory executes a particular procedure or method. It is intended to standardize the performance of the procedure.
Surrogates	- generally, organic compounds which are not target analytes, that are added to samples to assess analytical performance of a method. These compounds are spiked into all blanks, samples and spiked samples prior to analysis. Percent recoveries are calculated for each surrogate.
Trip Blank	- contaminant free water, or appropriate matrix, which accompanies bottles and samples during shipment to assess the potential for sample contamination during shipment. Trip blanks are not opened in the field.
Tuning	- a technique used in GC/MS procedures to verify that the instrument is properly calibrated to produce reliable mass spectral information.

2.3 Purpose

The IEA-Connecticut quality assurance program serves as an operational charter for the organization. It defines the purpose, organizational structure, and operating principles of the laboratory and presents an overview of the key elements of the quality assurance program. This quality assurance program will be reviewed and modified as necessary on an annual basis. Any deviation from this program must be approved in writing by the facility QA manager and copied to the President.

This quality assurance program has been prepared according to guidelines presented in the USEPA document entitled "Guidelines and Specifications for Preparing Quality Assurance Program Plans", Office of Monitoring Systems and Quality Assurance, Office of Research and Development, USEPA, (QAMS-004/80), EPA-600/8-83-024, June, 1983.

2.4 Scope

This QA program applies to the generation of analytical data at the IEA-Connecticut lab location. Since the vast majority of environmental client needs are driven by various federal and state regulations, the program has been designed to meet the requirements of the following programs:

Clean Water Act (CWA)
Clean Air Act (CAA)
Safe Drinking Water Act (SDWA)
Resource Conservation and Recovery Act (RCRA)
Comprehensive Environmental Response, Compensation and Liability Act (CERCLA)

This Quality Assurance Program Plan (QAPmP) covers laboratory operation at IEA-Connecticut. The purpose of this QAPmP is to provide information on laboratory operations as required for specific Quality Assurance Project Plans (QAPjPs), and to provide the basis for the Quality Assurance Program at IEA-Connecticut. This program is based on the IEA Corporate Quality Assurance Program Plan (Doc# QAQ00102.NET).

This QA program applies to the generation of analytical data utilized for environmental monitoring and assessment programs. The major types of laboratory support for government regulations are as follows:

- Analysis and characterization of environmental (soil, sediment, water and air) and waste samples per the Resource Conservation and Recovery Act (RCRA) for either compliance, disposal or delisting purposes.
- Analysis of drinking water samples in support of the Safe Drinking Water Act (SDWA).
- Analysis of environmental samples in accordance with contracts with the USEPA CLP program and various state agencies (CERCLA and NYSDEC) and government agencies, such as Army Corps of Engineers.
- Analysis of environmental samples in accordance with contracts through the AFCEE Program (Air Force) in accordance with the AFCEE QAP.
- Analysis of environmental samples (soil, sediment, water and air) for contaminants such as those compounds found on the EPA priority pollutant list, target compound list, etc. for site assessment purposes.
- Analysis of waste stream samples in accordance with NPDES requirements.

3.0 QUALITY ASSURANCE POLICY STATEMENT

It is the intention of IEA corporation to consistently produce analytical data of known and documented quality at all network laboratories which fully meet clients' data quality objectives.

The contents of the QA program describe the activities which are utilized in order to ensure this commitment is maintained.

IEA Quality Policy

"Management and staff are committed to maintaining a carefully controlled analytical environment in order to ensure the consistent generation of accurate data which meets or exceeds the data quality objectives of our clientele."

IEA recognizes that maintaining a proper ethical standard is an important element of an effective quality assurance program. In order to ensure that all personnel understand the importance the company places on maintaining high ethical standards at all times, IEA has established an "Ethics Policy" and it is presented for your information. This policy is used to set the standard within the organization for day-to-day performance. Each employee is requested to sign the ethics policy, signifying agreed compliance with it's stated purpose. Copies of all signed ethics policy statements are maintained in personnel files.

IEA ETHICS POLICY

The management of IEA corporation recognizes our responsibility to clients and fellow employees to ensure that fair and ethical business practices are followed at all facilities.

Our clients have placed their trust in our organization to continually provide high quality data which is valid, defensible and represents sound professional judgement at all times. In order to meet this responsibility it is imperative that high ethical standards be maintained at all times by all employees.

The management and staff are committed to maintaining a carefully controlled analytical environment which assures the consistent generation of accurate data which meets the data quality objectives of our clientele.

The following represents the IEA ethics policy which has been adopted to clearly identify the corporate position on ethical practices. Failure to comply with this policy cannot and will not be tolerated.

The Company and all its Employees will:

- Fully comply with all applicable federal, state, and local laws and regulations.
- Produce analytical products that are accurate, defensible and which represent sound professional judgement at all times.
- Provide employees with guidance and an understanding of the ethical and quality standards required in the environmental industry. In this regard, all employees should feel free to identify any ethical misconduct without fear of retribution. Any employee involved in any form of ethical misconduct will be subject to immediate disciplinary action including potential termination of employment.
- Present services to clients in a confidential, honest and forthright manner and strive to deliver quality products at a fair price.
- Treat employees equitably by compensating them fairly, acknowledging their scientific contributions, and providing them opportunities for professional growth and development.
- Offer employment opportunities to qualified candidates regardless of their race, creed, color, sex or age.
- Be a responsible corporate citizen of the community by operating in an environmentally sound manner at all times.
- Maintain all facilities in a safe and professional manner through maintenance of a safety awareness program and provide the necessary safety equipment and training to protect all employees from preventable injury and chemical exposure.

4.0 QUALITY ASSURANCE MANAGEMENT

4.1 Introduction

The management of IEA-Connecticut is committed to the execution of the quality assurance program described in this document. The officers of IEA as well as lab directors and lab managers are required to comply with the program's stated goals, requirements and responsibilities.

In addition, each staff member has a responsibility to ensure compliance at all times with the QA program.

4.2 Assignment of Responsibilities

The primary objective of the network quality assurance program is to ensure that systems are in place such that all network laboratories consistently generate high quality analytical data.

Additionally, the QA program provides a mechanism to identify and implement policies to improve the quality of products and services. Records must also be maintained to document the laboratory's performance.

Quality assurance at IEA is monitored at both the corporate and laboratory levels. IEA's network quality assurance program is led by the president of IEA. The QA program at each network lab is directed by the QA manager at that facility, who reports directly to the laboratory's director and indirectly to the president. Figure 4.2.1 presents the organizational structure of network quality assurance functions and Figure 4.2.2 illustrates the overall general management of the corporation.

The following provides a listing of responsibilities and authority of key managerial personnel. Section 5 of the Appendix presents the organizational structure of the IEA-Connecticut facility.

Vice President of Operations

Responsibility:

All corporate directors and managers comply with the quality assurance program and require similar compliance by all staff personnel.

Ensure that all laboratory operations under their control are active participants in attaining the network quality assurance objectives.

Ensure compliance with methods and procedures as written.

Timely compliance with any corrective action requirements.

Ensure that instrument tunings and calibrations are performed at the required frequency and that instrument maintenance and logbooks are maintained in an orderly manner.

Authority:

Maintain the authority to suspend or terminate employees for dishonesty, or non-compliance with established QA policies and procedures.

The directors' or managers' authority is granted from the president of IEA, to whom they report.

Laboratory Manager

Responsibility:

Ensure compliance with methods and procedures as written.

Ensure that analytical procedures are performed in accordance with the requested method and SOPs.

Oversee preparation of analytical reports and data review.

Authority:

Maintain the authority to suspend or terminate employees for dishonesty, or non-compliance with established QA policies and procedures.

Authority is granted from the Vice President of Operations, to whom they report.

Laboratory Quality Assurance Manager

Responsibility:

Responsible for recommending pertinent additions to the network QA program.

Responsible for monitoring and assessing compliance of the laboratory with the requirements contained in the QA program.

Function as a liaison between the corporate QA director and laboratory staff at their facility.

Represent the laboratory during all external audits conducted by clients or regulatory agencies.

Conduct audits and inspections to assess compliance with established methods, policies and procedures. Results of these audits are reported to the network QA director and the laboratory director.

Maintain a document control system containing current policies and procedures utilized by the laboratory.

Maintain various certification programs for the laboratory.

Review laboratory performance on various QC proficiency samples submitted to laboratories by state and federal agencies.

Inform local and corporate management of the status of the QA program at the particular facility through a monthly QA report.

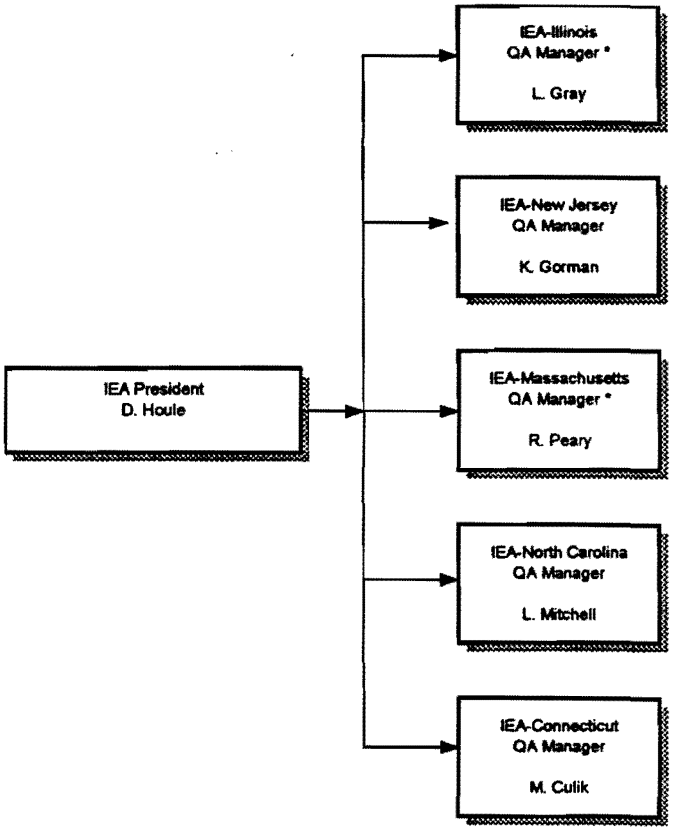
Investigate all inquiries relative to data quality issues and follow up on corrective action if necessary.

Authority:

The quality assurance staff has the authority to stop or change any analytical procedure in order to assure that data quality is maintained.

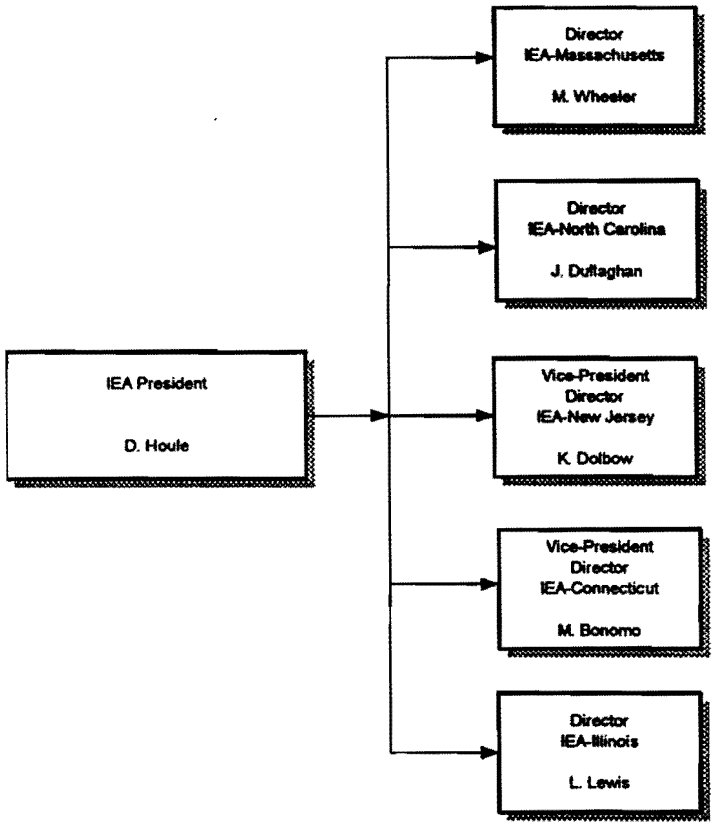
The authority of the QA staff is granted by the director of the facility.

FIGURE 4.2.1 NETWORK QUALITY ASSURANCE ORGANIZATIONAL CHART



* Above positions are part-time QA positions. These individuals also have operational responsibilities.

FIGURE 4.2.2 NETWORK ORGANIZATIONAL CHART



4.3 Communications

The quality assurance department communicates internally and externally through various means. Communication can take place via telephone, memoranda or take the form of audit reports. At the present time, the quality assurance staff participates conference calls to discuss relevant issues and disseminate information.

In addition, various quality assurance reports are routinely generated as discussed in section 4.5.

4.4 Document Control

A system of document control is essential to provide the framework necessary to ensure that methods and procedures are followed in a consistent manner.

IEA has developed a centralized document control system which is maintained for the entire network and is administered by the corporate staff located at the Cary, North Carolina facility. The document control system provides for the following:

- A unique document control number for each document
- A central location for all documents
- A systematic method for distribution of approved documents
- A tracking system for existing documents
- Identification of document revisions
- A mechanism for periodic review of documents
- Archival of outdated material
- A focal point for information exchange
- Facilitates the establishment of standardized methods and procedures

A detailed description of the document control system is contained in IEA document number QAS00101.NET. This document is available for inspection and review during a site visit. The Quality Assurance Manager is responsible for ensuring that the document control system is properly managed. Any new or revised document must be submitted to the QA Manager for review and distribution.

It is the responsibility of all members of the laboratory to maintain complete records of all operations performed. All records shall be neat and organized. All laboratory records are the property of the laboratory and shall not be removed from the premises without permission from supervisors. All records are considered confidential and must be safeguarded. Unauthorized changes, loss or destruction of records can be grounds for dismissal from the laboratory. Consult the IEA, Inc. Ethics Policy regarding integrity of data and employee conduct.

Measurement records must be recorded in pre-printed record logs or pre-printed measurement logs. This policy will facilitate the organization and archival of all laboratory data for future reference.

All injection forms, instrumentation forms, sample prep forms, QC forms, etc. which are used to process samples and measurement results are described and attached to each analytical SOP. The SOP specifies where these records and forms are cataloged and stored.

All measurement data is recorded in logbooks or on pre-printed log sheets in permanent ink. Transcriptions will be avoided whenever possible. The record will reflect the measurement performed and all appropriate details for conclusions related to the measurement. The record must be initialed and dated by the individual performing the measurement on the day the measurement is performed. Corrections shall be made by drawing a single line through the error, initialing and dating the error. All forms will be reviewed by the QA Manager annually. If it is found that the document does not meet the requirements of the SOP, the discrepancy is forwarded to the group/section leader through the corrective action process (reference SOP on Corrective Action Reports -QAS00501.CT). Further detail on laboratory document control is found in the SOP on Document Control - QAS00301.CT.

4.5 QA Program Assessment

The quality assurance program can only accomplish its objectives if management and staff are committed to adherence to the program. In order to assess continued compliance and to identify strong and weak points of the program, annual assessments are performed at each location.

Each quality assurance manager conducts an annual audit of the particular laboratory. This may include the entire lab or only particular departments depending on the frequency of external audits and their results. A copy of the audit along with any proficiency test results obtained are submitted to the president.

A written status report is prepared monthly by each of the facility QA managers. A copy of this report is issued to the facility laboratory director as well as the corporate president. The corporate staff provides a summary of these reports each month to upper management. A typical status report would include such information as:

- Changes in the quality assurance program
- Summary of proficiency results at each network lab
- Summary of on-time report issuance
- Changes in certification status
- Summary of system audits conducted at each network lab
- Significant QA concerns and recommendations for resolution
- Accomplishments since the previous report

4.6 Additional Lab Policies to Achieve QA Objectives

In addition to policies and procedures specified in other sections of this document there are numerous policies and standard procedures which have been implemented to ensure that data of known quality is continually generated by all network laboratories. Examples and a brief description of a few of these additional policies are presented below:

4.6.1 Participation in EPA Water Supply and Water Pollution Proficiencies

The USEPA currently operates a Water Supply (WS) and a Water Pollution (WP) proficiency program. Each program consists of the issuance of proficiency samples twice in a calendar year. Analysis of proficiency samples on the second set of samples in a year are only required by EPA for those parameters which the laboratory failed during the first round in a given year. As part of IEA's QA program, full participation and analysis of all appropriate parameters is required of all IEA labs regardless of past performance. This serves as an important indicator on the continuing quality of data being generated at each facility.

The laboratory also participates in the NYSDOH proficiency testing program for Potable Water, Hazardous Waste and CLP. The lab currently analyzes quarterly organic PE samples from EPA for the CLP program.

The laboratory recieves on an 18 month basis PE samples for the Army Corps of Engineers- MRD for certification as well as third party PE such as APG for various state certifcations.

4.6.2 Corporate Laboratory Performance Evaluation Program

In addition to participating in various agency sponsored performance evaluation programs such as Water Supply (WS) and Water Pollution (WP) studies, the corporate quality assurance office conducts additional performance evaluation studies through third party double blind studies.

Periodically, performance evaluation samples are submitted to each laboratory for parameters which are not addressed in other performance evaluation programs (ie. TCLP testing). In this type of testing, the laboratory is aware the samples are performance check samples but the "true" concentration values are

unknown. The results are submitted to corporate QA for evaluation and a report is issued on the findings. Corrective actions are taken if required, as a result of these test findings.

4.6.3 Routine Use of QC Check Samples

One of the most important goals of a strong quality assurance program is to ensure that data of known quality is consistently generated during day-to-day operations. IEA accomplishes this through the routine inclusion of a QC check sample in every inorganic analytical batch which includes metals and wet chemistries. For organic testing including GC and GC/MS a QC check sample is analyzed at the frequency required in the particular method. Section 8 in the Appendix provides QC check sample requirements for selected methods. A QC check sample is an artificially prepared sample which contains the analytes of interest. The source of the standards used for preparation of the check sample must be independent (either another vendor or a different lot from the same vendor) from those used to prepare a calibration curve. The QC check sample is an important mechanism to confirm the method is being executed properly during routine analysis. The QC check also serves as a useful tool in identifying possible problems such as matrix interference, degraded analytical standards, and inaccurate standard preparation.

In certain cases, reliable QC check samples are not available for a particular procedure. In such cases, the QA manager has the authority to waive this requirement for that particular test. The QA manager must document this waiver in writing.

4.6.4 Central Solvent Monitoring Program

IEA has established a central monitoring program for commonly used solvents within the corporation. Prior to use, a specific lot number of these solvents is provided to the laboratory for testing. The solvents are concentrated and tested for the presence of interfering substances relative to their intended use. If the particular lot of solvent passes the defined acceptance criteria, the vendor is notified and the solvent lot is reserved for use by the entire corporation. The approved lot numbers are provided to all laboratories and only approved solvents can be employed. IEA Document # QAS00400.NET describes the details of the solvent approval program and is available for review during a site audit.

4.6.5 Quality Assurance Final Report Review

An integral portion of the overall quality assurance program is the consistent monitoring of final reports as they leave IEA facilities. Each QA manager is responsible for reviewing 5 percent of the final data reports issued each month. The reports to be reviewed are picked at random. The reports are reviewed for typographical errors, technical clarity and overall presentation.

4.6.6 Lateness of Data Reports

IEA recognizes that one cannot overlook the timeliness of data generation when assessing the quality of our services from our client's perspective. High quality data, when delivered several weeks late is not acceptable. In recognition of this, IEA monitors the lateness of all reports on a monthly basis from each of its laboratory operations. The actual report shipment date is compared to the date originally projected to the client. This information is gathered monthly through the QA department and a monthly report is issued to each laboratory director and to corporate management. This monitoring program serves to identify service trends, and to ensure that corrective action will be taken before problems occur.

4.6.7 Method Detection Limit Verification

The laboratory performs method detection limit studies for all commonly performed test methods. The study must be performed during the initial setup and verification of the particular method. In addition, the

MDL study must be conducted in the event of a major change in the technique or instrumentation. The results of the MDL studies must be fully documented and available for review upon request. The quality assurance manager is responsible for maintaining such records. Specific state certification programs may require MDLs to be determined annually. If this is the case, the laboratory complies with this requirement.

4.6.8 Establishment of IEA Good Laboratory Practices

In order to ensure that various procedures are executed in a consistent and comprehensive manner, IEA has developed a series of procedures which fall into the category of "Good Laboratory Practices". These practices have been endorsed by the corporation for routine use at each laboratory facility and are defined in various standard operating procedures throughout the organization. Examples of a few of these "Good Lab Practices" are presented below for the reader's information:

A. Standardized logbook requirements (Doc# QAS01201.NET)

Preprinted pages
Prenumbered pages
Dedicated logbooks per test method
Bound logbooks
Use of black ink only
Document controlling of logbooks
Archival of old logbooks
Acceptance criteria in logbook
Making corrections
Secondary review of logbook entries

B. Balance calibration (Doc# QAS01002.NET)

Unique identifier for each balance
Balance must be checked daily with use and documented
Acceptance ranges are established for each balance
Balance must be checked in the weight range normally used
All balances must be professionally serviced and calibrated annually

C. Temperature monitoring requirements for lab apparatus (Doc# QAS00801.NET)

Refrigerators, freezers and lab ovens are checked each work day
Unique identifier assigned for each unit
Acceptance ranges are established for each unit
Thermometers used in monitoring must be calibrated to a NIST traceable thermometer annually, at a minimum. State certification requirements may require more frequent calibration
All thermometers are immersed in appropriate media to avoid temperature fluctuations during measurement

D. Correcting data and general laboratory records (Doc# QAS01300.NET)

All entries must be entered in black ink.
"White Out" is not to be used at any time within the laboratory for alteration or correction of lab documents
Corrections are made using a one-line strikeout
All corrections are initialed and dated by the data editor

E. Handling reagents and analytical standards (including the following)

Recording receipt and expiration dates
Documenting preparation of reagents and standards
Labelling requirements
Disposal

F. Cleaning procedures for sample containers and laboratory glassware (Doc# QAS01400.NET) (including the following)

Cleaning sample containers
Cleaning inorganic glassware
Procedures for cleaning organic glassware

G. Requirements for general lab calibration curves (including the following)

In cases where the referenced analytical method does not provide specific guidance or requirements for development of initial or continuing calibration curves, the following procedure is to be utilized by the laboratory.

All standard calibration curves must consist of a minimum of three points. Any deviation from this must be approved in writing by the facility QA manager.

All calibration points must be recalculated using the generated curve and all calibration points must be within 10% of the expected value for the curve to be considered acceptable.

Concentration of compounds or analytes must fall within the calibration range of the curve to be acceptable for quantitation for inorganic and organic methodology.

H. Method blank subtraction

Subtraction of method blanks from sample results is not permitted unless specifically authorized by the laboratory QA manager.

4.6.9 Quality Control Charts

Maintaining quality control charts is currently not mandatory under IEA's corporate quality assurance program, however, many state certification programs require them. As a result, laboratories are required to comply with such state certification requirements.

5.0 PERSONNEL QUALIFICATIONS

5.1 Introduction

IEA's management is very proud of its highly qualified and professional staff. The IEA-CT staff consists of over 50 professionals and support personnel which include:

Analytical Chemists
Quality Assurance Specialists
Computer Systems Analysts
Environmental Technicians
Customer service Staff
Account Executives

5.2 Education and Experience

In order to ensure that employees have sufficient education and experience to perform a particular task, requirements have been defined for each laboratory position.

The personnel who are responsible for operations of sample analyses and data validation are outlined in Section 5 of the Appendix. Section 1 of the appendix presents professional profiles of key personnel within the IEA-Connecticut organization. Profiles of additional IEA staff members are available for review during a facility visit or are available upon special request.

Throughout the years, IEA has performed sophisticated environmental analysis for a significant number of large corporations. Examples of relevant experience are available upon request.

5.3 Training

IEA is committed to furthering the technical and interpersonal skills of employees at all levels. Technical training is accomplished within each laboratory by management to ensure method comprehension. It is at these training sessions that staff is updated on all current technical advances. It is IEA policy that all new personnel must demonstrate competency in performing a particular method through the analysis of QC check samples prior to the analyst conducting analysis independently on client samples. New analysts may conduct analysis on client samples along with another experienced analyst prior to the completion of the training period. All laboratory personnel are required to acknowledge through signature that they have read and understood the SOP's that are appropriate for their particular area.

All laboratory personnel must have adequate education, training, and experience to carry out their responsibilities. The QA Manager and the Laboratory Management will periodically review the training needs of the staff and make recommendations for any additional training. Each department within the laboratory is responsible for personnel training. Training sessions are scheduled on a monthly basis. Each training session, whether it be individual or group training must be documented utilizing the forms attached to the corporate SOP for Employee Training QAS01600.NET. The completed forms must be submitted to the Human Resource department for placement into the employee training files. Included in the training process is analyst proficiency testing. A successful QC check sample must be analyzed and documented for each analyst. This information is on file with the QA Manager.

5.4 Certifications

Table 5.4.1 presents the state certifications held by the IEA-Connecticut laboratory. Many states certify laboratories for specific parameters or tests within a category (i.e. method 325.2 for wastewater). The information in the following table indicates the lab is certified in a general category of testing such as drinking water or wastewater analysis. The laboratory should be contacted directly if parameter-specific certification information is required.

IEA-CT currently participates in the USEPA Superfund Contract Laboratory Program (CLP). The lab is also approved to perform work for the Army Corps of Engineers which validates laboratories on a project-by-project basis.

This document is updated annually; therefore, it is likely that additional certifications, beyond those listed, may be currently available. This information can be obtained easily by calling the specific laboratory (See Table 2.2.1 for phone) and asking for the QA manager.

TABLE 5.4.1

STATE CERTIFICATIONS

In some instances it may be necessary for environmental data to be reported to a regulatory authority with reference to a certified laboratory. For your convenience, the laboratory identification numbers for the IEA-Connecticut laboratory are provided in the following table. Many states certify laboratories for specific parameters or tests within a category (i.e. method 325.2 for wastewater). The information in the following table indicates the lab is certified in a general category of testing such as drinking water or wastewater analysis. The laboratory should be contacted directly if parameter-specific certification information is required.

IEA-Connecticut
Certification Summary (as of February 1997)

State	Responsible Agency	Certification	Lab Number
Connecticut	Department of Health Services	Drinking Water, Wastewater	PH-0497
Kansas	Department of Health and Environmental Services	Drinking Water, Wastewater/Solid, Hazardous Waste	E-210/E-1185
Massachusetts	Department of Environmental Protection	Potable/Non-Potable Water	CT023
New Hampshire	Department of Environmental Services	Drinking Water, Wastewater	252891
New Jersey	Department of Environmental Protection	Drinking Water, Wastewater	46410
New York	Department of Health	CLP, Drinking Water, Wastewater, Solid/ Hazardous Waste	10602
North Carolina	Division of Environmental Management	Wastewater	388
North Dakota	Department of Health and Consolidated Laboratories	Non-Potable/Potable Hazardous Waste	R-138
Oklahoma	Department of Environmental Quality	General Water Qualtiy Sludge Testing	9614
Rhode Island	Department of Health	Chemistry...Non-Potable Water and Wastewater	A43
Washington	Department of Ecology	Wastewater/Hazardous Waste	C231
West Virginia	Division of Environmental Protection Office of Water Resources	Wasterwater/Hazardous Waste	263

6.0 FACILITIES, EQUIPMENT AND SERVICES

6.1 Introduction

The following describes the physical facility of the IEA-Connecticut laboratory.

6.2 Facilities

IEA-Connecticut

The laboratory currently maintains a staff of approximately 55 environmental professionals and occupies a facility of approximately 18,000 sq. ft. Separate laboratory areas are dedicated to GC instrumentation, GC/MS instrumentation, extractions for organic parameters, sample preparation for metals analysis, metals analysis and wet chemistries.

The volatiles analysis laboratory containing GC/MS instrumentation has a separate air handling system which is maintained at a positive pressure at all times. The organic sample preparation laboratory has a separate HVAC system that creates negative pressure in the area. This design results in a contaminant-free environment for trace-level volatiles analysis.

Critical instrumentation such as GC/MS units, ICP's, AA's, data systems and gas chromatographs are tied into an uninterruptable power supply system (UPS) to minimize instrument downtime and damage for short duration power interruptions.

The floor plan of the analytical laboratory is included in Section 4 of the Appendix.

Security of Facilities

The laboratory is secured by a card key access system. Only authorized IEA-CT personnel have access to the facility. All visitors must sign in with the receptionist and must be accompanied by an IEA-CT employee.

The sample receipt and storage area is under the responsibility of the sample custodian. This area is a locked, secure area opened by the sample control department each day. A walk-in refrigeration unit and 10 locked commercial refrigerator units are used to house samples waiting for analysis. Samples for volatile analysis are stored in separate units. Locked laboratory refrigerators, located throughout the laboratory, are used to maintain sample extracts or laboratory reagents. Each laboratory refrigerator is dedicated to sample, sample extract, or reagent storage.

6.3 Equipment

The following is a summary listing of equipment utilized at the IEA-CT facility. A more detailed listing is presented in Table 6.3.1.

