



**SPECTRUM ANALYTICAL, INC.**  
*Featuring*  
**HANIBAL TECHNOLOGY**  
**Rhode Island Division**

# QUALITY ASSURANCE PLAN

## 2011

Approved By:

\_\_\_\_\_  
Yihai Ding  
Laboratory Director

06/01/2011

Date

\_\_\_\_\_  
Edward A. Lawler  
Deputy Director for Quality Services

06/01/2011

Date

\_\_\_\_\_  
Sharyn B. Lawler  
Quality Assurance Director

06/01/2011

Date

EFFECTIVE DATE: 06/01/2011

175 Metro Center Boulevard · Warwick, RI 02886-1755 · 401-732-3400 · FAX 401-732-3499  
[www.spectrum-analytical.com](http://www.spectrum-analytical.com)

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### 3.0 INTRODUCTION

Spectrum Analytical, Inc. Featuring Hanibal Technology Rhode Island Division (formerly Mitkem Laboratories (MITKEM )and referenced as Spectrum Analytical, Inc. RI Division throughout this document going forward) is an environmental testing laboratory dedicated to providing high quality analytical data and exceptional customer service. During the transitional period of our name change, some documentation will still retain the laboratory identification as Mitkem Laboratories. We are making every effort to update all forms, laboratory logbooks and software references to our new name in a timely manner.

Spectrum Analytical, Inc. RI Division's senior managers have over 60 years combined experience in the industry and the company's highly qualified laboratory staff includes some of the most accomplished business and technical people in the field. Our Laboratory/Technical Director, Mr. Yihai Ding, has experience in a wide variety of analytical chemistry techniques, including GC, GC/MS, HPLC and FTIR. His expertise includes the operation, calibration and maintenance of sophisticated, computer controlled instrumentation. Mr. Ding has also taught chemistry and biochemistry laboratory courses at the university level. Mr. Ding is involved in daily lab operations and shares his expertise with the staff and our customers.

Opened in 1994, Spectrum Analytical, Inc. RI Division's laboratory facility consists of over 12,500 square feet of space and is located at 175 Metro Center Boulevard in Warwick, RI 02886. This facility is located within two miles of Providence's airport and two blocks of the regional Federal Express service center. Our close proximity to both the airport and Federal Express enables us to promptly receive samples for quick turn-around time analysis and data reporting. With over 400 linear feet of bench space, Spectrum Analytical, Inc. RI Division is designed for high throughput and efficient laboratory operations. Separate secure areas are dedicated to sample receipt and storage. Spectrum Analytical, Inc. RI Division has individual sample preparation laboratories for organic and inorganic analyses and individual instrumentation rooms for extractable organics, volatiles, metals and wet-chemistry analyses.

Spectrum Analytical, Inc. RI Division recognizes the importance of controlling in-house cross contamination. The organic preparation area and the volatile organic instrument room are in separate suites in the laboratory facility to minimize solvent contamination of the volatile analysis. It is not possible to get to the volatiles laboratory from the rest of the facility without going outside, and walking down the sidewalk. The air handling system in the volatiles laboratory is completely isolated from the remainder of the facility.

Spectrum Analytical, Inc. RI Division has placed a priority on obtaining and operating a large fleet of the latest, most sophisticated Hewlett-Packard, Thermo Scientific and Perkin-Elmer instruments. This emphasis on instrumentation enables the lab to operate at a high level of analytical efficiency.

Spectrum Analytical, Inc. RI Division specializes in performing laboratory analyses using the newest US EPA Contract Laboratory Program (CLP) *SOM* Organic and *ISM* Inorganic methods, as well as providing CLP-format data reports for virtually any test we perform. Spectrum Analytical, Inc. RI Division provides CLP-format reporting for EPA CLP, SW-846, MCAWW and Standard Methods analyses. Much of this work is performed by the laboratory under Department of Defense Quality Systems Manual (QSM) guidelines. Spectrum Analytical, Inc. RI Division has the flexibility to provide project-specific custom method modifications to meet the needs of a unique client or analytical requirement.

Spectrum Analytical, Inc. RI Division has participated in numerous environmental laboratory programs for both state and federal agencies including: the United States Navy, the United States Army Corps of Engineers, and the Air Force Center for Environmental Excellence. In addition Spectrum Analytical, Inc. RI Division is currently providing laboratory services under the United States Environmental Protection Agency Contract Laboratory Program. Spectrum Analytical, Inc. RI Division has been a contractor to the EPA under the CLP program continuously for over 12 years.

Spectrum Analytical, Inc. RI Division is a division of Spectrum Analytical, Inc. of Agawam, Massachusetts. Spectrum Analytical, Inc is an environmental laboratory company providing analyses of soil, water and air samples for a wide variety of private and government clients. Spectrum Analytical, Inc specializes in providing rapid turnaround data reports meeting the specific requirements of several Northeastern States, particularly for large volume programs.

This Quality Assurance Plan (QAP) describes the policies, organization, objectives, and quality control activities. It also specifies quality assurance functions employed at Spectrum Analytical, Inc. RI Division and demonstrates our dedication to the production of accurate, consistent data of known quality. This QAP is developed by following the guidelines discussed in the EPA Requirements for Quality Assurance Project Plans for Environmental Data Operations, EPA QA/R-5, Reissued May 2006: EPA Requirements for Quality Management Plans, EPA QA/R-2, Reissued May 2006: Department of Defense (DOD QSM) Quality Systems Manual for Environmental Laboratories Version 4.1: and the National Environmental Laboratory Accreditation Conference (NELAC) standards, June 5, 2003 (Effective July 1, 2003). Plans are underway to adopt The NELAC Institute (TNI) Standards by July 2011.

#### 4.0 QUALITY ASSURANCE POLICY STATEMENT

Spectrum Analytical, Inc. RI Division is firmly committed to the production of valid data of known quality through the use of analytical measurements that are accurate, reproducible and complete. To ensure the production of such data, we have developed a comprehensive Quality Assurance/Quality Control Program that operates throughout the entire organization.

Spectrum Analytical, Inc. RI Division Management considers Quality Assurance/Quality Control to be of the highest importance in the success of its Analytical Testing Laboratory and therefore fully supports the staff in the implementation and maintenance of a sound and thorough Quality Assurance Program.

Spectrum Analytical, Inc. RI Division's corporate success is based on its participation in the most rigorous and quality-focused environmental testing programs, such as the EPA Contract Laboratory Program, US Department of Defense programs, NELAC, and other nationwide and state-specific certification and approval programs. These programs require consistent application of the QA/QC procedures described in this document. Spectrum Analytical, Inc. RI Division's ability to demonstrate and document that analyses were performed in this manner is one of the foundations of its business. The other foundation of its business is to provide superior levels of customer service, above and beyond the norm for laboratories performing at this level of quality.

Spectrum Analytical, Inc. RI Division's approach to customer service is to aggressively meet or exceed customer expectations, particularly in terms of turnaround time for results. While the production of rapid turnaround time data may require lab employees to "go the extra mile" for the customer, without quality, the data are useless. Spectrum Analytical, Inc. RI Division constantly strives to manage its business to rapidly provide data to meet all the requirements of its quality program.

- Spectrum Analytical, Inc. RI Division management works to insure: that employees understand the primary importance of quality in its day to day operations,
- that employees will not be subjected to pressure to sacrifice quality for turnaround, financial or other considerations,
- that employees understand the importance of their ethical responsibilities in terms of data manipulation, falsification or other illegal or improper actions,
- that the company avoids involvement in activities that diminish its competence, impartiality, judgment or operational integrity.
- that employees maintain all client information in a confidential manner, and
- that employees understand that any short-term gain realized by disregarding the QA/QC program will be more than wasted by the serious penalties for these actions.
- That the laboratory has the technical personnel to identify occurrences of departure from the quality system and to initiate actions to prevent or minimize such departures.

All employees receive training in these issues as part of the initial orientation process, and are required to acknowledge that they understand their responsibilities in these areas.

These issues are also discussed among all laboratory staff at company meetings and re-training sessions. The QA Officer, Technical Director and other senior management are readily available to all staff through their daily presence, “open door” policy and approachable manner. This allows any employee to readily discuss any questions, concerns or issues that may occur.

Quality Control is defined as an organized system of activities whose purpose is to demonstrate that quality data are being produced through documentation. Quality Assurance is more broadly defined as a system of activities designed to ensure that the quality control program is actually effective in producing data of the desired quality.

Quality Control is included as part of Quality Assurance. In supporting government regulatory and enforcement proceedings, a high degree of attention to quality is essential. Thorough application of quality control principles and routine quality assurance audits is required.

The basic components of the Spectrum Analytical, Inc. RI Division’s QA/QC Program are control, evaluation and correction.

Control ensures the proper functioning of analytical systems through the implementation of an orderly and well-planned series of positive measures taken prior to and during the course of analysis including quality control practices, routine maintenance and calibration of instruments, and frequent validation of standards.

Evaluation involves the assessment of data generated during the control process. For example, precision and accuracy are determined from the results of duplicates and spikes, and other check samples. Long-term evaluation measures include performance and systems audit conducted by regulatory agencies, as well as the lab’s quality assurance department.

Correction includes the investigation, diagnosis and resolution of any problems detected in an analytical system. Proper functioning of the system may be restored through method re-evaluation, analysis of additional check samples, trouble-shooting and repair of instrumentation or examination and comparison with historical data. Corrective actions are documented and reviewed to make sure they are implemented.

Certain situations may occur when there are occasional departures or exceptions from documented policies and procedures or standard specifications due to client or project specific protocols, unusual sample matrix, or special non-target analyte or non-routine analyses. Spectrum Analytical, Inc. RI Division’s policy is to fully document all such procedures and their associated QC, and notify the client or regulatory agency. If the situation is to continue, a Standard Operating Procedure will be written and implemented.

## 5.0 QUALITY ASSURANCE MANAGEMENT, ORGANIZATION AND RESPONSIBILITY

Quality Assurance at Spectrum Analytical, Inc. RI Division is a company-wide function that depends on:

- (1) cooperative working relationships at all levels within the laboratory and
- (2) Multi-level review through all working levels of responsibility.

Responsibilities for QA/QC functions begin with the bench scientist and extend to the chief executive officer.

The primary level of quality assurance resides with the bench scientist. After completion of the documented training program, his/her responsibilities include:

- complying with all aspects of formally approved analytical methods and SOPs,
- carefully documenting each step of the analytical process,
- conscientiously obtaining peer review as required,
- promptly alerting laboratory supervisors and/or QA staff members to problems or anomalies that may adversely impact data quality, and
- participation in corrective actions as directed by the laboratory supervisor or QA Director.

The Manager of each laboratory department is responsible for ensuring thorough oversight of the quality of the data generated by the department supervisors, technicians and/or analysts. The Department Manager implements and monitors the specific QC protocols and QA programs with the laboratory to ensure a continuous flow of data meeting all control protocols and Spectrum Analytical, Inc. RI Division QA requirements. The Department Manager's responsibilities include providing the technicians and/or analysts with adequate resources to achieve the desired quality of performance.

The Spectrum Analytical, Inc. RI Division organizational structure is shown in the Organization Chart (Figure 5-1).

Spectrum Analytical, Inc. RI Division's lines of communication flow upward on the Organizational Chart. The open door policy allows all employees' access to anyone on the organization chart. If an employee has an issue with his/her immediate supervisor, he or she may, at any time, speak with someone in management higher up in the Organizational Chart.

Implementation of the entire Quality Assurance Program is the responsibility of the QA Director. While interacting on a daily basis with laboratory staff members, the QA Director remains independent of the laboratories and reports directly to the Laboratory Director. The QA Director evaluates laboratory compliance with respect to the QA

program through informal and formal systems and performance audits as described in Section 13. Remedial action, to alleviate any detected problems, is suggested and/or discussed with the appropriate parties and implemented when necessary.

With input from the appropriate staff members, the QA Director writes, edits and archives QA Plans, QC protocols, and Standard Operating Procedures (SOPs) in accordance with US EPA approved methodologies, and GLP procedures. If site-specific or project-specific QA Plans and/or QC protocols are required, these will be generated as needed.

An essential element of the QA program is record keeping and archiving all information pertaining to quality assurance including QA/QC data, pre-award check sample results, performance test sample results, scores, and follow-up; state certifications of the laboratory; external and internal audits with resolution of EPA and other audit team comments, recommendations and reports. The QA Director also plays an important role in the corrective action mechanism described in Section 16.

In addition, the QA Director works with laboratory staff and management to continuously upgrade procedures and systems to improve the laboratory's efficiency and data quality.

Ultimately, the success of the QA program depends on the cooperation and support of the entire organization. Spectrum Analytical, Inc. RI Division's most valuable resource is its staff of dedicated professionals who take personal pride in the quality of their performance.

Laboratory management works to ensure the competence of all who operate equipment, perform tests and calibrations, evaluate data and sign reports. When employees are in training, appropriate supervision will be provided until the employee has demonstrated the appropriate level of understanding, training, and skill.

Spectrum Analytical, Inc. RI Division's personnel job descriptions:

Responsibilities of each staff area in the laboratory include:

Sample Custodian:

- Receiving samples.
- Inspecting and documenting condition of sample shipping containers and samples.
- Signing documents shipped with samples.
- Verifying and recording agreement or non-agreement of information on sample documents.
- Initiating the paper work and establishing sample workorder files.
- Label samples with laboratory sample identification numbers and cross-referencing laboratory numbers to client numbers and sample tag numbers.
- Scanning samples into the IntCOC system.

- Placing samples and spent samples into appropriate storage and/or secure areas.
- Promptly alerting Department Manager and/or Project Managers to problems or anomalies that may adversely impact data quality.
- Routine housekeeping duties for the Log-in area.

Technician / Preparation Laboratory Areas:

- Analysis of samples through compliance with all aspects of formally approved analytical methods and laboratory SOPs.
- Carefully documenting each step of the analytical process.
- Noting in the appropriate logbook area any unusual occurrences or sample matrix problems.
- Conscientiously obtaining peer review as required.
- Promptly alerting laboratory supervisor, Department Managers and/or QA staff members to problems or anomalies that may adversely impact data quality.
- Routine housekeeping duties for their laboratory area.