Analytical instrumentation at IEA-Connecticut includes:

9 Gas Chromatographs/Mass Spectrometers (GC/MS)
6 Gas Chromatographs (GC)
3 Atomic Absorption Spectrometers (Graphite Furnace/AA)
2 Inductively Coupled Argon Plasma (ICP) Emission Spectrometer
2 Mercury analyzers
2 Gel Permeation Chromatographs
2 Infrared Spectrometer (IR)
1 Total Organic Halide (TOX) Analyzer
2 Total Organic Carbon Analyzer
2 Automated Analyzer for Wet Chemistries
1 LIMS (Laboratory Information System)
Automated Data Acquisition Management System (ADAM)

Table 6.3.1-Laboratory Equipment Listing

WET CHEMISTRY

Equipment Name	Manufacturer	Model Number	Serial Number
Centirfuge	DYNAC	0101	16846
Spectrophotometer, UV-VIS	Perkin-Elmer	35	34630
IR-Spectrophotometer	Perkin-Elmer	1310	134423
Turbidimeter	Hach Company	2100A	851017142
Turbidimeter	Orbeco/Hellige	965-10	2780
TOC Analyzer	Xertex-Dohrmann	DC-80	HF2029
TOC Analyzer	Xertex-Dohrmann	DC-190	96026010
TOX Analyzer	Xertex-Dohrmann	MC3 A,B	MF 2106
Fluorometer	Sequoia-Turner Corp.	112-003	D 01491
pH/ISE Meter	Orion	SA 720	SR45A
pH/ISE Meter	Beckman	12	0232578
Conductivity Meter	Cole-Parmer Instrument	1484-20	1421
Flash Point Apparatus	Precision Scientific	Pensky-Martin	10 Au-12
Oven	Fisher Scientific	55G	291
Oven	VWR	1320	0701090
Incubator	Blue M Electric	100 A	IN1-1362
Bio Refrigerator	Frost Queen	R20/L	00029
BOD Incubator (2)	Precision Scientific	FU199JRW2/FU178RRW2	FLC02662
Midi Distillation Setup (2)	Andrews Glass Co.	110-10-R	A4W0309/0209
D.O. Meter	YSI	51A	0241
COD Reactor	HACH	45600	920300006892
Muffle Furnace	Thermolyne	-	-
TKN block digestor	Scientific Instruments	AD-4020	8915049
Digital Hot Plate/Stirrer (2)	PMC	730	0298E
TCLP Spinners- 34 positions (4)	Dayton	3M137B/5K939B	-
Semiautomated Analyzer	LACHAT	Quikchem	125360

Table 6.3.1-Laboratory Equipment Listing

METALS

Equipment Name	Manufacturer	Model Number	Serial Number
Mercury Analyzer	Spectro-Products	HG4	4708
Mercury Analyzer	Jarrell-Ash	QS1	1210031
Autoclave	Market Forge	STM-E	034200
ICP-Trace	Jarell-Ash	JA61T	349490
ICP-Simultaneous	Jarrell-Ash	JA61	67782
Furnace AA	Perkin-Elmer	Z3030	3131
Furnace AA	Perkin-Elmer	Z5100	130911
Furnace AA	Perkin-Elmer	Z5100 PC	135141

ORGANIC EXTRACTIONS

Equipment Name	Manufacturer	Model Number	Serial Number
Gas Chromatograph	Perkin-Elmer	8320	83N546502
Gel Permeation Chromatograph	ABC	1002B	7323
Gel Permeation Chromatograph	ABC	AP1000	9228
Refrigerator	WW	4EF	F3978U
Oven	ASP	D 1142	144011
Oven	ASP	D 1162	149010
Sonicator	Sonics & Materials	SM500	6892
Sonicator	Sonics & Materials	VCX-400	00030C
Sonicator	Tekmar	TM500	7264
Auto Sampler	Perkin-Elmer	AS8300	95234
Rotary Evaporator	BUCH I	R-114	-
Seporatory Funnal Shaker	Glas-Col	Series 100	F715-10-B5J
Muffle Furnace	Wilt	M001210	91881

Table 6.3.1-Laboratory Equipment Listing

GC/MS VOLATILES

Equipment Name	Manufacturer	Model Number	Serial Number
Purge & Trap	Tekmar	LSC 2000	91318021
Purge & Trap	Tekmar	ALS 2016	91322002
Purge & Trap	Tekmar	LSC 2000	91203019
Purge & Trap	Tekmar	ALS 2016	91232007
Tube Desorber	Envirochem	810TD	268153
Computer/Data System	Hewlett Packard	425T	3048T147545
Terminal	Hewlett Packard	X-window	3048T18725
Terminal	Hewlett Packard	X-window	3048T18726
GC/MS	Hewlett Packard	5995B	2217A00358
GC/MS	Hewlett Packard	5995C	2413A00659
GC/MS	Hewlett Packard	5995C	2413A00430
GC/MS	Hewlett-Packard	5890 Series II/5972 MSD	-
GC/MS	Hewlett-Packard	5890 Series II/5972 MSD	-
Terminal	Hewlett Packard	45849A	2530A13541
Terminal	Hewlett Packard	35751	2643A07666
CRT	Hewlett Packard	35731A	8633K26810
Printers (partial list)	Hewlett Packard	2934A	2635A32940
Printers	Hewlett Packard	2934A	2715A43948
Printers	Hewlett Packard	2225A	2512S30379
Printers	Hewlett Packard	2225A	2510S32359
Terminal	Hewlett Packard	35751	2630A06622
CRT	Hewlett Packard	35731A	8610K20516
Magnetic Tape Unit	Hewlett Packard	7970E	N/A
Scanning Interface	Hewlett Packard	59824A	N/A
Scanning Interface	Hewlett Packard	59824A	N/A
Cart. Tape Unit	Hewlett Packard	7914	N/A
Cart. Tape Unit	Hewlett Packard	7914	N/A

Table 6.3.1-Laboratory Equipment Listing

GC/MS SEMI-VOLATILE

Equipment Name	Manufacturer	Model Number	Serial Number
Gas Chromatograph	Hewlett Packard	5890	7518A05422
Auto Sampler	Hewlett Packard	76732A	2441A03468
Mass Selective Detector	Hewlett Packard	5970	2513A00923
Gas Chromatograph	Hewlett Packard	5890	2728A14615
Auto Sampler	Hewlett Packard	76732A	2546A01489
Mass Selective Detector	Hewlett Packard	5970	2716A10638
Computer Terminal	Hewlett Packard	150 II	2720Y05798
Computer Terminal	Hewlett Packard	150 II	2720Y03266
Computer Terminal	Hewlett Packard	150 II	2530A13540
Scanning Interface (2)	Hewlett Packard	59824A	---
Tape Drive	Hewlett Packard	9144	---
Disc Drive	Hewlett Packard	7958	---
9 Track Magnetic Tape	Hewlett Packard	7970E	---
Computer	Hewlett Packard	HP1000A	---
Computer	Hewlett Packard	HP1000	---
Printer	Hewlett Packard	Laser Jet 4	--
Printer	Hewlett Packard	Laser Jet 4	--
Printer	Hewlett Packard	Laser Jet 4	--
Autosampler	Hewlett-Packard	7673A	2546A01489
GC	Hewlett-Packard	5890A	---
MSD	Hewlett-Packard	5971A	3040A01426
Autosampler	Hewlett-Packard	7673	3120A28431
Computer	HP Vectra	386/25 DX	365201578025
Terminal	Hewlett Packard	36731A	8635K28238
GC/MS/MSD	Hewlett-Packard	5890 SeriesII/5972MSD	-
Data System -Enviroquant	Hewlett-Packard	Vectra XM2	-
Data System -Enviroquant	Hewlett-Packard	Vectra XM2	-

Table 6.3.1-Laboratory Equipment Listing

GAS CHROMATOGRAPHY

Equipment Name	Manufacturer	Model Number	Serial Number
GC	Hewlett-Packard	5890	2541A06301
GC	Hewlett-Packard	5890	2750A14840
Autosampler	Hewlett-Packard	7673A	2546A00709
Autosampler	Hewlett-Packard	7673A	3123A25128
Autosampler	Hewlett-Packard	7673A	2718A0653A
Integrator	Hewlett-Packard	3396A	2804A01106
Integrator	Hewlett-Packard	3393A	2332A00D80
GC	Hewlett-Packard	5890 Series II	3121A35826
GC	Hewlett-Packard	5890 Series II	3235A44989
GC	Varian	3300	891094
Purge and Trap	Tekmar	4000	254
Purge and Trap	Tekmar	ALS	372
Data System	Hewlett-Packard	HP1000A	3020A05230
Terminals (3)	Hewlett-Packard	35741A	—
Printers (3)	Hewlett-Packard	35741A	—
Tape Drive	Hewlett Packard	9144	2724E13732
Data System-Enviroquant	Hewlett-Packard	Vectra XM2	-

6.4 Instrument Maintenance

Where it is economically feasible, the IEA-CT laboratory has service contracts for major instruments. These contracts provide routine preventive maintenance according to the manufacturer's requirements. Additionally the laboratory maintains an inventory of expendable parts and supplies to minimize downtime and to allow laboratory personnel to make minor repairs if necessary.

Each analytical measurement SOP lists the preventive maintenance schedule for each instrument which is to be followed by in-house and extramural repair contractors. In addition, each measurement group must maintain a log of all in-house and extramural preventive maintenance activities. Table 6.4.1 presents examples of general measures which are performed throughout the laboratory.

Table 6.4.1 Laboratory Preventative Maintenance

GC/MS SYSTEMS		
EQUIPMENT	ACTION PERFORMED	FREQUENCY
Hewlett-Packard 5995 GC/MS	Check oil level in mechanical pumps	Weekly
	Check water level and operating condition in the Neslab cooling units	Weekly
	Check compressed air gas supply	Daily
	Check helium gas supply	Daily
	Check carbon dioxide gas supply	Daily
	Change the oil in the mechanical pumps	Every 6 months
	Inspect the pump hoses and replace if required	Every 6 months
	Change oil in the diffusion pump	Every 6 months
	Change foreline and exhaust trap absorbent	Every 6 months
	Inspect and refill the calibration sample vial with PFTBA	Every 6 months
	Vacuum fan grills and filters	Every 6 months
	Check fore and separator pump pressures	Weekly
	Ion source cleaning and filament replacement	As needed
	Column replacement and conditioning	As needed
	Column cutting and reinstallation	As needed
	Manual tuning	As needed
	Change compressed air gas supply	As needed
	Change helium gas supply	As needed
	Change carbon dioxide gas supply	As needed
	Recharge Neslab cooling units	As needed
	Replace electron multiplier	As needed
	Remove and clean or replace jet separator	As needed

Table 6.4.1 Laboratory Preventative Maintenance

EQUIPMENT	ACTION PERFORMED	FREQUENCY
Hewlett-Packard 5970 MSD / 5971 MSD/5972 MSD	Check oil level in mechanical pumps	Weekly
	Change the oil in the mechanical pumps	Every 6 months
	Inspect the pump hoses and replace if required	Every 6 months
	Change oil in the turbo pump	Every 6 months
	Change exhaust trap absorbent	Every 6 months
	Inspect and refill the calibration sample vial with PFTBA	Every 6 months
	Vacuum fan grills and filters	Every 6 months
	Ion source cleaning and filament replacement	As needed
	Manual tuning	As needed
	Replace electron multiplier	As needed
	Clean out transfer line to GC	After every column removal
Hewlett-Packard 5890 GC	Check helium gas supply	Daily
	Change split vent trap	Every 3 months
	Column replacement and conditioning	As needed
	Column cutting and reinstallation	Daily or as needed
	Change helium gas cylinder	As needed
	Change liner and septum	Daily or as needed
	Clean injection port	As needed

Table 6.4.1 Laboratory Preventative Maintenance

EQUIPMENT	ACTION PERFORMED	FREQUENCY
Hewlett-Packard 7672A Autosampler	Inspect and correct injector alignment	After reseating
	Inspect syringe	Daily
	Check compressed air gas supply	Daily
	Inspect and adjust tension on sample tray	Daily
	Change rinse vials	Daily
	Change waste vials	Weekly
	Replace syringe	As needed
	Sand injector post	As needed
	Realign autosampler on brackets	As needed
	Change compressed air cylinder	As needed
Hewlett-Packard 7673A Autosampler	Inspect syringe	Daily
	Inspect seating of injector	Daily
	Change rinse vials	Daily
	Change waste vials	Weekly
	Replace syringe	As needed
	Reset control box	As needed
Tekmar Purge and Trap Sample Concentrators and Autosamplers	Inspect spargers and fittings	Daily
	Check purge flow	Daily
	Inspect line and valve temperatures	Daily
	Change and condition trap	As needed
	Adjust purge flow	As needed
	Rinse or clean sparging vessels	As needed
	Rinse sample lines	As needed
	Bake out trap	After each analysis, extend as needed
	Replace lines and fittings	As needed
	Adjust line and valve temperatures	As needed

Table 6.4.1 Laboratory Preventative Maintenance

EQUIPMENT	ACTION PERFORMED	FREQUENCY
Envirochem Air Sample Concentrator and Autosampler	Inspect fittings	Daily
	Check flows	Daily
	Inspect line and valve temperatures	Daily
	Change and condition internal traps	As needed
	Adjust flow	As needed
	Bake out trap	After each analysis, extend as needed
	Replace lines and fittings	As needed
	Adjust line and valve temperatures	As needed

GC SYSTEMS		
EQUIPMENT	ACTION PERFORMED	FREQUENCY
Hewlett-Packard 5890A GC (GC-1,4,5 Dual ECD)	Check gas supply	Daily
	Check breakdown criteria	As required by run sequence
	Vacuum filters and grills	Quarterly
	Column replacement and conditioning	As needed
	Column cutting and reinstallation	As needed
	Change gas cylinders	As needed
	Change liner and septum	As needed
	Replace guard column	As needed
	Clean injection port	As needed
	Recondition ECD	As needed
	Change ECD vent absorbent traps	Quarterly

Table 6.4.1 Laboratory Preventative Maintenance

EQUIPMENT	ACTION PERFORMED	FREQUENCY
Hewlett-Packard 5890A GC (GC-3 FID/NPD)	Check gas supply	Daily
	Vacuum filters and grills	Quarterly
	Column replacement and conditioning	As needed
	Column cutting and reinstallation	As needed
	Change gas cylinders	As needed
	Change liner and septum	As needed
	Clean injection port	As needed
	Replace or reactivate the NPD collector	As needed
Hewlett-Packard 7673A Autosampler	Inspect syringe	Daily
	Inspect seating of injector	Daily
	Inspect rinse and waste vials	Daily
	Vacuum filters and grills	Quarterly
	Replace syringe	As needed
	Change rinse and waste vials	As needed

Table 6.4.1 Laboratory Preventative Maintenance

EQUIPMENT	ACTION PERFORMED	FREQUENCY
Perkin-Elmer AS-100B Autosampler	Inspect syringe	Daily
	Inspect rinse and waste vials	Daily
	Check flushing efficiency	Daily
	Clean or replace syringe	As needed
	Change rinse and waste vials	As needed
	Change diverter valve septum	As needed
METALS SYSTEMS		
Graphite Furnace	Clean contact rings, furnace housing and quartz windows	Daily
	Inspect, clean or replace graphite tubes	As needed
	Replenish matrix modifiers	Daily
	Check lamp alignments and energies	Daily
	Clean mirrors for the optical sensors	Weekly
	Clean windows on furnace housing	Weekly
	Inspect contact rings for excessive wear	Monthly
Inductively Coupled Plasma	Change capillary and pump tubing	Twice weekly
	Replace liquid argon tank	As required
	Reprofile via slit micrometer	Per manual
	Replace and realign plasma torch	As needed
	Clean nebulizer and spray chamber	As needed
	Check primary imaging mirror	Weekly
Mercury Analyzer	Clean sample cell and tubing	Monthly
	Check sparger condition	Daily
	Check level of mercury scrubber solution	Daily
	Replace lamps	As required

WET CHEMISTRY SYSTEMS		
EQUIPMENT	ACTION PERFORMED	FREQUENCY
pH Meters	Clean electrode if calibration has deteriorated	As needed
	Store pH electrodes in pH 7.0 buffer	Daily
	Check ISE electrodes and meter	Per manual
Analytical Balances	Surfaces cleaned and covered	Daily
	Calibrated and cleaned by manufacturer	Semi-annually
	Accuracy checked by class "S" weights	Prior to use
Conductivity Meters	Instrument surfaces inspected and cleaned	Daily
	Calibrated using 0.01M potassium chloride	Daily
	Spare cells on inventory	As needed
Spectrophotometers	Instrument cleaned	Daily use
Total Organic Halogen Analyzer (TOX)	Instrument cleaned	Daily use
	Perform cell performance checks	Daily
	Flush cells and check heated tapes	Daily
	Inspect sample boats, inlet and exit tubes, o-rings and seals	Daily
Autoanalyzer Systems	Clean all components and flush system	Daily use
	Inspect all pump tubes and sample lines	Daily use
	Inspect line coils, heating baths and filters	Weekly
	Inspect all colorimeter filters	Weekly
	Inspect and clean chemical manifolds	Monthly

7.0 DATA GENERATION

7.1 Introduction

There are numerous policies and standard procedures which have been implemented to ensure that data of known quality is continually generated by the IEA-CT laboratory. The IEA Corporate and Laboratory Facility Quality Assurance Plans are examples of documents which are generated. Guidelines for the facility QA plans are detailed in section 7.2.1 of the Corporate Quality Assurance Program Plan Doc#QAQ00102.NET.

7.2 Quality Assurance Project Plans

Quality Assurance Project Plans (QAPjP) are developed to meet contract and agency requirements on a project specific basis. These plans discuss specific terms, policies, objectives and QA activities designed to achieve the data quality objectives of the project.

All QA project plans are written in accordance with the following USEPA Document: USEPA Guidelines and Specification for Preparing Quality Assurance Project Plans. QAMS-005/80, Washington DC: USEPA, Quality Assurance Management Staff, October 17, 1980.

Guidelines for preparing QA project plans are also detailed in the Corporate Quality Assurance Program Plan Doc#QAQ00102.NET.

7.3 Methods

IEA-CT utilizes a wide variety of analytical methods. A listing of general analytical capabilities is presented in Table 7.3.1. Section 8 of the Appendix lists the analytical method and detection limits associated with various analytical procedures.

Each department is required to have a written standard operating procedure (SOP) in use which describes how the requirements of the method are met. All SOPs must be prepared in accordance with IEA Doc.#QAS00200.NET.

Analytical methodologies and quality assurance protocols in use are based on the following guidelines:

"Methods of Organic Chemical Analysis of Municipal and Industrial Wastewater", Federal Register Vol. 49, No. 209, October 26, 1984;

"Test Methods for Evaluating Solid Waste", SW-846 Third Edition, September 1986, USEPA, plus updates;

"Standard Methods for the Examination of Water and Wastewater" 1985, 15th, 16th and 18th Edition;

"Methods for Chemical Analysis of Water and Wastes" March 1983, EMSL, EPA;

"Manual of Analytical Methods for the Analysis of Pesticides in Humans and Environmental Samples", EPA 600/8-80-038, June 1980;

Organic Analysis: Multi-media, Multi-Concentration-IFB-CLP, January 1991, Document Number OLM01.9 (plus revisions);

Organic Analysis: Multi-media, Multi-Concentration-IFB-CLP, Document Number OLM03.1

Inorganic Analysis: Multi-media, Multi-Concentration-IFB-CLP, Document Number ILM04.0;

"Handbook for Analytical Quality Control in Water and Wastewater Laboratories", EPA-600/4-79-019, March 1979;

National Enforcement Investigation Center Policies and Procedures Manual, EPA-330/9/78/001-R, Revised May 1986

"Manual for the Certification of Laboratories Analyzing Drinking Water", April 1990, EPA/570/9-90/008.

"EML Procedures Manual", HASL-300, November 1990, 27th Edition.

"New York State Department of Environmental Conservation Analytical Services Protocol, 10/95 Edition"

"U.S. Air Force Installation Restoration Program Qualtiy Assurance Project Plan, January 1997"

TABLE 7.3.1

IEA-CT ANALYTICAL CAPABILITIES

I. ORGANICS-GC/MS

Volatile Organics-524.2
Volatile Organics-CLP
Volatile Organics-8240
Volatile Organics-8260
Volatile Organics-T01/T02
Volatile Organics-Appendix IX
Acid & Base/Neutrals-8270
Acid & Base/Neutrals-CLP
Acid & Base/Neutrals-Appendix IX
Volatile Organics-624
Acid & Base/Neutrals-625

II. ORGANICS-GC

Misc. DAI - 8015
Organohalide Pesticides & PCBs-608
Organohalide Pesticides & PCBs-8081
Organohalide Pesticides & PCBs-CLP
Organophosphate Pesticides-8140
Organohalide Pesticides & PCBs-Appendix IX
Chlorinated Herbicides-8150
Chlorinated Herbicides-Appendix IX
Petroleum Hydrocarbons - GRO/DRO

III. INORGANIC METALS

ICP Metals
Furnace Metals
CLP Metals
Appendix IX Metals
TCLP Metals
Drinking Water Metals

V. INORGANIC WET CHEMISTRY*

Acidity
Alkalinity
Ammonia
Bicarbonate
Biochemical Oxygen Demand (BOD)
Bromide
Chloride
Chlorine Demand
Chlorine Residual
Chemical Oxygen Demand
Color
Conductivity
Chromium (VI)
Cyanide - Amenable
Cyanide - Total
Cyanide (CLP)
Dissolved Oxygen
Flashpoint
Fluoride
Grain Size
Hydrocarbon analysis
MBAS
Nitrate
Nitrite
Odor
Oil and Grease
Paint Filter Test
pH
Phenols
Phosphate
Phosphorus
Settleable Solids
Silica
Specific Gravity
Sulfate
Sulfide
Sulfite
Sludge Volume Index
Tannins and Lignins
Total Dissolved Solids
Total Kjeldahl Nitrogen
Total Organic Carbon
Total Organic Halides
Total Solids
Total Suspended Solids
Turbidity
Volatile Solids
Corrosivity Characteristics
Ignitability Characteristics
EPTOX
TCLP
SPLP
Extractable Organic Halides

Table 7.3.2 SUMMARY OF QC REQUIREMENTS FOR EPA VOLATILE ORGANIC ANALYSIS METHODS

Requirement	Drinking Water Analysis Method 524	Water and Wastewater Analysis Method 624	RCRA Solid Waste Analysis Methods 8240/8260	Superfund Hazardous Waste Analysis CLP SOW OLM01.9	NYSDEC
Tuning					
Frequency	25 ng BFB	50 ng BFB	50 ng BFB	50 ng BFB	50 ng BFB
Criteria	8 hrs	Daily	12 hrs	12 hrs	12 hrs
	See following page	See following page	See following page	See following page	See following page
Initial Calibration					
Maximum % RSD	3-5 standards	3 standards	5 standards	5 standards	5 standards
Minimum RRF	<20%	<35%	CCC <30%*	<20.5%*	<20.5%*
	NS	NS	SPCC >0.250-0.300*	0.01-0.500*	0.01-0.500*
					10 compounds with max 100% RSD
Continuing Calibration					
Frequency	8 hrs	Daily	12 hrs	12 hrs	12 hrs
Maximum %D	±30%	QC Limits	CCC ±25%*	±25.0%*	±25.0%*
Minimum RRF	NS	NS	SPCC >0.250-0.300*	0.01-0.500*	0.01-0.500*
IS Area	±30% of last CC or ±50% of IC	NS	-50 to +100% of last CC	NS	NS
					10 compounds with max 100% RSD
QC Check Sample/LCS					
Frequency	Quarterly	Daily	Each batch or if MS % recovery not in QC limits	NS	Each cat. B SDG
Criteria	QC Limits*	QC Limits*	QC Limits*	QC Limits*	QC Limits*
Method Blank					
Frequency	Daily	Daily	12 hrs	12 hrs	12 hrs
Criteria	<MDL	In control	In control	<CRQL*	<CRQL*
Spikes	Blank spike	Matrix spike	Matrix spike	Matrix spike	Matrix spike
Frequency	Daily or 5%	5%	5%	5% or 1/SDG	5% or 1/SDG
% Recovery	80-120%	QC Limits*	QC Limits*	QC Limits*	QC Limits*
Duplicates	BS duplicate	Field duplicate	MS duplicate or sample duplicate	MS duplicate	MS duplicate MSB required*
Frequency	Quarterly	NS	5%	5% or 1/SDG	5% or 1/SDG
Precision	<20% RSD	NS	SD Limits*	RPD Limits*	RPD Limits*
Sample Analysis					
Holding time	14 days	14 days	14 days	10 days from receipt	7 days from receipt
Internal standards	1 @ 2-10 ug/L	3 @ 30 ug/L	3-4 @ 50 ug/L	3 @ 50 ug/L	3 @ 50 ug/L
Criteria	NS	NS	NS	Area -50 to +100T	Area -50 to +100T
				RT ±30 sec	RT ±30 sec
Surrogate	2 @ 5 ug/L	3 @ 30 ug/L	3 @ 50 ug/L	3 @ 50 ug/L	3 @ 50 ug/L
Criteria	80-120%	NS	See following page	See following page	See following page
Analyte ID	RT ±3x SD window	RT ±30 sec	RRT ±0.06	RRT ±0.06	RRT ±0.06
	3 ions ±20%	3 ions ±20%	Ions >10% ±20%	Ions >10% ±20%	Ions >10% ±20%

*For complete information refer to method or protocol

SUMMARY OF VOLATILE SURROGATE RECOVERY LIMITS								
Compound	Method 524 (%)	Method 624 (%)	Method 8240 Water (%)	Method 8240 Soil (%)	Method 8260 Water (%)	Method 8260 Soil (%)	CLP SOW Water (%)	CLP SOW Soil (%)
4-Bromofluorobenzene	80-120	NS	86-115	74-121	86-115	72-121	86-115	59-113
1,2-Dichloroethane-d4	80-120	NS	76-114	70-121	NS	NS	76-114	70-121
Toluene-d8	NS	NS	86-110	81-117	88-110	81-117	88-110	84-138
Dibromofluoromethane	NS	NS	NS	NS	86-118	80-120	NS	NS

SUMMARY OF VOLATILE SPIKE RECOVERY LIMITS					
Method Compound	Method 524 (%)	Method 624 (%)	Method 8240 (%)	CLP SOW Soil (%)	CLP SOW Water (%)
Benzene	80-120	37-151	37-151	66-142	66-142
Chlorobenzene	80-120	37-160	37-160	75-130	60-133
1,1-Dichloroethane	80-120	59-155	59-155	61-145	59-172
Toluene	80-120	47-150	47-150	76-125	59-139
Trichloroethene	80-120	71-157	71-157	71-120	62-137

SUMMARY OF INSTRUMENT TUNING REQUIREMENTS					
BFB Ion Abundance Criteria	NYSDEC 1/91	Method 524 (%)	Method 624 (%)	Methods 8240/8260 (%)	CLP SOW OLM01.8 (%)
50 - % of mass 95	15-40	15-40	15-40	15-40	8.0-40.0
75 - % of mass 95	30-60	30-80	30-60	30-60	33.0-66.0
95	100	100	100	100	100.0
96 - % of mass 95	5-9	5-9	5-9	5-9	5.0-9.0
173 - % of mass 174	<2	<2	<2	<2	<2.0
174 - % of mass 95	>50	>50	>50	<50	50.0-120.0
175 - % of mass 174	5-9	5-9	5-9	5-9	4.0-9.0
176 - % of mass 174	95-101	95-101	95-101	95-101	95.0-101.0
177 - % of mass 176	5-9	5-9	5-9	5-9	5.0-9.0

TABLE 7.3.2 SUMMARY OF QC REQUIREMENTS FOR EPA SEMI-VOLATILE ORGANIC ANALYSIS METHODS

Requirement	Water and Wastewater Analysis Method 625	RCRA Solid Waste Analysis Method 8270A	Superfund Hazardous Waste Analysis CLP SOW OLM01.9	NYSDEC
Tuning	50 ng DFTPP	50 ng DFTPP	50 ng DFTPP	50 ng DFTPP
Frequency	Daily	12 hrs	12 hrs	12 hrs
Criteria	See following page	See following page	See following page	See following page
Initial Calibration	3 standards	5 standards	5 standards	5 standards
Maximum %RSD	<35%	CCC <30%*	<20.5%*	<20.5%*
Minimum RRF	NS	SPCC >0.050*	0.01-1.300*	0.01-1.300*
				20 compounds
				Max 100% RSD
Continuing Calibration				
Frequency	Daily	12 hrs	12 hrs	12 hrs
Maximum %D	±20%	CCC ±30%*	±25%*	±25%*
Minimum RRF	NS	SPCC >0.050*	0.01-1.300*	0.01-1.300*
IS Area	NS	-50 to +100% of last CC	NS	NS
				20 compounds
				Max 100% RSD
C Check Sample/LCS				
Frequency	≤5%	If MS % recovery	NA	Each SDG with cat. B
Criteria		not in QC limits		
	QC Limits*	QC Limits*		QC Limits*
Method Blank				
Frequency	1 per batch	1 per batch	1 per batch	1 per batch
Criteria	In control	In control	<CRQL*	<CRQL*
Spikes	Matrix spike	Matrix spike	Matrix spike	Matrix spike
Frequency	5%	5%	5% or 1/SDG	5% or 1/SDG
% Recovery	QC Limits*	QC Limits*	QC Limits*	QC Limits*
Duplicates	Field duplicates	MS duplicate or	MS duplicate	MS duplicate
Frequency		sample duplicate		MS blank
Precision	NS	5%	5% or 1/SDG	5% or 1/SDG
	NS	SD Limits*	RPD Limits*	RPD Limits*
Sample Analysis				
Holding Time				
Water extraction	7 days	7 days	5 days from receipt	completed within 5 days from receipt
Soil extraction	NA	14 days	10 days from receipt	completed within 5 days from receipt
Analysis	40 days from extraction	40 days from extraction	35 days from extraction	35 days from extraction
Internal standards	3	6 @ 40 ug/L	6 @ 20 ug/L	6 @ 20 ug/L
Criteria	NS	Area -50/+100%	Area -50/+100%	Area -50/+100%
		RT ±30 sec	RT ±30 sec	RT ±30 sec
Surrogate	3 @ 100 ug/L	6 @ 100-200 ug/L	8 @ 100-150 ug/L	8 @ 100-150 ug/L
Criteria	NS	See following page	See following page	See following page
Analyte ID	RT ±30 sec	RRT ±0.06	RRT ±0.06	RRT ±0.06
	3 ions ±20%	Ions >10% ±20%	Ions >10% ±20%	Ions >10% ±20%

*For complete information refer to method or protocol

SUMMARY OF SEMI-VOLATILE SURROGATE RECOVERY LIMITS					
Compound	Method 625 (%)	Method 8270 Water (%)	Method 8270 Soil (%)	NYSDEC '91 ASP CLP SOW Water (%)	NYSDEC '91 ASP CLP SOW Soil (%)
Nitrobenzene-d5	NS	35-114	23-120	34-114	23-120
2-Fluorobiphenyl	NS	43-116	30-115	43-116	30-115
p-Terphenyl-d14	NS	33-141	18-137	33-141	18-137
Phenol-d6	NS	10-94	24-113	10-110	24-113
2-Fluorophenol	NS	21-100	25-121	21-110	25-121
2,4,6-Tribromophenol	NS	10-123	19-122	10-123	19-122
1,2-Dichlorobenzene-d4	NS	NS	NA	16-110*	20-130*
2-Chlorophenol-d4	NS	NS	NA	33-110*	20-130*
Perylene-d12	NS	NS	NA	NA	NA

SUMMARY OF SEMI-VOLATILE SPIKE RECOVERY LIMITS				
Compound	Method 625 (%)	Method 8270 (%)	NYSDEC '91 ASP[1] CLP SOW Water (%)	NYSDEC '91 ASP CLP SOW Soil (%)
Acenaphthene	47-145	47-145	46-118	31-137
1,4-Dichlorobenzene	20-124	20-124	36-97	28-104
2,4-Dinitrotoluene	D-112	D-112	24-96	28-89
N-Nitroso-di-n-propylamine	D-230	D-230	41-116	41-126
Pyrene	52-115	52-115	26-127	35-142
1,2,4-Trichlorobenzene	44-142	44-142	39-98	38-107
4-Chloro-3-methylphenol	22-147	22-147	23-97	26-103
2-Chlorophenol	23-134	23-134	27-123	25-102
4-Nitrophenol	D-132	D-132	10-80	11-114
Pentachlorophenol	14-176	14-176	9-103	17-109
Phenol	5-112	5-112	12-110	26-90

SUMMARY OF GC/MS INSTRUMENT TUNING REQUIREMENTS					
DFTPP Ion Abundance Criteria	**NYSDEC**	**Method 525 (%)**	**Method 625 (%)**	**Method 8270 (%)**	**CLP SOW (%)**
51 - % of mass 198	30-60	10-80	30-60	30-60	30.0-80.0
68 - % of mass 69	<2	<2	<2	<2	<2.0
70 - % of mass 69	<2	<2	<2	<2	Present
127 - % of mass 198	40-60	10-80	40-60	40-60	25.0-75.0
197 - % of mass 198	<1	<2	<1	<1	<1.0
198	100	100	100	100	100
199 - % of mass 198	5-9	5-9	5-9	5-9	5.0-9.0
275 - % of mass 198	10-30	10-60	10-30	10-30	10.0-30.0
365 - % of mass 198	>1	>1	>1	>1	>0.75
441	<mass 443	<mass 443	<mass 443	<mass 443	<mass 443
442 - % of mass 198	40-110	>50	>40	>40	40.0-110.0
443 - % of mass 442	17-23	15-24	17-23	17-23	15.0-24.0

TABLE 7.3.2 SUMMARY OF QC REQUIREMENTS FOR PESTICIDE/PCB ANALYSIS METHODS

Requirement	Water and Wastewater Analysis Method 608	RCRA SW-846 Solid Waste Analysis Method 8080	Superfund Hazardous Waste Analysis CLP SOW OLM01.8	NYSDEC
Initial Calibration	3 standards	5 standards	3 standards (1 for multicomponent)	3 standards (1 for multicomponent)
Maximum % RSD	<10%	<20%	<10.0-15.0%*	<10.0-15.0%*
DDT/Endrin Breakdown	NS	<20%	<20.0%	<20.0%
Resolution	NS	NS	90-110%*	90-110%*
Continuing Calibration	Mid-level standard	Mid-level standard	Mid-level standard	Mid-level standard
Frequency	Daily	Daily	12 hrs	12 hrs
Maximum %D	±15%	±15%	±25.0%	±25.0%
RT Criteria	NS	NS	±0.05-0.07 min of mean RT	±0.05-0.07 min of mean RT
QC Check Sample/LCS				
Frequency	≤10%	If MS % recovery not in QC limits	NS	Each cat. B SDG
Criteria	QC Limits*	QC Limits*	QC Limits*	QC Limits*
Method Blank				
Frequency	1/batch	1/batch	1/batch	1/batch
Criteria	In control	In control	<CRQL	<CRQL
Spikes	Matrix spike	Matrix spike	Matrix spike	Matrix spike
Frequency	10%	5%	5% or 1/SDG	5% or 1/SDG
% Recovery	QC Limits*	QC Limits*	QC Limits*	QC Limits*
Duplicates	Field duplicate	MSD or sample duplicate	MSD	MSD/MS Blank
Frequency	NS	5%	5% or 1/SDG	5% or 1/SDG
Precision	NS	SD Limits*	RPD Limits*	RPD Limits*
Sample Analysis				
Holding Time				
Water extraction	7 days	7 days	5 days VTSR	Completed within 5 days VTSR
Soil extraction	NA	14 days	10 days VTSR	Completed within 5 days VTSR
Analysis	40 days	40 days	35 days	35 days
Analyte ID	RT within 3x SD of std. RT window	RT within 3x SD of std. RT window	RT ±0.05-0.07 min of std. RT on both columns; Conc. ±25.0%	RT ±0.05-0.07 min of std. RT on both columns; Conc. ±25.0%
Confirmation	2nd column for unknown samples	2nd column for positive ID	2 column required; GC/MS if >10 ng/uL	2 column required; GC/MS if >10 ng/uL