Analyst / Instrument Laboratory Areas:

- Analysis of samples through compliance with all aspects of formally approved analytical methods and laboratory SOPs.
- Routine maintenance of instrumentation.
- Preparation of analytical standards and spiking solutions which are documented and traceable to their original source.
- Carefully documenting each step of the analytical process.
- Noting in the appropriate logbook area any unusual occurrences or sample matrix problems.
- Conscientiously obtaining peer and Department Manager review as required.
- Promptly alerting the Department Manager and/or QA staff members to problems or anomalies that may adversely impact data quality.
- Documenting the initial review of analysis data to determine compliance with established company QA/QC protocols and any project-specific QA criteria, and noting any unusual occurrences or discrepancies on the data review checklist.
- Routine housekeeping duties for their laboratory area.

Data Reporting Specialists:

- Assemble CLP-format data reports by organizing data report forms and raw data in proper order to allow for technical data review.
- Enter data into LIMS or other data reporting computer programs, and print report forms as appropriate.
- Provide non-technical typographical review of data entered into computer systems by other individuals.
- Deliver data reports to customers by FAX or electronic mail.

- Paginate, photocopy, scan, save to CD (bookmark if required) and archive copies of customer reports or other documentation to be retained by the laboratory, or prepare paperless reports.
- Ship, or organize for courier delivery, final data reports to customers.
- Assist the QA Director in management of the document control system.
- Assist Project managers with bottle order requests and shipment of coolers.
- Assist Deputy Director for Quality Services in other tasks as required.

Department Manager/Supervisors:

- Oversight of supervisors (where applicable), technicians and/or analysts in their laboratory areas.
- Monitors the status of all work in their laboratory area to insure compliance with holding time and turnaround time requirements.
- Training new scientists in the appropriate procedures and methods in the laboratory.
- Works with Laboratory Director and the QA staff to review, revise and implement SOPs.
- Insures adequate resources to perform the needed tasks by working with administrative personnel to order needed supplies.
- Insures all supplies and reagents meet the QC requirements of their intended task prior to their use in the laboratory.
- Insures all staff are using proper safety protocols.
- Works with Laboratory Director on the annual review of personnel performance.
- Interviews prospective new employees to insure they have the minimal level of qualifications, experience, education and skills necessary to perform their tasks, as well as the appropriate work ethic and social skills necessary for proper teamwork and productivity.
- Review of analytical data to insure compliance with method/SOP requirements prior to release to the client.
- Documents any non-compliance or other unusual occurrences noted during sample analysis and data review such that these can be included in the report narrative and explained to the client.

Data Reviewer:

- Review of analytical data to insure compliance with method/SOP requirements prior to release to the client.
- Documents any non-compliance or other unusual occurrences noted during sample analysis and data review such that these can be included in the report narrative and explained to the client.
- Assist Laboratory Director, Deputy Director for Quality Services and Department Managers in other tasks as required.

Laboratory Director:

- Works with Department Managers to coordinate laboratory areas in the completion of analytical projects.
- Review of analytical data to insure compliance with method/SOP requirements prior to release to the client.
- Works with QA Director to implement new SOPs and to annually review and revise existing SOPs.
- Works with the QA Director, Department Managers and Supervisors to develop and implement corrective action when needed.
- Works with management and supervisory staff to continuously improve the quality and efficiency of all company procedures.
- Assists Department Managers in the annual review of personnel performance.
- Supervises all Management, QA and Supervisory staff to insure compliance with company QA policies and other company procedures.
- Provides technical assistance to all areas of the laboratory staff.
- Acts as technical consultant for chemistry related issues that arise in the lab.
- Provides assistance with instrument optimization or performance issues as needed.
- Offers input on the purchase and operation of new instrumentation.
- Trains other analysts in procedures and methodologies.

#### Deputy Director for Quality Services:

- Works with Laboratory Director, Department Managers and Supervisors to prioritize and coordinate laboratory areas in the timely completion of analytical projects.
- Review of analytical data to insure compliance with method/SOP requirements prior to release to the client.
- Writes project report narratives to document any unusual occurrences noted during sample analysis.
- Works with management and supervisory staff to continuously improve the quality and efficiency of all company procedures.
- Works with Project Management and Data Reporting staff to continuously improve the quality and efficiency of all company procedures.
- Works with clients to insure all questions and concerns are addressed and answered.
- Works with clients to insure their understanding of complex technical issues.
- Assists Laboratory Director and Department Managers in the annual review of personnel performance.

#### Project Manager:

- Works with the client to completely understand the requirements of all incoming work.

- Evaluates the client's requirements as compared to the abilities of the laboratory as stated in Standard Operating Procedure (SOP) #110.0023 Project Management.
- Works with the Data Reporting staff to continuously improve the quality and efficiency of all company procedures.
- Communicates the customer's requirements to all laboratory staff working on the project.
- Works with the customer to determine the number and type of sample containers required for the project.
- Works with the Sample Custodian to resolve and communicate to the client any problem or discrepancies with incoming samples.
- Maintains open, responsive and continuous communication with the customer.
- Follows up with the client to assess level of satisfaction, and insure all project goals have been accomplished.
- Assist Deputy Director for Quality Services in other tasks as required.
- Act as Document Control Officer for EPA SOM/ISM contract in organizing, assembling and shipping the complete SDG file in both hardcopy and PDF formats.

Information Technology Director:

- Oversees the operations of the three divisions of Spectrum Analytical, Inc. (MA, FL and RI). The IT Director's role is technical guidance, IT long term planning, coordination/communication between the divisions, oversees and makes the necessary decision to support the overall IT function.

Information Technology Manager:

Primary function is to oversee the operations of the Spectrum Analytical, Inc. RI Division's IT department.

- Oversee the operations of the network, including servers and workstations.
- Plan for hardware and software updates
  - 1) Support users IT needs.
  - 2) Support client IT needs.
  - 3) Oversee security of network
- Development and expansion of LIMS.
  - 1) Program new functionality into LIMS including program based protocols requirements
  - 2) hard copy reports
  - 3) electronic reports
  - 4) processing of data to web site
  - 5) tracking of data
  - 6) maintenance of LIMS
  - 7) security of LIMS
- Generate and troubleshoot more complex EDDs

- Provide backup for the Information Technology Specialist when out and support when it is needed.

Secondary function is to work with the other divisions to try and make transfer of information as seamless as possible. Lend technical support to other divisions and help to bring technical help from other divisions to Spectrum Analytical, Inc. RI Division IT department.

#### Information Technology Specialist:

- Primary duty is to generate and review EDDs using EDD module.
  - a) Generate and validate EDDs using EDD specific tools (CRA, Tetra Tech, CH2M Hill, etc...).
  - b) Generate all SEDD files for the EPA SOM contract, and work with the chemists to resolve any defects, if possible.
- Perform server room duties.
  - a) Monitor the servers and troubleshoot (if needed)
  - b) Perform backup/archive of data on servers
  - c) File grooming at the end of the month
  - d) Monitor event logs of the servers for issues.
  - e) Monitor status of centralized anti-viral program (AVG). Includes monitoring AVG status of workstations
  - f) Monitor centralized Windows System Update Server (WSUS). Includes monitoring WSUS status of workstations.
- Handles user issues with printer/scanner/copier systems from Ikon. Based on evaluation, schedule service calls or replace consumable parts.

#### Quality Assurance Director:

- Implements the entire QA program.
- Interacts on a daily basis with laboratory staff.
- Evaluates compliance with the QA program through formal and informal reviews of data and processes.
- Implements the corrective action system.
- Maintains a master list of all SOPs and monitors review schedules.
- Works with Department Managers and Supervisors to implement new SOPs and to annually review and revise existing SOPs.
- Controls all master and controlled-copies of SOPs and QAP as per SOP #80.0012; Production of Standard Operating Procedures.
- Distributes and archives all new and edited revisions of SOPs and QA manual as per SOP# 80.0012; Production of Standard Operating Procedures.
- Interfaces with certification authorities and agencies to maintain existing certifications and programs, and obtain new certifications.
- Maintains records of employee training and certification as per SOP# 80.0016; Training Procedures and Tracking.
- Instructs laboratory personnel on ethics in the workplace.

- Oversees analytical trends that need to be evaluated and corrected.
- Oversees the implementation of MDLs and control limit studies.
- Directs the internal audit program as per SOP# 80.0006; Internal Audits.
- Coordinates all external audit corrective action reports and investigations.
- Maintains certification of NIST thermometers and weights.
- Schedules annual hood inspections and balance calibrations.

In Spectrum Analytical, Inc. RI Division's organizational structure, the Laboratory Director has the ultimate authority for all chemistry-related aspects of the company.

The QA Director reports directly to the Laboratory Director. She has the authority within the management system to bring any issue to the highest levels of the company management and ownership, as well as to halt the release of data she believes to be questionable or suspend the performance of an analysis she believes to be unreliable.

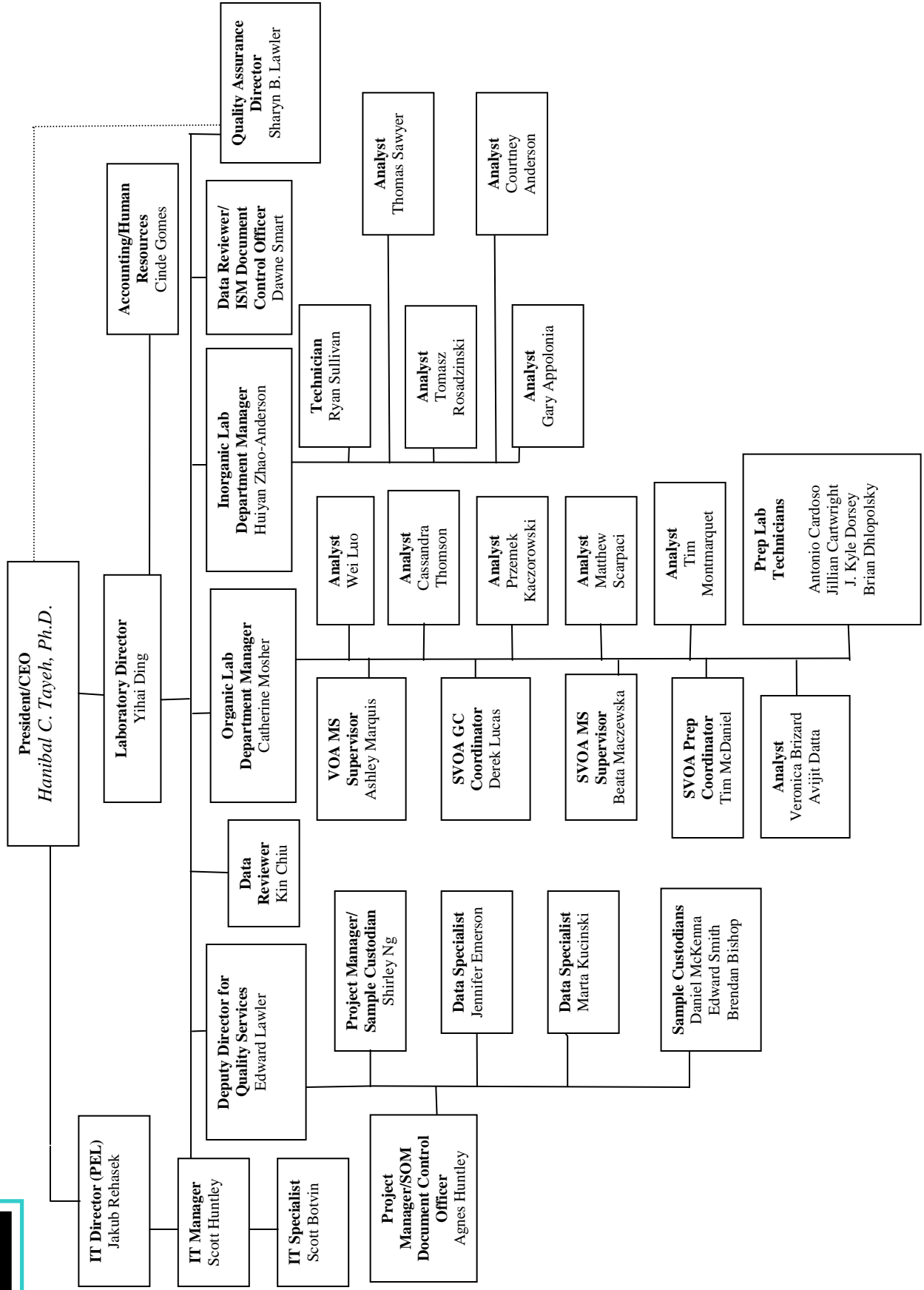
The Deputy Director for Quality Services works with the project management and marketing staff and with the Department Managers and Supervisors to prioritize and coordinate work within the laboratories.

The personnel training records are located in the QA department on-site as well as additional training documents being saved in pdf form on the Spectrum network. All individual training is documented including new employee training, individual training, annual retraining procedures, and Health and Safety training.

Figure 5-1  
SPECTRUM ANALYTICAL, INC. RI Division's Organizational Chart



# Organizational Chart



## 6.0 QUALITY ASSURANCE OBJECTIVES FOR MEASUREMENT DATA IN TERMS OF PRECISION, ACCURACY, REPRESENTATION, COMPLETENESS AND COMPARABILITY AND QA REPORTING

As part of the evaluation component of the overall QA Program, laboratory results are compared with the data quality indicators defined as follows:

- Precision: the agreement of reproducibility among individual measurements of the same property usually made under identical conditions.
- Accuracy: the degree of agreement of a measurement with the true or accepted value.
- Representation: the degree to which data accurately and precisely represent a characteristic of a population, parameter variations of a sample of a finite process condition, or of a finite environmental condition.
- Completeness: a measure of the amount of valid data obtained from a measurement system compared with the amount that was expected to be obtained under normal conditions.
- Comparability: an expression of the confidence with which one laboratory data set can be compared with another laboratory data set in regard to the same property and laboratory sample population.

Quality Assurance objectives may vary by project and requested parameters. The accuracy, precision, and representation of data will be functions of the origins of the sample material, the procedures used to analyze sample and generate data, and the specific sample matrices involved in each project. Quality control practices utilized in the evaluation of these data quality indicators include blanks, replicates, spikes, standards, check samples, calibrations and surrogates. The process for quantifying or assessing the above indicators for data quality is addressed in Section 15.

### 6.1 Precision and Accuracy:

For each parameter analyzed, the QA objectives for precision and accuracy will be determined from:

- Published historical data;
- Method validation studies;
- Spectrum Analytical, Inc. RI Division's experience with similar samples and/or;
- Project-specific requirements, such as those stipulated by the USEPA in the CLP protocols and control documents.