*For complete information refer to method or protocol

SUMMARY OF PESTICIDE SURROGATE RECOVERY LIMITS					
Compound	Method 508 (%)	Method 608	Method 8080	CLP SOW (%)	NYSDEC
Tetrachloro-m-xylene	NS	NS	Lab limits	60-150	60-150
Decachlorobiphenyl	NS	NS	Lab limits	60-150	60-150
Dibutylchlorendate	NS	NS	Lab limits	NS	

SUMMARY OF PESTICIDE SPIKE RECOVERY LIMITS					
Compound	Method 608 (%)	Method 8080 (%)	CLP SOW Water (%)	CLP SOW Soil (%)	NYSDEC
gamma-BHC (Lindane)	19-140	19-140	56-123	46-127	46-127
Aldrin	42-122	42-122	40-120	34-132	34-132
Dieldrin	36-146	36-146	52-126	31-134	31-134
4,4'-DDT	25-160	25-160	38-127	23-134	23-134
Endrin	30-147	30-147	56-121	42-139	42-139
Heptachlor	34-111	34-111	40-131	35-130	35-130

TABLE 7.3.2 SUMMARY OF QC REQUIREMENTS FOR EPA METALS ANALYSIS METHODS USING ATOMIC ABSORPTION (AA) SPECTROSCOPY

Requirement	Water and Wastewater Analysis Method 200.0	RCRA Solid Waste Analysis Method 7000	Superfund Hazardous Waste Analysis CLP SOW ILM04.0
Initial Calibration Frequency Criteria	3 standards and a blank Daily r ≥0.995	3 standards and a blank Daily r ≥0.995	3 standards and a blank Daily or every 24 hrs r ≥0.995
Calibration Verification Frequency Criteria	A standard at or near MCL After initial calibration and every 20 samples 90-110% recovery	Mid-range standard Every 10 samples ICV: 90-110% recovery CCV: 80-120% recovery	Mid-range standard Beginning, end, and every 10 samples or every 2 hrs 90-110% recovery Hg: 80-120% recovery
Detection Limits **Standard** Frequency Criteria	NS NS NS	NS NS NS	Standard at the CRDL or IDL Beginning of each sample run EPA QC limits
Calibration Blanks Frequency Criteria	After each calibration NS	After each calibration NS	Beginning, end, and every 10 samples or every 2 hrs All analytes ≤ CRDL
Preparation Blanks Frequency Criteria	Each digestion batch NS	Each digestion batch NS	1 per SDG or digestion batch All analytes ≤ CRDL
QC Check Sample/LCS Frequency Criteria	NS NS	1 per batch NS	1 per matrix per SDG or digestion batch 80-120% recovery
Matrix Spike Samples Frequency Criteria	10% or 1 per batch NS	5% or 1 per batch NS	5% or 1 per SDG per matrix per level (predigestion) 75-125% recovery
Duplicate Samples Frequency Criteria	10% or 1 per batch NS	5% or 1 per batch NS	5% or 1 per SDG per matrix per level (predigestion) ≤20% RPD for values ≥5 x CRDL ±1 x CRDL for values <5 x CRDL
Furnace Quality Control Frequency Criteria	MSA as needed NS	MSA as needed Serial dilution: 1 per batch per matrix MSA: r≥0.995 5¥ dilution within ±10%	Duplicate injections on all; Post digestion spikes on all samples, blanks, and LCS; MSA as needed Duplicate injections: ≤20% RSD/CV Spikes: 85-115% recovery MSA: r≥0.995

TABLE 7.3.2 SUMMARY OF QC REQUIREMENTS FOR EPA METALS ANALYSIS METHODS USING INDUCTIVELY COUPLED PLASMA (ICP) SPECTROSCOPY

Requirement	Water and Wastewater Method 200.7	RCRA Solid Waste Analysis Method 6010	Superfund Hazardous Waste Analysis CLP SOW ILM04.0
Initial Calibration			
Frequency	1 standard and a blank Daily	1 standard and a blank Daily	1 standard and a blank Daily or every 24 hrs
Calibration Verification			
Frequency	Mid-range standard Every 10 samples	Mid-range standard Every 10 samples and at end	Mid-range standard Beginning, end, and every 10 samples or every 2 hours
Criteria	95-105% recovery	90-110% recovery	90-110% recovery
Other Standards			
Frequency	Highest mixed standard Before sample analyses	Highest mixed standard Before sample analyses	Standard at 2 x CRDL or IDL Beginning and end of each run or 2 every 8 hrs
Criteria	95-105% recovery	95-105% recovery	EPA QC Limits
Interference Check Sample			
Frequency	Beginning, end, and periodic intervals	Beginning and end of each run or every 8 hours	Beginning and end of each run or 2 every 8 hrs
Criteria	±1.5 x SD of mean value	80-120% recovery	80-120% recovery
Calibration Blanks			
Frequency	Every 10 samples	Every 10 samples and at end	Beginning, end, and 10% of samples or every 2 hrs
Criteria	±2 x SD of mean value	±3 x SD of mean value	All analytes ≤CRDL
Preparation Blanks			
Frequency	1 per batch	1 per batch	1 per SDG or digestion batch
Criteria	NS	NS	All analytes ≤CRDL
QC Check Sample/LCS			
Frequency	Each IC and weekly	Each IC and weekly	1 per SDG or digestion batch for each matrix
Criteria	95-105% recovery	90-110% recovery	80-120% recovery
Matrix Spike Samples			
Frequency	1 every new sample matrix	5% or 1 per batch	5% or 1 per SDG per matrix per level (predigestion)
Criteria	90-110% recovery	75-125% recovery	75-125% recovery
Duplicate Samples			
Frequency	NS	5% or 1 per batch	5% or 1 per SDG per matrix per level (predigestion)
Criteria	NS	≤20% RPD for values > 10 x IDL	≤20% RPD for values ≥5 x CRDL ±1 x CRDL for values <5 x CRDL
Serial Dilution			
Frequency	1 every new sample matrix	1 every new sample matrix	1 per SDG per matrix per level
Criteria	Dilution within ±5%	4 x dilution within ±10%	5 x dilution within ±10%

TABLE 7.3.2 SUMMARY OF QC REQUIREMENTS FOR EPA MERCURY ANALYSIS METHODS USING COLD VAPOR ATOMIC ABSORPTION (AA) SPECTROSCOPY

Requirement	Water and Wastewater Analysis Method 245.1/245.5	RCRA Solid Waste Analysis Method 7470/7471	Superfund Hazardous Waste Analysis CLP SOW ILM04.0
Method Detection Limit	0.2 ug/L	0.2 ug/L	CRDL: 0.2 ug/L
Holding Time	28 days	28 days	26 days
Initial Calibration Frequency Criteria	6: blank and 5 standards Daily r ≥0.995	6: blank and 5 standards Daily and every hour of analysis r ≥0.995	5: blank and 4 standards Daily or every 24 hours r ≥0.995
Calibration Verification Frequency Criteria	A standard at or near MCL After initial calibration and every 20 samples 90-110% Recovery	Mid-range standard Every 10 samples 80-120% Recovery	Independent standard CCV: diff. conc. than ICV, or at near the mid-range ICV: After initial calibration CCV: 10% or every 2 hours 80-120% Recovery
Calibration Blanks Frequency Criteria	After each calibration NS	After each calibration NS	Beginning, end, and every 10 samples or every 2 hours ≤CRDL
Preparation Blanks Frequency Criteria	1 per digestion batch NS	1 per digestion batch NS	1/SDG/digestion batch ≤CRDL
QC Check Sample/LCS Frequency Criteria	Blind performance sample 1 per year (Optional: 1 per quarter) EPA control limits	Independent standard Every 15 samples 80-120% Recovery	EPA standard 1/SDG/batch (solid samples only) 80-120% Recovery
Matrix Spike Samples Frequency Criteria	NS NS	5% or 1 per batch NS	5% or 1/SDG/matrix/level 75-125% Recovery
Duplicate Samples Frequency Criteria	10% or 1 per batch EPA control limits	5% or 1 per batch NS	5% or 1/SDG/matrix/level ≤20% RPD
Other Method Criteria Frequency Criteria	Method of standard addition As needed NS	Method of standard addition As needed NS	Standard at the CRDL or IDL Beginning of each sample run EPA control limits

TABLE 7.3.2 SUMMARY OF QC REQUIREMENTS FOR EPA CYANIDE ANALYSIS METHODS

Requirement	Water and Wastewater Analysis Method 335.4	RCRA Solid Waste Analysis Method 9012	Superfund Hazardous Waste Analysis CLP SOW ILM04.0
Method Detection Limit	Titration: 1 mg/L Colorimetric: 0.02 mg/L	Titration: 0.1 mg/L Colorimetric: 0.02 mg/L	CRDL: 10 ug/L
Holding Time	14 days (24 hours when sulfide is present)	14 days	12 days from sample receipt
Initial Calibration[1] Frequency	6 standards and a blank Daily	6 standards and a blank Daily	3 standards and a blank (one standard at the CRDL) Daily
Calibration Verification[2] Frequency Criteria	NS NS NS	Mid-range standard Every 15 samples 85-115% Recovery	CCV: Mid-range standard Beginning, end, and every 10 samples or 2 hours 85-115% Recovery
Other Standards (Distilled) Frequency Criteria	High and low standard 1 each per batch 90-110% Recovery	High and low standard 1 each per batch 90-110% Recovery	Mid-level standard 1 per batch 85-115% Recovery
Calibration Blanks Frequency Criteria	Colorimetric: 1 per batch Use in initial calibration	Colorimetric: 1 per batch Use in initial calibration	Colorimetric: Beginning, end and every 10 samples or 2 hours ≤CRDL
Preparation Blanks Frequency Criteria	Titration: 1 per batch Colorimetric: Not specified Titration: Use in calculation Colorimetric: Not specified	Titration: 1 per batch Colorimetric: Not specified Titration: Use in calculation Colorimetric: Not specified	Titration: 1 per batch Colorimetric: 1 per batch Titration: Use in calculation Colorimetric: ≤CRDL
Laboratory Control Standard Frequency Criteria	NS NS NS	Independent check standard 1 per batch 85-115% Recovery	Distilled independent standard (ICV) 1 per batch 85-115% Recovery
Matrix Spike Samples Frequency Criteria	1 per batch to check distillation efficiency NS	Matrix spike and matrix spike duplicate per batch NS	1 per matrix per concentration level per batch 75-125% Recovery
Duplicate Samples Frequency Criteria	NS NS	1 matrix spike duplicate per batch NS	1 per matrix per concentration level per batch ≤20% RPD for values >5 x CRDL
Other Method Criteria	Verify sample pH ≥12; Check for oxidizing agents and sulfides	Verify sample pH ≥12; Check for oxidizing agents and sulfides	Verify sample pH ≥12; Check for oxidizing agents and sulfides

KEY TO CHART	
BS	Blank Spike
CC	Continuing Calibration
CCC	Calibration Check Compounds
CCV	Continuing Calibration Verification
CRDL	Contract-Required Detection Limit
CRQL	Contract-Required Quantitation Limit
CV	Coefficient of Variation
D	Detected
IC	Initial Calibration
ICV	Initial Calibration Verification
IDL	Instrument Detection Limit
IS	Internal Standard
LCS	Laboratory Control Sample
MCL	Maximum Contaminant Level
MDL	Method Detection Limit

KEY TO CHART	
MS	Matrix Spike
MSA	Method of Standard Additions
NA	Not Applicable
NS	Not Specified
%D	Percent Difference
%Rec.	Percent Recovery
PQL	Practical Quantitation Limit
r	Correlation Coefficient
RF	Response Factor
RPD	Relative Percent Difference
RRT	Relative Retention Time
RSD	Relative Standard Deviation
RT	Retention Time
SD	Standard Deviation
SDG	Sample Delivery Group
SPCC	System Performance Check Compounds

NOTES

(1) Calibration standards must be distilled for EPA Methods 335.4 and 9012 when sulfides are present in the samples.

(2) CLP SOW specifies that the initial calibration verification standard (ICV) be distilled and analyzed as the laboratory control standard (LSC).

7.4 Standard Operating Procedures

All laboratory activities, from sample receipt to analysis to final report generation, must adhere to the laboratory Standard Operating Procedures (SOPs) which have been developed to provide quality environmental data with adequate documentation to be of known quality and hence of maximum use to our clients. All SOPs provide complete documentation as to how each sample is measured for each parameter. Reference corporate document QAS00200.NET for the IEA corporate format for generating SOPs. Each SOP shall have a unique code in accordance with the IEA corporate document control procedure as outlined in the corporate SOP on document control.

On a regular basis the QA Manager will review data to check for compliance to SOPs. Additionally the QA Manager will review SOPs to ensure they meet the requirements of the methodologies and applicable regulations. If it is found that the document does not meet the requirements, the discrepancy is forwarded to the group/section leader through the corrective action process. (reference SOP on Corrective Action Reports -QAS00501.CT).

In addition to method SOPs, at minimum the laboratory is required to have on file SOPs for the following operations. Many of these SOPs have been generated by the IEA corporate QA department.

Sample Receipt, Logging and Disposal
Chain-of-Custody Procedures
Sample Storage
Security of Samples and Laboratory Facility
Purity of Standards and Standards Preparation Documentation
Maintaining Laboratory Records and Logbooks
Sample Analysis and Data Control Systems
Sample Bottle and Glassware Cleaning Procedures
Laboratory Waste Disposal

An example listing of laboratory SOPs is presented in Section 7 of the Appendix. A complete list of all laboratory SOPs is available upon request.

7.5 Chain-of-Custody

Samples are physical evidence and are handled at IEA according to certain procedural safeguards. For the purposes of legal proceedings, a demonstration to the court that the laboratory is a secure area may be all that is required for the analyzed evidence to be admitted. However, in some cases, the court may require a presentation of the hand-to-hand custody of the samples while they were at the laboratory. In the event that a client requires such a comprehensive chain-of-custody demonstration, upon special request, IEA is capable of producing documentation that traces the in-house custody of the samples from the time of receipt to completion of analysis.

The National Enforcement Investigations Center (NEIC) of EPA defines custody of evidence in the following ways:

- It is in your actual possession; or
- It is in your view, after being in your physical possession; or
- It was in your possession and then you locked or sealed it up to prevent tampering; or it is in a secure area

At IEA-CT, chain of custody begins with shipment of the sample bottles and coolers. IEA-CT has a printed external chain-of-custody form that accompanies each sample shipment. An example of this form is found in Section 2 of the appendix.

Upon receipt of the samples in the laboratory the sample custodian and the sample control group are responsible for obtaining all necessary shipping documentation and verification of all data entered into the laboratory sample custody records. The internal laboratory custody form is generated at this point.

All samples and projects entering the laboratory are identified with a job/project number. Individual samples are then identified using the job number and sample counter. The samples are then stored according to the requirements of the analytical protocols (refrigeration) and preservative type.

Preliminary sample receipt notifications are distributed to each department to notify department of sample arrival and facilitate the analysis of parameters with short holding times. Each department has a system of tracking sample analysis throughout their respective departments to ensure protocol holding times are met.

All documentation received with samples is reviewed by the sample custodian at the time of receipt. The project manager then reviews the paperwork again at the time of log-in to the computer system. If there are any discrepancies noted by the sample custodian, a corrective action report is filled out and submitted to the project manager. The client is then contacted for resolution.

The specific procedures and requirements for receiving samples are specified in the SOP for sample control - "Sample Processing Methods Performed at Sample Arrival" (Doc# SMS00401.CT). IEA's chain-of-custody record is designed to meet the legal requirements of federal, state and local government agencies and the courts of law. The record covers:

- Labeling of sample bottles, packing the shipping container and transferring the shipping container under seal to the custody of a shipper;
- Outgoing shipping manifests;
- The chain-of-custody form completed by the person(s) breaking the shipping container seal, taking the sample, resealing the shipping container and transferring custody to a shipper;
- Incoming shipping manifests;
- Breaking the shipping container's reseal;
- Storing each labeled sample bottle in a secured area;
- Disposition of each sample to an analyst or technician; and
- The use of the sample in each bottle in a testing procedure appropriate to the intended purpose of the sample.

For each link in this process the records indicate the following:

- The person with custody; and
- The time and date each person accepted or relinquished custody.

IEA has implemented the following standard operating procedures with regard to laboratory chain-of-custody:

- Samples are stored in a secure area;
- Non-employee access to the laboratories are controlled through the use of limited access points at each facility. Outside personnel can access the facility either through the front receptionist or the sample receipt area. Other access doors to the laboratory are maintained in a secure manner at all times;
- All visitors to each facility are required to sign-in at the reception area and must be escorted by an IEA representative at all times while in the laboratory;
- Refrigerators, freezers, and other sample storage areas are kept locked, when not in use;
- The designated sample custodian and supervisory personnel control access to the sample storage area(s); and
- Samples remain in secured sample storage until removed for sample preparation or analysis; and
- Upon special request, all transfers of samples into and out of storage are documented through an internal chain-of-custody procedure.

7.6 Analytical Calibration Standards

The calibration standards used for instruments and equipment are described in the specific analytical methods, or instrument manufacturers' operational guides. All standard preparations are recorded in a bound "Standards Preparation Log Book" with the lot number, method of preparation, date and analyst's initials. This log provides the internal documentation which traces the internal working standards to primary and secondary (purchased) stocks.

The stock solutions are all kept in a daily monitored 4o C refrigerator with the exception of the organic stock solutions which are kept in a 0o C freezer. Stock calibration standards are coded in the "Prep Log" mentioned above with the analyte, concentration, date prepared, initials, and referenced to the book and page where a description of the preparation can be found and traced. No samples are maintained in the same areas as the standards.

Records on the traceability of the standards are maintained in the office of the Quality Assurance Manager. These records include sources, dates of receipt, lot numbers (if Applicable) and expiration dates (if applicable).

Table 7.6.1 provides an overview of the standard sources, types and preparation by instrument group.

Metals Calibration Standards

Commercially available at 1000 ppm levels from Inorganic Ventures and prepared from primary standard material traceable to EPA A2LA standards. Stock standards solutions are prepared every six months or when needed as multi-element stocks.

Inorganic Calibration Standards

Most calibration standards described in the methodology used ACS Reagent Grade materials. Some reference materials are available from NIST to standardize titrating solutions. Stock solutions are prepared every three months while diluted working standards are prepared daily at the time of analysis. Spike solution preparation is also documented in the solution/standard log book.

Organic Calibration Standards

Pure compounds for organic calibration materials are available through EPA EMSL in Cincinnati, EPA in Research Triangle Park, EPA Las Vegas, Supelco, Inc., Restek, Inc. and Accustandard, Inc. Organic stocks are prepared every six months and diluted working standards are prepared weekly. Stock non-volatile solutions can be prepared every six months and diluted working standards are prepared weekly. Stock non-volatile solutions can be prepared every six months with working standards made weekly. Organic spike solutions are prepared from neat solutions and documented.

pH Calibration Standards

Calibration materials which are certified by the manufacturer to be standardized against NIST Standards are commercially available and are used by the laboratory. Three standards - 4,7, and 10 are used daily to calibrate the pH meters.

Weighing Calibration Standards

Analytical balances are certified annually. Calibration is performed on a weekly or daily basis using class "S" weights (0.50, 5.00, and 50g).

Oven Calibration Standards

Daily calibration by monitoring oven temperature with a thermometer calibrated annually with a NIST Certified Thermometer.

Conductivity Calibration Standard

Conductivity solutions are described in Standard Methods, 15th edition, Section 502.

Turbidity Standards

Formazin solution prepared from CMS neat standard according to EPA Method 180.1-2. Four standards are used to prepare a calibration curve and are made fresh daily. The stock formazin standard is prepared every three months and kept under refrigeration.

Photometer Calibration Standard

Spectronic Standards - Catalog #331-31-50 (wavelength calibration).

Refrigerators

All refrigerators are checked daily for temperature stability. Yearly, the refrigerator thermometers are calibrated against an NIST thermometer. Daily readings are recoreded in a bound logbook.

TABLE 7.6.1 STANDARD SOURCES AND PREPARATION

Inst. Group	Source	Form Received	Storage	Preparation from Source	Laboratory Stock Storage	Preparation Frequency
GC/MS	Restek, Inc. EPA Supelco Accustandard	Neat Solutions> 1000 ppm	Frozen Frozen	Primary stocks are prepared from source stocks Intermediate stocks are prepared from primary or source stocks Working stocks are prepared from intermediates	Frozen Refrigerator N/A	Semi-annual Weekly Weekly
GC	Restek, Inc. EPA RTP Supelco Accustandard	Neat Solutions >1000 ppm	Frozen Frozen	Primary stocks are prepared from source stocks Intermediate stocks are prepared from primary or source stocks Working stocks are prepared from intermediates	Frozen Refrigerator N/A	Semi-annual Semi-annually Semi-annually
GFAA; ICP	Inorganic Ventures	Solutions of 1000ppm	Room temp.	Primary stocks (1 - 10 ppm) are prepared from source Intermediate stocks (1ppb - 1 ppm) Working stocks	O.15% HNO_3 at room temperature 0.15% HNO_3 at room temperature 0.15% HNO_3 at room temperature	Annually Semi-annually or as needed Daily

7.7 Instrument Calibration Procedures

The proper calibration of instrumentation and equipment is a key element in the quality of the analysis done by the laboratory. Each type of instrumentation and each EPA approved method has specific requirements for the calibration procedures, depending on the analytes of interest and the medium of the sample.

Tables 7.7.1 list in tabular form the procedures which are followed by IEA Connecticut. The calibration protocols meet or exceed the minimum method criteria requirements. If a method calibration requirement, outlined in a project specific QA Plan, is more stringent than those listed in the Quality Assurance Plan, the more stringent will be followed in each case.

Documentation and records on calibrations are maintained in instrument logs and also with the data sets of the samples which are analyzed and related to them. In addition, laboratory department managers monitor the results of the calibration program to ensure the proper implementation at the analyst level.

TABLE 7.7.1 INSTRUMENT CALIBRATION SUMMARY

Analysis	Cal. Type	# Standards	Type of curve	Acceptance/rejection criteria	Frequency
GC Pesticides Herbicides OP pesticides	Initial	5 concentration levels	Linear	≤ 20% RSD	continuing calibration fails
	Continuing	1 standard (mid)		+/- 15% Difference	Daily and every 10 samples
GC/MS quadrupole	Initial	5 concentration levels; tuning with BFB/DFTPP	Linear; tuned to manufacturer's specifications	≤ 20% RSD	continuing calibration failure
	Every 12 hours	1 standard; tuning with BFB/DFTPP		+/- 15% Diff	Daily
AAS Graphite	Initially	5 concentration levels	Linear	>.995 coefficient of variation	continuing calibration failure
	Continuing	1 standard		+/- 95% of value	Every 10 samples
ICP	Initially	5 concentration levels	Linear	According to instrument manufactures's instructions	Quarterly
	Daily	2 levels			
	Continuing	1 standard			Every 10 samples
Lachat Analysis	Initially,Daily	5 concentration levels	Linear	<.995 coefficient of variation	continuing calibration failure
	Continuing	1 standard			Every 10 samples
pH Meters	Initially and daily	2 standards (pH 7 and 4 or 10)	Linear	+/- 95% of value	Daily
	Continuing	1 standard			Every 10 samples

TABLE 7.7.1 INSTRUMENT CALIBRATION SUMMARY

Analysis	Cal. Type	# Standards	Type of curve	Acceptance/rejection criteria	Frequency
Spectrophoto-meter	Initially and daily	5 concentration levels plus set %T with no cuvette in holder	Linear	<.995 coefficient of variation	Daily
	Continuing	1 standard		+/- 95% of value	Every 10 samples
Infrared Spectrophoto-meter	Initially and monthly	5 concentration levels	Linear	<.995 coefficient of variation	Daily
	Continuing	1 level		+/- 95% of value	Every 10 samples
Conductivity meter	Daily	3 concentration levels	Linear	<.995 coefficient of variation	Daily
	Continuing	3 concentration levels		+/- 95% of value	Every 10 samples
Turbidimeter	Daily	3 concentration levels	Linear	<.995 coefficient of variation	Daily
	Continuing	3 concentration levels		+/- 95% of value	Every 10 samples
Balance	Daily	3 levels Class "S" weights	Point		Check single weight upon use

8.0 DATA PROCESSING

8.1 Introduction

Data processing is defined as the mechanisms employed for collecting, reviewing, transcribing, reporting and storing of analytical data and related information.

Because of the critical relationship between instrument calibration, the accuracy of the analytical data generated, and specific method protocols that determine data quality, IEA maintains strict controls on the calibration procedures for the various types of analytical equipment. Each type of instrumentation is calibrated prior to sample analysis according to method criteria. Specific criteria for the instrument calibrations must be met before samples may be processed. Corrective action must be taken to remedy any out of control situations.

8.2 Collection

Data in the environmental laboratory make take several forms. Some are manually generated, while others are automated computer outputs. Some examples of typical data are:

Field measurements or observations made on-site during the sample collection effort as part of a monitoring program.

Information provided on chain-of-custody forms such as sampler, sampling date, sample location, sample identification, weather observations and custody transfer information.

Recordkeeping information such as instrument run logs, standards traceability, sample preparation logbooks and balance calibrations which represent information not normally required for inclusion in client reports.

Analytical data produced by various instrumentation such as GC/MS units, gas chromatographs, atomic absorption spectrophotometers, and automated analyzers. This includes various associated outputs such as chromatograms, strip chart recordings and computer tape readouts.

Records of standard calibration curves as well as associated quality control data such as method blanks, matrix spikes, matrix spike duplicate, replicate and QC check samples.

Consistent data collection is achieved through the existence and use of standard operating procedures at each facility. For example, chain-of-custody forms are routinely checked for completeness and if omissions occur, the sampler is contacted for the missing information.

Laboratory data sheets or logbooks have a standard format to ensure that all pertinent information is recorded consistently. These items are regularly monitored to ensure compliance with established requirements.

Outputs from all instruments are monitored for readability and consistency. If clarity is less than desired, corrective actions are undertaken to rectify the output based on instrument manufacturers' recommendations.

The following sections will describe the general procedures which are employed at the IEA-CT laboratory. More specific detail can be found in the standard operating procedures.

Gas Chromatography

Data from the Gas Chromatographs is collected through interfaces and processed by a Hewlett Packard computer system (HP-1000) with RTE-A operating system and 3550A LAS software and or HP Chemstation with Enviroquant software. Data is reviewed at the bench level by the analyst. If all required QC is met then the data is reviewed for chromatographic scaling and dilutions. If necessary reintegrations and rescalings

are done using the LAS system or Enviroquant software. The binary result files are then converted to ASCII report files for transfer to the Seedpak system for data report forms generation.

GC/Mass Spectrometry

GC/MS data is collected utilizing Hewlett Packard 1000 RTE, RTA or DOS chemstation computer systems with Aquarius or Environquant software. This software allows for the comparison of sample non-target spectrum against reference library spectra. The most recent NIST/EPA mass spectral library supported by the system must be used. Data is reviewed by the analyst. If the data meets QC requirements, then binary data files are then converted to ASCII report files for transfer to the Seedpak II computer system via the network for data report forms generation.

Atomic Absorption

ICAP metals are analyzed by a Thermo-Jarrel Ash 61 or 61E. The data collected is transfered via a network system to the Seedpak system. Furnace data analyzed by the Perkin Elmer 5100s are collected on PCs and also transferred to the network to the Seedpak system for forms generation. Mercury data is analyzed on the TJA mercury analyzer and entered into Seedpak.

Classical Chemistry

Routine wet chemistry analyses have pre-printed logbooks, such as distillation logs and digestion logs. The less frequent analyses are recorded in analysts' notebooks. Raw data is then entered into the LIMS computer for data calculation. This includes the calibration curve data which may have been previously entered. Semi-automated analyses performed on the Lachat produce calculated final results. These results are then entered into LIMS. Any raw data produced is stored in a central file. Quality control data is manually calculated. Results data is reported off LIMS in the required format.

8.3 Review

Data review can be defined as the process whereby data is accepted or rejected based on specific criteria in order to ensure that the data are adequate for the intended purpose. In most cases, the criteria is defined by the particular analytical method.

Data review is performed prior to release of the data to the client. It is performed as soon as possible after data acquisition in order to provide sufficient time for corrective action if required.

In general, the procedure presented in Figure 8.3.1 is utilized by laboratory personnel throughout the network for data review purposes.

There are numerous policies and standard procedures which have been implemented to ensure that data of known quality is continually generated by the IEA-CT laboratory.

Each analytical SOP details the type and frequency of quality control checks. This includes such items as analysis of client reference standards, matrix spikes, blanks, the use of internal standards and surrogate spikes, etc. All calibrations are checked before sample analysis can begin. If the analytical system does not pass the initial QC limits, then the system is determined to be "out of control", and the cause of the problem must be determined and corrected before measurements can continue. Once the problem is corrected, QC measurements are repeated to verify the calibration. If the system is still out of control, the system is re-examined until the problem is corrected. General requirements are listed below:

Organics

- A minimum of one method blank is analyzed per 20 samples (or batch) per matrix, per concentration level or extraction procedure. A method blank is required every 12 hours for volatile analysis. Blanks and samples are analyzed on the same instrumentation. Pesticides/PCB's also require instrument blanks.
- Holding blanks are placed in volatile refrigerators on a weekly basis. For EPA CLP SOW volatile analysis, holding blanks are analyzed once per SDG.
- A matrix spike/matrix spike duplicate is analyzed at a frequency of one per 20 samples per matrix, per concentration level or per SDG, whichever is more frequent.
- Prior to sample processing, surrogates are added to all samples and method blanks. GC/MS analyses also require the use of internal standards.
- Multi-level initial calibration curves are performed with continuing calibration standards analyzed every 12 hours. Recalibration is required if criteria cannot be met.
- GC/MS system tuning is verified every 12 hours.

Inorganics

- Multi-level calibration is performed on required instrumentation and verified as required.
- Calibration and prep blanks are analyzed at required frequencies.
- A matrix spike and sample duplicate are analyzed every 20 samples/SDG per matrix type.
- A Laboratory Control Sample is analyzed every 20 samples or per batch.
- Multi-level calibrations are performed for all manual and semi-automated wet chemistry methods and verified as required (if applicable).
- Method blanks are analyzed at required frequencies.

The precision and accuracy control limits employed by IEA are based primarily on limits contained in the published methods or required by the U.S. Environmental Protection Agency's Contract Laboratory Program (CLP). When warranted by IEA's historical data, more restrictive control limits are set than those cited by the method or the CLP.

When the CLP protocol is not applicable to analysis of samples, the precision and accuracy requirements for each analytical method are included in the individual laboratory Standard Operating Procedure (SOPs). Examples of data acceptance criteria is detailed in table 7.3.2.

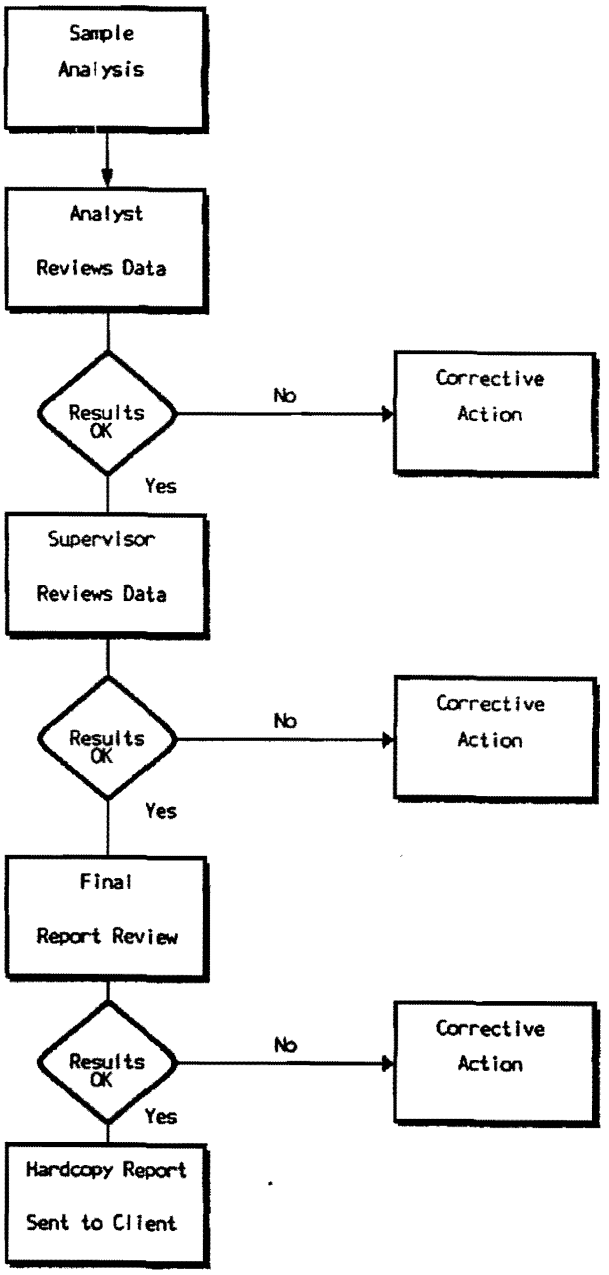
At a minimum, all data will be subject to analyst and supervisory review. Sensitive data requires higher level review and release. Final releases of data must be in writing.

Each analytical group in the laboratory is responsible for generating the data for all analyses the group performs. In general the data must first meet all the specific QA/QC associated with the SOP that was used for the analysis prior to any release of the data. The analytical group leader (supervisor) is responsible for the final verification of the data from the analysis.

The laboratory employs a system of QA sign-off sheets called QC Batch Approval Forms and Quality Control Approval Reports (QCAR's), where each analyst must sign off that their respective part of the analysis is complete and meets the QA/QC requirements of the governing SOP. Both the Volatile and semi-volatile RTE computer systems produce batch-specific QC summary reports to check various analytical parameters. Analysis QCAR's are filled with the analysis batches while the final deliverable QCAR's are signed and placed in each job folder along with any Corrective Action Forms (CAF) which details any problems which were encountered in the measurement of samples. Any deviations from SOPs are noted on CAF's and explained in the SDG narrative which is incorporated into the final report. The group leader has final sign-off responsibility on the QCAR and is responsible for assuring the overall quality of the data.

The laboratory Quality Assurance Manager periodically examines data packages at random to ensure that all QCAR's are present and to ascertain that the data package meets the requirements as stated in the SOP. These findings are transmitted to laboratory management via progress reports.

FIGURE 8.3.1 NETWORK DATA REVIEW PROCESS (GENERAL)



8.4 Data and Report Storage

Unless specified otherwise by the client, all analytical data and associated information is stored for a minimum period of three years. Local state data storage requirements may vary from the corporate requirement and must be met by the laboratory if they are more stringent.

Stored information may consist of hardcopy or electronic data stored on a magnetic media.

All hardcopy information is stored at the laboratory that generated the data or off-site at a commercial document storage facility equipped with a professional security system.

All electronic data is stored on-site at the laboratory that generated the data or off-site at a commercial document storage facility equipped with a professional security system and a controlled environment suitable for storage of magnetic media.

Access to archived information is controlled by the appropriate data management custodian or facility manager.

At IEA-Connecticut, reports for the current year are filed by the data management department. The report files along with any data package are then stored in numbered boxes. The number of the box is recorded into the cross reference logs and then stored in the locked storage area in the basement. All jobs must be signed out in a logbook if being removed from the data management area.

8.5 Transcription

Whenever possible, manual data transcription is avoided through the use of electronic data transfer within the laboratories. In cases where manual transcription is employed, information is checked and verified by the supervisor or designee within the department.

It may not be possible to totally eliminate transcription related errors, however, section 4.6.9, paragraphs A and D, list procedures which are designed to minimize their occurrence and impact on data quality.

8.6 Data Reduction

Data reduction includes all processes that change either the form of expression (i.e., the units of measure) or the quantity of data values (rounding). It often involves statistical and mathematical analysis of data and usually results in a reduced subset of the original data set. Data reduction is performed either manually by the analyst or by computer systems interfaced to the analytical instruments. Whenever such procedures are employed within the laboratory network, mathematical procedures have been verified for accuracy of computation.

An example of this would be for "CLP like" data packages, the data is transferred directly onto the Seedpak II system computer software from the Metals, GC and GC/MS systems via the network. The data is further processed and stored in the database. Other relevant data is transfered via the network at this point such as client ID's, etc. All calculations and final results are performed by the Seedpak II software. Many of these calculations are also done at the instrumentation level as a secondary review. Data in the database is sorted by client delivery group for easy retrieval. CLP type forms are generated after all data is entered and reviewed. The forms and raw data are compiled into a data package. Data summary results are also generated at this point for level I reports, reducing the occurance of typographic errors.

The data associated with each analysis is hardcopied for permanent storage either through the printing of computer files or through hand entry into bound laboratory notebooks. All notebook entries are dated and signed by the analyst.

Job packages which include 20 samples or samples received by the laboratory during a one week time frame will comprise an "SDG". All organic parameter results will be reported in ug/L for aqueous samples and ug/Kg dry weight for soil/sediment samples. Inorganic result units vary according to the methodology.

It is laboratory policy that any and all problems related to client samples and the measurement of client samples be documented in the SDG narrative of the final laboratory report which goes to the client. The mechanism for documenting problems which shall be included in the SDG narrative is described in Section 10.0. It is the responsibility of the data management group to see that information on CAR's is included in the final SDG narrative.

After final review by the department manager, the data is placed in Data Management for tracking on the project status sheet. If possible the data is placed into the job folder. When all parameters are complete the project is now ready for assembly by the data management department. It is the responsibility of the data management group to make sure that all the data is present and deliverable requirements are complete. This may include chain of custody forms, special instructions, and case narratives. The data is then compiled and sent to the report production group for word processing.

9.0 DATA QUALITY ASSESSMENT

Data quality is assessed based on five main characteristics:

Precision
Accuracy
Completeness
Representativeness
Comparability

Each of these characteristics have been previously defined in section 2.2 of this document.

Laboratory Quality Assurance Objectives

Precision:

The objective of the network laboratories concerning precision is to equal or exceed the precision demonstrated in the published analytical method on similar samples. Relative Percent Difference (RPD) is used as the measure of precision sample duplicates. The formula utilized to calculate RPD is as follows:

Relative Percent Difference (RPD)

$$RPD = \frac{(\text{Sample Result - Duplicate Result})}{\text{Mean of Sample and Duplicate Results}} \times 100$$

Note: RPD is expressed as the absolute value obtained from the above formula.

Accuracy:

The objective of the network laboratories concerning accuracy is to equal or exceed the accuracy demonstrated in the published analytical method on similar samples. Accuracy is determined on matrix spikes and/or blank spikes and is calculated as follows:

$$\text{Percent Recovery} = \frac{(\text{Observed-Sample}) \text{ Concentration}}{\text{Spiked Concentration}} \times 100$$

Completeness:

IEA's objective for completeness is to be able to provide analytical data for 100 % of samples received intact and have sufficient sample volume for conducting re-analysis if initial analysis does not meet QC acceptance criteria.

Representativeness:

Representativeness of the analytical data is primarily a function of the sampling procedures and techniques employed in the field. As such, the sampling plan must be designed to provide representative samples to the laboratory. Once received at the laboratory, samples are homogenized in an effort to yield representative data on the sample submitted for analysis.

Comparability:

IEA's objective for comparability is that all data be fully comparable with data from other network laboratories. This is accomplished through use of the following practices:

- Demonstrate traceability of standards to NIST or EPA sources
- Use of standard and approved methodologies
- Standardized units of measure
- Standardized QC acceptance criteria
- Participation in interlaboratory studies to demonstrate laboratory performance

9.1 Content of Analytical Reports

Laboratory customers have a wide variety of analytical needs. In order to meet these varied requirements, the laboratory offer several levels of data reporting options ranging from very simple format to an extreme level of documentation. Table 9.1.1 presents the contents of various levels of reports offered by the laboratory. Custom reporting beyond those listed is usually available but may require additional cost. The information provided in Table 9.1.1 is a summary only. In some cases, individual methods may not include the indicated items. For example, in metals graphite furnace analysis an ICP interference check would not be included since it is inappropriate for that method.

Table 9.1.1 Report Content Options

	Data Reporting Options		
Wet Chemistry	Level 1	Level 2	Level 3 (CLP)
Case narrative	Yes	Yes	Yes
Sample Results	Tabular	Tabular	Form I
Method Blank	Yes	Yes	Yes
External Chain of Custody	Yes	Yes	Yes
Internal Chain of Custody	Yes	Yes	Yes
Duplicate	-	Yes	Yes
Matrix Spike	-	Yes	Yes
Initial Calibration Verification (ICV)	-	-	Yes
Continuing Calibration Verification (CCV)	-	-	Yes
Laboratory Control Sample (LCS)	-	-	Yes
EPA Forms 1-14	-	-	Yes
Metals	Level 1	Level 2	Level 3 (CLP)
Case Narrative	Yes	Yes	Yes
Sample Results	Tabular	Tabular	Form I
Method Blank	Yes	Yes	Yes
External Chain of Custody	Yes	Yes	Yes
Internal Chain of Custody	Yes	Yes	Yes
Duplicate	-	Yes	Yes
Matrix Spike	-	Yes	Yes
Initial Calibration Verification (ICV)	-	-	Yes
Continuing Calibration Verification (CCV)	-	-	Yes
Laboratory Control Sample (LCS)	-	-	Yes
ICP Interference Check	-	-	Yes
ICP Linear Range	-	-	Yes
ICP Post Spike	-	-	Yes
EPA Forms 1-14	-	-	Yes
Organics	Level 1	Level 2	Level 3 (CLP)
Case Narrative	Yes	Yes	Yes
Sample Results	Tabular	TAbular	Form I
Method Blank	Yes	Yes	Yes
External Chain of Custody	Yes	Yes	Yes
Internal Chain of Custody	Yes	Yes	Yes
Matrix Spike	-	Yes	Yes
Matrix Spike Duplicate	-	Yes	Yes
Laboratory Control Sample (LCS)	-	-	as needed
Surrogate Recovery Information	-	Yes	Yes
Tuning Data (GC/MS only)	-	-	Yes
Initial Calibration Information	-	-	Yes
Continuing Calibration Information	-	-	Yes
Run Sequence Logs	-	-	EPA only
Sample Preparation Logs	-	-	Yes
Chromatograms and Mass Spectra	-	-	Yes
EPA Forms 1-8	-	-	Yes

10.0 CORRECTIVE ACTION

10.1 Introduction

The Corrective action report form (CAR), presented in Section 7 of the Appendix, provides a routine written communication vehicle to describe most types of problems which may occur throughout the laboratory or as a result of a client inquiry. Problems described in SDG narratives should be supported by a CAR.

Corrective actions can be initiated at several operational levels; however they must always involve the QA Manager. Corrective actions are reviewed, documented and distributed to the appropriate personnel through the QA department. Responses are returned to QA for review and redistributed in a specified time frame.

Examples of three types of corrective actions which may be initiated are as follows:

Sample problems

Individual samples or matrix problems may cause documented corrective actions such as re-extraction, reanalysis, cleanups or dilutions.

QC problems

Corrective action may occur on entire batches of samples when QC criteria cannot be achieved.

Systematic problems

Specific project issues and procedural issues may require corrective actions. These are handled by laboratory management and the QA department.

The QA Manager will monitor and log the progress of CAR's and will report in the QA Progress Report the status of major corrective actions taken in the past month. It is the QA Manager's responsibility to see that laboratory problems are documented and solved in a timely manner. This system is outlined in the SOP for Corrective Action Reports - QAS00501.CT.

10.2 System Audit

A system audit is an inspection and review of the entire data generation and support system of a laboratory. Activities related to the established requirements in the quality assurance program are reviewed for compliance. A typical system audit includes an evaluation of the following:

- Assessment of degree of compliance with the quality assurance program
- Continuing compliance with corrective actions identified in a previous audit of the facility
- Calibration procedures and documentation
- Sample handling procedures including chain-of-custody
- Experience of laboratory personnel
- Existence and routine use of standard operating procedures
- Analytical data review and validation procedures
- Data storage and recordkeeping

A system audit is performed by the on-site quality assurance manager at each facility annually.

As previously indicated, all system audits are conducted utilizing a comprehensive standardized checklist (IEA Doc.# QAS00300.NET). Copies of the system audits conducted by the QA managers are submitted to the appropriate laboratory director/manager and president for review.

The auditor will identify any deficiencies in the audit report which is to be generated within a week of the actual audit. The laboratory director/manager is required to respond, in writing, no later than 30 days from issuance of the audit report. The response must address each of the items contained in the audit. If corrective action cannot be taken immediately, the anticipated date of compliance must be presented. If the auditor identifies issues which are significant (in their opinion), a follow-up audit can be conducted prior to the regularly scheduled audit.

A summary of the audit report findings is included in the quality assurance status report provided to management by the corporate quality assurance director.

10.3 Performance Audits

A performance audit is a quantitative check on the accuracy and/or precision of analytical data.

IEA network laboratories participate in a number of contracts and certification programs (see Table 5.4.1). Many of the certification programs employ rigorous performance evaluations which take the form of proficiency samples submitted to the laboratories on a regular basis. The following represents typical examples of routine proficiency programs.

All network laboratories are active participants in EPA Water Pollution (WP) and Water Supply (WS) proficiency programs which issue performance check samples on a semi-annual frequency.

IEA-CT participates in a number of contracts and certification programs. Many of these programs employ performance evaluations which take the form of proficiency samples submitted to the laboratory on a regular basis.

Bi-annually, the laboratory participates in the USEPA Water Supply (WS) and Water Pollution (WP) proficiency programs. IEA-CT also participates in the NYSDOH proficiency testing program for Potable Water, Hazardous Waste and CLP. The lab currently analyzes quarterly organic PE samples from EPA for the CLP program.

A copy of all analytical results associated with any proficiency samples is submitted to the operations director and president by each laboratory. The corporate office reviews this information and will utilize it in performing the regularly scheduled system audits at each lab. If results indicate a significant problem may exist, the network QA director will investigate accordingly.

In addition to participating in the above performance evaluation programs, the corporate office conducts additional performance evaluation studies.

Periodically, performance evaluation samples are submitted to each laboratory for parameters which are not addressed in other performance evaluation programs (ie. TCLP testing). In this type of testing the laboratory is aware the samples are performance check samples but the "true" concentrations are unknown. The results are submitted to corporate QA for evaluation and a report is issued on the findings. Corrective actions are taken if required, as a result of these test findings.

10.4 Independent Audits

IEA network laboratories are routinely audited by state and federal agencies for compliance with government regulations. In addition, several industrial clients conduct systems and performance audits of the facilities prior to project plan approval.

10.5 Subcontracted Services

IEA network laboratories occasionally choose to send selected analyses to a subcontract laboratory outside of the IEA organization. The most common reason for utilization of a subcontract facility is that the procedure is not routinely performed by an IEA network laboratory and the subcontractor has greater experience in day-to-day execution of the method. In such cases, although an IEA lab could in all likelihood conduct the analysis, it is more cost effective for both IEA and the client to utilize a subcontract lab as necessary. All subcontract laboratories utilized by IEA on a continuing basis require approval of the QA department prior to use.

IEA's clients are notified whenever another IEA laboratory or a subcontract laboratory is to be utilized for any portion of the analytical requirements. Although all analytical data appears in the IEA report, all data produced by another IEA laboratory or a subcontract laboratory is identified. In specific cases, states (ie. New Jersey) may have specialized requirements concerning the reporting of subcontracted analyses. In such cases, the laboratory will comply with the stated requirements. Subcontractors are not utilized when specifically restricted in a client's quality assurance project plan.

APPENDIX, Section 1

PROFESSIONAL PROFILES OF KEY PERSONNEL

The following professional profiles are presented alphabetically and represent the key quality assurance and laboratory management personnel for the network organization. Additional professional profiles are available for review during a site visit to any of our laboratory facilities.

PROFESSIONAL PROFILE
Michael V. Bonomo

TITLE: Director - Northern Region - IEA/American Environmental Network

ACADEMIC ACCOMPLISHMENTS:

Fordham University - Bronx, New York
B.S. Biology

Pace University - White Plains, New York
M.B.A Marketing

MAJOR AREA OF EXPERTISE:

Environmental Regulations
(RCRA, CERCLA, CWA, SDWA, ECRA)

Sampling and Analysis Plan Design

Data Management

SUMMARY OF EXPERIENCE:

Mr. Bonomo has over 19 years experience in environmental monitoring programs. He has functioned in numerous roles including director, co-director, sales manager, project manager, field and laboratory scientist, consultant, and seminar instructor. He has assisted many Fortune 500 companies and consultant/engineers in the design and implementation of sampling and analysis project plans. He has been involved in a wide spectrum of environmental programs for groundwater, soil, and sludge testing as well as monitoring various aquatic biota. Mr. Bonomo is also experienced in data management requirements for large analytical projects. He was instrumental in developing and implementing a data collection through data reporting system that was successfully utilized on many projects. He has also served as a seminar instructor for groundwater monitoring sampling and data tracking for a major waste management company.

PROFESSIONAL EXPERIENCE:

1992 to Present — IEA, Inc.
Monroe, CT

Position — Vice-President, Director of Operations

Responsibility

Responsible for overall operations and profitability.

1991 to 1992 — IEA, Inc.
Monroe, CT

Position — Co-Director

Responsibilities

Co-responsibility for the profitability and management. Duties included business development, marketing, financial and budget management, sales management, strategic planning and monitoring operations.

1990 to 1991 IEA, Inc.
Monroe, CT

Position Sales and Marketing Manager

Responsibilities

Responsible for the sales staff, corporate strategic planning, sales management and marketing in the New Jersey, Connecticut, Massachusetts and Vermont laboratories.

1989 to 1990 York Wastewater Consultants (YWC)
Monroe, Connecticut

Position Executive Director

Responsibilities

Assisted in the growth of an unknown Connecticut based company to one that covered all of the Eastern United States. Participated in a 6-month strategic planning process that provided insight into the tools needed to run a successful company. In spite of severe banking problems, a 2-year Federal EPA investigation, and a downturn in the market, saw YWC through successful acquisition by Aquarion.

1987 to 1989 York Wastewater Consultants (YWC)
Monroe, Connecticut

Position Sales and Marketing Manager

Responsibilities

Managed three York Laboratories division of YWC, Inc. Responsible for the Northeast, Mid-Atlantic and Midwest United States. Built sales and marketing program where non had existed before. Helped to assimilate five disjointed businesses into a single working division with resource sharing, cross training, budget management, and team building.

1982 - 1987 ETC

Position National Account Executive

Responsibilities

Developed and managed new business for analytical and data management services. Responsible for marketing to many Fortune 500 chemical, petrochemical, waste, and electronics firms. Efforts included major projects throughout the United States. Worked with clients in regulatory compliance, project design, and data use and interpretation. Developed a client base that was involved in RCRA groundwater monitoring, CERCLA site Remedial Investigation Feasibility Studies, New Jersey ECRA investigations, and Clean Water Act compliance. Involved in one of the first petroleum refinery land treatment demonstrations in the United States. Served as the Chairman of the ETC Technical Product Development Committee.

IEA
An Aquarion Company

PROFESSIONAL PROFILE
Jeffrey C. Curran

TITLE: Laboratory Manager

ACADEMIC ACCOMPLISHMENTS:

Southern Connecticut State University - New Haven, Connecticut
B.A. Chemistry, 1975
M.S. Chemistry, 1978

MAJOR AREA OF EXPERTISE:

Quality Control/Quality Assurance
Hazardous Waste Analyses
Classical and Wet Chemistry Analyses
PCB Analysis
Capillary GC/MS Analysis
Industrial Hygiene

Certified Laboratory Director for the States of Connecticut, New York, New Jersey and Massachusetts.

SUMMARY OF EXPERIENCE:

Mr. Curran has extensive experience in analytical chemistry specializing in environmental analysis. He has worked in all areas of the laboratory and has hands-on expertise in general wet chemistry techniques, atomic spectroscopy, gas chromatography, infrared spectroscopy and gas chromatography/mass spectrometry.

PROFESSIONAL EXPERIENCE:

Present

IEA, Inc. - Connecticut

Position Laboratory Manager

Responsibilities

For the past 15 years Mr. Curran has directed and participated in a variety of projects. Some highlights are listed below:

Hazardous Waste Site, East Windsor, CT

At a major Connecticut Hazardous Waste site Mr. Curran participated in the sampling analysis of buried drums of hazardous waste during a state-supervised cleanup project.

Ethylene Oxide Emissions Testing, Sherburn, New York

At a major EtO user in Upstate New York, Mr. Curran directed an on-site testing program for measuring EtO emissions using gas chromatography. Mr. Curran also worked on a testing program in conjunction with the NYSDEC for testing pollutant control equipment for EtO sterilizers.

Canadian Tariff Board Hearings

Mr. Curran provided expert witness testimony at a Canadian Tariff Board Hearing concerning chemical composition of foam packaging material.

Worker Exposure Study, Lynchburg, Virginia

Mr. Curran directed an on-site industrial hygiene study to monitor employee exposure to various solvents and chemicals. Mr. Curran was also part of the team which analyzed the various samples collected using gas chromatography, atomic spectroscopy, and UV-VIS spectroscopy in accordance with NIOSH protocols.

Food Processing Plant, Rochester, New York

Mr. Curran conducted an investigation to determine the cause of stainless steel tubing failures for a national food process company. The results of this study were used in determining alternatives to the current materials used in the process.

Hazardous Breakdown Product Study

Mr. Curran designed a system to identify and measure potentially hazardous breakdown products resulting from the pyrolysis of plastic materials for an international aircraft manufacturer. Results of this study were used to identify what materials were responsible for and how to alleviate the problem.

PROFESSIONAL AFFILIATIONS:

Member of the American Chemical Society

1980 - 1982 Lawler, Matusky and Skelly Engineers

Position Assistant Project Manager

Responsibilities

Project Manager for environmental monitoring programs related to the electric utility industry. Responsible for proposal writing, program design, management of staff scientists and technicians for projects, budget control, report writing and technical presentations. These projects included water chemistry, fish population studies, lower trophic level monitoring, and mitigation of the impact of power plants on the river environment. Designed and implemented groundwater sampling programs with customized equipment for a major site in New York State.

1978 - 1980 Lawler, Matusky and Skelly Engineers

Position Project Scientist

Responsibilities

Crew Chief for field survey including sampling of biota, water, soil and sludge. Responsible for maintaining field control of samples, including documentation, custody, and proper sampling techniques. Performed laboratory analysis including wet chemistry procedures, fish taxonomy and other biological studies. Responsible for writing Standard Operating Procedures for laboratory operations.

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PROFESSIONAL PROFILE
Marsha Culik

TITLE: QA Manager

ACADEMIC ACCOMPLISHMENTS:

S.U.N.Y. at Alfred - Alfred, New York
A.A.S. Medical, 1976
Laboratory Technology

MAJOR AREA OF EXPERTISE:

Extensive development and "hands on" experience with Gas Chromatography, Atomic Absorption Spectrophotometry, Auto Analyzer, and some computer data stations.

SUMMARY OF EXPERIENCE:

Ms. Culik has over 12 years experience in the environmental laboratory field. Experience ranges from analysis of drinking water with a Grade 3 Water Treatment Plant Operator to gas chromatography chemist with environmental samples. Ms. Culik has experience as supervisor of the Gas Chromatography department.

PROFESSIONAL EXPERIENCE:

1/91 to Present

IEA, Inc. - Connecticut

Position QA Manager

Responsibilities

Quality Assurance Manager, responsible for monitoring the continuing compliance with the Corporate QA Program and to be a liaison between Corporate QA and laboratory staff.

Additional responsibilities include maintaining certification programs, coordination of external and internal audits, coordinate all inquiries relative to quality issues and follow-up on corrective actions as necessary, maintain files of all QA related documentation include review and approval of all SOP's.

1986 to 1991

Position GC Group Leader

Responsibilities

Supervisor of GC Group, responsible for analysis of environmental samples for pesticides/PCB's according to EPA/NYSDEC CLP Protocols, SW846 Methods and EPA "600" Series Methods. Additional responsibilities include analysis of samples via purge & trap/GC according to various protocols.

Other duties include analysis of air samples, charcoal absorbent tubes and other miscellaneous samples for any parameters requiring gas chromatography analysis. She is also responsible for supervision of the group including sample tracking, data review, etc.

1984 to 1986

Position Chemist

Responsibilities

Experience in sample prep and GC analyses of Pesticides/PCB's in water, oil and soil samples.

1981 to 1984

Position Laboratory Analyst - American Waterworks Service Company

Responsibilities

Experience performing complete laboratory analysis or raw, potable, and waste water including all miscellaneous include Volatile Organics, Trihalomethanes and Aromatics using Purge and Trap techniques; Pesticides and Herbicides by GLC; Transition and Heavy Metals by Flame and Graphite Furnace Atomic Absorption; and Nutrients by Automated and other various wet chemistry procedures. Assisted Lab Director in the development of many methods used in these analyses. Responsible for collection and interpretation of all quality control data.

1978 to 1981

Position Lab Technician - Suffolk County Water Authority

Responsibilities

Laboratory experience in the analysis of potable water for a large water utility. Cooperative studies done in conjunction with state and local health agencies concerning water and wastewater quality. Also monitoring the chemical quality of water and seawater programs for the U.S.G.S. Primary responsibilities were for the analysis of Halogenated and Aromatic organic compounds by Purge and Trap Gas Chromatography. Other areas of experience include the analyses of nutrients by Technicon Auto Analyzer, metals by Flame and Graphite Furnace Atomic Absorption, and microbiological testing using Millipore System.

1976-1978

Position Lab Technician - Hooker Chemicals & Plastics

Responsibilities

Responsible for the analysis of vinyl chloride monomer in PVC Compounds, Resins and Food Packageability studies utilizing Gas Chromatography. Responsible for monitoring the air quality of the plant environment.

SPECIALIZED TRAINING:

1984 Certified Grade 3 Water Treatment Plant Operator

1977 ASCP Registered MLT

Environmental Laboratory Management
Two day seminar on Environmental Laboratory Management
John H. Taylor, Analytical Technology.

Performance Management Workshop
One day seminar
Cynthia Barnet, Human Resources Consultant

Interview Skills Workshop
One day seminar
Cynthia Barnet, Human Resources Consultant

Leadership Development Workshop
Four day workshop
William Frackler, Ingoldsby, Inc.

Mass Spectral Data Interpretation
One day seminar
Dr. Frank Rutecek, Cornell University

Introduction to Analytical Separations
Four day seminar
Dr. Dhea Habboush, Sacred Heart University

ASQC Course
Auditing of Quality Systems

ASQC Course
Introduction to SPC

IEA
An Aquarion Company

PROFESSIONAL PROFILE
Lawrence H. Decker

TITLE: GC/MS Manager

ACADEMIC ACCOMPLISHMENTS:

Franklin Pierce College - Rindge, New Hampshire
B.A. Biology 1982

MAJOR AREA OF EXPERTISE:

Final Data Review
Coordination of sample analysis for the GC/MS group
Organics analysis by GC/MS

SUMMARY OF EXPERIENCE:

Lawrence Decker has eight years of GC/MS experience. He has been responsible for operations of the GC/MS group for five years. Presently functioning as the Volatile Group Leader.

PROFESSIONAL EXPERIENCE:

5/92 to Present

IEA, Inc. - Connecticut

Position GC/MS Manager

Responsibilities

Responsible for the volatile group operations. Duties include: Scheduling workforce, ordering supplies, final data package review, employee reviews, overseeing sample analysis and sample prioritizing, adhering to forecasted budget, dealing with client requests, training employees, updating sample/job status with client service and laboratory directors. Tracking workflow through group.

10/91 to 5/92

Position GC/MS Section Leader

Responsibilities

Responsibilities included: Sample analysis for both semi-volatile and volatile samples, tracking and scheduling sample analysis, troubleshooting instrumentation, final data package preparation and review. Unknown compound determination (TIC's). Assisting Group Leader with selected tasks. Responsible for tracking and prioritizing sample analysis, reviewing both initial sample batches and final reports, troubleshooting instruments and monitoring of GC/MS operations.

4/86 to 9/90

Position GC/MS Operator

Responsibilities

Running samples, calibrating instruments, tracking samples, screening, total solids standard preparation, paperwork. Familiarity with EPA/NYSDEC CLP, SW846 and EPA "6--" Series VOA and BNA methods and routine analysis of aqueous and soil samples for VOA and BOA target and non-target (TIC) compounds. Experience in the data review process which involves monitoring surrogate recoveries, internal standard areas, target compounds concentration ranges and matrix spike/matrix spike duplicate performance parameters.

SPECIALIZED TRAINING:

Mass Spectroscopy Data Interpretation
One day Seminar
Dr. Frank Turecek (Cornell University)

Course description included close examination of mass spectra pertaining to identification of molecular ion, stability structure relationship, characteristic ion group effects, fragmentation and identifiable isotope clusters. Further concepts discussed include the nitrogen rule, the picket fence (alkane) series, and common fragment ions.

RTE-VI Procedures File Workshop
Four day seminar
GC/MS HP Aquarius Software Training
Mark Harwick (HP Instructor)

Course description included detailed examination of GC/MS Hardware, theory and function of mass spectroscopy, data acquisition and interpretation. Course emphasized software manipulation to enhance the overall quality and quantity of accurate and legible data.

Hewlett-Packard User I Course
Five day seminar
Hewlett-Packard, Paramus, New Jersey

Course description included a general overview of the HP computer system, mass spectrometer theory, instrument tuning and utility programs.

Introduction to Analytical Separations

Introduction to Chemical Analysis

Terms associated with chemical analysis; a review of the important considerations in analytical chemistry; sensitivity and detection limit; evaluation of results.

Analytical Separation

Solvent extraction; emulsions, completeness of extraction; extraction of organic compounds; pH effect; extraction with metal chelator.

Chromatography (General Principles)

Chromatographic behavior of solutes; column efficiency and resolution.

Gas Chromatography

Gas chromatograph; gas chromatographic columns; liquid phases and column selection; detectors for gas chromatography; optimization of experimental conditions; interfacing gas chromatography with mass spectrometry.

IEA
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PROFESSIONAL PROFILE
John Bennett, Jr.

TITLE: GC/MS Semi-Volatiles Supervisor

ACADEMIC ACCOMPLISHMENTS:

Southern Connecticut State University - New Haven, CT
B.S. Biology 1978 (Chemistry Minor)

MAJOR AREA OF EXPERTISE:

Classical Chemistry
Atomic Spectroscopy
Organic Extractions
Gas Chromatography
Microbiology

SUMMARY OF EXPERIENCE:

An extensive background in all phases of laboratory operations. Was responsible for designing, specifying, and hiring staff for a state of the art environmental laboratory. Had day to day responsibility for all phases of operation of the lab. Responsible for writing and conducting performance reviews for staff. Implemented stringent QA/QC program in the lab following USEPA CLP protocols. Had direct responsibility for inorganics section of the laboratory. Functioned as a resource person and problem solver for staff.