## 6.2 Representation:

Analytical data should represent the sample analyzed regardless of the heterogeneity of the original sample matrix. In most cases, representation is achieved by mixing the laboratory sample well before removing a portion for analysis. On occasion, multi-phase laboratory samples may require that each phase be analyzed individually and reported in relation to its proportion in the whole sample.

## 6.3 Completeness:

The completeness goal is 100% in all cases and includes:

- Analysis of all samples;
- Generation and analysis of all required QC samples;
- Sufficient documentation of associated calibration, tuning and standardization;
- Records of data reduction processes, including manual calculations.

While the laboratory staff is responsible for achieving the completeness objective stated above, assigning each project a specific project manager whose functions include sample management and tracking ensures completeness.

## 6.4 Comparability:

To assure comparability, Spectrum Analytical, Inc. RI Division employs established and approved analytical methods (e.g. USEPA protocols), consistent analytical bases (dry weight, volume, etc.) and consistent reporting units (mg/Kg, µg/L, etc.). Where data from different samples must be comparable, the same sample preparation and analysis protocols are used for all of the samples of interest.

## 6.5 QA Reporting

General QA procedures require that an MS/MSD or DUPLICATE/MS be reported with each sample batch up to 20 samples. In addition, each batch requires a method blank (MB) and laboratory control sample (LCS).

An acceptance criterion for the MB depends upon the method criteria. In-house control limits dictate the acceptability of the LCS in many methods. Several methods have set LCS control limits. A high bias LCS is considered acceptable if the analyte is not present in the samples above the reporting limit. A low bias LCS will require re-extraction (if sample volume allows) and re-analysis.

DUP, MS, and MSD recoveries and calculated RPD are specified in the analytical methods. Recoveries outside the limits require some form of corrective action,

whether that includes a post-digestion/distillation spike, re-extraction, re-analysis and/or notification to the client in the project narrative.

LIMS will flag any QA samples outside method criteria on the reporting forms. Formal written corrective action reports are required for any incident that does not meet method criteria and cannot be remedied or explained by the laboratory. The QA Officer signs off on any corrective actions and can also track QA trends in this manner.

## 7.0 SAMPLING PROCEDURES

For most projects, outside sampling teams deliver or send samples to Spectrum Analytical, Inc. RI Division's. When sampling by Spectrum Analytical, Inc. RI Division's personnel is required, the sampling team follows the sampling procedures outlined in the EPA *Test Methods for Evaluating Solid Wastes*, SW-846, 3<sup>rd</sup> Edition, or procedures found in the EPA "Handbook for Sampling and Sample Preservation of Water and Wastewater".

Appropriately prepared sample containers are supplied by Spectrum Analytical Inc., RI Division at clients' request. When required, preservatives are added to the sample containers. Tables 7-1 through 7-3 provide the Spectrum Analytical Inc., RI Division Recommended Container, Preservation Techniques and Holding Times. Additional sample volumes may be required if additional QC functions are to be performed.

Holding times for SW846, CLP Methods, Standard Methods and certain USEPA methods are different and are presented in Tables 7-1 to 7-3. Holding times for most methods are calculated from the date of sample collection. Holding times for CLP methods are calculated from the Validated Time of Sample Receipt (VTSR). It should be noted that the CLP analysis program combines chemical analyses and contract compliance procedures in one document. For laboratory analysis and contract compliance purposes, holding times are calculated from VTSR, while post-analysis data usability and validation (generally performed by the client or a third party) compares holding times to the SW-846 method holding times calculated from date of sample collection.

Representative portions of samples are taken for analysis by following Spectrum Analytical, Inc. RI Division's SOP #110.0039 Standard Operating Procedure for Sub-Sampling.

Table 7-1

Recommended Container, Preservation Techniques and Holding Times  
For  
SW-846 Analyses

<u>Analytes</u>	<u>Method</u>	<u>Containers</u>	<u>Required* Volume</u>	<u>Preservation</u>	<u>Holding Times</u>
Volatiles Organics					
Solid	8260, 5030	Amber glass jar with Teflon lining	Minimal head-space in jar	4°C	14 days
Solid <sup>a</sup>	8260, 5035	40mL vial or Encore with Teflon lining	5.0gram ± 0.5	4°C, unpreserved 48 hours	
				DI Water -10 to -20°C	14 days
				Sodium bisulfate -10 to -20°C, 4°C	14 days
				Methanol 4°C	14 days
Aqueous	8260, 5030	40mL VOA Vials with Teflon septum	40mL	4°C HCl, pH<2	14 days
Semivolatile Organics					
Solid	3540, 3550 8270	Amber glass jar with Teflon lining	30gram	4°C	Extraction within 14 days Analysis within 40 days
Aqueous	3510, 3520 8270	Amber glass bottles with Teflon lining	1L	4°C	Extraction within 7 days Analysis within 40 days
Polychlorinated Biphenyls					
Solid	3540, 3550 8082	Amber glass jar with Teflon lining	30gram	4°C	Extraction within 14 days Analysis within 40 days
Aqueous	3510, 3520 8082	Amber glass bottle with Teflon lining	1L	4°C	Extraction within 7 days Analysis within 40 days
Organochlorine Pesticides					
Solid	3540, 3550 8081	Amber glass jar with Teflon lining	30gram	4°C	Extraction within 14 days Analysis within 40 days
Aqueous	3510, 3520 8081	Amber glass bottle with Teflon lining	1L	4°C	Extraction within 7 days Analysis within 40 days
Chlorinated Herbicides					
Solid	8151	Amber glass jar with Teflon lining	30gram	4°C	Extraction within 14 days Analysis within 40 days
Aqueous	8151	Amber glass bottle with Teflon lining	1L	4°C	Extraction within 7 days Analysis within 40 days

Table 7-1 (cont'd)

Recommended Containers, Preservation Techniques and Holding Times  
For  
SW846 Analyses

<u>Analytes</u>	<u>Method</u>	<u>Containers</u>	<u>Required* Volume</u>	<u>Preservation</u>	<u>Holding Times</u>
Total Petroleum Hydrocarbons					
Gasoline Range Organics, including Maine-GRO**					
Solid	8015, 5030 ME 4.1.17	Amber glass jar With Teflon lining	Minimal head- space in jar	4°C	14 days
Solid <sup>a</sup>	8015, 5035	40mL vial or Encore with Teflon lining	5.0gram ± 0.5	4°C, unpreserved	48 hours
				4°C, Methanol	14days
Aqueous	8015, 5030 ME 4.1.17	40mL VOA vials With Teflon septum	40mL	4°C HCl, pH<2	14 days
Diesel Range Organics, including Maine-DRO					
Solid	3540, 3550 8015 ME 4.1.25	Amber glass jar with Teflon lining	30gram	4°C	Extraction within 14 days Analysis within 40 days
Aqueous	3510, 3520 8015 ME 4.1.25	Amber glass bottle with Teflon lining	1L	4°C H <sub>2</sub> SO <sub>4</sub> , pH<2	Extraction within 7 days Analysis within 40 days
Total Metals except Mercury and Chromium (VI)					
Solid	3050 6010	Amber glass jar with Teflon lining	10g	4°C	180 days
Aqueous	3005, 3010	Polyethylene bottle	100mL	HNO <sub>3</sub> , pH<2	180 days
Chromium (VI)					
Solid	3060, 7196	Amber glass jar with Teflon lining	10g	4°C	Digestion within 30 days Analysis within 96 hours
Aqueous	7196	Polyethylene bottle	25mL	4°C	24 hours
Mercury					
Solid	7471	Amber glass jar	10g	4°C	28 days
Aqueous	7470	Polyethylene bottle	100mL	4°C HNO <sub>3</sub> , pH<2	28 days
Cyanide					
Solid	9012	Amber glass jar with Teflon lining	10g	4°C	14 days
Aqueous	9012	Polyethylene bottle	50mL	4°C NaOH, pH≥12	14 days
Flashpoint					
Aqueous	1010	Amber glass bottle	30mL	4°C	28 days

Table 7-1 (cont'd)

Recommended Containers, Preservation Techniques and Holding Times  
For  
SW846 Analyses

<u>Analytes</u>	<u>Method</u>	<u>Containers</u>	<u>Required* Volume</u>	<u>Preservation</u>	<u>Holding Times</u>
Chloride					
Aqueous	9056	Polyethylene bottle	50mL	4°C	28 days
Nitrate					
Aqueous	9056	Polyethylene bottle	50mL	4°C	48 hours
Nitrite					
Aqueous	9056	Polyethylene bottle	50mL	4°C	48 hours
Orthophosphate					
Aqueous	9056	Polyethylene bottle	50mL	4°C	48 hours
Sulfates					
Aqueous	9056	Polyethylene bottle	50mL	4°C	28 days

Table 7-2

Recommended Container, Preservation Techniques and Holding Times  
For  
CLP/ASP Analyses

<u>Analytes</u>	<u>Method</u>	<u>Containers</u>	<u>Required* Volume</u>	<u>Preservation</u>	<u>Holding Times</u>	
Volatile Organics						
	Solid	CLP/ASP	Amber glass jar with Teflon lining	Minimal head- space in jar	4°C	10 days from VTSR
	Aqueous	CLP/ASP	40mL VOA vials with Teflon septum	40mL	4°C HCl, pH<2	10 days from VTSR
		CLP Low	40mL VOA vials with Teflon septum	40mL	4°C HCl, pH<2	10 days from VTSR
Semivolatile Organics						
	Solid	CLP/ASP	Amber glass jar with Teflon lining	30gram	4°C	10 days from VTSR Analysis within 40 days
	Aqueous	CLP/ASP	Amber glass bottle with Teflon lining	1L	4°C	5 days from VTSR Analysis within 40 days
		CLP Low	Amber glass bottle with Teflon lining	1L	4°C	5 days from VTSR Analysis within 40 days
Organochlorine Pesticide/PCB						
	Solid	CLP/ASP	Amber glass jar with Teflon lining	30gram	4°C	10 days from VTSR Analysis with 40 days
	Aqueous	CLP/ASP	Amber glass bottle with Teflon lining	1L	4°C	5 days from VTSR Analysis within 40 days
		CLP Low	Amber glass bottle with Teflon lining	1L	4°C	5 days from VTSR Analysis within 40 days
Cyanide						
	Solid	CLP/ASP	Amber glass jar	10gram	4°C	12 days from VTSR
	Aqueous	CLP/ASP	Polyethylene bottle	50mL	4°C NaOH, pH>12	12 days from VTSR
Total Metals except Mercury						
	Solid	CLP/ASP	Amber glass jar	10gram	4°C	180 days from VTSR
	Aqueous	CLP/ASP	Polyethylene bottle	100mL HNO <sub>3</sub> , pH<2	4°C	180 days from VTSR

Table 7-2 (cont'd)

Recommended Container, Preservation Techniques and Holding Times  
For  
CLP/ASP Analyses

<u>Analytes</u>	<u>Method</u>	<u>Containers</u>	<u>Required* Volume</u>	<u>Preservation</u>	<u>Holding Times</u>
Mercury					
Solid	CLP/ASP	Amber glass jar	10gram	4°C	26 days from VTSR
Aqueous	CLP/ASP	Polyethylene bottle	100mL	4°C HNO <sub>3</sub> , pH<2	26 days from VTSR

Table 7-3

Recommended Containers, Preservation Techniques and Holding Times  
for  
Other Analyses

<u>Analytes</u>	<u>Method</u>	<u>Containers</u>	<u>Required* Volume</u>	<u>Preservation</u>	<u>Holding Times</u>
Volatile Organics Aqueous	624	40mL VOA vials with Teflon septum	40mL	4°C HCl, pH<2	14 days
Semivolatile Organics Aqueous	3510, 3520 625	Amber glass bottle with Teflon lining	1L	4°C	Extraction within 7 days Analysis within 40 days
Organochlorine Pesticide/PCB Aqueous	3510, 3520 608	Amber glass bottle with Teflon lining	1L	4°C	Extraction within 7 days Analysis within 40 days
EDB/DBCP Aqueous	8011	40mL VOA vials with Teflon septum	35mL	4°C HCl, pH<2	28 days
MA Extractable Petroleum Hydrocarbons (EPH) Solid	3540, 3550 MADEP	Amber glass jar with Teflon lining	10gram	4°C	Extraction within 7 days Analysis within 40 days
Aqueous	3510, 3520 MADEP	Amber glass bottle with Teflon lining	1L	4°C HCl, pH<2	Extraction within 14 days Analysis within 40 days
MA Volatile Petroleum Hydrocarbons (VPH) Solid	MADEP	Amber glass jar with Teflon lining	10gram	4°C 10mL Methanol	14 days
Aqueous	MADEP	40mL VOA vial with Teflon lining	40mL	4°C HCl, pH<2	14 days
Oil & Grease Aqueous	1664	Amber glass bottle with Teflon lining	1L	4°C HCl, pH<2	28 days
Alkalinity Aqueous	SM2320B	Polyethylene bottle	100mL	4°C	14 days
Ammonia Aqueous	SM4500NH3B	Polyethylene bottle	100mL	4°C H <sub>2</sub> SO <sub>4</sub> , pH<2	28 days
Chloride Aqueous	SM4500 CL E	Polyethylene bottle	100mL	4°C	28 days

Table 7-3 (cont'd)

Recommended Containers, Preservation Techniques and Holding Times  
for  
Other Analyses