Wide ranging experience in the analysis of environmental and hazardous waste samples using EPA, APHA, and ASTM methodologies. Experienced in the analysis of contaminants from stationary sources. Has performed industrial hygiene surveys fore a variety of contaminants, and is familiar with the NIOSH procedures for their analysis. Instrumental expertise is ICP spectroscopy, as well as flame and furnace atomic absorption spectroscopy. In addition, has extensive experience with all basic laboratory apparatus and gas chromatography.

A broad background in microbiology including the identification and enumeration of microorganisms from a wide variety of sources. Familiar with USP and APHA procedures of analysis. Performed studies on the effects of point source contamination of water supplies and has performed characterization of problem microorganisms in sewage treatment plants. Developed a novel procedure for determining the microbial kill effectiveness of ethylene oxide sterilization cycles..

PROFESSIONAL EXPERIENCE:

1988 to Present

IEA, Inc. - Connecticut

Position GC/MS Semi-volatiles Supervisor

Responsibilities

Responsible for daily operations of organics extractions group. Interacted with other departments in the laboratory concerning the status of client samples. Responsible for the supervision of six staff members. Responsible for the quality of work produced by group as well as meeting turnaround goals.

1987 to 1989

Position Laboratory Director - Chemrox, Inc.

Responsibilities

State of Connecticut Certified Laboratory Director for Chemrox Laboratory Services. Had overall responsibility for the operation of the laboratory, as well as the development of the business. Supervised 10 staff members. Interacted with other departments in the company, as well as outside clients on technical aspects of laboratory analyses. Participated in seminars to educate various groups about environmental issues.

1985 to 1987

Position Senior Chemist

Responsibilities

Responsible for ethylene oxide associated analyses. Performed pilot scale testing on a variety of medical devices to determine optimal de-gassing conditions. Aided in the design and construction of a pilot ethylene oxide. Was a member of the AAMI committee that developed reference test methods for ethylene oxide residues in medical services.

1980 to 1985

Position Senior Microbiologist/Associate Chemist - YWC, Inc.

Responsibilities

Responsible for performing non-routine microbiological analyses as well as providing technical guidance to technicians performing routine work. Instituted strict quality control procedures on all reagents, media and organisms. Was responsible for routine and non-routine chemical analyses on environmental samples. Was heavily involved in atomic spectroscopy analysis. Also performed evaluations on consumer products ranging from air cleaners to home water purification units.

1978 to 1980

Position Senior Chemist - Nutmeg Chemical Company

Responsibilities

Promoted to Assistant Director of Laboratory. Supervised staff in absence of Director. Served as liaison between director and staff. Performed non-routine water and oil analysis, quality control companies products as well as routine water, oil and deposit analysis. Also performed microbiological analysis of water samples.

1978 to 1979

Position Laboratory Technician

Responsibilities

Responsibilities included routine water and oil analyses and quality control of products.

SPECIALIZED TRAINING:

Basic Atomic Spectroscopy
Perkin Elmer
Norwalk, Connecticut 1979

ICP Spectroscopy
Spectra Inc.
Pompton Lakes, New Jersey 1988

Graphite Furnace Atomic Absorption Spectroscopy
Spectra Inc.
Pompton Lanes, New Jersey 1988

Interpretation of Low Resolution Mass Spectra
YWC
Whippany, New Jersey 1989

IEA
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PROFESSIONAL PROFILE
Daniel W. Helfrich

TITLE: Inorganics Manager

ACADEMIC ACCOMPLISHMENTS:

Quinnipiac College
Sacred Heart University
M.S. Chemistry
M.B.A.
B.A. Biology
B.S. Biology, 1985

MAJOR AREA OF EXPERTISE

Four years running ICP on environmental samples.
Two years running Furnace analysis.
Four years sample prep in environmental area.
Three years CLP Data Review.
OSHA trained and certified.
Familiar with EPA & NYSDEC protocols and SW846 Methods relating to inorganic metals analysis.

SUMMARY OF EXPERIENCE:

Mr. Helfrich has over 4 years experience in environmental analysis. He has functioned in numerous analytical roles including: Sample prep, Furnace analysis, ICP analysis and hazardous waste coordinator. Experienced in data review, and familiar with EPA and NYSDEC protocols. OSHA trained and experienced.

PROFESSIONAL EXPERIENCE:

1992 to Present

IEA, Inc. - Connecticut

Position Group Leader

Responsibilities

Manage daily flow of work, set priorities.
Monitor productivity of group.
CLP data review ensuring QA/QC protocols are followed.
Manage the collection and removal of all hazardous waste generated by IEA-CT.

1989 to 1992

Position Senior Chemist - IEA,Inc. CT

Responsibilities

ICP & Furnace Operator, manage flow of work, CLP data review ensuring QA/QC protocols are followed.

1987 to 1989

Position Lab Manager - PGP Industries

Responsibilities

ICP Operator and Health & Safety Manager

1984 to 1987

Position Senior Chemist - Handy & Harmon

Responsibilities

ICP Operator

SPECIALIZED TRAINING:

OSHA Seminar - 40 hour training + 28 hour update

Clean Harbours - Hazardous Waste Seminar

IEA
An Aquarion Company

PROFESSIONAL PROFILE
Kimberly A. Maturo

TITLE: GC/Semi-VOA Group Leader

ACADEMIC ACCOMPLISHMENTS:

Southern Connecticut State University - New Haven, Connecticut
B.S. Biology, 1985

SUMMARY OF EXPERIENCE:

Ms. Maturo has over 7 years experience in the environmental field. She started in the organic extractions department as a lab technician and worked her way up to supervisor. From there, she transferred to the Gas Chromatography Department in order to expand her knowledge by learning more about the analysis of environmental samples. She is now Group Leader of the GC Department and is experienced in Pesticide and PCB residue analysis.

PROFESSIONAL EXPERIENCE:

3/91 to Present

IEA, Inc. - Connecticut

Position GC Group Leader

Responsibilities

Supervisor of GC Group, responsible for analysis of environmental samples for pesticides/PCB's according to EPA/NYSDEC CLP Protocols, SW846 Methods and EPA "600" Series Methods. Additional responsibilities include analysis of samples via purge & trap/GC according to various protocols.

Other duties include analysis of air samples, charcoal absorbent tubes and other miscellaneous samples for any parameters requiring gas chromatography analysis. She is also responsible for supervision of the group including sample tracking, data review, etc.

10/88 to 3/91

Position GC- Senior Lab Technician

Responsibilities

Ms. Maturo's primary duties are the operation of the gas chromatographs for a variety of analyses. She has experience in pesticide/PCB determinations as well as other miscellaneous analytes such as alcohols, herbicides and solvents in general.

Ms. Maturo's other duties include computer data entry, sample tracking and monitoring QC samples for the group.

10/85 to 10/87

Position Extractions Group

Responsibilities

Over this time period Ms. Maturo was a member of the extractions group and supervised the operations and staff for the last year. Her duties were primarily extraction of environmental samples for semi-volatile organics, pesticides/PCB's and herbicides. She also was responsible for screening of organic extracts via gas chromatography.

IEA
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PROFESSIONAL PROFILE
John Mercure

TITLE: Systems Manager

ACADEMIC ACCOMPLISHMENTS:

Western Connecticut State University
Danbury, CT
Morse School of Business
Hartford, CT

MAJOR AREA OF EXPERTISE:

Computer packages, Assembler Programming, Database Management, BASIC Programming, Debugging, OS/JCL, QUEST Troubleshooting, Maintaining and upgrading PC's, "C" Programming, UNIX, etc.

SUMMARY OF EXPERIENCE

Mr. Mercure has written a Laboratory Invoicing System on the Concurrent 3210 using Fortran and Perkin Elmer Fortran 7 User Interface. A Mercury Program which generates Mercury Analysis Results from Peak Height numbers. Written on Concurrent 3210 using Fortran and Perkin Elmer.

PROFESSIONAL EXPERIENCE:

10/87 to present

IEA, Inc. - Connecticut

Position Systems Analyst II

Responsibilities

Mr. Mercure is now Senior Programmer Analyst (from 10/87 to 9/93 was Programmer/Analyst) his experience his experience includes Application Program Development and Maintenance. User support on minicomputer and microcomputer platforms. Hardware: Concurrent 3210, IBM PC and Compatibles. Operating Systems: OS/32 Concurrent 3210, DOS 3.3x - PC's. Concurrent Software: DMA/32, LIMS Database, Perkin Elmer Fortran 7 User Interface. PC Software: DMA/32, LIMS Database, Perkin Elmer, Fortran 7 User Interface. PC Software: WordPerfect 5.x, Lotus 2.x, Quattro Pro 4.0. Home Computer: Compaq DeskPro 286e, Compaz DOS 5.00, WordStar 7.0, Quattro Pro 2.0, PC Tools, Paradox 4.0, Turbo Pascal 6.0, Turbo C/CH 3.0.

IEA
An Aquarion Company

PROFESSIONAL PROFILE
Stephanie N. Plunkett

TITLE: Client Services Manager

ACADEMIC ACCOMPLISHMENTS:

BA - Biology, 1986
Hartwich College, Oneonta, NY
Four year recipient - Hartwick College Merit Scholarship

PROFESSIONAL EXPERIENCE:

1/93 to Present

IEA, Inc. - Connecticut

Position Client Services Manager

Responsibilities

Responsible for client service representatives/project managers functions. Aid in solving client problems and questions. Discuss technical issues and manage clients through sampling programs. Assist Account Executives on sales calls and project kick-off meetings.

7/91 to 9/91

IEA, Inc. - Monroe, CT

Position Inside Sales/Project Manager

Responsibilities

While continuing to perform project management services for an established list of laboratory clients, I also assumed marketing and sales responsibilities. Duties include surveying various trade journals, The Federal Register, etc. identifying regulatory trends and predicting future business opportunities. Follow-up includes determining which industries would most likely be impacted by pending legislation and the development of a marketing strategy. Strategies implemented include telemarketing campaigns, mass mailings, and a seminar series. Also responsible for surveying existing clients periodically to assess IEA's strengths and weaknesses. The results of these surveys are compiled, graphically displayed and distributed to all employees.

10/88 to 3/90 IEA, Inc., Monroe, CT

Client Services/Project Manager

Operational responsibilities in environmental investigations, including scheduling and workload projection, technical supervision, sample tracking, contract compliance, data review, and report preparation and interpretation. Interface with clients on project design, including sampling and analytical program requirements, responsible for coordinating project specific quality control/methodology compliance requirements.

1987/1988 Massachusetts General Hospital
Boston, MA

Technologist

Cardiac Unit/Department of Molecular Research. Utilized knowledge of genetics and biology in the screening of DNA libraries with variously prepared probes, plasmid construction, and single and double stranded Sangar dideoxy chain termination sequencing. Duties also include maintaining laboratory supplies and the training of new personnel.

Related Course Work

Chemistry, Organic Chemistry, Environmental History, Ecology

IEA
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PROFESSIONAL PROFILE
Diane Turro

TITLE: Metals Analyst IV
Section Leader

ACADEMIC ACCOMPLISHMENTS:

University of Hartford
1987 B.S. Chemistry

MAJOR AREA OF EXPERTISE:

Graphite Furnace Operation environmental samples
I-CAP Operation
Hg (Mercury) cold vapor prep and analysis
Smartlog operation (CLP Data Package Production)
Data Review

PROFESSIONAL EXPERIENCE:

May 2, 1988 to Present

IEA, Inc. - Connecticut

Position Metals Analyst IV

Position Assistant Group Leader

PROFESSIONAL AFFILIATIONS

American Chemical Society

APPENDIX, Section 2

IEA CHAIN-OF-CUSTODY FORM

IEA
An Aquarion Company

200 Monroe Turnpike
Monroe, CT 06468
203-261-4458

CHAIN OF CUSTODY RECORD

PAGE OF | NO.

EA JOB #:

LIENT:

ROJECT ID:

EA PROJECT MGR:

RUSH ☐ YES ☐ NO | DUE DATE

TITLE T #	CLIENT SAMPLE ID	DATE / TIME SAMPLED	MATRIX	LAB ID	QC Y / N	TESTS								GENERAL REMARKS
						BOTTLE TYPE AND PRESERVATIVE								
						FIELD FILTERED - CIRCLE Y or N								SAMPLE REMARKS
						Y / N	Y / N	Y / N	Y / N	Y / N	Y / N	Y / N	Y / N	

MATRIX CODES

- AIR
- AQUEOUS
- COMPLEX
- DRUM WASTE
- OIL
- S - SOIL
- SL - SLUDGE
- W - WIPE
- O - OTHER
- FB - FIELD BLANK
- TB - TRIP BLANK

BOTTLES PREPPED BY	DATE / TIME	BOTTLES REC'D BY	DATE / TIME
SIGNATURE		SIGNATURE	
SAMPLES COLLECTED BY	DATE / TIME	RECEIVED IN LAB BY	DATE / TIME
SIGNATURE		SIGNATURE	

REMARKS ON SAMPLE RECEIPT

☐ BOTTLES INTACT ☐ CUSTODY SEALS
☐ PRESERVED ☐ SEALS INTACT
☐ CHILLED ☐ SEE REMARKS

LABORATORY COPY

APPENDIX, Section 3

IEA NETWORK SAMPLE PRESERVATION AND HOLDING TIME REQUIREMENTS

Metals in Water					
Parameter[1]	Technique	Method	Holding Time	Container	Preservation
Aluminum	flame	202.1	6 Months	500 ml P,G	HNO3 to pH<2
	furnace	202.2	6 Months	500 ml P,G	HNO3 to pH<2
	ICP	200.7	6 Months	500 ml P,G	HNO3 to pH<2
	flame	7020	6 Months	500 ml P,G	HNO3 to pH<2
	ICP	6010	6 Months	500 ml P,G	HNO3 to pH<2
Antimony	flame	204.1	6 Months	500 ml P,G	HNO3 to pH<2
	furnace	204.2	6 Months	500 ml P,G	HNO3 to pH<2
	ICP	200.7	6 Months	500 ml P,G	HNO3 to pH<2
	flame	7040	6 Months	500 ml P,G	HNO3 to pH<2
	furnace	7041	6 Months	500 ml P,G	HNO3 to pH<2
	ICP	6010	6 Months	500 ml P,G	HNO3 to pH<2
Arsenic	furnace	206.2	6 Months	500 ml P,G	HNO3 to pH<2
	AA, hydride	206.3	6 Months	500 ml P,G	HNO3 to pH<2
	ICP	200.7	6 Months	500 ml P,G	HNO3 to pH<2
	AA, hydride	7061	6 Months	500 ml P,G	HNO3 to pH<2
	furnace	7060	6 Months	500 ml P,G	HNO3 to pH<2
	ICP	6010	6 Months	500 ml P,G	HNO3 to pH<2
Barium	flame	208.1	6 Months	500 ml P,G	HNO3 to pH<2
	furnace	208.2	6 Months	500 ml P,G	HNO3 to pH<2
	ICP	200.7	6 Months	500 ml P,G	HNO3 to pH<2
	flame	7080	6 Months	500 ml P,G	HNO3 to pH<2
	furnace	7081	6 Months	500 ml P,G	HNO3 to pH<2
	ICP	6010	6 Months	500 ml P,G	HNO3 to pH<2
Beryllium	flame	210.1	6 Months	500 ml P,G	HNO3 to pH<2
	furnace	210.2	6 Months	500 ml P,G	HNO3 to pH<2
	ICP	200.7	6 Months	500 ml P,G	HNO3 to pH<2
	flame	7090	6 Months	500 ml P,G	HNO3 to pH<2
	furnace	7091	6 Months	500 ml P,G	HNO3 to pH<2
	ICP	6010	6 Months	500 ml P,G	HNO3 to pH<2
Boron	colorimetric	212.3	6 Months	500 ml P,G	HNO3 to pH<2
	ICP	6010	6 Months	500 ml P,G	HNO3 to pH<2

Metals in Water-Continued...					
Parameter [5]	Technique	Method	Holding Time	Container	Preservation
Cadmium	flame	213.1	6 Months	500 ml P,G	HNO3 to pH<2
	furnace	213.2	6 Months	500 ml P,G	HNO3 to pH<2
	ICP	200.7	6 Months	500 ml P,G	HNO3 to pH<2
	flame	7130	6 Months	500 ml P,G	HNO3 to pH<2
	furnace	7131	6 Months	500 ml P,G	HNO3 to pH<2
	ICP	6010	6 Months	500 ml P,G	HNO3 to pH<2
Calcium	ICP	200.7	6 Months	500 ml P,G	HNO3 to pH<2
	flame	215.1	6 Months	500 ml P,G	HNO3 to pH<2
	titrimetric	215.2	6 Months	500 ml P,G	HNO3 to pH<2
	flame	7140	6 Months	500 ml P,G	HNO3 to pH<2
	ICP	6010	6 Months	500 ml P,G	HNO3 to pH<2
Chromium	flame	218.1	6 Months	500 ml P,G	HNO3 to pH<2
	furnace	218.2	6 Months	500 ml P,G	HNO3 to pH<2
	ICP	200.7	6 Months	500 ml P,G	HNO3 to pH<2
	flame	7190	6 Months	500 ml P,G	HNO3 to pH<2
	furnace	7191	6 Months	500 ml P,G	HNO3 to pH<2
	ICP	6010	6 Months	500 ml P,G	HNO3 to pH<2
Chromium Hexavalent	Coprecipitation	7195	24 Hours	500 ml P,G	Cool, 4 C.
	colorimetric	7196	24 Hours	500 ml P,G	Cool, 4 C.
	flame	7197	24 Hours	500 ml P,G	Cool, 4 C.
	DPP	7198	24 Hours	500 ml P,G	Cool, 4 C.
Cobalt	flame	219.1	6 Months	500 ml P,G	HNO3 to pH<2
	furnace	219.2	6 Months	500 ml P,G	HNO3 to pH<2
	ICP	200.7	6 Months	500 ml P,G	HNO3 to pH<2
	flame	7200	6 Months	500 ml P,G	HNO3 to pH<2
	furnace	7201	6 Months	500 ml P,G	HNO3 to pH<2
	ICP	6010	6 Months	500 ml P,G	HNO3 to pH<2

Metals in Water-Continued...					
Parameter [1]	Technique	Method	Holding Time	Container	Preservation
Copper	ICP	200.7	6 Months	500 ml P,G	HNO3 to pH<2
	flame	7210	6 Months	500 ml P,G	HNO3 to pH<2
	furnace	7211	6 Months	500 ml P,G	HNO3 to pH<2
	ICP	6010	6 Months	500 ml P,G	HNO3 to pH<2
Iron	flame	236.1	6 Months	500 ml P,G	HNO3 to pH<2
	furnace	236.2	6 Months	500 ml P,G	HNO3 to pH<2
	ICP	200.7	6 Months	500 ml P,G	HNO3 to pH<2
	flame	7380	6 Months	500 ml P,G	HNO3 to pH<2
	furnace	7381	6 Months	500 ml P,G	HNO3 to pH<2
	ICP	6010	6 Months	500 ml P,G	HNO3 to pH<2
Lead	flame	239.1	6 Months	500 ml P,G	HNO3 to pH<2
	furnace	239.2	6 Months	500 ml P,G	HNO3 to pH<2
	ICP	200.7	6 Months	500 ml P,G	HNO3 to pH<2
	flame	7420	6 Months	500 ml P,G	HNO3 to pH<2
	furnace	7421	6 Months	500 ml P,G	HNO3 to pH<2
	ICP	6010	6 Months	500 ml P,G	HNO3 to pH<2
Magnesium	flame	242.1	6 Months	500 ml P,G	HNO3 to pH<2
	furnace	239.2	6 Months	500 ml P,G	HNO3 to pH<2
	flame	7450	6 Months	500 ml P,G	HNO3 to pH<2
	furnace	7421	6 Months	500 ml P,G	HNO3 to pH<2
Manganese	flame	243.1	6 Months	500 ml P,G	HNO3 to pH<2
	furnace	243.2	6 Months	500 ml P,G	HNO3 to pH<2
	ICP	200.7	6 Months	500 ml P,G	HNO3 to pH<2
	flame	7460	6 Months	500 ml P,G	HNO3 to pH<2
	furnace	7461	6 Months	500 ml P,G	HNO3 to pH<2
	ICP	6010	6 Months	500 ml P,G	HNO3 to pH<2
Mercury	cold vapor-manual	245.1	28 Days	500 ml P,G	HNO3 to pH<2
	cold vapor-automated	245.2	28 Days	500 ml P,G	HNO3 to pH<2
	cold vapor-manual	7470	28 Days	500 ml P,G	HNO3 to pH<2

Metals in Water-Continued...					
Parameter [3]	Technique	Method	Holding Time	Container	Preservation
Molybdenum	flame	246.1	6 Months	500 ml P,G	HNO3 to pH<2
	furnace	246.2	6 Months	500 ml P,G	HNO3 to pH<2
	ICP	200.7	6 Months	500 ml P,G	HNO3 to pH<2
	flame	7480	6 Months	500 ml P,G	HNO3 to pH<2
	furnace	7481	6 Months	500 ml P,G	HNO3 to pH<2
	ICP	6010	6 Months	500 ml P,G	HNO3 to pH<2
Nickel	flame	249.1	6 Months	500 ml P,G	HNO3 to pH<2
	furnace	249.2	6 Months	500 ml P,G	HNO3 to pH<2
	ICP	200.7	6 Months	500 ml P,G	HNO3 to pH<2
	flame	7520	6 Months	500 ml P,G	HNO3 to pH<2
	furnace	7521	6 Months	500 ml P,G	HNO3 to pH<2
	ICP	6010	6 Months	500 ml P,G	HNO3 to pH<2
Potassium	flame	258.1	6 Months	500 ml P,G	HNO3 to pH<2
	ICP	200.7	6 Months	500 ml P,G	HNO3 to pH<2
	flame	7610	6 Months	500 ml P,G	HNO3 to pH<2
	ICP	6010	6 Months	500 ml P,G	HNO3 to pH<2
Selenium	ICP	200.7	6 Months	500 ml P,G	HNO3 to pH<2
	furnace	270.2	6 Months	500 ml P,G	HNO3 to pH<2
	AA, hydride	270.3	6 Months	500 ml P,G	HNO3 to pH<2
	ICP	6010	6 Months	500 ml P,G	HNO3 to pH<2
	furnace	7740	6 Months	500 ml P,G	HNO3 to pH<2
	AA, hydride	7741	6 Months	500 ml P,G	HNO3 to pH<2
Silica	ICP	200.7	6 Months	500 ml P,G	HNO3 to pH<2
Silver	flame	272.1	6 Months	500 ml P,G	HNO3 to pH<2
	furnace	272.2	6 Months	500 ml P,G	HNO3 to pH<2
	ICP	200.7	6 Months	500 ml P,G	HNO3 to pH<2
	flame	7760	6 Months	500 ml P,G	HNO3 to pH<2
	furnace	7761	6 Months	500 ml P,G	HNO3 to pH<2
	ICP	6010	6 Months	500 ml P,G	HNO3 to pH<2

Metals in Water-Continued...					
Parameter [3]	Technique	Method	Holding Time	Container	Preservation
Sodium	flame	273.1	6 Months	500 ml P,G	HNO3 to pH<2
	ICP	200.7	6 Months	500 ml P,G	HNO3 to pH<2
	flame	7770	6 Months	500 ml P,G	HNO3 to pH<2
	ICP	6010	6 Months	500 ml P,G	HNO3 to pH<2
Thallium	flame	279.1	6 Months	500 ml P,G	HNO3 to pH<2
	furnace	279.2	6 Months	500 ml P,G	HNO3 to pH<2
	ICP	200.7	6 Months	500 ml P,G	HNO3 to pH<2
	flame	7840	6 Months	500 ml P,G	HNO3 to pH<2
	furnace	7841	6 Months	500 ml P,G	HNO3 to pH<2
	ICP	6010	6 Months	500 ml P,G	HNO3 to pH<2
Tin	flame	282.1	6 Months	500 ml P,G	HNO3 to pH<2
	furnace	282.2	6 Months	500 ml P,G	HNO3 to pH<2
	flame	7870	6 Months	500 ml P,G	HNO3 to pH<2
Titanium	flame	283.1	6 Months	500 ml P,G	HNO3 to pH<2
	furnace	283.2	6 Months	500 ml P,G	HNO3 to pH<2
Vanadium	flame	286.1	6 Months	500 ml P,G	HNO3 to pH<2
	furnace	286.2	6 Months	500 ml P,G	HNO3 to pH<2
	ICP	200.7	6 Months	500 ml P,G	HNO3 to pH<2
	flame	7910	6 Months	500 ml P,G	HNO3 to pH<2
	furnace	7911	6 Months	500 ml P,G	HNO3 to pH<2
	ICP	6010	6 Months	500 ml P,G	HNO3 to pH<2
Zinc	flame	289.1	6 Months	500 ml P,G	HNO3 to pH<2
	furnace	289.2	6 Months	500 ml P,G	HNO3 to pH<2
	ICP	200.7	6 Months	500 ml P,G	HNO3 to pH<2
	flame	7950	6 Months	500 ml P,G	HNO3 to pH<2
	furnace	7951	6 Months	500 ml P,G	HNO3 to pH<2
	ICP	6010	6 Months	500 ml P,G	HNO3 to pH<2

Wet Chemistries in Water					
Parameter [3]	Technique	Method	Holding Time	Container	Preservation
Acidity	titrimetric	305.1	14 Days	100 ml P,G	Cool 4 C.
Alkalinity	titrimetric	310.1	14 Days	100 ml P,G	Cool 4 C.
Biochemical Oxygen Demand (BOD)	5 days, 20 C.	405.1	48 Hours	1000 ml P,G	Cool 4 C.
Bromide	titrimetric	320.1	28 Days	100 ml P,G	none required
Chemical Oxygen Demand (COD)	titrimetric, mid-level	410.1	28 Days	50 ml P,G	Cool 4 C, H2SO4 to pH <2
	titrimetric, low-level	410.2	28 Days	50 ml P,G	Cool 4 C, H2SO4 to pH <2
	titrimetric, high-level	410.3	28 Days	50 ml P,G	Cool 4 C, H2SO4 to pH <2
	automated-colorimetric	410.4	28 Days	50 ml P,G	Cool 4 C, H2SO4 to pH <2
Chloride	colorimetric	325.2	28 Days	50 ml P,G	none required
	colorimetric	9250	28 Days	50 ml P,G	none required
	titrimetric	9252	28 Days	50 ml P,G	none required
	colorimetric	9257	28 Days	50 ml P,G	none required
Cyanide	amenable to chlorine	335.1	14 Days [2]	500 ml P,G	Cool 4 C, NaOH to pH>12 Ascorbic Acid [1]
	spectrophotometric	335.2	14 Days [2]	500 ml P,G	Cool 4 C, NaOH to pH>12 Ascorbic Acid [1]
	Total, UV	335.3	14 Days [2]	500 ml P,G	Cool 4 C, NaOH to pH>12 Ascorbic Acid [1]
	colorimetric	9012	14 Days [2]	500 ml P,G	Cool 4 C, NaOH to pH>12 Ascorbic Acid [1]
Fluoride	distillation	340.1	28 Days	500 ml P,G	none required
	ion selective electrode	340.2	28 Days	500 ml P,G	none required
	colorimetric	340.3	28 Days	500 ml P,G	none required
Hardness, Total	colorimetric	130.1	6 Months	100 ml P,G	HNO3 to pH<2
	titrimetric	130.2	6 Months	100 ml P,G	HNO3 to pH<2
Iodide	titrimetric	345.1	24 Days	100 ml P,G	Cool 4 C.
Methylene Blue Active Substances	colorimetric	425.1	48 Hours	500 ml P,G	Cool 4 C.
Nitrogen Ammonia	colorimetric,phenate	350.1	28 Days	500 ml P,G	Cool 4 C, H2SO4 to pH <2
	distillation	350.2	28 Days	500 ml P,G	Cool 4 C, H2SO4 to pH <2
	ion selective electrode	350.3	28 Days	500 ml P,G	Cool 4 C, H2SO4 to pH <2

Wet Chemistries in Water-Continued...					
Parameter [3]	Technique	Method	Holding Time	Container	Preservation
Nitrogen-TKN	colorimetric,phenate	351.1	28 Days	500 ml P,G	Cool 4 C, H2SO4 to pH <2
	block digestor	351.2	28 Days	500 ml P,G	Cool 4 C, H2SO4 to pH <2
	colorimetric	351.3	28 Days	500 ml P,G	Cool 4 C, H2SO4 to pH <2
	ion selective electrode	351.4	28 Days	500 ml P,G	Cool 4 C, H2SO4 to pH <2
Nitrate	colorimetric, brucine	352.1	48 Hours	100 ml P,G	Cool 4 C.
	colorimetric, brucine	9200	48 Hours	100 ml P,G	Cool 4 C.
Nitrate-Nitrite	colorimetric, hydrazine	353.1	28 Days	100 ml P,G	Cool 4 C, H2SO4 to pH <2
	cadmium reduction, auto	353.2	28 Days	100 ml P,G	Cool 4 C, H2SO4 to pH <2
	cadmium reduction, manual	353.3	28 Days	100 ml P,G	Cool 4 C, H2SO4 to pH <2
Nitrite	spectrophotometric	354.1	48 Hours	100 ml P,G	Cool 4 C.
Oil & Grease, Total	gravimetric	413.1	28 Days	1000 ml G only	Cool 4 C, HCL or H2SO4 to pH <2
	IR	413.2	28 Days	1000 ml G only	Cool 4 C, HCL or H2SO4 to pH <2
	gravimetric	9070	28 Days	1000 ml G only	Cool 4 C, HCL or H2SO4 to pH <2
	gravimetric-sludge	9071	28 Days	1000 ml G only	Cool 4 C, HCL or H2SO4 to pH <2
Petroleum Hydrocarbons	IR	418.1	14 Days	1000 ml G only	Cool 4 C, HCL to pH <2
pH	electrode	150.1	in-field	50 ml P,G	not applicable
	electrode	9040	in-field	50 ml P,G	not applicable
	test paper	9041	in-field	50 ml P,G	not applicable
Phenolics, T-Recoverable	spectrophotometric	420.1	28 Days	500 ml G only	Cool 4 C, H2SO4 to pH <2
	colorimetric	420.2	28 Days	500 ml G only	Cool 4 C, H2SO4 to pH <2
	4AAP, Manual, Distillation	9065	28 Days	500 ml G only	Cool 4 C, H2SO4 to pH <2
	4AAP, Auto, Distillation	9066	28 Days	500 ml G only	Cool 4 C, H2SO4 to pH <2
	MBTH, Distillation	9067	28 Days	500 ml G only	Cool 4 C, H2SO4 to pH <2
Phosphorus, Ortho	colorimetric, auto	365.1	48 Hours	50 ml P,G	Filter immediately, Cool 4 C
	colorimetric, single	365.2	48 Hours	50 ml P,G	Filter immediately, Cool 4 C
	colorimetric-dual	365.3	48 Hours	50 ml P,G	Filter immediately, Cool 4 C
	total, auto, block digester	365.4	48 Hours	50 ml P,G	Filter immediately, Cool 4 C

Wet Chemistries in Water-Continued...					
Parameter [3]	Technique	Method	Holding Time	Container	Preservation
Phosphorus, Total	colorimetric, auto	365.1	28 Days	50 ml P,G	Cool 4 C, H2SO4 to pH <2
	colorimetric, single	365.2	48 Hours	50 ml P,G	Cool 4 C, H2SO4 to pH <2
	colorimetric-dual	365.3	48 Hours	50 ml P,G	Cool 4 C, H2SO4 to pH <2
	total, auto, block digester	365.4	48 Hours	50 ml P,G	Cool 4 C, H2SO4 to pH <2
Residue (Solids)	filterable (TDS)	160.1	7 Days	100 ml P,G	Cool 4 C.
	non-filterable (TSS)	160.2	7 Days	100 ml P,G	Cool 4 C.
	total (TS)	160.3	7 Days	100 ml P,G	Cool 4 C.
	volatile	160.4	7 Days	100 ml P,G	Cool 4 C.
	settleable	160.5	48 Hours	100 ml P,G	Cool 4 C.
Specific Conductance	meter	120.1	28 Days	100 ml P,G	Cool 4 C.
	meter	9050	28 Days	100 ml P,G	Cool 4 C.
Sulfate	ion chromatography	300.0	28 Days	50 ml P,G	Cool 4 C.
	colorimetric	375.1	28 Days	50 ml P,G	Cool 4 C.
	gravimetric	375.3	28 Days	50 ml P,G	Cool 4 C.
	turbidimetric	375.4	28 Days	50 ml P,G	Cool 4 C.
	colorimetric	9035	28 Days	50 ml P,G	Cool 4 C.
	colorimetric	9036	28 Days	50 ml P,G	Cool 4 C.
	turbidimetric	9038	28 Days	50 ml P,G	Cool 4 C.
Sulfide	titrimetric	376.1	7 Days	500 ml P,G	Cool 4 C, ZnAc/NaOH to pH>9
	colorimetric	376.2	7 Days	500 ml P,G	Cool 4 C, ZnAc/NaOH to pH>9
	colorimetric	9030	7 Days	500 ml P,G	Cool 4 C, ZnAc/NaOH to pH>9
Total Organic Carbon (TOC)	combustion or oxidation	415.1	28 Days	50 ml P,G	Cool 4 C, HCL or H2SO4 to pH <2
	combustion or oxidation	9060	28 Days	50 ml P,G	Cool 4 C, HCL or H2SO4 to pH <2
Total Organic Halides (TOX)	titrimetric	9020	28 Days	1000 ml G only [4] No Headspace	Cool 4 C, H2SO4 to pH <2 [5] Sodium Sulfite
Turbidity	nephelometric	180.1	48 Hours	100 ml P,G	Cool 4 C

Parameters by Gas Chromatography in Water					
Parameter [5]	Technique	Method	Holding Time	Container	Preservation
Halogenated Volatile Organics	gas chromatography	601	14 Days	3x40 ml vials	Cool 4 C.,Thiosulfate [6]
	gas chromatography	8010	14 Days	3x40 ml vials	Cool 4 C.,Thiosulfate [6]
Non-Halogenated Volatile Organics	gas chromatography	8015	14 Days	3x40 ml vials	Cool 4 C., HCL to pH<2, Thiosulfate [6]
Purgeable Aromatics	gas chromatography	602	7/14 Days [7]	3x40 ml vials	Cool 4 C., HCL to pH<2, Thiosulfate [6]
	gas chromatography	8020	7/14 Days [7]	3x40 ml vials	Cool 4 C., HCL to pH<2, Thiosulfate [6]
Acrolein & Acrylonitrile	gas chromatography	603	14 Days	3x40 ml vials	Cool 4 C., HCL to pH 5, Thiosulfate [6]
	gas chromatography	8030	14 Days	3x40 ml vials	Cool 4 C., HCL to pH 5, Thiosulfate [6]
Phenols	gas chromatography	604	ext.-7 Days anal.-40 Days	1 L, Amber G	Cool 4 C.,Thiosulfate [6]
	gas chromatography	8040	ext.-7 Days anal.-40 Days	1 L, Amber G	Cool 4 C.,Thiosulfate [6]
Phthalate Esters	gas chromatography	606	ext.-7 Days anal.-40 Days	1 L, Amber G	Cool 4 C.,Thiosulfate [6]
	gas chromatography	8060	ext.-7 Days anal.-40 Days	1 L, Amber G	Cool 4 C.,Thiosulfate [6]
Nitrosamines	gas chromatography	607	ext.-7 Days anal.-40 Days	1 L, Amber G	Cool 4 C.,Thiosulfate [6]
Organochlorine Pesticides and PCB's	gas chromatography	608	ext.-7 Days anal.-40 Days	1 L, Amber G	Cool 4 C.,Thiosulfate [6]
	gas chromatography	8080	ext.-7 Days anal.-40 Days	1 L, Amber G	Cool 4 C.,Thiosulfate [6]
Polynuclear Aromatic Hydrocarbons (PNA's)	gas chromatography/LC	610	ext.-7 Days anal.-40 Days	1 L, Amber G	Cool 4 C.,Thiosulfate [6]
	gas chromatography	8100	ext.-7 Days anal.-40 Days	1 L, Amber G	Cool 4 C.,Thiosulfate [6]
	HPLC	8310	ext.-7 Days anal.-40 Days	1 L, Amber G	Cool 4 C.,Thiosulfate [6]

Parameters by Gas Chromatography in Water-Continued...					
Parameter [5]	Technique	Method	Holding Time	Container	Preservation
Haloethers	gas chromatography	611	ext.-7 Days anal.-40 Days	1 L, Amber G	Cool 4 C.,Thiosulfate [6]
Chlorinated Hydrocarbons	gas chromatography	612	ext.-7 Days anal.-40 Days	1 L, Amber G	Cool 4 C.,Thiosulfate [6]
	gas chromatography	8120	ext.-7 Days anal.-40 Days	1 L, Amber G	Cool 4 C.,Thiosulfate [6]
Organophosphorus Pesticides	gas chromatography	8140	ext.-7 Days anal.-40 Days	1 L, Amber G	Cool 4 C.,Thiosulfate [6]
Chlorinated Herbicides	gas chromatography	8150	ext.-7 Days anal.-40 Days	1 L, Amber G	Cool 4 C.,Thiosulfate [6]

Parameters by GC/MS in Water					
Parameter [5]	Technique	Method	Holding Time	Container	Preservation
Purgeables	GC/MS-624 list	624	7/14 Days [7]	3x40 ml vials	Cool 4 C., HCL to pH<2, Thiosulfate [6]
	Priority Pollutant list	624	7/14 Days [7]	3x40 ml vials	Cool 4 C., HCL to pH<2, Thiosulfate [6]
	Hazardous Substance list	624	7/14 Days [7]	3x40 ml vials	Cool 4 C., HCL to pH<2, Thiosulfate [6]
	Target Compound list (TCL)	624	7/14 Days [7]	3x40 ml vials	Cool 4 C., HCL to pH<2, Thiosulfate [6]
	Appendix IX list	624	7/14 Days [7]	3x40 ml vials	Cool 4 C., HCL to pH<2, Thiosulfate [6]
	Priority Pollutant list	8240	7/14 Days [7]	3x40 ml vials	Cool 4 C., HCL to pH<2, Thiosulfate [6]
	Hazardous Substance list	8240	7/14 Days [7]	3x40 ml vials	Cool 4 C., HCL to pH<2, Thiosulfate [6]
	Target Compound list (TCL)	8240	7/14 Days [7]	3x40 ml vials	Cool 4 C., HCL to pH<2, Thiosulfate [6]
	Appendix IX list	8240	7/14 Days [7]	3x40 ml vials	Cool 4 C., HCL to pH<2, Thiosulfate [6]

Parameters by GC/MS in Water					
Parameter [3]	Technique	Method	Holding Time	Container	Preservation
Base-Neutral & Acid Extractables	625 list	625	ext.-7 Days anal.-40 Days	1 L, Amber G	Cool 4 C.,Thiosulfate [6]
	Priority Pollutant list	625	ext.-7 Days anal.-40 Days	1 L, Amber G	Cool 4 C.,Thiosulfate [6]
	Hazardous Substance list	625	ext.-7 Days anal.-40 Days	1 L, Amber G	Cool 4 C.,Thiosulfate [6]
	Target Compound list (TCL)	625	ext.-7 Days anal.-40 Days	1 L, Amber G	Cool 4 C.,Thiosulfate [6]
	Appendix IX list	625	ext.-7 Days anal.-40 Days	1 L, Amber G	Cool 4 C.,Thiosulfate [6]
	Priority Pollutant list	8250	ext.-7 Days anal.-40 Days	1 L, Amber G	Cool 4 C.,Thiosulfate [6]
	Hazardous Substance list	8250	ext.-7 Days anal.-40 Days	1 L, Amber G	Cool 4 C.,Thiosulfate [6]
	Target Compound list (TCL)	8250	ext.-7 Days anal.-40 Days	1 L, Amber G	Cool 4 C.,Thiosulfate [6]
	Appendix IX list	8250	ext.-7 Days anal.-40 Days	1 L, Amber G	Cool 4 C.,Thiosulfate [6]
	Priority Pollutant list	8270	ext.-7 Days anal.-40 Days	1 L, Amber G	Cool 4 C.,Thiosulfate [6]
	Hazardous Substance list	8270	ext.-7 Days anal.-40 Days	1 L, Amber G	Cool 4 C.,Thiosulfate [6]
	Target Compound list (TCL)	8270	ext.-7 Days anal.-40 Days	1 L, Amber G	Cool 4 C.,Thiosulfate [6]
	Appendix IX list	8270	ext.-7 Days anal.-40 Days	1 L, Amber G	Cool 4 C.,Thiosulfate [6]

Metals in Soil					
Parameter [3]	Technique	Method	Holding Time	Container	Preservation
Aluminum	flame	7020	6 Months	100 g P,G	Cool 4 C.
	ICP	6010	6 Months	100 g P,G	Cool 4 C.
Antimony	flame	7040	6 Months	100 g P,G	Cool 4 C.
	furnace	7041	6 Months	100 g P,G	Cool 4 C.
	ICP	6010	6 Months	100 g P,G	Cool 4 C.
Arsenic	ICP	6010	6 Months	100 g P,G	Cool 4 C.
	furnace	7060	6 Months	100 g P,G	Cool 4 C.
	AA, hydride	7061	6 Months	100 g P,G	Cool 4 C.
Barium	flame	7080	6 Months	100 g P,G	Cool 4 C.
	furnace	7081	6 Months	100 g P,G	Cool 4 C.
	ICP	6010	6 Months	100 g P,G	Cool 4 C.
Beryllium	flame	7090	6 Months	100 g P,G	Cool 4 C.
	furnace	7091	6 Months	100 g P,G	Cool 4 C.
	ICP	6010	6 Months	100 g P,G	Cool 4 C.
Boron	ICP	6010	6 Months	100 g P,G	Cool 4 C.
Cadmium	flame	7130	6 Months	100 g P,G	Cool 4 C.
	furnace	7131	6 Months	100 g P,G	Cool 4 C.
	ICP	6010	6 Months	100 g P,G	Cool 4 C.
Calcium	flame	7140	6 Months	100 g P,G	Cool 4 C.
	ICP	6010	6 Months	100 g P,G	Cool 4 C.
Chromium	flame	7190	6 Months	100 g P,G	Cool 4 C.
	furnace	7191	6 Months	100 g P,G	Cool 4 C.
	ICP	6010	6 Months	100 g P,G	Cool 4 C.

Metals in Soil-Continued...					
Parameter [3]	Technique	Method	Holding Time	Container	Preservation
Cobalt	flame	7200	6 Months	100 g P,G	Cool 4 C.
	furnace	7201	6 Months	100 g P,G	Cool 4 C.
	ICP	6010	6 Months	100 g P,G	Cool 4 C.
Copper	flame	7210	6 Months	100 g P,G	Cool 4 C.
	furnace	7211	6 Months	100 g P,G	Cool 4 C.
	ICP	6010	6 Months	100 g P,G	Cool 4 C.
Iron	flame	7380	6 Months	100 g P,G	Cool 4 C.
	furnace	7381	6 Months	100 g P,G	Cool 4 C.
	ICP	6010	6 Months	100 g P,G	Cool 4 C.
Lead	flame	7420	6 Months	100 g P,G	Cool 4 C.
	furnace	7421	6 Months	100 g P,G	Cool 4 C.
	ICP	6010	6 Months	100 g P,G	Cool 4 C.
Magnesium	flame	7450	6 Months	100 g P,G	Cool 4 C.
	ICP	6010	6 Months	100 g P,G	Cool 4 C.
Manganese	flame	7460	6 Months	100 g P,G	Cool 4 C.
	furnace	7461	6 Months	100 g P,G	Cool 4 C.
	ICP	6010	6 Months	100 g P,G	Cool 4 C.
Mercury	cold vapor-manual	7470	28 Days	100 g P,G	Cool 4 C.
	cold vapor-manual	7471	28 Days	100 g P,G	Cool 4 C.
Molybdenum	flame	7480	6 Months	100 g P,G	Cool 4 C.
	furnace	7481	6 Months	100 g P,G	Cool 4 C.
	ICP	6010	6 Months	100 g P,G	Cool 4 C.
Nickel	flame	7520	6 Months	100 g P,G	Cool 4 C.
	furnace	7521	6 Months	100 g P,G	Cool 4 C.
	ICP	6010	6 Months	100 g P,G	Cool 4 C.
Potassium	flame	7610	6 Months	100 g P,G	Cool 4 C.
	ICP	6010	6 Months	100 g P,G	Cool 4 C.
Selenium	ICP	6010	6 Months	100 g P,G	Cool 4 C.
	furnace	7740	6 Months	100 g P,G	Cool 4 C.
	AA, hydride	7741	6 Months	100 g P,G	Cool 4 C.

Metals in Soil-Continued...					
Parameter [3]	Technique	Method	Holding Time	Container	Preservation
Silver	flame	7760	6 Months	100 g P,G	Cool 4 C.
	furnace	7761	6 Months	100 g P,G	Cool 4 C.
	ICP	6010	6 Months	100 g P,G	Cool 4 C.
Sodium	flame	7770	6 Months	100 g P,G	Cool 4 C.
	ICP	6010	6 Months	100 g P,G	Cool 4 C.
Thallium	flame	7840	6 Months	100 g P,G	Cool 4 C.
	furnace	7841	6 Months	100 g P,G	Cool 4 C.
	ICP	6010	6 Months	100 g P,G	Cool 4 C.
Tin	flame	7870	6 Months	100 g P,G	Cool 4 C.
Vanadium	flame	7910	6 Months	100 g P,G	Cool 4 C.
	furnace	7911	6 Months	100 g P,G	Cool 4 C.
	ICP	6010	6 Months	100 g P,G	Cool 4 C.
Zinc	flame	7950	6 Months	100 g P,G	Cool 4 C.
	furnace	7951	6 Months	100 g P,G	Cool 4 C.
	ICP	6010	6 Months	100 g P,G	Cool 4 C.

Wet Chemistries in Soil					
Parameter [8]	Technique	Method	Holding Time	Container	Preservation
Cyanide	spectrophotometric	9010	14 Days	100 g P,G	Cool 4 C.
	colorimetric	9012	14 Days	100 g P,G	Cool 4 C.
Sulfate	colorimetric	9035	28 Days	100 g P,G	Cool 4 C.
	colorimetric	9036	28 Days	100 g P,G	Cool 4 C.
	turbidimetric	9038	28 Days	100 g P,G	Cool 4 C.
Sulfide	colorimetric	9030		100 g P,G	Cool 4 C

Parameters by Gas Chromatography in Soil					
Parameter [3]	Technique	Method	Holding Time	Container	Preservation
Halogenated Volatile Organics	gas chromatography	8010	14 Days	3x40 ml vials[4]	Cool 4 C.
Non-Halogenated Volatile Organics	gas chromatography	8015	14 Days	3x40 ml vials[4]	Cool 4 C.
Purgeable Aromatics	gas chromatography	8020	14 Days	3x40 ml vials[4]	Cool 4 C.
Acrolein & Acrylonitrile	gas chromatography	8030	14 Days	3x40 ml vials[4]	Cool 4 C.
Phenols	gas chromatography	8040	ext.-14 Days anal.-40 Days	100 g ,G	Cool 4 C.
Phthalate Esters	gas chromatography	8060	ext.-14 Days anal.-40 Days	100 g ,G	Cool 4 C.
Nitrosamines	gas chromatography	8070	ext.-14 Days anal.-40 Days	100 g ,G	Cool 4 C.
Organochlorine Pesticides and PCB's	gas chromatography	8080	ext.-14 Days anal.-40 Days	100 g ,G	Cool 4 C.
Polynuclear Aromatic Hydrocarbons (PNA's)	gas chromatography	8100	ext.-14 Days anal.-40 Days	100 g ,G	Cool 4 C.
	HPLC	8310	ext.-14 Days anal.-40 Days	100 g ,G	Cool 4 C.
Chlorinated Hydrocarbons	gas chromatography	8120	ext.-14 Days anal.-40 Days	100 g ,G	Cool 4 C.
Organophosphorus Pesticides	gas chromatography	8140	ext.-14 Days anal.-40 Days	100 g ,G	Cool 4 C.
Chlorinated Herbicides	gas chromatography	8150	ext.-14 Days anal.-40 Days	100 g ,G	Cool 4 C.

Parameters by GC/MS in Soil					
Parameter [3]	Technique	Method	Holding Time	Container	Preservation
Volatile organics	packed column	8240	14 Days	3x40 ml vials[8]	Cool 4 C.
	capillary column	8260	14 Days	3x40 ml vials[8]	Cool 4 C.
Base-Neutral & Acid Extractables	semi-vol packed	8250	ext.-14 Days anal.-40 Days	100 g ,G	Cool 4 C.
	semi-vol capillary	8270	ext.-14 Days anal.-40 Days	100 g ,G	Cool 4 C.

Footnotes

[1] If residual chlorine is present in the sample, 0.6 g of ascorbic acid is utilized. Ascorbic acid is only used if residual chlorine is present.

[2] Maximum holding time is 24 hours when sulfide is present. Optionally, all samples may be tested with lead acetate paper before pH adjustments in order to determine if sulfide is present. If sulfide is present, it can be removed by the addition of cadmium nitrate powder until a negative spot test is obtained. The sample is filtered and then NaOH is added to pH 12.

[3] The following information is based upon EPA requirements as outlined in Table II, Part 136, Title 40 of the Code of Federal Regulations, July 1991. This reference should be consulted if further clarification is desired. Various state agencies have differing requirements for both holding times and preservation from those listed above. In such cases, the local requirements supercede the EPA information.

[4] All samples should be collected in bottles with teflon septa and be protected from light. If this is not possible, use 250 ml bottles fitted with teflon lined caps. Samples should contain no headspace.

[5] If samples contain residual chlorine, it must be removed in the field by adding sulfite to the sample bottle (5 mg sodium sulfite crystals per liter of sample).

[6] If samples contain residual chlorine, 0.008% sodium thiosulfate must be added at the time of sampling and should only be used if residual chlorine is present.

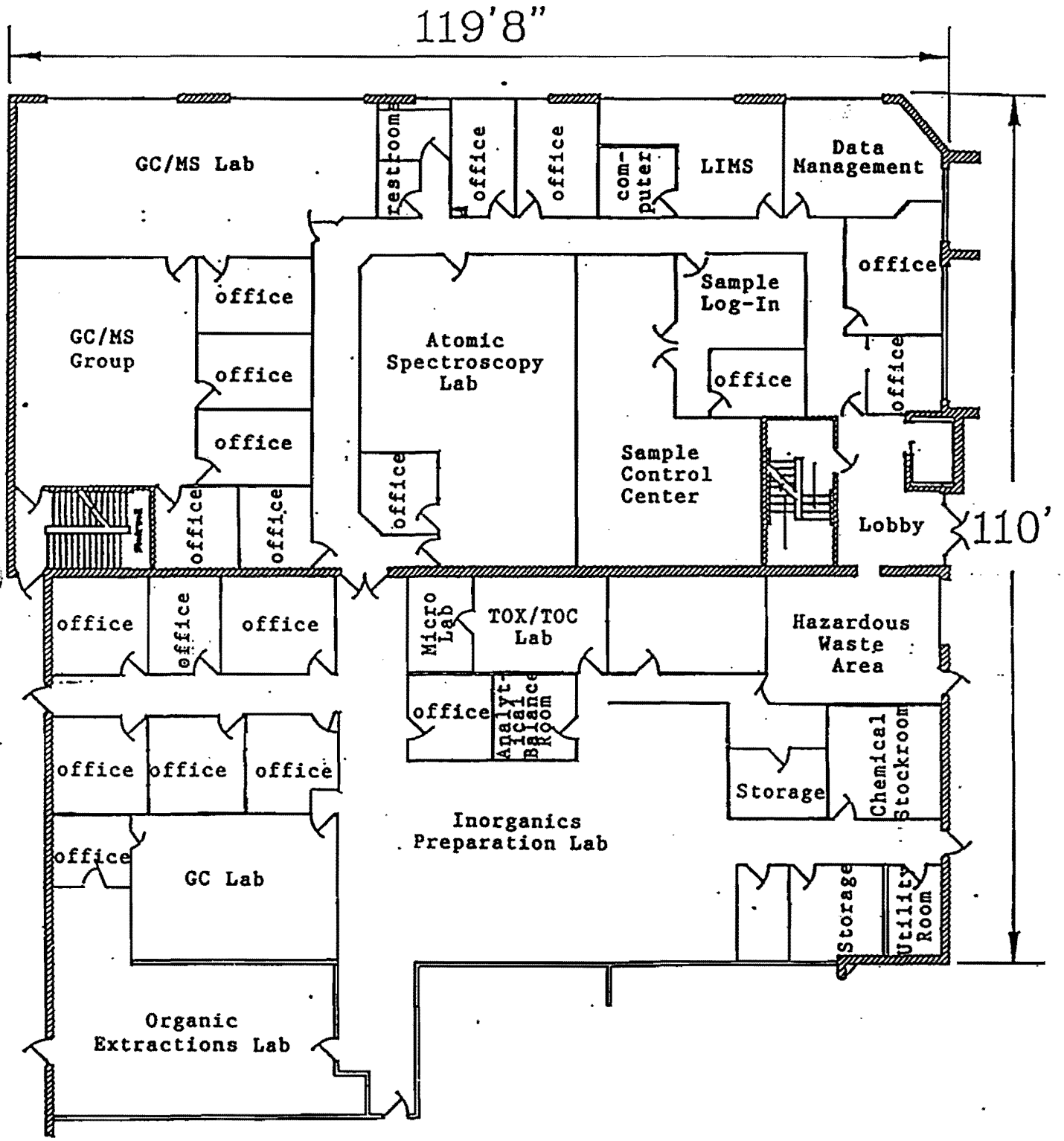
[7] If samples do not receive pH adjustment, the holding time is 7 days. With pH adjustment, the holding time is 14 days.

[8] Alternatively, wide mouth glass jars designed for volatile samples may be utilized with teflon lined caps.

APPENDIX, Section 4

LABORATORY FLOOR PLAN

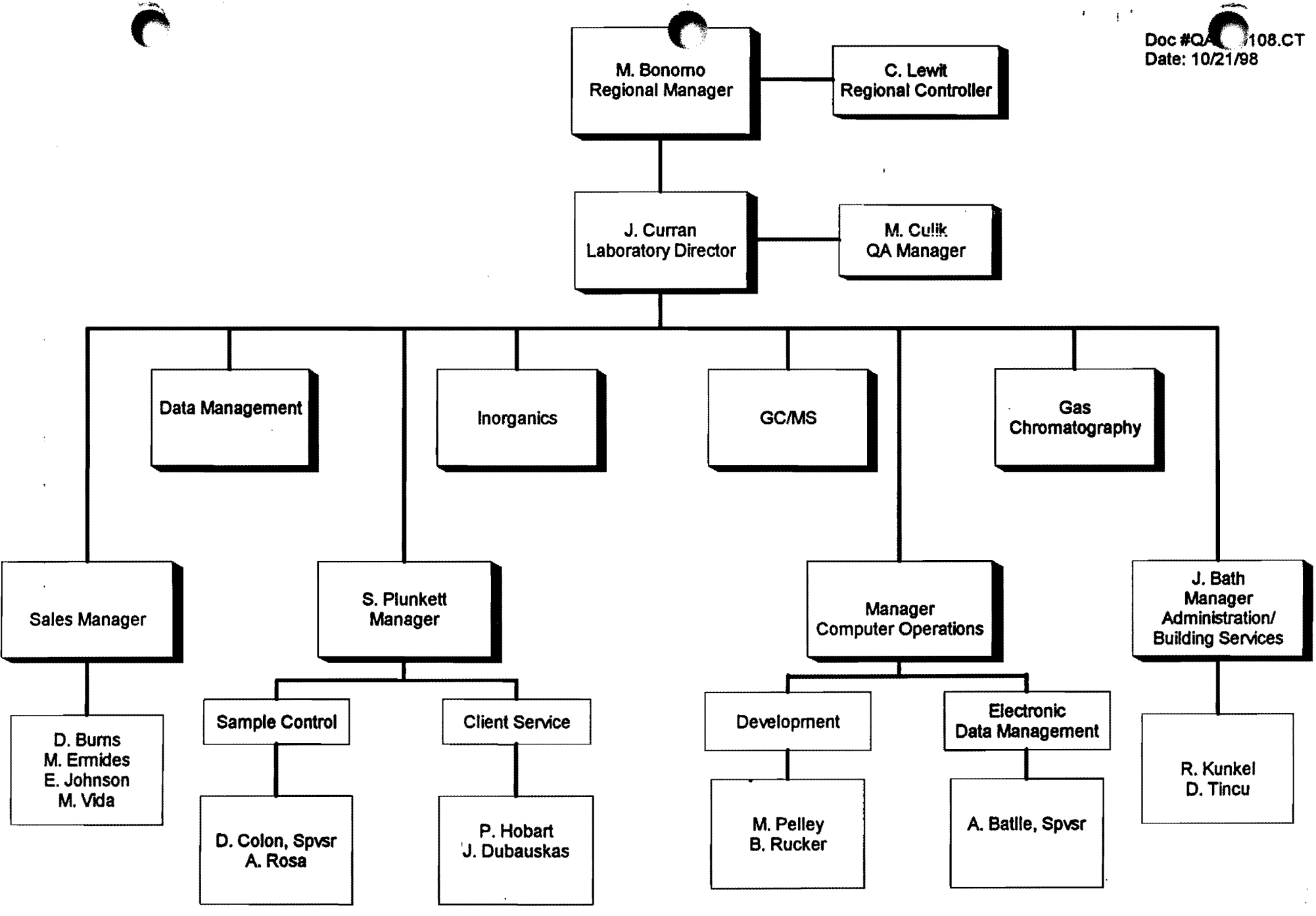
Fig. 3.0



IEA, INC.-CONNECTICUT
200 MONROE TURNPIKE MONROE, CONNECTICUT 06468
FLOOR PLAN 5/91

APPENDIX, Section 5

ORGANIZATIONAL CHART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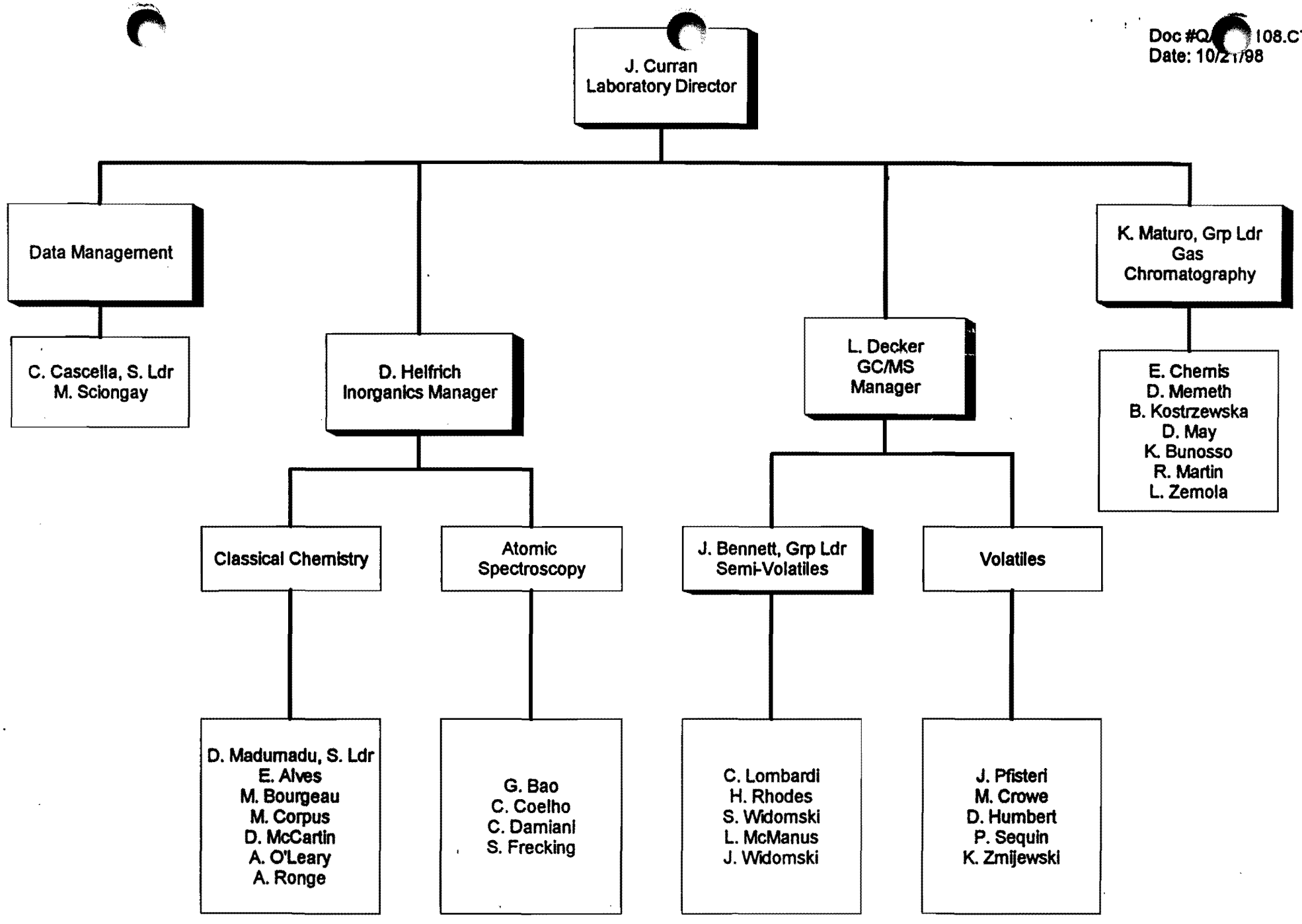
C. Lewit
Regional Controller
J. Curran
Laboratory Director
M. Culik
QA Manager
Data Management
Inorganics
GC/MS
Gas
Chromatography
Sales Manager
S. Plunkett
Manager
Manager
Computer Operations
J. Bath
Manager
Administration/
Building Services
D. Burns
M. Ermides
E. Johnson
M. Vida
Sample Control
Client Service
Development
Electronic
Data Management
R. Kunkel
D. Tincu
D. Colon, Spvsr
A. Rosa
P. Hobart
J. Dubauskas
M. Pelley
B. Rucker
A. Batlle, Spvsr


J. Curran
Laboratory Director
Data Management
C. Cascella, S. Ldr
M. Sciongay
D. Helfrich
Inorganics Manager
Classical Chemistry
D. Madurnadu, S. Ldr
E. Alves
M. Bourgeau
M. Corpus
D. McCartin
A. O'Leary
A. Ronge
Atomic
Spectroscopy
G. Bao
C. Coelho
C. Damiani
S. Frecking
L. Decker
GC/MS
Manager
J. Bennett, Grp Ldr
Semi-Volatiles
C. Lombardi
H. Rhodes
S. Widomski
L. McManus
J. Widomski
Volatiles
J. Pfisteri
M. Crowe
D. Humbert
P. Sequin
K. Zmijewski
K. Maturo, Grp Ldr
Gas
Chromatography
E. Chernis
D. Memeth
B. Kostrzewska
D. May
K. Bunosso
R. Martin
L. Zemola

APPENDIX, Section 6

CORRECTIVE ACTION FORM

IEA
An Aquarion Company

CORRECTIVE ACTION FORM

A. Originator Information

Client Inquiry_____

Client:_____ Job/Case:_____

Date/time:_____ Sample Number(s):_____

Client/Lab Contact: _____ Date/Time Response Due:_____

Detailed Description of Potential Problem:_____

B. Quality Assurance Information

Corrective Action ID# _____

Recommended Corrective Action:_____

Groups Involved:

__Sample Control __ Wet Chemistry __ Metals

__Gas Chromatography __ Mass Spectrometry __ Report Generation

__Client Service __ Sample Preparation

C. Final Resolution

Describe What Happened and Long Term Corrective Action Taken:_____

Supervisor Signature: _____ Date_____ Date/Time Client Notified:_____

D. Quality Assurance Final Approval (QA Manager use only)

Corrective Action Approved: _____

Date Finalized: _____

Was a problem identified? Yes / No

APPENDIX, Section 7

EXAMPLE LISTING OF LABORATORY

STANDARD OPERATING PROCEDURES (SOPs)

SAMPLE CONTROL

Standard Operating Procedure	Code	Date Generated
SOP for Bottle Order Preparation	SMS00100.CT	02/15/95
SOP for Sample Processing and Sample Arrival	SMS00402.CT	05/15/92
SOP for Log-in of CLP Samples	SMS00502.CT	05/15/92
SOP for Storing Water and Soil Samples	SMS00602.CT	05/12/92
SOP for Generating Labels/Labeling Containers	SMS00700.CT	05/15/92
SOP for Documenting Sample Removal from Laboratory	SMS00802.CT	05/15/92
SOP for Securing the Laboratory and Samples	SMS00903.CT	05/15/92
SOP for Temperature Control Requirements	SMS01001.CT	05/15/92
SOP for Compositing Samples	SMS01100.CT	06/16/94
SOP for Sample Receipt (NJDEPE)	SMS01200.CT	01/24/95
SOP for Operating and Maintaining Fume Hoods	SFS00202.CT	05/15/92
SOP for Hazardous Waste Disposal	SFS00100.CT	05/06/92
SOP for Emergency Procedures	SFS00300.CT	06/21/94
SOP for Hazardous Waste Minimization Plan	SFS00500.CT	07/25/94
SOP for Tracking and Collection of Mixed Waste	RAS00100.CT	02/06/94
SOP for Radioactivity Swpie Tests	RAS00200.CT	08/17/94
SOP for Radiation Screening	RAS00300.CT	08/15/94
SOP for Management/Disposal of Mixed Waste	RAS00400.CT	08/24/94

DATA MANAGEMENT/HANDLING

Standard Operating Procedure	Code	Date Generated
SOP for Preparation/ Review of Laboratory Reports	RPS00300.CT	02/16/94
SOP for Documentation Policy/Procedures	DM:090191:2	09/01/91
SOP for Data Reduction, Mgt, and Handling - CLP	RPS00200.CT	05/05/92
SOP for Sample Tracking	QAS00200.CT	01/13/92
SOP for Data validation/Self Inspection - CLP	QAS00100.CT	05/06/92
SOP for Data Validation/Self Inspection - OLM02.1	QAS00600.CT	01/17/94
SOP for Data Validation	QAS00700.CT	dft

EXTRACTIONS

Standard Operating Procedure	Code	Date Generated
SOP for CLP Aqueous BNA Preparation	SPS00303.CT	08/20/91
SOP for CLP Aqueous Pesticide/PCB Preparation	SPS00403.CT	08/19/91
SOP for CLP Soil BNA Preparation	SPS00102.CT	08/23/91
SOP for CLP Soil Pesticide/PCB Preparation	SPS00202.CT	08/26/91
SOP for CLP Extractions Standard Prep	SPS00702.CT	05/07/92
SOP for CLP BNA extract Screening	SPS00803.CT	05/12/92
SOP for CLP GPC BNA Extracts	SPS00502.CT	08/29/91
SOP for CLP GPC Pesticide/PCB Extracts	SPS00602.CT	04/02/92
SOP for Cleaning Glassware	SPS00901.CT	05/13/92
SOP for Hydrocarbon Sample Prep	SPS01000.CT	dft
SOP for Aqueous Herbicides Method 509B	SPS01100.CT	dft
SOP for Preparation of Chlorinated Herbicides (W) - 8150	SPS02800.CT	09/06/94
SOP for Aqueous BNA Methods 3510/3520	SPS01300.CT	09/10/93
SOP for Aqueous Pest/PCB Methods 3510/3520	SPS01200.CT	09/15/93
SOP for Soil BNA Method 3550	SPS01400.CT	12/10/93
SOP for Soil Pest/PCB Method 3550	SPS01600.CT	01/21/94
SOP for Aqueous OP Pesticides Methods 3510/3520	SPS01700.CT	06/15/94
SOP for SW846 GPC of BNA extracts	SPS01801.CT	12/17/94
SOP for GPC of Pesticide/PCB extracts method 3640	SPS01900.CT	03/04/94
SOP for Soil OP Pesticides Method 3550	SPS02700.CT	03/07/94
SOP for Waste dilution - BNA	SPS03000.CT	03/08/94
SOP for Waste dilution - Pesticides/PCB	SPS03100.CT	03/04/94
SOP for Pesticide/PCB extraction method 608	SPS03201.CT	08/24/94
SOPs for extractions CLP OLM02.1	SPS02000.CT-SPS02600.CT	dft
SOP for Extraction Standard Prep	SPS01500.CT	dft

EXTRACTIONS - cont.