<u>Analytes</u>	<u>Method</u>	<u>Containers</u>	<u>Required Volume</u>	<u>Preservation</u>	<u>Holding Times</u>
Chloride	E300.0	Polyethylene bottle	50mL	4°C	28 days
COD					
Aqueous	SM5220D	Amber VOA vial	40mL	4°C H <sub>2</sub> SO <sub>4</sub> , pH<2	28 days
Color					
Aqueous	SM2120B	Polyethylene bottle	50mL	4°C	Immediate
Nitrate/Nitrite					
Aqueous	E353.2	Polyethylene bottle	50mL	4°C H <sub>2</sub> SO <sub>4</sub> , pH<2	28 days
Nitrate					
Aqueous	E300.0	Polyethylene bottle	50mL	4°C	48 hours
Nitrite					
Aqueous	E300.0	Polyethylene bottle	50mL	4°C	48 hours
Orthophosphate					
Aqueous	SM4500-P, E E300.0	Polyethylene bottle	50mL	4°C	48 hours
Total phosphate					
Aqueous	SM4500-P B5,E	Polyethylene bottle	50mL 50mL	4°C H <sub>2</sub> SO <sub>4</sub> , pH<2	28 days
Phenols					
Aqueous	SM5530B E420.1	glass	250mL	4°C H <sub>2</sub> SO <sub>4</sub> , pH<2	28 days
Sulfates					
Aqueous	SM426 15 <sup>th</sup> Ed. E300.0	Polyethylene bottle	50mL	4°C	28 days
Sulfide					
Total					
Aqueous	SM4500-S-D	Polyethylene bottle	50mL	4°C NaOH, pH>12 ZnAc	28 days
Reactivity					
Solid	Chapter 7 SW846	Amber glass jar	10gram	4°C	28 days
Aqueous	Chapter 7	Polyethylene bottle	250mL	4°C	28 days
Total Organic Carbon (TOC)					
Solid	Lloyd Kahn Walkley-Black	Amber glass jar	10g	4°C	14 days

Table 7-3 (cont'd)

Recommended Containers, Preservation Techniques and Holding Times  
For  
Other Analyses

<u>Analytes</u>	<u>Method</u>	<u>Containers</u>	<u>Required* Volume</u>	<u>Preservation</u>	<u>Holding Times</u>
Total Organic Carbon					
Aqueous	SM5310B	40mL VOA vials	40mL	4°C H <sub>3</sub> PO <sub>4</sub> , pH<2	28 days
TKN					
Aqueous	SM4500Norg C	Polyethylene bottle or Amber glass bottle	50mL	4°C H <sub>2</sub> SO <sub>4</sub> , pH<2	28 days
Total Solids (TS)					
Aqueous	SM2540B	Polyethylene bottle	200mL	4°C	7 days
Total Dissolved Solids (TDS)					
Aqueous	SM2540C	Polyethylene bottle	200mL	4°C	7 days
Total Suspended Solids (TSS)					
Aqueous	SM2540D	Polyethylene bottle	200mL	4°C	7 days
Settleable Solids					
Aqueous	SM2540F	Polyethylene bottle	200mL	4°C	48 hours
Chromium (VI)					
Aqueous	SM3500	Polyethylene bottle	25mL	4°C	24 hours

\* These represent minimum required volume. Additional sample volumes should be collected to minimize headspace loss for volatile analysis. Additional sample aliquot are also required to perform QA/QC functions (e.g. spikes, duplicates), % moisture for solid samples and sample re-analysis (if needed).

<sup>a</sup> For Massachusetts analyses, the Volatile Organics soil samples are preserved in Methanol in the field.

EPA SW-846 Method 5035 provides several options for preservation of soil samples for volatile organics. Certain state jurisdictions (NY for example) have not adopted these options to-date, and continue to recommend the collection of unpreserved soil sample aliquots for volatiles analysis. Spectrum Analytical Inc., RI Division's preference for low-level analysis is to collect approximately 5 grams of soil into 5mL of organic-free DI water and to preserve by freezing within 48hours of collection. A separate container with approximately 5 grams of soil into 5mL of methanol is also collected for potential medium-level analysis. A separate container of unpreserved soil also must be collected to perform percent moisture analysis.

\*\* Maine GRO soil analysis requires a medium level methanol extraction. A 10 gram sample and 10mL methanol volume is used.

## 8.0 SAMPLE CUSTODY

### 8.1 Chain of Custody:

Samples are physical evidence collected from a facility or the environment. In hazardous waste investigations, sample data may be used as evidence in (EPA) enforcement proceedings. In support of potential litigation, laboratory chain-of-custody procedures have been established to ensure sample traceability from time of receipt through the disposal of the sample.

A sample is considered to be in the custody under the following conditions:

- It is in an authorized person's actual possession, or
- It is in an authorized person's view, after being in that person's physical possession, or
- It was in an authorized person's possession and then was locked or sealed to prevent tampering, or
- It is in a secure area.

Chain-of-custody originates as samples are collected. Chain-of-custody documentation accompanies the samples as they are moved from the field to the laboratory with shipping information and appropriate signatures indicating custody changes along the way.

Laboratory chain-of-custody is initiated as samples are received and signed for by the Sample Custodian or his/her designated representative at Spectrum Analytical, Inc. RI Division. Documentation of sample location continues as samples are signed in and out of the designated storage facility for analysis in the several laboratory departments, using the Internal Chain of Custody (IntCOC) barcode system. After analysis, any remaining sample is held in the designated storage area to await disposal. Spectrum Analytical, Inc. RI Division's policy is to hold spent samples for a period of at least thirty days from submittal of final report, unless other arrangements are agreed upon with the client. USEPA samples and empty containers are held for 60 days. Refer to SOP #30.0030 ICOC for details on using the IntCOC barcode system and program.

### 8.2 Laboratory Security:

Samples and all data generated from the analyses of samples at Spectrum Analytical, Inc. RI Division are kept within secure areas during all stages of residence, including the periods of time spent in preparation for analysis, while undergoing analysis, and while in storage.

The entire laboratory is designated as a secure area. The doors to the laboratory are under continuous surveillance, are kept locked after regular business hours

and may only be accessed by key or keypad entry. Only authorized personnel are allowed to enter the secure areas. The central laboratory facility and IT office are only accessed through keypad entry. A Spectrum Analytical, Inc. RI Division staff member must accompany visitors to the laboratory. Refer to SOP #110.0012 Laboratory Security for further detail.

### 8.3 Duties and Responsibilities of Sample Custodian:

Duties and responsibilities of the Sample Custodian include:

- 8.3.1 Receiving samples.
- 8.3.2 Inspecting and documenting sample shipping containers for presence/absence and condition of:
  - 8.3.2.1 Custody seals, locks, “evidence tape”, etc.;
  - 8.3.2.2 Container breakage and/or container integrity, including air space in aqueous samples, or proper preservation for soil samples for Volatiles analysis.
- 8.3.3 Recording condition of both shipping containers and sample containers (cooler temperature, bottles, jars, cans, etc.).
- 8.3.4 Signing documents shipped with samples (i.e. air bills, chain-of-custody record(s), Sample Management Office (SMO) Traffic Reports, etc.)
- 8.3.5 Verifying and recording agreement or non-agreement of information on sample documents (i.e. sample tags, chain-of-custody records, traffic reports, air bills, etc.). If there is non-agreement, recording the problems, contacting the project manager for direction, and notifying appropriate laboratory personnel. (Client’s corrective action directions shall be documented in the case file.)
- 8.3.6 Initiating the paper work for sample analyses on laboratory documents (including establishing sample workorder files) as required for analysis or according to laboratory standard operating procedures.
- 8.3.7 Label samples with laboratory sample identification numbers and cross-referencing laboratory numbers to client numbers and sample tag numbers.
- 8.3.8 Scanning samples into the IntCOC system.
- 8.3.9 Placing samples and spent samples into appropriate storage and/or secure areas.

- 8.3.10 Where applicable, making sure that sample tags are removed from the sample containers and included in the workorder file.
- 8.3.11 Where applicable, accounting for missing tags in a memo to the file or documenting that the sample tags are actually labels attached to sample containers or were disposed of, due to suspected contamination.
- 8.3.12 Monitoring storage conditions for proper sample preservation and prevention of cross-contamination.
- 8.3.13 Sending shipping containers with prepared sample bottles and sample instructions to clients who request them.
- 8.3.14 Calibrating the non-contact infrared temperature gun quarterly.
- 8.3.15 Disposal of samples after a specified time period determined by contract or client request.

#### 8.4 Sample Receipt:

The Sample Custodian or his/her designated representative receives sample shipments at Spectrum Analytical, Inc. RI Division. Unless the shipment is a continuation of a previous workorder, a new workorder file is started for the sample.

The cooler is inspected for the following (if applicable) and findings are documented on the Sample Login Form (Figure 8.4-1) for USEPA CLP samples, and on the Sample Condition Form (Figure 8.4-2) for all other samples:

- Custody seal (conditions and custody number)
- Air bill (courier and air bill #)

The cooler is then opened and the following items are checked (in order). Make sure the hood is turned on when the cooler is opened.

- Chain of custody (COC) records (or traffic report). These are usually taped to the inside of the cooler cover.
- Radioactivity using the Geiger counter, which continuously monitors the receiving area for radiation
- Cooler temperature using the non-contact infrared temperature gun. Record the temperature of a temperature blank if available, using a calibrated thermometer. Record each temperature on the COC.

The Sample Custodian will perform the following:

- Remove the sample containers and arrange them in the same order as documented in the chain of custody report.
- Inspect condition of the sample containers.
- Assign laboratory sample ID and cross-reference the laboratory ID to the client ID.
- Remove tags and place in the workorder file.
- Check preservative and document in the Sample Condition Form (Figure 8.4-2) if needed. If additional preservative is needed, it is added at this time.
- Check for air bubbles in aqueous samples and for proper preservation and immersion of soil samples designated for volatile organic analysis.
- Ensure peer review occurs for proper cross-referencing and labeling of sample containers.

Any discrepancies or problems are noted in the Sample Condition Notification Form (Figure 8.4-3).

The sample custodian conveys the information to the project manager who will in turn inform the client, or may directly inform the client of the discrepancies.

Samples can be rejected at Spectrum Analytical, Inc. RI Division for any of the following reasons:

1. Complete and proper documentation was not sent with the samples.
2. Sample labels cannot be identified because indelible ink was not used during the sampling procedure.
3. Hold times had already been exceeded when samples arrived at the laboratory.
4. Inadequate sample volume.
5. Potential cross-contamination has occurred among samples.
6. Samples are inadequately preserved.
7. The samples or shipping container is badly destroyed during shipping.
8. The samples are potentially radioactive.
9. The samples represent untreated fecal waste for which Spectrum Analytical, Inc. RI Division employees are currently not inoculated against.

In all instances, the client is contacted initially before any action is taken at Spectrum Analytical, Inc. RI Division.

The Sample Custodian signs the Sample Receipt Form and originates a file folder for the set of samples. The following forms are included in the file: the Sample Receipt Form, chain of custody records, shipping information, and an orange Sample Condition Notification Form if any problems or discrepancies need to be addressed.

When the Sample Custodian is not available to receive samples, another lab staff member will sign for the sample container. The time, date and name of the person receiving the container are recorded on the custody records. In addition, the cooler temperature is measured and recorded on the Sample Condition Form. The samples are then stored in the centralized walk-in refrigerator in the sample receipt area. The sample receipt area is located in the secure central storage facility of the laboratory. VOA samples are stored in the VOA analysis laboratory. The samples are officially received and documented by the Sample Custodian or designee before the next business day.

At times, samples will be sent to another lab for analysis not performed at Spectrum Analytical, Inc. RI Division. These subcontracted analyses are performed by laboratories certified to perform the analyses. The use of a subcontractor laboratory is discussed with the client prior to sending samples, per Spectrum Analytical, Inc. RI Division's Project Management Standard Operating Procedure.

These samples are packed to prevent breakage and stored in a cooler in the walk-in or stored in the small refrigerator in the central storage facility. The samples are either hand delivered to a local sub-contract lab, or shipped with sufficient coolant to maintain a 4 degree temperature by air courier under Spectrum Analytical, Inc. RI Division's chain-of-custody (Figure 8.4-4). Refer to SOP #30.0003 Sample Receipt, Storage, Tracking and Disposal for more detail.

## 8.5 Sample Log-in Identification:

### 8.5.1 Sample Identification:

To maintain sample identity, each sample received at Spectrum Analytical, Inc. RI Division is assigned unique sample identification (Sample ID) numbers. Samples are logged into the laboratory via the Omega Laboratory Information Management System (LIMS).

After inspecting the samples, the Sample Custodian logs each sample into the Omega LIMS, which assigns a lab Sample ID Number. These Numbers are assigned sequentially in chronological order. Spectrum Analytical, Inc. RI Division Sample Identification Numbers appear in the following format:

**YXXXX-NNF**

In which: Y – represents the current year with A for 2002, B for 2003, C for 2004, etc.

XXXX – represents a four-digit work order number that is assigned sequentially to each submittal of samples

NN – represents the sample number within the group or workorder.

F – represents the fraction. All sample portions that are received in identical bottles with identical preservatives are grouped into one fraction.

For example, the first fraction of the fifth sample of the 20<sup>th</sup> workorder of 2003 would have the number: B0020-05A

The Sample ID Number is recorded on the Sample Login Form (Figure 8.4-1) for USEPA CLP samples, and on the Sample Condition Form (Figure 8.4-2) for all other samples. Information on these forms cross-reference the Sample ID Numbers with SDG numbers, sample tag numbers and/or other client identifiers. Each sample is clearly labeled with its lab Sample ID Number by the Sample Custodian. The same sample ID Number appears on the LIMS status report, on each sample preparation container and extract vial associated with the sample. Refer to SOP #20.0003 Logging Workorders into Omega for further information.

#### 8.5.1.1 Sample Extract Identification:

As described in Section 8.5.1, a sample extract is identified with the same unique sample identification number as the sample from which it derives

#### 8.5.2 Sample Login:

The sample login system at Spectrum Analytical, Inc. RI Division consists of computerized entry using Omega LIMS (Figure 8.5-1). The information recorded onto the Workorder Report includes:

- Workorder number
- Client name
- Project name and location
- Final data report format
- Date of receipt
- Date sample collected
- Due date, fax and/or hardcopy
- EDD requirements
- Comments or notes on the workorder
- Lab Sample Identification numbers
- Client Sample Identification numbers
- Sample matrix
- Analyses required
- Case number, where used by the client
- SDG number, where used by the client

#### 8.5.3 Sample Information:

After sample information is properly recorded and the samples have been properly logged into the LIMS, bottle labels are generated and applied to the sample containers. The Sample Custodian notifies the Project Manager or peer or supervisor to review the sample bottle labeling. This person reviews all the information associated with the samples. He/she verifies (by initialing) the correctness of the information on the Sample Condition Form or Sample Log-In Form. Sample login information is available through the Omega LIMS to all appropriate laboratory staff. The Sample Custodian then scans the samples into the IntCOC system and posts the samples.

The Sample Custodian initiates a red workorder file. This file contains the original Sample Log-In Form or Sample Condition Form, air bills, SMO traffic reports, sample tags, workorder reports and all correspondence with the Client or SMO or others. The red workorder file is forwarded to the Project Manager for review of the login paperwork, and for updating status of the workorder in the LIMS. Once the login information is thoroughly reviewed for correctness, the red workorder file is stored in the data reporting area. Analytical data are placed in this as analyses are completed and data are reviewed.