Standard Operating Procedure	Code	Date Generated
SOP for Prep Soil/Sediment samples for CLP P/P OLM03.1	SPS03300.CT	11/11/94
SOP for GPC of Pesticide extracts OLM03.1	SPS03400.CT	11/11/94
SOP for Prep Soil/Sed samples for CLP BNA's OLM03.1	SPS03500.CT	11/11/94
SOP for GPC of Semivolatile extracts OLM03.1	SPS03600.CT	11/11/94
SOP for Prep of Aqueous samples for CLP BNA's OLM03.1	SPS03700.CT	11/11/94
SOP for Prep of Aqueous samples for CLP P/P OLM03.1	SPS03800.CT	11/11/94
SOP for Prep of Low Level PCB Analysis 3510A	SPS03900.CT	11/29/94
SOP for Alumina Column C/U Method 3611A	SPS02901.CT	03/23/95
SOP for Prep Aqueous SV OLC10/92	SPS04000.CT	Dft
SOP for Prep of Semivolatiles in Tissue samples	SPS04200.CT	10/21/95
SOP for Prep of Pesticides/PCBs in Tissue samples	SPS04300.CT	10/21/95
SOP for Prep of Chlorinated Herbicides -Method 8150 (S)	SPS04400.CT	Dft
SOP for Prep of PUF Samples for Pesticides/PCB T04	SPS04500.CT	07/15/96
SOP for Prep of PUF Samples for Semi-volatiles T013	SPS04600.CT	07/15/96
Sop for Prep of SV Method 625 (Water)	SPS04700.CT	4/23/96
SOP for Prep of Wipe Samples Pesticides/PCBs	SPS04800.CT	Dft
SOP for Florisil Cartridge clean-up P/P extracts	SPS04900.CT	11/8/96

GC/MS

Standard Operating Procedures	Code	Date Generated
SOP for CLP Volatiles (GC/MS)	MSS00601.CT	09/04/91
SOP for Semi-volatile CLP OLM01.8	MSS01001.CT	09/10/91
SOP for Volatile Std Prep CLP	MSS00100.CT	05/05/92
SOP for Semi-volatile Std Prep CLP	MSS00200.CT	05/05/92
SOP for Cleaning AS vials	MSS01200.CT	02/15/93
SOP for Analysis of BNA Method 8270A	MSS00700.CT	05/23/94
SOP for Analysis of Volatiles Method 8240A	MSS00400.CT	04/30/93
SOP for Volatile Standard Prep	MSV:120588:1	12/05/88
SOP for BNA standard Prep	MSSV:112686:2	11/26/86
SOP for GC/MS Semi-volatiles CLP OLM02.1	MSS00800.CT	01/14/94
SOP for GC/MS Volatiles CLP OLM02.1	MSS00900.CT	01/14/94
SOP for Volatile Std Prep CLP OLM02.1	MSS01300.CT	01/14/94
SOP for Semi-volatile Std Prep CLP OLM02.1	MSS01400.CT	01/14/94
SOP for GC/MS Volatiles in Air	MSS00300.CT	dft
SOP for GC/MS Volatile in Air - Summa Canister	MSS01100.CT	dft
SOP for GC/MS Volatile 524.2 Rev. 3	MSS01500.CT	dft
SOP for GC/MS Semivolatiles OLM03.1	MSS01600.CT	11/12/94
SOP for GC/MS Semivolatile Standard Prep OLM03.1	MSS01700.CT	11/12/94
SOP for GC/MS Volatiles OLM03.1	MSS01800.CT	11/12/94
SOP for GC/MS Volatile Standard Prep OLM03.1	MSS01900.CT	11/12/94
SOP for GC/MS Analysis Method 625	MSS02002.CT	07/13/94
SOP for GC/MS Analysis Method 624	MSS02100.CT	02/27/95
SOP for GC/MS Semivolatile OLC10/92	MSS02200.CT	Dft
SOP for GC/MS Semivolatile Method T013	MSS02300.CT	09/27/96
SOP for GC/MS Semivolatiles Method 8270B	MSS02400.CT	10/2/96

GAS CHROMATOGRAPHY

Standard Operating Procedures	Code	Date Generated
SOP for GC CLP OLM01.8	GCS00200.CT	09/11/91
SOP for Standard Prep CLP- Pesticides	GCS00100.CT	05/05/92
SOP for Sulfur Removal	GCS00300.CT	04/30/93
SOP for Pest/PCB Method 8080A	GCS00600.CT	02/15/94
SOP for Analysis of OP Pesticides Method 8141	GCS00500.CT	02/28/94
SOP for HP3350A LAS System	GCS00400.CT	06/08/93
SOP for Misc. Volatiles Method 8015 (DAI)	GCS00700.CT	02/14/94
SOP for Herbicide analysis Method 8150	GCS00800.CT	02/14/94
SOP for Analysis of Hydrocarbon Fingerprinting	GCS01300.CT	08/02/94
SOP for GC/ECD Pesticides/PCB CLP OLM02.1	GCS00900.CT	01/14/94
SOP for Pesticide/PCB Standard Prep OLM02.1	GCS01000.CT	01/14/94
SOP for Pesticides/PCB Method 608	GCS01100.CT	02/15/94
SOP for Sulfur Removal - CLP OLM01.8	GCS01200.CT	06/10/94
SOP for GC/ECD Pesticides/PCB analysis OLM03.1	GCS01400.CT	11/11/94
SOP for Pesticide/PCB Standard Prep OLM03.1	GCS01500.CT	11/11/94
SOP for Low Level Pesticide/PCB analysis - 8080	GCS01600.CT	11/29/94
SOP for Pesticide/PCB analysis - Method 8081	GCS01700.CT	12/28/95
SOP for Diesil Range Organics - Method 8015B	GCS01800.CT	02/07/96
SOP for Gasoline Range Organics - Method 8015B	GCS01900.CT	02/07/96
SOP for Pesticide/PCB analysis - Method T04	GCS02000.CT	07/15/97

METALS

Standard Operating Procedures	Code	Date Generated
SOP for SW846 Method 3005	MES00800.CT	04/21/93
SOP for SW846 Method 3010	MES00900.CT	04/21/93
SOP for SW846 Method 3020A	MES00701.CT	04/21/93
SOP for SW846 Method 3050	MES01001.CT	04/21/93
SOP for CLP SOW Digestion (S)	MES01100.CT	04/21/93
SOP for CLP SOW Digestion (W)	MES01200.CT	04/21/93
SOP for Method 200.7 with TJA 61 Operation	MES00600.CT	04/16/93
SOP for GFAAS 200 series methods	MES00501.CT	04/16/93
SOP for Tracking Metals and Cyanide Samples	IN:050189:1	05/01/89
SOP for Standards Preparations	AS:092988:1	09/29/88
SOP for Determination of Mercury in Water ILM03.0	MES01300.CT	06/10/94
SOP for Determination of Mercury in Soils ILM03.0	MES01400.CT	06/10/94
SOP for Determination of Mercury in Water - 7470A	MES01501.CT	09/12/94
SOP for Determination of Mercury in Soils - 7471A	MES01601.CT	09/12/94
SOP for Method 6010A with TJA 61	MES00400.CT	09/12/94
SOP for GFAAS SW846 series methods	MES00300.CT	09/12/94
SOP for Microwave Digestion Method 3015 (W)	MES01700.CT	04/20/95
SOP for Microwave Digestion Method 3051 (S)	MES01800.CT	04/20/95
SOP for Digestion of AS/SE (GFAA)	MES01900.CT	10/02/95
	MES02000.CT	
SOP for Microwave Digestion ILM03.0	MES02100.CT	04/20/95

METALS (cont.)

Standard Operating Procedure	Code	Date Generated
SOP for Metals Digestion ILM04.0 (Water)	MES02200.CT	07/31/96
SOP for Metals Digestion ILM04.0 (Soil)	MES02300.CT	07/31/96
SOP for Determination of Mercury in Water ILM04.0	MES02400.CT	07/31/96
SOP for Determination of Mercury in Soil ILM04.0	MES02500.CT	07/31/96
SOP for Determination of Metals - ILM04.0 TJA-61E Trace	MES02600.CT	08/1/96
SOP for Determination of Metals - 200.7 TJA 61E Trace	MES02700.CT	08/1/96
SOP for Determination of Mercury in Water Method 245.1	MES02800.CT	08/1/96
SOP for Metals Digestion of Wipe Samples	MES02900.CT	Dft

COMPUTER SYSTEMS

Standard Operating Procedures	Code	Date Generated
SOP for PCB EPA CLP Forms and Disk File	SYS00100.CT	05/25/89
SOP for LIMS Data Entry	SYS00201.CT	02/23/89
SOP for LIMS Data Entry Errors	SYS00301.CT	05/12/92
SOP for LIMS Data Base Security and Backup	SYS00400.CT	08/24/91
SOP for Testing, Modifying and Implementing Changes to Existing Computer Systems	SYS00502.CT	08/25/91
SOP for System Maintenance Operations and Response Time	SYS00600.CT	08/26/91
SOP for Lotus Diskette Deliverable	SYS00700.CT	02/25/92
SOP for Volatile Data Filter Program	SSY00800.CT	03/25/92
SOP for Metals Data Filter Program	SYS00900.CT	03/26/92
SOP for Classical Chemistry Results Program	SYS01000.CT	03/24/92
SOP for LIMS to PC File Transfer	SYS01100.CT	03/27/92
SOP for Classical Chemistry Completion Date Entry Program	SYS01200.CT	03/31/92
SOP for Hamilton Standard Diskette Deliverable	SYS01300.CT	04/01/92
SOP for Envision Software - Organic Deliverables	SYS01400.CT	03/27/92
SOP for Acres Diskette Deliverable	SYS01501.CT	12/01/92
SOP for Control Charts	SYS01600.CT	dft
SOP for CH2MHILL Diskette Deliverable	SYS01701.CT	02/23/93
SOP for AAS File Filter Program	SYS01800.CT	dft

QUALITY ASSURANCE

Standard Operating Procedures	Code	Date Generated
SOP for Document Control	QAS00301.CT	05/08/92
SOP for Corrective Action Reports	QAS00501.CT	07/08/91
SOP for Internal Quality Assurance	QAS00400.CT	07/18/92

SALES/MARKETING

Standard Operating Procedures	Code	Date Generated
SOP for Taking Client Orders	MKS00100.CT	02/21/94
SOP for LIMS Log-in	MKS00200.CT	02/21/94
SOP for Preparation for Price Quotations	MKS00300.CT	02/14/94
SOP for Telephone Logs	MKS00400.CT	06/22/94

CLASSICAL CHEMISTRY

Standard Operating Procedures	Code	Date Generated
Analysis of Tannins and Ligins in Environmental Samples	WC:042091:0	04/20/91
Analysis of Acidity (Method 305.2)	WC:033191:0	03/31/91
Analysis of Acidity (Method 305.1)	CVS00800.CT	03/24/94
Bromide (Method 405)	WC040791:0	04/07/91
Analysis of Hydrocarbons (418.1)	WC:041891:0	04/18/91
Analysis of Oil & Grease (Gravimetric)- 413.1	CVS01001.CT	03/29/94
Analysis of Salinity in Water	WC:070891:0	07/08/91
Analysis of Temperature in Water	WC:070591:0	07/05/91
Analysis of Grain Size	WC:071591:0	07/15/91
Measurement of Conductivity	CVS04300.CT	08/21/90
Analysis of Dissolved Oxygen in Water	WC:071691:0	07/16/91
Analysis of Phosphorus in Water	WC:053191:0	05/31/91
Analysis of Alkalinity in Water - 310.1	CVS00700.CT	02/22/94
Analysis of Ammonia (method 350.1) in Water	WC:070791:0	07/07/92
Analysis of MBAS in Water	CVS00600.CT	03/31/94
Measurement of pH	CVS00900.CT	03/31/94
Analysis of Sulfide (376.1)	CVS01700.CT	01/08/97
Analysis of Biochemical Oxygen Demand	CVS00500.CT	02/22/94
Analysis of COD (Method 410.4)	CVS01201.CT	08/17/94
Analysis of Hexavalent Chromium in cromite ore samples	WC:911205:0	12/05/91
Analysis of Samples for Total Cyanide CLP Protocol	CVS01100.CT	07/01/87
Analysis of Fluroide in Water (Method 340.2)	WC:051590.0	05/15/90
Total Organic Halides Analysis in Water Samples	CVS03801.CT	05/14/90
Analysis of Total Organic Carbon in Water	CVS02200.CT	DFT

CLASSICAL CHEMISTRY (cont.)

Standard Operating Procedures	Code	Date Generated
Analysis of Hexavalent Chromium Colorimetric	WC:090192:0	09/01/92
Analysis of Hexavalent Chromium Alkaline digestion of Soil Samples	WC:083192:0	08/31/92
Analysis of TOC Soil Samples	CVS03400.CT	dft
Analysis of TKN in Environmental Samples	WC:081090:0	08/10/90
Analysis of Hardness in Water	CVS03100.CT	Dft
Analysis of Chloride (325.2) in Water	CVS03900.CT	08/11/90
Analysis of Chloride (325.3) in Water	WC:040991:0	04/09/91
Analysis of Ammonia-Nitrogen in Environmental Samples	WC:021690:0	02/16/90
Standard Operating Procedure for Reactivity	CVS01900.CT	09/29/94
Standard Operating Procedure for Corrosivity	WC:011069:0	01/10/69
Standard Operating Procedure for Ignitability	CVS02300.CT	08/01/96
Manual Spectrophotometric Method for Hexavalent Chromium	WC:110889:4	11/08/89
Analysis of Total Suspended Solids in Water	CVS00200.CT	08/21/93
Analysis of Sulfate in Water (Method 375.3)	CVS01300.CT	03/04/89
Analysis of Sulfate in Water (Method 375.4)	CVS01400.CT	Dft
EPTOX Leachate Procedure in Environmental Samples	WC:081090:0	08/10/90
Analysis of Total Dissolved Solids in Water	CVS00100.CT	08/16/93
Analysis of Nitrate and Nitrite for Water Samples (Method 353.2)	CVS02500.CT	05/03/90
Gravimetric Determination of Lube Oils in Solids	WC:062889:0	06/28/89
SOP for the Analysis of Total Recoverable Phenols	CVS03600.CT	10/09/96
Analysis of Environmental Samples for T- Phenols	WC:080186:1	08/01/86
Analysis of Samples for Chloride (SM407A)	WC:031189:0	03/11/89
Analysis of Environmental Samples for Formaldehyde	WC:072489:0	07/24/89
SOP for Total Cyanide - Method 335.4	CVS02000.CT	10/04/94
SOP for Amenable Cyanide - Method 335.1	CVS02100.CT	10/04/94
SOP for Toxicity Characteristic Leaching Procedure - 1311	CVS01500.CT	09/28/94

CLASSICAL CHEMISTRY (cont.)

Standard Operating Procedures	Code	Date Generated
Measurement of Turbidity in Water Samples	CVS04000.CT	08/21/90
Shake Extraction of Solids for Wet Chemistry Analysis	WC:041391:0	04/13/91
COD (410.1)	WC:082290:1	08/22/90
SOP for WC Data Reporting/Validation	CVS00400.CT	08/29/93
SOP for Total Solids	CVS00300.CT	08/21/93
SOP for Flashpoint - Method 1010	CVS01600.CT	09/28/94
SOP for Waste Extraction Test (WET) Procedure	CVS01800.CT	09/28/94
SOP for Cation/Anion Balance	CVS02800.CT	3/20/95
SOP for CEC Method 9081	CVS02900.CT	3/20/95
SOP for Soil Homogenization	CVS03000.CT	3/20/95
SOP for AVS/SEM	CVS03500.CT	07/09/96
SOP for Oxidation -Reduction Potential	CVS03301.CT	04/29/96
SOP for The Determination of Hydrazine	CVS03200.CT	04/26/96
SOP for The Determination of Ferrous Iron	CVS03700.CT	10/10/96
SOP for Phenols Distillation	CVS02400.CT	09/27/96

APPENDIX, Section 8

LISTING OF ANALYTICAL METHODS

AND ASSOCIATED DETECTION LIMITS

Metals

COMPONENT	SAMPLE MATRIX	ANALYTICAL METHOD	PRECISION %RSD	ACCURACY % RECOVERY	UNITS	PQL
Aluminum	Water	200.7	0-20	90-110	ug/l	200
	Water	6010	0-20	90-110	ug/L	200
	Soil	6010	0-20	90-110	mg/Kg	40
Antimony	Water	200.7	0-20	90-110	ug/L	60
	Water	6010	0-20	90-110	ug/L	60
	Soil	6010	0-20	90-110	mg/Kg	12
	Water	204.2	0-20	80-120	ug/L	1.0
	Soil	7412	0-20	80-120	mg/Kg	1.0
Arsenic	Water	200.7	0-20	90-110	ug/L	10
	Water	6010	0-20	90-110	ug/L	10
	Water	206.2	0-20	80-120	ug/L	10
	Water	7060	0-20	80-120	ug/L	10
	Soil	7060	0-20	80-120	mg/Kg	2.0
	Soil	6010	0-20	90-110	mg/Kg	2.0
Barium	Water	200.7	0-20	90-110	ug/l	200
	Water	6010	0-20	90-110	ug/L	200
	Soil	6010	0-20	90-110	mg/Kg	40
Beryllium	Water	200.7	0-20	90-110	ug/l	5.0
	Water	6010	0-20	90-110	ug/L	5.0
	Soil	6010	0-20	90-110	mg/Kg	1.0
Cadmium	Water	200.7	0-20	90-110	ug/l	5.0
	Water	6010	0-20	90-110	ug/L	5.0
	Soil	6010	0-20	90-110	mg/Kg	1.0
Calcium	Water	200.7	0-20	90-110	ug/l	5000
	Water	6010	0-20	90-110	ug/L	5000
	Soil	6010	0-20	90-110	mg/Kg	1000
Cobalt	Water	200.7	0-20	90-110	ug/l	50
	Water	6010	0-20	90-110	ug/L	50
	Soil	6010	0-20	90-110	mg/Kg	10
Chromium	Water	200.7	0-20	90-110	ug/l	10
	Water	6010	0-20	90-110	ug/L	10
	Soil	6010	0-20	90-110	mg/Kg	2.0

Date: 02/14/97

Metals

COMPONENT	SAMPLE MATRIX	ANALYTICAL METHOD	PRECISION %RSD	ACCURACY % RECOVERY	UNITS	PQL
Copper	Water	200.7	0-20	90-110	ug/l	25
	Water	6010	0-20	90-110	ug/L	25
	Soil	6010	0-20	90-110	mg/Kg	5.0
Iron	Water	200.7	0-20	90-110	ug/l	100
	Water	6010	0-20	90-110	ug/L	100
	Soil	6010	0-20	90-110	mg/Kg	20
Lead	Water	200.7	0-20	90-110	ug/L	3.0
	Water	239.2	0-20	80-120	ug/L	3.0
	Water	7421	0-20	80-120	ug/l	3.0
	Water	6010	0-20	90-110	ug/L	3.0
	Soil	6010	0-20	90-110	mg/Kg	0.6
	Soil	7421	0-20	90-110	mg/Kg	0.6
Magnesium	Water	200.7	0-20	90-110	ug/l	5000
	Water	6010	0-20	90-110	ug/L	5000
	Soil	6010	0-20	90-110	mg/Kg	1000
Manganese	Water	200.7	0-20	90-110	ug/L	15
	Water	6010	0-20	90-110	ug/L	15
	Soil	6010	0-20	90-110	mg/Kg	3.0
Molybdenum	Water	200.7	0-20	90-110	ug/l	20
	Water	6010	0-20	90-110	ug/L	20
	Soil	6010	0-20	90-110	mg/Kg	4.0
Mercury	Water	245.1	0-20	80-120	ug/L	0.2
	Water	7470	0-20	80-120	ug/L	0.2
	Soil	7471	0-20	80-102	mg/Kg	0.1
Nickel	Water	200.7	0-20	90-110	ug/L	40
	Water	6010	0-20	90-110	ug/L	40
	Soil	6010	0-20	90-110	mg/Kg	8.0
Potassium	Water	200.7	0-20	90-110	ug/L	5000
	Water	6010	0-20	90-110	ug/L	5000
	Soil	6010	0-20	90-110	mg/Kg	1000
Selenium	Water	200.7	0-20	90-110	ug/L	5.0
	Water	270.2	0-20	80-120	ug/L	5.0
	Water	6010	0-20	90-110	ug/L	5.0

Date: 02/14/97

Metals						
COMPONENT	SAMPLE MATRIX	ANALYTICAL METHOD	PRECISION %RSD	ACCURACY % RECOVERY	UNITS	PQL
	Water	7740	0-20	80-120	ug/L	5.0
	Soil	7740	0-20	80-120	mg/Kg	1.0
	Soil	6010	0-20	90-110	mg/Kg	1.0
Silver	Water	200.7	0-20	90-110	ug/L	10
	Water	6010	0-20	90-110	ug/L	10
	Soil	6010	0-20	90-110	mg/Kg	2.0
Sodium	Water	200.7	0-20	90-110	ug/L	5000
	Water	6010	0-20	90-110	ug/L	5000
	Soil	6010	0-20	90-110	mg/Kg	1000
Thallium	Water	200.7	0-20	90-110	ug/L	10
	Water	6010	0-20	90-110	ug/L	10
	Water	279.2	0-20	80-120	ug/L	10
	Water	7841	0-20	80-120	ug/L	10
	Soil	7841	0-20	80-120	mg/Kg	2.0
	Soil	6010	0-20	90-110	mg/Kg	2.0
Tin	Water	200.7	0-20	90-110	ug/L	50
	Water	6010	0-20	90-110	ug/L	50
	Soil	6010	0-20	90-110	mg/Kg	10
Titanium	Water	200.7	0-20	90-110	ug/L	20
	Water	6010	0-20	90-110	ug/L	20
	Soil	6010	0-20	90-110	mg/Kg	4.0
Zinc	Water	200.7	0-20	90-110	ug/L	20
	Water	6010	0-20	90-110	ug/L	20
	Soil	6010	0-20	90-110	mg/Kg	4.0
Vanadium	Water	200.7	0-20	90-110	ug/L	50
	Water	6010	0-20	90-110	ug/L	50
	Soil	6010	0-20	90-110	mg/Kg	10

(1) Acceptance limits are those indicated in the published method data.

Date: 02/14/97

Wet Chemistry						
COMPONENT	SAMPLE MATRIX	ANALYTICAL METHOD	PRECISION %RSD	ACCURACY % RECOVERY	UNITS	PQL
Acidity	Water	305.1	0-20	NA	mg/L	1.0
Alkalinity	Water	310.1	0-20	NA	mg/L	2.0
Ammonia-N	Water	350.1	0-20	75-125	mg/L	0.04
Bicarbonate	Water	406C	0-20	NA	mg/L	1.0
Biochemical Oxygen Demand (BOD)	Water	405.1	0-20	75-125	mg/L	2.0
Bromide	Water	320.1	0-20	75-125	mg/L	2.0
Bromide	Water	405	0-20	75-125	mg/L	0.50
Chloride	Water	325.2	0-20	75-125	mg/L	3.0
Chlorine Demand	Water	3-364	0-20	NA	mg/L	1.0
Chlorine Residual	Water	330.5	0-20	NA	mg/L	0.1
Chemical Oxygen Demand (COD)	Water	410.4	0-20	75-125	mg/L	10.0
Color	Water	110.2	0-20	NA	Pt-Co	5.0
Conducitivity	Water	120.1	0-20	NA	umho/cm	NA
Chromium (VI)	Water	7196	0-20	75-125	mg/L	0.01
Cyanide-Total	Water	335.4	0-20	75-125	ug/L	10.0
Cyanide-Total	Water	9012	0-20	75-125	ug/L	10.0
Cyanide-Amenable	Water	335.1	0-20	75-125	ug/L	10.0
Cyanide-CLP	Water	ILM04	0-20	75-125	ug/L	10.0
Dissolved Oxygen	Water	360.1	0-20	NA	mg/L	0.1
Flashpoint	Water	1010	0-20	75-125	-	-
Fluoride	Water	340.2	0-20	75-125	mg/L	0.10
Grain Size	Water	D442-63	0-20	NA	-	-
Hardness	Water	130.2	0-20	75-125	mg/L	1.0
Hydrocarbons (Grav.)	Water	503E	0-20	75-125	mg/L	1.0
Hrdrocarbons (IR)	Water	418.1	0-20	75-125	mg/L	1.0
MBAS	Water	425.1	0-20	75-125	mg/L	0.04
Nitrate-Nitrite-N	Water	353.2	0-20	75-125	mg/L	0.10
Nitrate-N	Water	353.2	0-20	75-125	mg/L	0.005
Odor	Water	140.1	0-20	NA	NA	-
Oil & Grease (Grav.)	Water	413.1	0-20	75-125	mg/L	1.0
Oil &Grease (IR)	Water	413.2	0-20	75-125	mg/L	1.0

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GC/MS Extractable Organics					
	QC CHECK/LCS % RECOVERY LIMIT	MATRIX SPIKE % RECOVERY LIMIT	RELATIVE % DIFFERENCE (RPD) LIMIT	MDL (ug/Kg)	PQL (ug/Kg)
2,4-Dimethylphenol	64-238	-	-	39.5	330
Dimethyl phthalate	D-112	-	-	9.88	330
4,6-Dinitro-2-methylphenol	D-362	-	-	11.9	1600
2,4-Dinitrophenol	D-382	-	-	105	1600
2,4-Dinitrotoluene	39-139	28-89	47	11.6	330
2,6-Dinitrotoluene	50-138	-	-	14.0	330
Di-n-octylphthalate	4-146	-	-	14.8	330
Fluoranthene	26-137	-	-	11.8	330
Fluorene	59-121	-	-	18.2	330
Hexachlorobenzene	D-132	-	-	13.8	330
Hexachlorobutadiene	24-116	-	-	15.4	330
Hexachlorocyclopentadiene	D-59	-	-	12.8	330
Hexachloroethane	40-113	-	-	21.5	330
Indeno(1,2,3-cd)pyrene	D-171	-	-	17.1	330
Isophorone	21-196	-	-	14.9	330
2-Methylnaphthalene	D-127	-	-	14.8	330
2-Methylphenol (o-cresol)	40-189	-	-	11.2	330
4-Methylphenol (p-cresol)	28-198	-	-	17.8	330
Naphthalene	21-133	-	-	13.0	330
2-Nitroaniline	D-127	-	-	49.2	1600
3-Nitroaniline	D-91	-	-	342	1600
4-Nitroaniline	D-108	-	-	138	1600
Nitrobenzene	35-180	-	-	13.5	330
2-Nitrophenol	58-364	-	-	13.5	330
4-Nitrophenol	D-264	10-80	50	128	1600
N-Nitroso-di-n-propylamine	D-230	41-126	38	13.1	330
N-Nitrosodiphenylamine	D-114	-	-	17.1	330
Pentachlorophenol	28-352	17-109	47	76.7	1600
Phenanthrene	54-120	-	-	12.5	330
Phenol	10-224	26-90	35	10.6	330
Pyrene	52-113	35-142	36	18.2	330
1,2,4-Trichlorobenzene	44-142	38-107	23	15.2	330

Date: 02/14/97

GC/MS Extractable Organics					
	QC CHECK/LCS % RECOVERY LIMIT	MATRIX SPIKE % RECOVERY LIMIT	RELATIVE % DIFFERENCE (RPD) LIMIT	MDL (ug/Kg)	PQL (ug/Kg)
2,4,5-Trichlorophenol	82-354	-	-	56.1	1600
2,4,6-Trichlorophenol	74-288	-	-	28.9	330

Date: 02/14/97

Wet Chemistry						
COMPONENT	SAMPLE MATRIX	ANALYTICAL METHOD	PRECISION %RSD	ACCURACY % RECOVERY	UNITS	PQL
Oil & Grease (Grav.)	Water	1664	0-20	80-120	mg/L	5.0
Paint Filter Test	Water	9095	0-20	75-125	NA	-
pH	Water	150.1	0-20	NA	NA	-
pH	Water	9040	0-20	NA	NA	-
Phenols	Water	420.2	0-20	75-125	mg/L	0.005
Phenols	Water	9066	0-20	75-125	mg/L	0.005
Phosphorus	Water	365.2	0-20	75-125	mg/L	0.10
Phosphate (Ortho)	Water	365.2	0-20	75-125	mg/L	0.10
Settable solids	Water	160.5	0-20	NA	mL/L	1.0
Silica	Water	370.1	0-20	75-125	mg/L	1.0
Specific Gravity	Water	3-61	0-20	75-125	NA	-
Sulfate	Water	375.3	0-20	75-125	mg/L	10.0
Sulfate	Water	375.4	0-20	75-125	mg/L	10.0
Sulfide	Water	376.1	0-20	75-125	mg/L	1.0
Sulfite	Water	377.1	0-20	-	mg/L	1.0
Sludge Volume Index	Water	213C	0-20	-	ml/mg	1.0
Total Kjeldahl Nirogen	Water	351.2	0-20	75-125	mg/L	1.0
Total Soilds	Water	160.3	0-20	N/A	mg/L	1.0
Total Dissolved Solids	Water	160.1	0-20	N/A	mg/L	5.0
Total Suspended Solids	Water	160.2	0-20	N/A	mg/L	5.0
Total Volatile Solids	Water	160.4	0-20	N/A	mg/L	1.0
Total Organic Carbon	Water	415.2	0-20	75-125	mg/L	1.0
Total Organic Halides	Water	9020	0-20	75-125	ug/L	10.0
Turbidity	Water	180.1	0-20	-	NTU	0.10
Cyanide	Soil	ILM04	0-20	75-125	mg/Kg	0.5
Total Organic Carbon	Soil	9060M	0-20	75-125	mg/Kg	100
Corrosivity Char.	Soil	9045	-	-	-	-
Ignitability Char.	Soil	BRT	-	-	-	-
TCLP	W/S	1311	-	-	-	-
SPLP	W/S	1312	-	-		

* Acceptance limits are those indicated in the published method data.