#### 8.6 Sample Storage and Disposal:

Samples at Spectrum Analytical, Inc. RI Division are stored in a central storage facility or in satellite designated areas, (see SOP #30.0003 Sample Receipt, Storage, Tracking and Disposal). After sample receipt and login procedures are completed, the Sample Custodian places the samples in the centralized walk-in refrigerator. Volatile Organic sample aliquots are released to the volatile organic lab with documentation (Figure 8.6-1).

The central storage facility is for samples only; no standards or reagents are to be stored there. Access to the centralized sample storage facility is limited by keypad entry at all times. All sample storage areas are within the secure laboratory facility.

All sample/extract refrigerators are maintained at  $4^{\circ}\text{C} \pm 2^{\circ}\text{C}$ . Standards are kept in freezers maintained at  $-10$  to  $-20^{\circ}\text{C}$ . The temperature is recorded electronically using temperature probes that are affixed inside all refrigerator and freezer units (see SOP #80.0020 Temperature Monitoring Systems).

When analysis is complete, any remaining sample is retained in the designated storage facility until it may be removed for disposal. Broken and damaged samples are promptly disposed in a safe manner. Unless there is a specific request by the client, excess, unused sample aliquots are stored for at least 30 days after the submission of compliant data (USEPA is 60 days for samples and empty containers). The samples are then disposed after such period. USEPA and NYS

ASP extracts are stored under refrigeration for at least one year. Other extracts are stored under refrigeration for up to three months, unless there is a specific agreement with the client. After such time, the extracts are disposed. All disposals are performed in a manner compliant with federal and state regulations. International samples require special disposal procedures associated with the USDA Soil Permit. (see SOP #30.0024 Sample Disposal for more details).

#### 8.6.1 Extract Transfer:

The extracts generated during the preparation for the organic analyses are transferred from the Organic Prep Lab to the Analysis Labs. The transfer of extracts for Semivolatiles, TPH, Pesticides and PCBs, are documented electronically in the Prep Batch Log with the storage location (refrigerator ID).

Metals analysis samples that are transferred from the prep area to the analysis room are also documented in the Prep Batch Log with the storage location (ICP or Hg lab).

There is no extract transfer that occurs with either Wet Chemistry or VOA samples.

#### 8.6.2 Extract Storage:

Semivolatile, Pesticide/PCB, and TPH extracts, which are contained in crimp top vials or screw cap vials with Teflon lined septa, are stored at  $4^{\circ}\text{C} \pm 2^{\circ}\text{C}$ . Semivolatile and Pesticide/PCB extracts are stored in refrigerators in the Semivolatiles Analysis room. They are catalogued numerically by workorder number that approximates chronological order, according to date of receipt. USEPA CLP extracts are stored separately within the refrigerator from sample extracts of other clients.

Excess Pesticide extracts, not analyzed, are stored in screw cap vials with Teflon lined septa in the Organic Prep Lab. In most instances, they consist of the remaining 8-9 mL aqueous and soil sample extracts and are stored chronologically by workorder.

#### 8.7 Sample Tracking:

When a sample is removed from storage, the analyst must scan each jar or bottle taken, using the IntCOC program and their user ID. When the sample(s) are returned to the central storage facility, the analyst must scan the samples back into the system using the IntCOC program and their user ID, and return the physical samples to their original storage location. In addition to the individual's initials, the date and time is recorded. This system maintains the location of the sample at any point in time.

Chain-of-custody of a sample ensures that the sample is traceable from the field, where it was taken, through laboratory receipt, preparation, analysis and finally disposal. The primary chain-of-custody documents are used to locate a sample at any point in time.

1. The chain-of-custody form from the field describes the origin and transportation of a sample;
2. The IntCOC document acceptance of a sample by Spectrum Analytical Inc., RI Division; and
3. The IntCOC documents which analyst has custody of the sample after removal from storage.
4. The sample preparation batch log document when the extracts or digestates were received by the analytical labs and where they are stored in the refrigerator.

Figure 8.4-1  
USEPA CLP Sample Login Form

SAMPLE LOG-IN SHEET  
FORM DC-1

Lab Name				Page ___ of ___	
Received By (Print Name)				Log-in Date	
Received By (Signature)					
Case Number		Sample Delivery Group No.			Mod. Ref. No.
Remarks:		Corresponding			Remarks: Condition of Sample Shipment, etc.
		EPA Sample #	Sample Tag #	Assigned Lab #	
1. Custody Seal(s)	Present/Absent* Intact/Broken				
2. Custody Seal Nos.	_____				
3. Traffic Reports/ Chain of Custody Records (TR/COCs) or Packing Lists	Present/Absent*				
4. Airbill	Airbill/Sticker Present/Absent*				
5. Airbill No.	_____				
6. Sample Tags	Present/Absent*				
Sample Tag Numbers	Listed/Not Listed on Chain-of- Custody				
7. Sample Condition	Intact/Broken*/ Leaking				
8. Cooler Temperature Indicator Bottle	Present/Absent				
9. Cooler Temperature	_____				
10. Does information on TR/COCs and sample tags agree?	Yes/No*				
11. Date Received at Laboratory	_____				
12. Time Received	_____				
Sample Transfer					
Fraction	Fraction				
Area #	Area #				
By	By				
On	On				

\* Contact SMO and attach record of resolution.

Reviewed By	Logbook No.
Date	Logbook Page No.

Figure 8.4-2  
Sample Condition Form



Figure 8.4-3  
Sample Condition Notification Form

# Spectrum Analytical, Inc. RI Division Sample Condition Notification

Project#: \_\_\_\_\_

Date of Receipt: \_\_\_\_\_

Client: \_\_\_\_\_

Received By: \_\_\_\_\_

Client project #/name: \_\_\_\_\_

### Unusual Occurance Description:

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### Client Contacted:

Contacted via: Phone/Fax/E-mail

Date: \_\_\_\_\_ Time: \_\_\_\_\_

Contacted By: \_\_\_\_\_

Name of person contacted: \_\_\_\_\_

### Client Response:

Responded via: Phone/Fax/E-mail

Date: \_\_\_\_\_

Name of person responding: \_\_\_\_\_

Responding to: \_\_\_\_\_

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### Action Taken:

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Form ID: QAF.0005

Figure 8.4-4  
Spectrum Analytical, Inc. RI Division Chain-of-custody Form



Figure 8.5-1  
Workorder Information Form

**WorkOrder: J2694**

**01/07/2011 09:12**

**Mitkem Laboratories**

**Client ID:** MITKEM\_WARWICK  
**Project:** INTERNAL TESTING  
**WO Name:** WW 12/30  
**Location:** WATER\_TESTING,  
**Comments:** N/A

**Case:**  
**SDG:**  
**PO:** INTERNAL TESTING

**HC Due:** 01/13/11  
**Fax Due:**  
**Fax Report:**

**Report Level:** LEVEL 2  
**Special Program:**  
**EDD:**

Lab Samp ID	Client Sample ID	Collection Date	Date Recv'd	Matrix	Test Code	Samp / Lab Test Comments	HF	HT	MS	SEL	Storage
J2694-01A	WW-G-12/30	12/30/2010 00:00	12/30/2010	Aqueous	E624	/					VOA
J2694-01B	WW-G-12/30	12/30/2010 00:00	12/30/2010	Aqueous	E335.4	/					D2
J2694-01C	WW-G-12/30	12/30/2010 00:00	12/30/2010	Aqueous	E625	/					D2
J2694-02A	WW-C-12/30	12/30/2010 00:00	12/30/2010	Aqueous	SM5220	/					G2
J2694-02B	WW-C-12/30	12/30/2010 00:00	12/30/2010	Aqueous	E200.7	/ Cd, Cr, Cu, Pb, Ni, Ag, Zn				Y	M3

HF = Fraction logged in but all tests have been placed on hold

HT = Test logged in but has been placed on hold

Figure 8.6-1  
Volatiles Receiving Logbook Form



## 9.0 CALIBRATION PROCEDURES AND FREQUENCIES

All purchased equipment, materials, and services must meet specific method requirements, standard requirements, or project specific requirements. These requirements are documented in the individual analytical or project SOPs.

### 9.1 Instruments:

Specific calibration and check procedures are given in the analytical methods referenced in Section 10. The frequencies of calibration and the concentrations of calibration standards are determined by the cited methods and any special project or contract-specific requirements. Standard calibration curves of signal response versus concentration are generated on each analytical instrument used for a project, prior to analysis of samples. A calibration curve of the appropriate linear range is established for each parameter that is included in the analytical procedure employed and is verified on a regular basis with check standards as specified in the appropriate CLP Protocols. For non-CLP work, Spectrum Analytical, Inc. RI Division adheres to the calibration criteria specified by SW-846 and/or Standard Methods for both organic and inorganic analyses. Where requested, other method specific calibration criteria are used. Refer to the individual Standard Operating Procedures listed in Figure 11.7-1 of this QAP for the specific calibration and check procedures as well as concentration and frequency requirements.

For organic analyses whenever possible, unless otherwise specified in the individual methods, the initial calibration standards (ICAL), continuing calibration verification standards (CCV), laboratory control sample spike (LCS) and matrix spike (MS) will all be from the same source. The initial calibration verification (ICV) standards are prepared from a separate source. Refer to the Standard Operating Procedures listed in Figure 11.7-1 of this QAP for the specific calibration source and procedural requirements of each method. The following are examples of calibration procedures for various instrumental systems:

**GC/ECD and GC/FID** – An initial calibration is performed using five different concentration levels for each parameter of interest for SW-846 analyses. The initial calibration is done on each column and each instrument, and is repeated each time a new column is installed or whenever a major change is made to the chromatographic system.

Initial calibration verification (ICV), near mid level concentration for all analytes, is performed immediately after the calibration. If the ICV does not meet method specific criteria, a new calibration curve is generated and an ICV is analyzed. If repeated ICV failures are encountered, the system is checked to find the cause of these failures, and the problem is corrected. For certain GC/FID analyses (i.e. GRO /DRO), the instrument

is calibrated using individual compounds while the laboratory control sample or ICV uses a product (diesel or gasoline).

Continuing calibration verification (CCV), near a mid-level concentration for all analytes, is run at intervals determined by sample number or time allowed, as required by the individual methods. If CCV values are determined outside the upper limit of the method specified range and if no analytes were detected in the samples, the run will be accepted as valid and 'Non Detects' reported for the sample. If an analyte is detected and the CCV is out at the high end, the problem will be identified and corrected and the affected samples will be re-analyzed with a compliant CCV.

If a CCV value is out of the method specified limits at the lower limit, the cause of the problem will be identified and corrected, and all samples affected by the out of control CCV will be rerun with a compliant CCV.

For CLP-type analyses, the continuing calibration takes place at the beginning of the analytical sequence and once every twelve (12) hours throughout the analytical sequence, and again at the end of the sequence. The percent difference in calibration factors for each standard must not exceed the criteria specified by the method.

If a CCV fails to meet criteria limits, a new calibration curve will be generated and all samples affected will be re-analyzed.

**GC/MS** – For CLP methods, a minimum of five-level calibration (four-level for selected semivolatile compounds) is carried out for each analyte per system before analysis of samples take place.

Continuing calibrations, near midpoint levels, are analyzed every twelve hours of instrument analysis time for CLP analyses.

Re-calibration takes place whenever a major change occurs in the system, such as a column change in the GC or a source cleaning of the mass spectrometer or when the continuing calibration fails to meet method specific requirements.

Tunes are performed once every twelve (12) hours of instrument run time for all CLP-type and SW846 analyses. The GC/MS system is tuned to USEPA specifications for bromofluorobenzene (BFB) or decafluorotriphenylphosphine (DFTPP) for volatile and semivolatile analyses, respectively. Extended tune time is allowed in CLP SOM protocols where an ending CCV is acceptable as an opening CCV.

More detailed instrument and method-specific calibration procedures and criteria are described in the individual analysis SOPs.

**ICP/AES** – Instrument calibration, for each wavelength used, occurs at the start of each analysis. The calibration curve is constructed per method specification.

An initial calibration verification and initial calibration blank (ICB) are analyzed before analysis of samples. If the ICV and ICB do not meet method specific criteria for an analyte, the analyte is re-analyzed with a new calibration.

During the analysis, a continuing calibration verification (CCV) and continuing calibration blank (CCB) is analyzed at least every ten (10) samples or 2 hours for EPA ISM sequences. If either the CCV or CCB fails to meet method specific criteria for an analyte, the source of the problem is investigated. If it can be determined that the failed CCV and/or CCB is not representative (such as for instrument carryover from previous sample or from an empty auto-sampler tube), the CCV and/or CCB are re-analyzed and the reason for the failure documented. If a failure still occurs, further corrective action is performed, and the analyte is re-analyzed with a new calibration. For SW-846 and EPA 200.7 Methods, the ICV/CCV is obtained from a source independent from that of the calibration standards. The concentrations for the different analytes are at method specified levels. For EPA ISM Methods, only the ICV is obtained from a source independent from that of the calibration standards.

**ICP/MS** – Instrument calibration, for each mass used, occurs at the start of each analysis. The calibration curve is constructed per method specification.

An initial calibration verification and initial calibration blank (ICB) are analyzed before analysis of samples. If the ICV and ICB do not meet method specific criteria for an analyte, the analyte is re-analyzed with a new calibration. The ICV is obtained from a source independent from that of the calibration standards.

During the analysis, a continuing calibration verification (CCV) and continuing calibration blank (CCB) is analyzed at least every ten (10) samples or 2 hours for EPA ISM sequences. If either the CCV or CCB fails to meet method specific criteria for an analyte, the source of the problem is investigated. If it can be determined that the failed CCV and/or CCB is not representative (such as for instrument carryover from previous sample or from an empty auto-sampler tube), the CCV and/or CCB are re-analyzed and the reason for the failure documented. If a failure still occurs, further corrective action is performed, and the analyte is re-analyzed with a new calibration.

**The Flow Injection Mercury System (FIMS)** - Instrument calibration occurs at the start of each analysis. The calibration curve is constructed per method specification.

An initial calibration verification (ICV) and initial calibration blank (ICB) are analyzed before analysis of samples. If the ICV and ICB do not meet method specific criteria for Mercury, re-calibration and reanalysis are required.

During the analysis, a continuing calibration verification (CCV) and continuing calibration blank (CCB) is analyzed at least every ten (10) samples or 1 hour for EPA ISM sequences. If either the CCV or CCB fails to meet method specific criteria for Mercury, the source of the problem is investigated. If it can be determined that the failed CCV and/or CCB is not representative (such as for instrument carryover from previous sample or from an empty autosampler tube), the CCV and/or CCB are re-

analyzed and the reason for the failure documented. If a failure still occurs, further corrective action is performed, and the analyte is re-analyzed with a new calibration.

For SW-846 and EPA 245.1 Methods, the ICV/CCV is obtained from a source independent from that of the calibration standards. The concentrations for the different analytes are at method specified levels. For EPA ISM Methods, only the ICV is obtained from a source independent from that of the calibration standards.

Other instrumentation:

**IC-** The Ion Chromatograph is calibrated each day of use. Calibration verification is analyzed at the beginning, end, and at least every 10 samples. The verification standard is from an independent source. If the calibration verification does not meet method specific criteria for an analyte, it is re-analyzed once. If failure still occurs, a new calibration curve is established and any affected samples are reanalyzed.

**pH-** the meter is calibrated at two pH levels (4.0 and 10.0) before analyses of samples. The pH 7.0 buffer is analyzed as an LCS and recovery is calculated.

**Lachat 8000-** automated flow-through spectrophotometer is calibrated per method specification before the analyses of samples.

An initial calibration verification and initial calibration blank (if required) are analyzed before analysis of samples. If the ICV and/or ICB do not meet method specific criteria for an analyte, re-calibration must occur. The ICV is obtained from a source independent from that of the calibration standards.

During the analyses, continuing calibration verification and continuing calibration blanks are analyzed at least every ten (10) samples or 1 hour for EPA ISM sequences. If either the CCV or CCB fails to meet specified criteria for an analyte, the source of the problem is investigated. If it can be determined that the failed CCV and/or CCB is not representative (such as for instrument carryover from previous sample or from an empty autosampler tube), the CCV and/or CCB are re-analyzed and the reason for the failure documented. If a failure still occurs, further corrective action is performed, and the analyte is re-analyzed with a new calibration. The CCV concentration is at method specified levels.

**SpecGenesys-** manual spectrophotometer is calibrated per method specification.

Calibration curve calibration verification is analyzed at the beginning, end, and at least every 10 samples. The verification standard is from an independent source. If the calibration verification does not meet method specific criteria for an analyte, it is re-analyzed once. If failure still occurs, a new calibration curve is established and any affected samples are reanalyzed. Calibration curves are established at least quarterly.

Annual calibration and preventative maintenance is required by an outside vendor unless calibration can be performed in-house using a calibration kit.

**Balances:** are calibrated by an outside source on an annual basis.

The balances are calibrated externally each day of use by a lab technician with NIST traceable Class “1” or “2” weights. The weights are certified by an outside service on a regular basis, not to exceed five years.

**Liquid in Glass Thermometers** are calibrated once a year against a NIST-verified thermometer or as they are replaced. The NIST-verified thermometers are certified by an outside certified service annually.

**Gel Permeation Chromatography** is used to clean samples according to CLP and client requirements. GPCs are calibrated using a calibration standard provided by Ultra Scientific, Cat. # CLP-340. Once a successful calibration is achieved it is valid for a period of seven days.

## 9.2 Standards and Reagents:

**Standard** reference materials used for routine calibration, calibration checks, and accuracy are obtained from commercial manufacturers. These reference materials are traceable to the source and readily compared to EPA references. All standards come with a Certificate of Analysis which is kept on record in the appropriate laboratories or saved electronically to the central database. When a chemical standard can not be purchased in solution form, a neat source may be bought. The lab must attempt to obtain the highest purity available. If the lab can not find a neat source with at least 97% purity, the laboratory must document why. In addition, the impurity correction factor must be used when calculating the standard concentration. While most standards are traceable to NIST; however, certain projects, especially those involving pesticide registration, may necessitate the use of reference standards supplied by the client. New standards are also routinely validated against known standards that are traceable to EPA or NIST reference materials. See SOP #80.0001, Standard Preparation, Equivalency and Traceability, for more details.

Organic Preparatory Lab Surrogate and Matrix spikes are prepared in the appropriate instrument labs and then QA'd by diluting the standard and analyzing it on the GC or GC/MS. Criteria for the diluted spike analysis must meet the method or in-house criteria. If acceptable, the spike is able to be used. If unacceptable, another standard is prepared and the same steps repeated. Data from the QC analysis is retained in the laboratory for reference and traceability.

Primary, intermediate and working standards are all named using specific nomenclature as designated in the QA Department SOP# 80.0001, Standard Preparation, Equivalency and Traceability.

Standards are dated and labeled upon arrival. Any material exceeding its shelf life as described by the methods in QAP Section 10 is discarded and replaced. Standards are

periodically analyzed for concentration changes/degradation and inspected for signs of deterioration such as color change and precipitate formation. Standards Logbooks, which contain all pertinent information regarding the source and preparation of each analytical standard, are maintained by each of the laboratory departments in the LIMS.

See individual analytical SOPs (listed in Figure 11.7-1), sections 7 and 8 for standards preparation procedures.

**Solvents** are tested for purity prior to use to ensure there is no external source of contamination. For organic solvents, each lot number of solvent is QC'd prior to use. This is accomplished by concentrating an aliquot of solvent or extracting with reagent media (such as sodium sulfate) in the same manner as the samples and analyzing it for contamination by GC/MS. Any detectable analyte could render the solvent or reagent unsuitable for use. Supervisors make the final decision as to the suitability of the solvent or reagent, and whether the lot may be used for standard or sample preparation.

**Chemicals and Reagents** are stored in the respective laboratories during use. Backup supplies are stored in the stockroom. Reagent grade chemicals are used in all tests. All dry chemicals and reagents are given a 5-year expiration period unless designated otherwise by the manufacturer. Sometimes the viability of the reagent does not remain throughout the entire 5-year period (as determined through investigation following poor results in a preparation method blank or bench analysis, for example). In this case, the chemical or reagent is readily discarded. Acids/caustics are given a 3-year expiration period unless designated otherwise by the manufacturer. Solvents are given a 1-year expiration period unless designated otherwise by the manufacturer.

Chemicals and reagents are logged into the laboratory and each bottle is given a unique ID. The ID is based upon the date of its arrival at the laboratory. The only exceptions include cases/cycletainers of solvents and cases of acids. For solvents and acids, the boxes/cases are labeled with received date to insure first in/first out usage. All other chemicals and reagents are named using specific nomenclature as designated in the QA Department SOP # 80.0013, Reagent Purchasing and Tracking. Refer to this SOP for more detail regarding Solvents and Reagents.

When a bottle is opened in the laboratory, it is inspected to ensure it meets the requirements of the method. The analyst records his or her initials on the bottle along with the date opened and the ID. Any applicable certificates of analysis (COA) are scanned and archived. They may also be stored in the individual laboratories or in the QA Department.

## 10.0 ANALYTICAL PROCEDURES

Spectrum Analytical, Inc. RI Division uses the methods specified in Tables 10-1 through 10-5 unless otherwise specified by the client. Spectrum Analytical, Inc. RI Division performs analyses on non-potable waters and soil/solids. The RI Division does not perform regulatory potable (drinking) water analyses or environmental lead (paint chips, wipes, etc. for RIDOH compliance) testing. Associated Standard Operating Procedures related to these analytical procedures can be found in Figure 11.7-1 of this QAP.

Table 10-1  
Non-potable Water Analytical Methods

Parameter	Method Description	Method Reference
Metals	ICP-AES	200.7
Mercury	Cold Vapor	245.1
Cyanide	Midi-distillation Automated	EPA 335.4
Alkalinity	Titration	SM2320B
Anions	Ion Chromatography	EPA 300.0
Chloride		
Sulfate		
Nitrate		
Nitrite		
Orthophosphate		
Bromide		
Fluoride		
Volatile Fatty Acids	Ion Chromatography	EPA 300.0 Mod
Acetic		
Butyric		
Lactic		
Propionic		
Pyruvic		
pH	Electrode	SM4500 H+ B
Sulfate	Turbidimetric	426C SM 15 <sup>th</sup> Ed.
Ammonia	Distillation/Nesslerization	SM4500-NH3 B,C
Orthophosphate	Ascorbic, Manual	SM4500-P E
Total phosphate	Persulfate, Manual	SM4500-P B5 & E

Table 10-1  
Non-potable Water Analytical Methods (cont.)

<u>Parameter</u>	<u>Method description</u>	<u>Method Reference</u>
Chemical Oxygen Demand	Spectrophotometric(Closed Reflux)	SM5220-D
Total Organic Carbon	Combustion	SM5310 B
Phenols	Distillation, 4-AAP, Direct Photometric	SM5530 B E420.1
Total Dissolved Solids	Gravimetric	SM2540 C
Total Solids	Gravimetric	SM2540 B
Total Suspended Solids	Gravimetric	SM2540 D
Total Settleable Solids	Imhoff cones	SM2540 F
Hexavalent Chromium	Diphenyl Carbazide Colorimetric	SM 3500Cr D
Volatile Organics		
Halocarbons	Purge & Trap, GC/MS	624
Aromatics	Purge & Trap, GC/MS	624
Semivolatile Organics	Extraction, GC/MS	625
Organochlorine Pesticides/ PCBs	Extraction, GC/ECD	608
Oil & Grease (HEM, SGT)	Extraction, Gravimetric	1664A

Table 10-2  
SW-846 Inorganic Analytical Methods

<u>Parameter</u>	<u>Method Description</u>	<u>Method Reference</u>
Metals		
Aqueous	Acid digestion ICP/AES ICP/MS	Method 3005A/3010A Method 6010C Method 6020A
Solid	Acid digestion ICP/AES ICP/MS	Method 3050B Method 6010C Method 6020A
Mercury		
Aqueous	Permanganate digestion Cold Vapor analysis	Method 7470A
Solid	Permanganate digestion Cold Vapor analysis	Method 7471B
Hexavalent Chromium		
Solid	Acid Digestion colorimetric	Method 3060A/7196A
Cyanide		
Solid & Aqueous	Midi-distillation Automated	Method 9012B
pH		
Solid Waste	Electrode	Method 9045C Method 9040B
Ignitability (Flashpoint)		
Aqueous	Pensky-Martens closed cup	Method 1010
Solid	Pensky-Martens closed cup	Method 1010 Mod.
Reactive Cyanide		
Solid & Aqueous	Distillation Automated	SW 846 7.3.3.2
Reactive Sulfide		
Solid & Aqueous	Distillation Colorimetric	SW 846 7.3.4.2

Table 10-2  
SW-846 Inorganic Analytical Methods (cont.)

<u>Parameter</u>	<u>Method Description</u>	<u>Method Reference</u>
Anions	Ion Chromatography	SW 846 9056A
Chloride		
Sulfate		
Nitrate		
Nitrite		
Orthophosphate		
Bromide		
Fluoride		
Toxicity Characteristic Leaching Procedure (TCLP)		
Aqueous	Leachate by Filtration	Method 1311
Solid	Leachate Generation	Method 1311
Synthetic Precipitation Leaching Procedure (SPLP)		
Aqueous	Leachate by Filtration	Method 1312
Solid	Leachate Generation	Method 1312

Table 10-3  
SW-846 Organic Analytical Methods

<u>Parameter</u>	<u>Sample Preparation</u>	<u>Sample Analysis</u>
Volatile Organic Compounds		
Aqueous	Method 5030	Method 8260C
Solid	Method 5035	Method 8260C
1,2-Dibromo-3-chloropropane 1,2-Dibromomethane	Micro extraction GC\ECD Analysis	Method 8011
Semivolatile Organic Compounds		
Aqueous	Method 3510C Method 3520C	Method 8270D
Solid	Method 3540C Method 3550B Method 3545 Method 3570	Method 8270D
Organochlorine Pesticides		
Aqueous	Method 3510C Method 3520C	Method 8081B
Solid	Method 3540C Method 3550B Method 3545 Method 3570	Method 8081B
Polychlorinated Biphenyls (Aroclors and Congeners)		
Aqueous	Method 3510C Method 3520C	Method 8082A
Solid	Method 3540C Method 3550B Method 3545 Method 3570	Method 8082A
Total Petroleum Hydrocarbons		
Aqueous	Method 3510C Method 3520C	Method 8015B,D
Solid	Method 3540C Method 3550B	Method 8015B,D

Table 10-3  
SW-846 Organic Analytical Methods (cont.)