Date: 02/14/97

COMPONENT	ACCURACY % RECOVERY	MATRIX SPIKE % RECOVERY	MDL ug/L	PQL ug/L
Method 608 Organochlorine Pesticides in Water				
alpha-BHC	37-134	26-126	.001	.001
beta-BHC	17-147	54-140	.001	.001
delta-BHC	19-140	3-113	.001	.001
gamma-BHC (Lindane)	32-127	47-123	.001	.001
Heptachlor	34-111	26-119	.001	.001
Aldrin	42-122	53-104	.001	.001
Heptachlor epoxide	37-142	59-125	.001	.001
Endosulfan I	45-153	69-138	.002	.002
Dieldrin	36-146	50-136	.002	.002
4,4'-DDE	30-145	73-104	.002	.002
Endrin	30-147	52-154	.002	.002
Endosulfan II	D-202	18-124	.015	.015
4,4' DDD	31-141	10-163	.006	.006
Endosulfan sulfate	26-144	59-152	.006	.006
4,4'-DDT	25-160	51-140	.005	.005
Methoxychlor	62-181	62-181	.006	.006
Toxaphene	41-126	-	2.1	2.1
Aroclor 1016	50-114	-	.159	.159
Aroclor 1221	15-178	-	1.76	1.76
Aroclor 1232	10-215	-	.418	.418
Aroclor 1242	39-150	-	.407	.407
Aroclor 1248	38-158	-	.118	.118
Aroclor 1254	29-131	-	.282	.282
Aroclor 1260	8-127	-	.144	.144
Chlordane (technical)	45-119	-	0.076	0.076
Endrin aldehyde	30-164	30-164	.008	.008
Endrin ketone	30-150	30-150	.006	.006

COMPONENT	LCS/QC CHECK % RECOVERY	MATRIX SPIKE % RECOVERY LIMIT	RELATIVE % DIFFERENCE (RPD)	MDL ug/L	PQL ug/L
Method 8081 Organochlorine Pesticides/PCBs in Water					
alpha-BHC	37-134	-	-	.001	0.05
beta-BHC	17-147	-	-	.001	0.05
delta-BHC	19-140	-	-	.001	0.05
gamma-BHC (Lindane)	32-127	56-123	20	.001	0.05
Heptachlor	34-111	40-131	20	.001	0.05
Aldrin	42-122	40-120	20	.001	0.05
Heptachlor epoxide	37-142	-	-	.001	0.05
Endosulfan I	45-153	-	-	.002	0.05
Dieldrin	36-146	52-126	20	.002	0.1
4,4'-DDE	30-145	-	-	.002	0.1
Endrin	30-147	56-121	20	.002	0.1
Endosulfan II	D-202	-	-	.015	0.1
4,4' DDD	31-141	-	-	.006	0.1
Endosulfan sulfate	26-144	-	-	.006	0.1
4,4'-DDT	25-160	38-127	20	.005	0.1
Methoxychlor	50 - 168	-	-	.006	0.5
Toxaphene	-	-	-	2.1	0.5
Aroclor 1016	-	-	-	.159	1.0
Aroclor 1221	-	-	-	1.76	2.0
Aroclor 1232	-	-	-	.418	1.0
Aroclor 1242	33-128	-	-	.407	1.0
Aroclor 1248	-	-	-	.118	1.0
Aroclor 1254	-	-	-	.282	1.0
Aroclor 1260	41-116	15-175	20	.144	1.0
Chlordane (technical)	-	-	-	0.076	0.2
Endrin aldehyde	44-154	-	-	.008	0.1
Endrin ketone	30-150	-	-	.006	0.1
Method 8150 Chlorinated Herbicides in Water					
2,4-D	50-176	10-200	20	0..201	0.50
Silvex (2,4,5-TP)	10-134	10-197	20	0.028	0.50
2,4,5-T	10-146	-	-	0.023	0.50

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COMPONENT	LCS/QC CHECK % RECOVERY	MATRIX SPIKE % RECOVERY LIMIT	RELATIVE % DIFFERENCE (RPD)	MDL ug/Kg	PQL ug/Kg
Method 8081 Organochlorine Pesticides/PCBs in Soil					
alpha-BHC	37-134	-	-	0.295	1.7
beta-BHC	17-147	-	-	0.652	1.7
delta-BHC	19-140	-	-	0.481	1.7
gamma-BHC (Lindane)	32-127	46-127	20	0.350	1.7
Heptachlor	34-111	35-130	20	0.281	1.7
Aldrin	42-122	40-120	20	0.138	1.7
Heptachlor epoxide	37-142	-	-	0.268	1.7
Endosulfan I	45-153	-	-	0.616	1.7
Dieldrin	36-146	31-134	20	0.425	3.3
4,4'-DDE	30-145	-	-	0.478	3.3
Endrin	30-147	42-139	20	0.425	3.3
Endosulfan II	D-202	-	-	0.906	3.3
4,4' DDD	31-141	-	-	1.915	3.3
Endosulfan sulfate	26-144	-	-	1.967	3.3
4,4'-DDT	25-160	23-134	20	2.144	3.3
Methoxychlor	50 - 168	-	-	11.61	17
Toxaphene	-	-	-	17.2	17
Aroclor 1016	-	-	-	8.62	33
Aroclor 1221	-	-	-	5.62	67
Aroclor 1232	-	-	-	16.7	33
Aroclor 1242	33-128	-	-	5.72	33
Aroclor 1248	-	-	-	9.97	33
Aroclor 1254	-	-	-	7.11	33
Aroclor 1260	41-116	10-175	20	9.18	33
Chlordane (technical)	-	-	-	4.92	6.7
Endrin aldehyde	44-154	-	-	2.81	3.3
Endrin ketone	30-150	-	-	3.98	3.3
Method 8150 Chlorinated Herbicides in Soil					
2,4-D	50-176	10-200	20	7.43	20
Silvex (2,4,5-TP)	10-134	10-197	20	1.10	20
2,4,5-T	10-146	-	-	1.68	5.0

GC/MS Volatile Organics					
	LFB % RECOVERY LIMIT	LAB MATRIX SPIKE % RECOVERY LIMIT	RELATIVE % DIFFERENCE (RPD) LIMIT	MDL (ug/l)	PQL (ug/l)
Method 524.2 Low Level Purgeables in Water					
Benzene	80-120	80-120	13	0.20	1.0
Bromobenzene	80-120	80-120	13	0.12	1.0
Bromochloromethane	80-120	80-120	13	0.12	1.0
Bromodichloromethane	80-120	80-120	13	0.26	1.0
Bromoform	80-120	80-120	13	0.11	1.0
Bromomethane	80-120	80-120	13	0.19	1.0
n-Butylbenzene	80-120	80-120	13	0.20	1.0
sec-Butylbenzene	80-120	80-120	13	0.14	1.0
tert-Butylbenzene	80-120	80-120	13	0.12	1.0
Carbon tetrachloride	80-120	80-120	13	0.18	1.0
Chlorobenzene	80-120	80-120	13	0.14	1.0
Chloroethane	80-120	80-120	13	0.34	1.0
Chloroform	80-120	80-120	13	1.54	1.0
Chloromethane	80-120	80-120	13	0.29	1.0
2-Chlorotoluene	80-120	80-120	13	0.24	1.0
4-Chlorotoluene	80-120	80-120	13	0.14	1.0
Dibromochloromethane	80-120	80-120	13	0.16	1.0
1,2-Dibromo-3-chloropropane	80-120	80-120	13	0.46	1.0
1,2-Dibromoethane	80-120	80-120	13	0.46	1.0
Dibromomethane	80-120	80-120	13	0.13	1.0
1,2-Dichlorobenzene	80-120	80-120	13	0.10	1.0
1,3-Dichlorobenzene	80-120	80-120	13	0.11	1.0
1,4-Dichlorobenzene	80-120	80-120	13	0.11	1.0
Dichlorodifluoromethane	80-120	80-120	13	0.16	1.0
1,1-Dichloroethane	80-120	80-120	13	0.24	1.0
1,2-Dichloroethane	80-120	80-120	13	0.14	1.0
1,1-Dichloroethene	80-120	80-120	13	0.21	1.0
cis-1,2-Dichloroethene	80-120	80-120	13	0.23	1.0
trans-1,2-Dichloroethene	80-120	80-120	13	0.21	1.0

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GC/MS Volatile Organics					
	LFB % RECOVERY LIMIT	LAB MATRIX SPIKE % RECOVERY LIMIT	RELATIVE % DIFFERENCE (RPD) LIMIT	MDL (ug/l)	PQL (ug/l)
1,2-Dichloropropane	80-120	80-120	13	0.21	1.0
1,3-Dichloropropane	80-120	80-120	13	0.16	1.0
2,2-Dichloropropane	80-120	80-120	13	0.17	1.0
1,1-Dichloropropene	80-120	80-120	13	0.10	1.0
Ethylbenzene	80-120	80-120	13	0.20	1.0
Hexachlorobutadiene	80-120	80-120	13	0.41	1.0
Isopropylbenzene	80-120	80-120	13	0.15	1.0
p-Isopropyltoluene	80-120	80-120	13	0.12	1.0
Methylene chloride	80-120	80-120	13	1.0	1.0
Naphthalene	80-120	80-120	13	0.32	1.0
n-Propylbenzene	80-120	80-120	13	0.12	1.0
Styrene	80-120	80-120	13	0.16	1.0
1,1,1,2-Tetrachloroethane	80-120	80-120	13	0.21	1.0
1,1,2,2-Tetrachloroethane	80-120	80-120	13	0.16	1.0
Tetrachloroethene	80-120	80-120	13	0.16	1.0
Toluene	80-120	80-120	13	0.15	1.0
1,2,3-Trichlorobenzene	80-120	80-120	13	0.31	1.0
1,2,4-Trichlorobenzene	80-120	80-120	13	0.21	1.0
1,1,1-Trichloroethane	80-120	80-120	13	0.22	1.0
1,1,2-Trichloroethane	80-120	80-120	13	0.13	1.0
Trichloroethene	80-120	80-120	13	0.17	1.0
Trichlorofluoromethane	80-120	80-120	13	0.18	1.0
1,2,3-Trichloropropane	80-120	80-120	13	0.31	1.0
1,2,4-Trimethylbenzene	80-120	80-120	13	0.14	1.0
1,3,5-Trimethylbenzene	80-120	80-120	13	0.19	1.0
Vinyl chloride	80-120	80-120	13	0.30	1.0
o-Xylene	80-120	80-120	13	0.21	1.0
m/p-xylene	80-120	80-120	13	0.41	1.0

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GC/MS Volatile Organics				
	QC CHECK/LCS % RECOVERY LIMIT	MATRIX SPIKE % RECOVERY LIMIT	MDL (ug/l)	PQL (ug/l)
Method 624 Purgeables in Water				
Benzene	37-151	78-122	0.52	0.52
Bromodichloromethane	35-155	82-117	0.47	0.47
Bromoform	45-169	61-136	0.81	0.81
Bromomethane	d-242	67-122	1.6	1.6
Carbon tetrachloride	70-140	76-127	0.66	0.66
Chlorobenzene	37-160	78-117	0.41	0.41
Chloroethane	14-230	79-118	2.09	2.09
2-Chloroethylvinyl ether	D-305	10-305	1.23	1.23
Chloroform	51-138	83-114	0.53	0.53
Chloromethane	D-273	35-152	1.0	1.0
Dibromochloromethane	53-149	78-122	0.69	0.69
1,2-Dichlorobenzene	18-190	18-190	0.65	0.65
1,3-Dichlorobenzene	59-156	59-156	0.37	0.37
1,4-Dichlorobenzene	18-190	60-145	0.43	0.43
1,1-Dichloroethane	59-155	81-181	0.82	0.82
1,2-Dichloroethane	49-155	80-123	0.41	0.41
1,1-Dichloroethene	D-234	79-121	0.82	0.82
1,2-Dichloroethene (total)	54-156	85-113	1.0	1.0
1,2-Dichloropropane	D-210	77-124	0.52	0.52
cis-1,3-Dichloropropene	D-227	75-110	0.54	0.54
trans-1,3-Dichloropropene	17-183	73-132	0.53	0.53
Ethylbenzene	37-162	83-112	0.54	0.54
Methylene chloride	D-221	83-115	1.14	1.14
1,1,2,2-Tetrachloroethane	46-157	69-138	1.11	1.11
Tetrachloroethene	64-148	76-121	0.48	0.48
Toluene	47-150	77-117	0.51	0.51
1,1,1-Trichloroethane	52-162	72-130	0.39	0.39
1,1,2-Trichloroethane	52-150	71-126	0.81	0.81

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GC/MS Volatile Organics				
	QC CHECK/LCS % RECOVERY LIMIT	MATRIX SPIKE % RECOVERY LIMIT	MDL (ug/l)	PQL (ug/l)
Trichloroethene	71-157	82-116	0.27	0.27
Trichlorofluoromethane	17-181	17-181	0.72	0.72
Vinyl Chloride	D-251	65-127	1.07	1.07

GC/MS Volatile Organics					
	QC CHECK/LCS % RECOVERY LIMIT	MATRIX SPIKE % RECOVERY LIMIT	RELATIVE % DIFFERENCE (RPD) LIMIT	MDL (ug/L)	PQL (ug/l)
Method 8260 Purgeables in Water					
Acetone	28-374	-	-	6.7	10
Benzene	37-151	76-127	11	0.5	5
Bromodichloromethane	35-155	-	-	0.5	5
Bromoform	45-169	-	-	1.1	5
Bromomethane	d-242	-	-	0.5	10
2-Butanone	D-502	-	-	6.2	10
Carbon disulfide	D-95	-	-	0.4	5
Carbon tetrachloride	70-140	-	-	0.9	5
Chlorobenzene	37-160	75-130	13	0.4	5
Dibromochloromethane	53-138	-	-	0.3	5
Chloroethane	14-230	-	-	1.5	10
2-Chloroethylvinyl ether	D-305	-	-	0.7	10
Chloroform	51-138	-	-	1.0	5
Chloromethane	59-155	-	-	0.4	10
1,1-Dichloroethane	D-234	61-145	14	0.3	5
1,2-Dichloroethane	49-155	-	-	0.5	5
1,1-Dichloroethene	D-234	-	-	0.6	5
1,2-Dichloroethene (total)	54-156	-	-	0.9	5
1,2-Dichloropropane	D-210	-	-	0.5	5
cis-1,3-Dichloropropene	D-227	-	-	0.3	5
trans-1,3-Dichloropropene	17-183	-	-	0.4	5
Ethylbenzene	37-162	-	-	0.4	5
2-Hexanone	11 - 68	-	-	1.4	10
Methylene chloride	D-221	-	-	1.1	5
4-Methyl-2-pentanone	31-154	-	-	1.0	10
Styrene	61-126	-	-	0.4	5
1,1,2,2-Tetrachloroethane	46-157	-	-	2.2	5
Tetrachloroethene	64-148	-	-	0.5	5
Toluene	47-150	76-125	13	0.4	5
1,1,1-Trichloroethane	52-162	-	-	0.5	5

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GC/MS Volatile Organics					
	QC CHECK/LCS % RECOVERY LIMIT	MATRIX SPIKE % RECOVERY LIMIT	RELATIVE % DIFFERENCE (RPD) LIMIT	MDL (ug/L)	PQL (ug/l)
1,1,2-Trichloroethane	52-162	-	-	0.4	5
Trichloroethene	71-157	71-120	14	0.4	5
Vinyl acetate	16-235	-	-	4.5	10
Vinyl chloride	D-251	-	-	0.6	10
Xylenes (total)	33-103	-	-	0.5	5

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GC/MS Volatile Organics					
	QC CHECK/LCS % RECOVERY LIMIT	MATRIX SPIKE % RECOVERY LIMIT	RELATIVE % DIFFERENCE (RPD) LIMIT	MDL (ug/Kg)	PQL (ug/Kg)
Method 8260 Purgeables in Soil					
Acetone	14-187	-	-	8.3	10
Benzene	70-145	66-142	21	0.97	5
Bromodichloromethane	70-125	-	-	0.95	5
Bromoform	45-169	-	-	0.97	5
Bromomethane	13-145	-	-	2.62	10
2-Butanone	D-251	-	-	2.30	10
Carbon disulfide	D-475	-	-	1.27	5
Carbon tetrachloride	70-140	-	-	1.16	5
Chlorobenzene	90-135	60-133	21	1.03	5
Dibromochloromethane	70-130	-	-	1.09	5
Chloroethane	14-230	-	-	2.42	10
2-Chloroethylvinyl ether	D-305	-	-	0.9	10
Chloroform	80-135	-	-	2.92	5
Chloromethane	D-273	-	-	2.36	10
1,1-Dichloroethane	75-135	59-172	22	1.38	5
1,2-Dichloroethane	65-135	-	-	1.05	5
1,1-Dichloroethene	70-125	-	-	0.96	5
1,2-Dichloroethene (total)	68-132	-	-	1.36	5
1,2-Dichloropropane	75-145	-	-	1.05	5
cis-1,3-Dichloropropene	70-113	-	-	0.79	5
trans-1,3-Dichloropropene	70-113	-	-	1.19	5
Ethylbenzene	75-130	-	-	0.88	5
2-Hexanone	28-170	-	-	1.28	10
Methylene chloride	50-160	-	-	1.57	5
4-Methyl-2-pentanone	60-170	-	-	1.26	10
Styrene	80-120	-	-	0.93	5
1,1,2,2-Tetrachloroethane	65-130	-	-	1.27	5
Tetrachloroethene	64-148	-	-	1.34	5
Toluene	31-130	50-139	21	1.07	5
1,1,1-Trichloroethane	80-120	-	-	1.28	5

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GC/MS Volatile Organics					
	QC CHECK/LCS % RECOVERY LIMIT	MATRIX SPIKE % RECOVERY LIMIT	RELATIVE % DIFFERENCE (RPD) LIMIT	MDL (ug/Kg)	PQL (ug/Kg)
1,1,2-Trichloroethane	85-130	-	-	1.24	5
Trichloroethene	70-135	62-137	24	0.82	5
Vinyl acetate	8-118	-	-	1.16	10
Vinyl chloride	1-240	-	-	1.21	10
Xylenes (total)	55-172	-	-	0.97	5

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GC/MS Extractable Organics				
	QC CHECK/LCS % RECOVERY LIMIT	MATRIX SPIKE % RECOVERY LIMIT	MDL (ug/L)	PQL (ug/l)
Method 625 Extractables in Water				
Acenaphthene	47-145	47-123	0.6	0.6
Acenaphthylene	33-145	33-145	0.4	0.4
Anthracene	27-133	27-133	0.5	0.5
Benzidine			79	80
Benzo(a)anthracene	33-143	33-143	1.2	1.2
Benzo(a)pyrene	17-163	17-163	0.9	0.9
Benzo(b)fluoranthene	24-139	24-139	0.7	0.7
Benzo(g,h,i)perylene	D-219	D-219	0.6	0.6
Benzo(k)fluoranthene	11-162	11-162	1.2	1.2
bis(2-Chloroethoxy)methane	33-184	33-184	0.7	0.7
bis(2-Chloroethyl)ether	12-138	12-138	0.6	0.6
bis(2-Chloroisopropyl)ether	36-166	36-166	0.5	0.5
bis(2-Ethylhexyl)phthalate	8-138	8-138	1.8	1.8
4-Bromophenyl phenyl ether	53-127	53-127	0.5	0.5
Benzyl butyl phthalate	D-132	D-132	1.4	1.4
2-Chloronaphthalene	60-118	60-118	0.8	0.8
4-Chlorophenyl phenyl ether	25-138	25-138	0.6	0.6
Chrysene	17-168	17-168	1.1	1.1
Dibenzo(a,h)anthracene	D-227	D-227	0.6	0.6
1,2-Dichlorobenzene	32-129	32-129	0.7	0.7
1,3-Dichlorobenzene	D-172	D-172	0.6	0.6
1,4-Dichlorobenzene	20-124	20-124	0.6	0.6
3,3'-Dichlorobenzidine	D-262	D-262	1.0	1.0
Diethyl phthalate	D-114	D-114	0.6	0.6
Dimethyl phthalate	D-112	D-112	0.5	0.5
Di-n-butylphthalate	1-118	1-118	1.1	1.1
2,4-Dinitrotoluene	39-139	39-139	0.6	0.6
2,6-Dinitrotoluene	50-138	50-138	0.5	0.5
Di-n-octylphthalate	4-146	4-146	0.7	0.7
Fluoranthene	26-137	26-137	0.7	0.7
Fluorene	59-121	59-121	0.6	0.6

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GC/MS Extractable Organics				
	QC CHECK/LCS % RECOVERY LIMIT	MATRIX SPIKE % RECOVERY LIMIT	MDL (ug/L)	PQL (ug/l)
Hexachlorobenzene	D-132	D-132	0.5	0.5
Hexachlorobutadiene	24-116	24-116	0.6	0.6
Hexachlorocyclopentadiene	D-59	D-59	1.0	1.0
Hexachloroethane	40-113	19-89	0.5	0.5
Indeno(1,2,3-cd)pyrene	D-171	D-171	0.6	0.6
Isophorone	21-196	21-196	0.6	0.6
Naphthalene	21-133	21-133	0.6	0.6
Nitrobenzene	35-180	35-180	0.6	0.6
N- Nitrosodimethylamine			0.4	0.4
1,2 diphenylhydrazine			0.6	0.6
N-Nitroso-di-n-propylamine	D-230	D-230	0.6	0.6
N-Nitrosodiphenylamine	D-114	D-114	1.4	1.4
Phenanthrene	54-120	54-120	0.5	0.5
Pyrene	52-113	52-113	1.4	1.4
1,2,4-Trichlorobenzene	44-142	44-142	0.5	0.5
4-Chloro-3-methylphenol	44-294	44-294	0.6	0.6
2-Chlorophenol	46-268	44-123	0.5	0.5
2,4-Dichlorophenol	78-270	78-270	0.6	0.6
2,4-Dimethylphenol	64-238	64-238	0.5	0.5
2,4-Dinitrophenol	D-382	D-382	1.7	1.7
2-Methyl-4,6-dinitrophenol	D-362	D-362	0.6	0.6
2-Nitrophenol	58-364	58-364	0.5	0.5
4-Nitrophenol	D-264	D-264	0.3	0.3
Pentachlorophenol	28-352	28-352	0.8	0.8
Phenol	10-224	10-224	0.3	0.3
2,4,6-Trichlorophenol	74-288	74-288	0.6	0.6

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GC/MS Extractable Organics					
	QC CHECK/LCS % RECOVERY LIMIT	MATRIX SPIKE % RECOVERY LIMIT	RELATIVE % DIFFERENCE (RPD) LIMIT	MDL (ug/l)	PQL (ug/l)
Method 8270 Extractables in Water					
Acenaphthene	47-145	46-118	31	0.6	10
Acenaphthylene	33-145	-	-	0.4	10
Anthracene	27-133	-	-	0.5	10
Benzoic acid	D-473	-	-	7.8	50
Benzo(a)anthracene	33-143	-	-	1.2	10
Benzo(b)fluoranthene	24-139	-	-	0.7	10
Benzo(k)fluoranthene	11-162	-	-	1.2	10
Benzo(g,h,i)perylene	D-219	-	-	0.6	10
Benzo(a)pyrene	17-163	-	-	0.9	10
Benzyl alcohol	D-130	-	-	1.5	10
bis(2-Chloroethoxy)methane	33-184	-	-	0.7	10
bis(2-Chloroethyl)ether	12-138	-	-	0.6	10
bis(2-Chloroisopropyl)ether	36-166	-	-	0.5	10
bis(2-Ethylhexyl)phthalate	8-138	-	-	1.8	10
4-Bromophenyl phenyl ether	53-127	-	-	0.5	10
Benzyl butyl phthalate	D-132	-	-	1.4	10
4-Chloroaniline	1-78	-	-	0.7	10
2-Chloronaphthalene	60-118	-	-	0.8	10
4-Chloro-3-methylphenol	44-294	23-97	42	0.6	10
2-Chlorophenol	46-268	27-123	40	0.5	10
4-Chlorophenyl phenyl ether	25-138	-	-	0.6	10
Chrysene	17-168	-	-	1.1	10
Dibenzo(a,h)anthracene	D-227	-	-	0.6	10
Dibenzofuran	D-170	-	-	0.6	10
Di-n-butylphthalate	1-118	-	-	1.1	10
1,3-Dichlorobenzene	D-172	-	-	0.6	10
1,4-Dichlorobenzene	20-124	36-97	28	0.6	10
1,2-Dichlorobenzene	32-129	-	-	0.7	10
3,3'-Dichlorobenzidine	D-52	-	-	1.0	10
2,4-Dichlorophenol	78-270	-	-	0.6	10
Diethyl phthalate	D-114	-	-	0.6	10

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GC/MS Extractable Organics					
	QC CHECK/LCS % RECOVERY LIMIT	MATRIX SPIKE % RECOVERY LIMIT	RELATIVE % DIFFERENCE (RPD) LIMIT	MDL (ug/l)	PQL (ug/l)
2,4-Dimethylphenol	64-238	-	-	0.5	10
Dimethyl phthalate	D-112	-	-	0.5	10
4,6-Dinitro-2-methylphenol	D-362	-	-	0.6	25
2,4-Dinitrophenol	D-382	-	-	1.7	25
2,4-Dinitrotoluene	39-139	24-96	38	0.6	10
2,6-Dinitrotoluene	50-138	-	-	0.5	10?
Di-n-octylphthalate	4-146	-	-	0.7	10
Fluoranthene	26-137	-	-	0.7	10
Fluorene	59-121	-	-	0.6	10
Hexachlorobenzene	D-132	-	-	0.5	10
Hexachlorobutadiene	24-116	-	-	0.6	10
Hexachlorocyclopentadiene	D-59	-	-	1.0	10
Hexachloroethane	40-113	-	-	0.5	10
Indeno(1,2,3-cd)pyrene	D-171	-	-	0.6	10
Isophorone	21-196	-	-	0.6	10
2-Methylnaphthalene	D-127	-	-	0.6	10
2-Methylphenol (o-cresol)	40-189	-	-	0.6	10
4-Methylphenol (p-cresol)	28-198	-	-	0.5	10
Naphthalene	21-133	-	-	0.6	10
2-Nitroaniline	D-127	-	-	0.5	25
3-Nitroaniline	D-91	-	-	0.6	25
4-Nitroaniline	D-108	-	-	0.8	20
Nitrobenzene	35-180	-	-	0.6	10
2-Nitrophenol	58-364	-	-	0.5	10
4-Nitrophenol	D-264	10-80	50	0.3	25
N-Nitroso-di-n-propylamine	D-230	41-116	38	0.6	10
N-Nitrosodiphenylamine	D-114	-	-	1.4	10
Pentachlorophenol	28-352	9-103	50	0.8	25
Phenanthrene	54-120	-	-	0.5	10
Phenol	10-224	12-110	42	0.3	10
Pyrene	52-113	26-127	31	1.4	10
1,2,4-Trichlorobenzene	44-142	39-98	28	0.5	10

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GC/MS Extractable Organics					
	QC CHECK/LCS % RECOVERY LIMIT	MATRIX SPIKE % RECOVERY LIMIT	RELATIVE % DIFFERENCE (RPD) LIMIT	MDL (ug/Kg)	PQL (ug/Kg)
Method 8270 Extractables in Soil					
Acenaphthene	47-145	31-137	19	12.6	330
Acenaphthylene	33-145	-	-	12.8	330
Anthracene	27-133	-	-	13.0	330
Benzoic acid	D-473	-	-	100	1600
Benzo(a)anthracene	33-143	-	-	14.6	330
Benzo(b)fluoranthene	24-139	-	-	23.0	330
Benzo(k)fluoranthene	11-162	-	-	16.5	330
Benzo(g,h,i)perylene	D-219	-	-	17.8	330
Benzo(a)pyrene	17-163	-	-	16.2	330
Benzyl alcohol	D-130	-	-	13.4	330
bis(2-Chloroethoxy)methane	33-184	-	-	11.7	330
bis(2-Chloroethyl)ether	12-138	-	-	17.0	330
bis(2-Chloroisopropyl)ether	36-166	-	-	15.2	330
bis(2-Ethylhexyl)phthalate	8-138	-	-	20.8	330
4-Bromophenyl phenyl ether	53-127	-	-	12.9	330
Benzyl butyl phthalate	D-132	-	-	11.4	330
4-Chloroaniline	1-78	-	-	47.8	330
2-Chloronaphthalene	60-118	-	-	14.3	330
4-Chloro-3-methylphenol	44-294	26-103	33	14.5	330
2-Chlorophenol	46-268	25-102	50	12.0	330
4-Chlorophenyl phenyl ether	25-138	-	-	14.6	330
Chrysene	17-168	-	-	15.1	330
Dibenzo(a,h)anthracene	D-227	-	-	18.5	330
Dibenzofuran	D-170	-	-	16.7	330
Di-n-butylphthalate	1-118	-	-	16.8	330
1,3-Dichlorobenzene	D-172	-	-	14.0	330
1,4-Dichlorobenzene	20-124	28-104	27	16.7	330
1,2-Dichlorobenzene	32-129	-	-	13.4	330
3,3'-Dichlorobenzidine	D-52	-	-	10.4	660
2,4-Dichlorophenol	78-270	-	-	10.2	330
Diethyl phthalate	D-114	-	-	17.1	330

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GC/MS Extractable Organics					
	QC CHECK/LCS % RECOVERY LIMIT	MATRIX SPIKE % RECOVERY LIMIT	RELATIVE % DIFFERENCE (RPD) LIMIT	MDL (ug/l)	PQL (ug/l)
2,4,5-Trichlorophenol	82-354	-	-	0.6	25
2,4,6-Trichlorophenol	74-288	-	-	0.5	25