<u>Parameter</u>	<u>Sample Preparation</u>	<u>Sample Analysis</u>
Herbicides Solid & Aqueous	Method 8151A	Method 8151A
Gel Permeation Chromatography (GPC) Solid & Aqueous	Method 3640A	Various
Florisil Cleanup Solid & Aqueous	Method 3620B	Various
Silica Gel Cleanup Solid & Aqueous	Method 3630C	Various
Sulfur Cleanup Solid & Aqueous	Method 3660B	Various
Sulfuric Acid Cleanup Solid & Aqueous	Method 3665A	Various

<u>Parameter</u>	<u>Method Description</u>	<u>Method Reference</u>
Toxicity Characteristic Leaching Procedure (TCLP)		
Aqueous	Leachate by Filtration	Method 1311
Solid	Leachate Generation	Method 1311
Synthetic Precipitation Leaching Procedure (SPLP)		
Aqueous	Leachate by Filtration	Method 1312
Solid	Leachate Generation	Method 1312

Table 10-4  
CLP-Type Analytical Methods

<u>Parameter</u>	<u>Method Reference</u>
USEPA CLP Organics	OLM04.3, SOM01.2
USEPA CLP Inorganics	ILM05.4, ISM01.2
USEPA Low Level Organics	OLC03.2
NYS-ASP CLP Organics	ASP 2000/2005 SOW
NYS-ASP CLP Organics	ASP 2000/2005 SOW

Table 10-5  
Other Analytical Methods

<u>Parameter</u>	<u>Method Reference</u>
Volatile Petroleum Hydrocarbons	
Aqueous	MADEP VPH 1.1
Solid	MADEP VPH 1.1
Extractable Petroleum Hydrocarbons	
Aqueous	MADEP EPH 1.1
Solid	MADEP EPH 1.1
New York State Total Petroleum Hydrocarbon	
Solid	310.13 Mod.
Extractable Total Petroleum Hydrocarbons	
Aqueous	CT ETPH 99-3
Solid	CT ETPH 99-3
Deisel Range Organics	
Aqueous	ME 4.1.25
Solid	ME 4.1.25
Gasoline Range Organics	
Aqueous	ME 4.2.17
Solid	ME 4.2.17

## 10.1 Analytical References

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10. Methods of Soil Analysis. Part 2, Chemical and Microbiological Properties, Second Edition, American Society of Agronomy, Inc., Soil Science Society of America, Inc., Madison, WI, 1982.
11. Standard Methods for the Examination of Water and Wastewater, 18<sup>th</sup> Edition, APHA, Washington, D. C., 1992.
12. Standard Methods for the Examination of Water and Wastewater, 20<sup>th</sup> Edition, APHA, Washington, D. C., 1998.
13. Test Methods for Evaluating Solid Waste-Physical/Chemical Methods, SW-846, 3<sup>rd</sup> Edition Final Updates I through IV. Office of Solid Waste and Emergency Response, USEPA, Washington, D. C., 1998. Status table found at <http://www.epa.gov/epawaste/hazard/testmethods/sw846/pdfs/methstat.pdf>

14. USEPA Contract Laboratory Program. Statement of Work for Organic Analysis, USEPA, OLM04.3, OLC03.2, and SOM01.2.
15. USEPA Contract Laboratory Program. Statement of Work for Inorganic Analysis, USEPA ILM05.4, and ISM01.2.
16. Maine Health and Environmental Testing Laboratory. Modified GRO and DRO Methods, Method 4.2.17 and 4.1.25, September 6<sup>th</sup> 1995.
17. EPA Methods and Guidance for Analysis of Water, Version 2.0. includes MCAWW Methods and most current EPA Methods @ <http://www.epa.gov/ost/methods/>

## 11.0 DATA COLLECTION, REDUCTION, VALIDATION AND REPORTING

### 11.1 Data Collection:

Most of the lab's data is uploaded into the LIMS systems directly from the instruments. The exception is the GC's and GC/MS's in which data is first processed in Target and then uploaded into the LIMS.

Either the instrument analyst or data reporting group will upload the data into the LIMS. The person who performs the upload does a technical review to ensure recoveries of CCVs, MS, MSD, and LCS all seem to be correct. A completeness review is done at this time to ensure all applicable samples have been uploaded for all the necessary analytes.

Next, an employee with a technical background will perform the QA process of the uploaded data. This person is either a supervisor or someone with extensive experience in environmental chemistry. Corrections to the run are made at this step if necessary. When the review is complete, this technical person authorizes the data to be reported by "QA-ing" the run in the LIMS. For a more detailed view of the LIMS uploading/review procedure, see SOP # 110.0028, Data Validation/Self Inspection Procedures.

### 11.2 Data Reduction:

Instrument printouts, computer terminal displays, chromatograms, strip chart recordings and physical measurements provide raw data that are reduced to concentrations of analytes through the application of the appropriate calculations.

Equations are generally given within the analytical methods referenced in Section 10. Data reduction may be performed automatically by computerized data systems on the instrument, manually by the analyst, or by PCs using spreadsheet and/or data base software. See SOPs #110.0034 Sample Data Control for Inorganic CLP and #110.0035 Sample Data Control for CLP SOM01.2 for additional detail regarding the ISM/SOM programs.

### 11.3 Data Verification:

The verification process requires the following checks to be made on data before they are submitted to the client:

- A completeness inspection is required which ensures that all required data are included in the data packages submitted to the client and that the appropriate signatures are present on the data packages.

- A contract compliance screening to ensure that contractual requirements have been satisfied.
- A consistency check to ensure that nominally identical or similar data appearing in different places within a data package are consistent with respect to value and units.
- All manual integrations are properly performed and documented.
- A correctness check to ensure that reported data have been calculated correctly or transcribed correctly.

#### 11.4 Data Validation:

Data validation is an essential element of the QA evaluation system. Validation is the process of data review and subsequent acceptance or rejection based on established criteria.

The following analytical criteria are employed by Spectrum Analytical, Inc. RI Division in the technical evaluation of data:

- Accuracy requirements.
- Precision requirements.
- Detection limits requirements.
- Documentation requirements.

As in the case of EPA/CLP procedures, data acceptance limits may be defined within the method. As one means of tracking data acceptability, quality control charts are plotted for specific parameters determined in similar, homogeneous matrices. Control limits for non-CLP methods are statistically determined as analytical results are accumulated unless provided by method or program.

Upon completion of the evaluation, the evaluator dates and initials the data review checklist as described in Section 11.5 below.

#### 11.5 Data Interpretation and Reporting:

Interpretation of raw data and calculation of results are performed by a scientist experienced in the analytical methodology. Upon completion of data reduction, the scientist signs for the reported results on the data review checklist. For GC/ECD, GC/FID and GC/MS, a technical peer review is performed using the data processing software prior to form generation.

The laboratory supervisor is responsible for the data generated in that department. The supervisor or other senior technical staff performs an independent review of

data and completed report forms. Members of the QA staff also check the results on selected sets of data (usually 10%).

#### 11.5.1 Report Formats:

Spectrum Analytical, Inc. RI Division uses a flexible data reporting system where final report format is based on the requirements of the client. The two most common types of data reports generated by the Spectrum Analytical, Inc. RI Division are Level 2 or “commercial-format” and Level 4 or “CLP-format”. The lab adapts its data report format, wherever possible, to meet customer requirements. Occasionally reports are generated that are a compromise between “commercial” and CLP-format deliverables or are designed to meet the needs of a particular regulatory format or sampling program.

Commercial data reports are generated using the LIMS. All instrumental analysis data are uploaded from instruments to the LIMS by electronic data transfer. Non-instrumental analysis data or sample preparation data are manually entered into the LIMS. All manual data entry steps are double-checked to insure they are correct, and instrumental data are spot-checked to insure the proper functioning of the data upload system. All data receive a 100% review before they are released to the client as final.

CLP data reports are generated using specialized CLP report modules in the LIMS for all inorganic and most organic analyses. These reports also undergo a 100% review before they are released to the client in their final form.

Records are maintained for all data, even those results that are rejected as invalid.

#### 11.6 Levels of Data Review:

Spectrum Analytical, Inc. RI Division employs five (5) levels of data review. These are based on requirements outlined in several government and other environmental analysis programs including the U. S. Army Corps of Engineers, Air Force Center for Environmental Excellence (AFCEE), Naval Facilities Engineering Service Center (NFESC), HAZWRAP, Department of Defense ELAP (QSM), EPA Contract Laboratory Program (SOM/ISM), as well as commercial engineering firm programs.

The data review and evaluation process is structured to insure that all data reported to customers has been thoroughly reviewed and approved using a multi-step process designed to identify and correct any error. At any step in the data evaluation and review process, the reviewer has the responsibility and authority to return any data not meeting requirements back to the previous step for re-analysis

or correction. No reports are released to the client as final data without successfully passing through each step in the data evaluation and review process. The steps of the data review process are documented, generally using a checklist. Several checklists are used, depending on the type and format of analysis data being reviewed. Any data released prior to the completion of the full review process are released with the statement that the data is preliminary pending final review. The word "Preliminary" is automatically printed on the bottom of all data sheets that are generated prior to completion of data review.

The five levels of data review are detailed in SOP # 110.0028 Data Validation/Self Inspection Procedures. A Flow chart of the data review process follows in Figure 11.6-1.

#### 11.7 Document Control:

All login sheets, Chains-of-Custody (COC) and Sample Condition Forms (SCF) and other sample transmittal documentation are generated in Sample Receiving. A red Workorder File is initiated to contain all workorder-specific hard copy documents. Samples are signed in/out of the sample receiving area by analysts. In the Prep lab, samples and all pertinent information is recorded into logbooks. Once samples are moved to the instrument lab, the transfer of extracts is documented in the electronic transfer logbook (ICOC). In the instrument lab, the analysis of extracts is recorded in the instrument run log. All analysis data, including ICAL, CAL and raw data are acquired using computer-controlled instruments, and stored on the hard drive of the computer performing data acquisition. Data are automatically copied to the company file server after acquisition. Organics analysis data are processed using Thru-Put Systems' Target software. This system creates a folder on the file server for each analysis fraction for each work order or SDG. This folder contains raw data, processed analysis results, instrument tune, initial calibration and continuing calibration results as well as a copy of the data processing method used. This allows for long-term archiving and complete reconstruction of the data at any time in the future. Organic data files are also uploaded into LIMS so reporting forms can be printed. The raw data are printed and arranged with all appropriate sample-preparation and instrument run logbook page copies for technical review.

Inorganic data files are uploaded into LIMS and reporting forms are printed. The original instrument data files and the processed SDG are stored on the file server where they can later be archived by the LIMS Administrator. Hard copy or digital printouts for reporting forms, instrument data output and all associated preparation logbook page copies are assembled for technical data review.

Efforts are in progress to move to some paperless reporting schemes where no raw data or report will be printed in hardcopy. We expect to be primarily utilizing a paperless system by mid 2011 with the exception of our EPA CLP reports.

See SOP # 110.0029, Electronic Data Management for a detailed description of data management activities used to support laboratory activities.

Following technical review and generation of the report narrative, results go into the workorder file in data reporting. The original copy or CD (dependent on client requirements) of the report is sent to the client. The report is also scanned into an optical file database for long-term archiving. All other information associated with the report, including data review checklists are kept in the red workorder file. The non-reported data (NRD) is scanned into the optical file database for long-term archiving as was the client report. As documents are scanned into the database they are recorded for permanent storage on hard drives within the fileserver. The archived electronic data is kept for a minimum of ten (10) years or according to contract/program requirements. Prior to the use of the optical file database, hardcopy reports and NRD were shipped to an offsite storage area where they will remain for a minimum of ten (10) years. After this time, these older files will be destroyed.

#### 11.7.1 Logbooks:

All logbooks are issued and controlled by the QA Department. Logbooks are given a unique ID that includes the mm/yy the logbook was printed. Laboratory personnel must sign for the logbook when it has been released by the QA Department. When logbooks are complete, the analyst returns them to the QA Department for archiving unless still needed for reference in the lab. A new logbook is released. The archived logbooks are stored in an on-site storage box for approximately 4-6 months and then are stored in an off-site storage facility or may remain on-site depending on storage space. Refer to SOP # 80.0040, Logbook Use, Review, and Control for more detail. In addition, refer to SOP # 110.0027, Documentation Policy and Procedures for details on Spectrum Analytical, Inc. RI Division's Logbook policies. Logbooks are archived for a minimum of ten (10) years or according to contract/program requirements. Please note that during the transition period of changing our name, logbooks will still be in use that have the name Mitkem Laboratories on them. After June 1, 2011 all newly issued logbooks will have Spectrum Analytical, Inc. RI Division.

#### 11.7.2 Workorder/Data Files:

Spectrum Analytical, Inc. RI Division is a secured, limited access building. The doors are secured with a keypad entry system. All hard copy information pertaining to the analysis of samples is maintained and stored in a workorder file folder. This information includes all login sheets, COC, SCF, bench sheets and analytical data. Electronic data are also stored by laboratory workorder number on the company file server, and in the optical file database of completed reports and NRD as mentioned in section 11.7. File folders containing any remaining

workorder information are stored in an off-site storage facility or may remain on-site for a total of 10 years.

The off-site storage facility referred to in the above sections is a locked storage area. Access is limited to the Laboratory Director or his designee and request to retrieve a file will be made to this person.

In the event Spectrum Analytical, Inc. RI Division changes ownership, the maintenance, control, storage and eventual disposal at the end of the appropriate time period, of all records, including client data and QA/QC files, will transfer to the new owners.

In the event Spectrum Analytical, Inc. RI Division decides to cease operations, clients will be notified prior to the cessation of operations and their files/records will be made available to them. Within a designated time period after notification, the client will be responsible for taking custody and the future maintenance of their records. If the client determines they do not want to maintain the records, these will be disposed of properly.

#### 11.7.3 Standard Operating Procedures (SOPs):

SOPs are prepared by the Lab Supervisor and laboratory personnel in conjunction with the QA Director. The QA Director/Staff downloads a copy of the current SOP to the network at Public on 'Bernoulli' (Q:). The SOPs can be found in Q:\QA\_SOPs. In addition a .pdf file of the SOP is located in Q:\QA\_PUBLIC\PDF-MITKEM SOPs. A list of the current SOPs in use at Spectrum Analytical, Inc. RI Division is given in Figure 11.7-1.

The laboratory staff revises the SOPs by making changes to the document that is then reviewed by the department supervisor only if the supervisor is not the party responsible for the revisions. Any additional changes are made at this point.

The QA Department is notified that revisions are completed. The QA Director/Staff moves the revised copy of the SOP to the QA directory, QA Safety/SOPs Needing QA Revision. The QA Director makes changes to the document to include revision number and date and title clarification, if necessary.

The QA Director prints a copy of the SOP that is then signed by the Laboratory or Technical Director, and the QA Director. The effective date is then added to the SOP and the full document is pdf'd. If an older version of the SOP exists, it is moved to its archive location. The new version will be moved into the Spectrum Analytical, Inc. RI Division Intranet SOP Database for access by all laboratory personnel. Each

analyst who performs any duties related to the SOP must review the new version and enter electronically that he or she has read and understands the material there.

SOP review/revisions occur on an annual basis. The procedure for preparing, reviewing, approving, revising and distributing SOPs as well as the SOP Revision Schedule are described in SOP #80.0012 Standard Operating Procedure Production.

Please note that during the transition period of changing our name, SOPs will still be in use that have the name Mitkem Laboratories on them. After June 1, 2011 all newly revised SOPs will be titled with Spectrum Analytical, Inc. RI Division.

Minor changes to the hardcopy SOP between revision dates can be done by making hand-written changes to the document and its copies. The changes must be initialed by the QA Director and incorporated into the next version SOP. Minor changes are recorded in the Revision Record that is a part of the master copy.

#### 11.7.4 Quality Assurance Manual:

The lab will review the QA Manual annually at a minimum. Past versions of the QA Manual are maintained and archived by the QA Director in the same manner as SOPs. Edits to the QA Manual are made by the QA Director in conjunction with the laboratory management. Spectrum Analytical, Inc. RI Division will amend the QAP and any affected SOPs within 14 days when technical changes (or any of the circumstances outlined in the USEPA SOW for SOM or ISM, Exhibit E, section 5.3.2) occur. Amendments are made to correct or explain discrepancies identified by outside agencies (identified during on-site, data or electronic audits) or internally during laboratory audits or corrective action investigations as examples. Edits are also initiated due to personnel, equipment or capability changes. Procedural or method changes may also need to be reflected in a revised QAP. The revised QAP with visible markups will be sent to the USEPA as per section 5.3.2.1.

#### 11.7.5 Method Updates:

In most cases it is the laboratory's policy to implement new revisions of frequently used methods within six months of the date the method revision is promulgated or published as a final method (non-CLP methods, for CLP methods see below). The QA Director, Deputy Director for Quality Services, Technical Director and Laboratory Director make the final decision on when a method revision will be adopted by the laboratory. Additionally, if a client specifically requests or mandates that an "older"

method, Spectrum Analytical, Inc. RI Division will advise the client that it is not the most recent method. If the client still insists upon the older method, the lab will comply and make a note in the narrative.

When the laboratory is in the middle of a client's project, the lab will continue using the same revision for the entire sampling event unless advised otherwise by the client. Consequently, once the laboratory has formally adopted a new method revision, both the old and new revision may be in use at the same time, depending on the project.

If a client should not specify which methods to be used, the methods employed by the laboratory shall be fully documented and validated. Additionally, the methods shall be published in a reputable technical journal or text or by a reputable technical organization or instrument manufacturer.

Revisions to USEPA CLP methods are required to be implemented within 14 days of notification when the EPA modifies the technical requirements of the statement of work, or the contract. At this same time, the QAP will be amended as necessary as noted in section 11.7.4.

Laboratory-developed methods can be used as long as they have been documented and validated by qualified personnel. In all cases the client should be notified.

Figure 11.6-1  
Data Review Flow Diagram

Spectrum Analytical, Inc. RI Division  
Review Process Flow Diagram

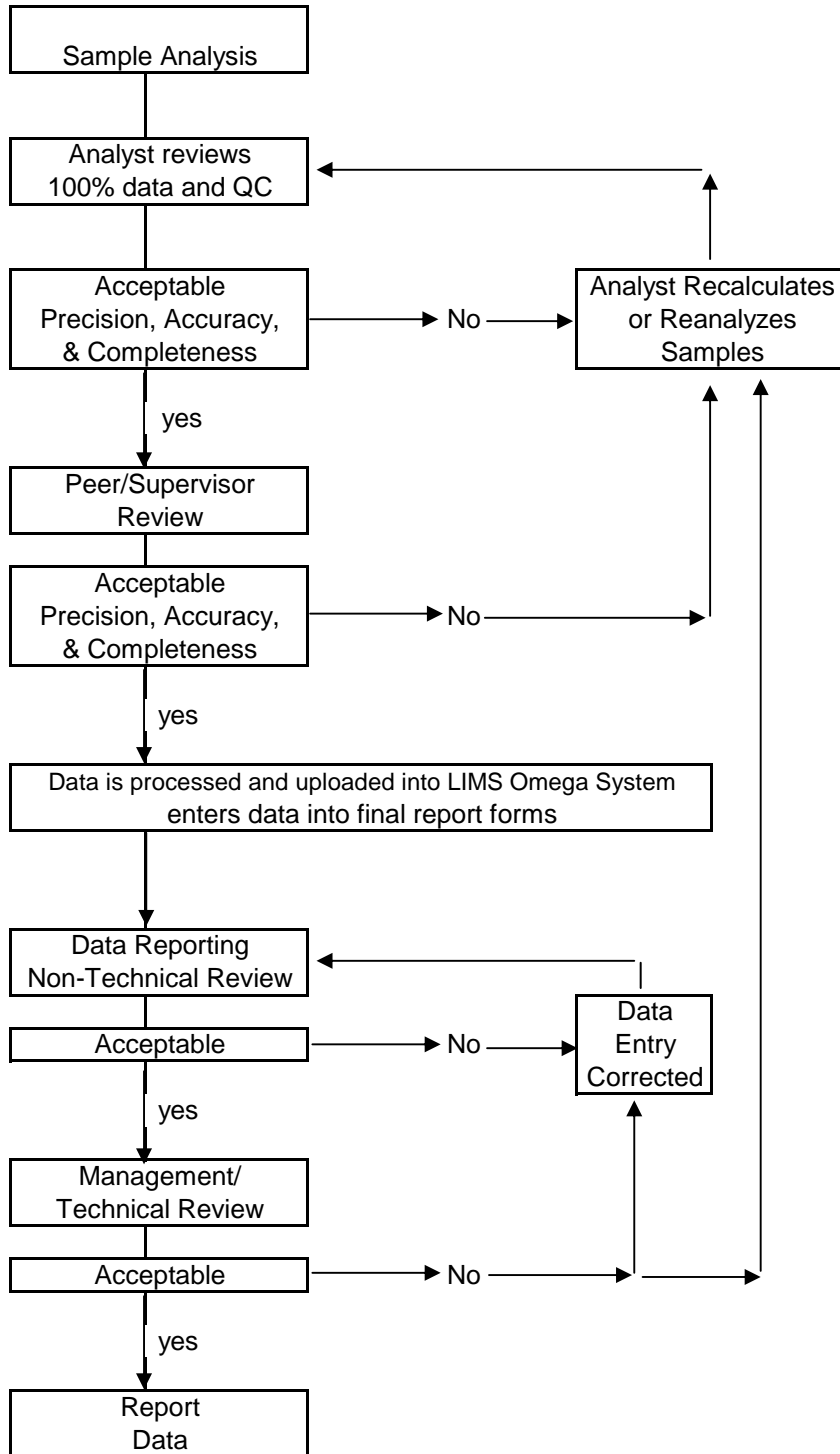


Figure 11.7-1  
Standard Operating Procedures (SOPs)

**Spectrum Analytical, Inc. Rhode Island Division**  
**Standard Operating Procedures (SOPs)**  
**Master List**

SOP #	Title
10.0009	Application Xtender
10.0016	Assembly of Inorganic CLP and CLP-type Reports
10.0017	Assembly of Organic CLP and CLP-type Reports
10.0018	Assembly of Commercial Data Reports
10.0021	Data Report Options
10.0036	EPA/SOM Organic Data PDF Bookmarking
10.0037	EPA/ISM Inorganic Data PDF Bookmarking
20.0003	Logging Workorders into Omega
20.0005	Level 2 LIMS report preparation
30.0002	Bottle order preparation
30.0003	Sample Receipt, Storage, Tracking and Disposal
30.0024	Sample and Waste Disposal
30.0030	ICOC Procedures using IntCOC program
50.0004	Glassware Cleaning - Organics
50.0027	Organic Preparation of Aqueous/Soil Samples for Chlorinated Herbicides by SW-846 Method 8151A
50.0030	SOM01.2 Sulfur Cleanup
50.0031	SW-846 Method 3665A Acid Cleanup
50.0032	Gel Permeation Chromatography by SW-846 Method 3640A
50.0033	SW-846 Method 3620B Florisil Cleanup
50.0034	SW-846 Method 3630C Silica Gel Cleanup
50.0035	Oil&Grease (HEM&SGT) by Method 1664 Revision A

**Spectrum Analytical, Inc. Rhode Island Division**  
**Standard Operating Procedures (SOPs)**  
**Master List**

SOP #	Title
50.0036	SW-846 Method 3660B Sulfur Cleanup
50.0050	Organic Preparation of Aqueous Samples by Continuous Liquid-Liquid (Method 3520)
50.0051	Organic Preparation of Aqueous Samples by Separatory Funnel (Method 3510)
50.0052	Organic Preparation of Soil Samples by Sonication (Method 3550)
50.0053	Organic Preparation of Soil Samples by Soxhlet (Method 3540)
50.0054	Organic Extract Filtration and Concentration Techniques
50.0060	Organic Preparation of Aqueous Samples by Continuous Liquid-Liquid for Pesticides/Aroclors for SOM01.2
50.0061	Organic Preparation of Aqueous Samples by Separatory Funnel for Pesticides/Aroclors for SOM01.2
50.0062	Organic Preparation of Solid Samples by Sonication for Pesticides/Aroclors for SOM01.2 by Method 3550B
50.0063	Organic Preparation of Aqueous Samples by Continuous Liquid-Liquid for Semivolatiles for SOM01.2
50.0064	Organic Preparation of Solid Samples by Sonication for Semivolatiles for SOM01.2
50.0100	Preparation of Soil Samples by MSE by Method 3570
50.0101	Preparation of Soil Samples by PFE by Method 3545
50.0102	Percent Lipid Determination in Tissue Samples
60.0002	Pesticide/PCB Analysis by EPA Method 608
60.0003	Determination of Polychlorinated Biphenyls by Gas Chromatography/Electron Capture Detector Analysis by SW.846 Method.8082A
60.0006	Determination of Pesticides by Gas Chromatography/Electron Capture Detector Analysis by SW.846 Method 8081B

**Spectrum Analytical, Inc. Rhode Island Division**  
 Standard Operating Procedures (SOPs)  
 Master List

SOP #	Title
60.0007	EDB/DBCP by EPA Method 504.1 and SW-846 8011
60.0034	Determination of Chlorinated Herbicides by Gas Chromatography/Electron Capture Detector Analysis by SW846
60.0048	Aroclor Analysis GC/ECD by USEPA SOW SOM01.2
60.0049	Pesticide Analysis GC/ECD by USEPA SOW SOM01.2
60.0050	Total Petroleum Hydrocarbons by GC-FID using EPA SW-846 Methods 8015/State Methods
60.0053	PCB Congeners by SW-846 Method 8082 (MOD)
60.0054	PCB Homologs by E680 GC/MS SIMS
70.0011	Determination of Semivolatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS) Analysis by SW846 Method 8270D
70.0030	Screening for Semivolatile Organic Analysis by Gas Chromatography/Mass Spectrometry for SOM01.2
70.0033	SIM Analysis by GC/MS (Modified EPA Method 8270D)
70.0035	Semivolatile Organic Analysis by SIM Gas Chromatography/Mass Spectrometry for SOM01.2
70.0048	Semivolatile Organic Analysis by Gas Chromatography/Mass Spectrometry for SOM01.2
70.0051	Semivolatile Organics by GC/MS for Aqueous Samples by EPA Method 625
80.0001	Standard Equivalency/Traceability
80.0002	Client Complaint Policies
80.0004	QA Data Pkg Review
80.0005	Method Detection Limit Determination
80.0006	Internal Audit Procedures

**Spectrum Analytical, Inc. Rhode Island Division**  
 Standard Operating Procedures (SOPs)  
 Master List

SOP #	Title
80.0007	Corrective Action Procedures
80.0009	Newly Implemented Methods (Demonstration of Acceptable Performance)
80.0010	Control Chart Generation and Use
80.0012	The Production of Standard Operating Procedure
80.0013	Reagent Purchasing & tracking
80.0016	Training Procedures and Tracking
80.0020	Temperature Monitoring Systems
80.0030	Labware Volume Verification
80.0040	Logbook Use, Review, and Control
80.0050	Performance Testing Procedures
90.0012	Determination of Volatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS) Analysis by SW846 Method 8260C
90.0035	Low/Med Volatile Organics Analysis GC/MS by USEPA SOM01.2
90.0036	Trace Volatile Organics Analysis GC/MS for USEPA SOM01.2
90.0038	Gasoline Range Organics by GC/FID using Methods SW-846 8015 and Maine 4.2.17
90.0040	Trace Volatile Organics Analysis GC/MS using SIM for USEPA SOM01.2
90.0052	Volatile Organics by GC/MS for Aqueous Samples by EPA Method 624
90.0060	Methane, Ethane, and Ethene by GC/FID Method RSKSOP-175
100.0001	Glassware Cleaning - Inorganics
100.0002	Alkalinity (by Standard Method 2320)
100.0003	Sample Preparation of Aqueous Samples by Acid Digestion ICP (3005/3010)

**Spectrum Analytical, Inc. Rhode Island Division**  
**Standard Operating Procedures (SOPs)**  
**Master List**

SOP #	Title
100.0004	Total Cyanide by Automated Colorimetric with Midi-distillation by SW846 9012B
100.0005	Determination of Metals and Trace Elements in Water and Waste by ICP - Atomic Emission Spectrometry by EPA Method 200.7
100.0006	ICAP 3000XL/4300DV Operation
100.0010	Nitrite Analysis by Standard Method 4500-NO2 B
100.0011	pH Value by Standard Methods 4500-H+ B
100.0012	Mercury Analysis in Aqueous Samples by Flow Injection Analysis System for Atomic Analysis by Method 7470A/7471B
100.0013	Total and Ortho Phosphate using Ascorbic Acid Method by Standard Method 4500-PE
100.0014	Mercury (Manual Cold Vapor Technique) by EPA Method 245.1
100.0015	The Preparation of Waste Samples for reactive Cyanide and Sulfide; Determination of Reactive Cyanide by Automated Colorimetric Method and Reactive Sulfide by Spectrophotometric Method SW-846 Methods 7.3.3.2 and 7.3.4.2
100.0016	Preparation of Soil Samples for Sulfide Analysis by Modified SW-846 Method 9031
100.0017	Inorganic Analysis of Sulfates in Aqueous Samples by SM 426 C 15th Ed.
100.0018	Inorganic Analysis of Sulfides in Aqueous Samples (Methylene blue method)
100.0019	Total Dissolved Solids Dried at 180°C by Standard Method 2540 C
100.0020	Total Solids Dried at 103-105°C by Standard Method 2540 B
100.0021	Total Suspended Solids Dried at 103-105°C by Standard Method 2540 D
100.0022	TKN Distillation and Determination by Manual Spectrophotometric Analysis by Standard Method 4500-N
100.0023	Color Analysis by Visual Comparison by Modified Standard Methods 2120B
100.0024	Flashpoint Analysis by SW846 Method 1010

**Spectrum Analytical, Inc. Rhode Island Division**  
**Standard Operating Procedures (SOPs)**  
**Master List**

SOP #	Title
100.0025	Total Organic Carbon by Methods SW-846 9060A and SM5310B
100.0026	Settleable Solids by Standard Method 2540 F
100.0027	Paint Filter Liquids Test by SW-846 Method 9095A
100.0028	Carbon Dioxide (CO2) and Forms of Alkalinity by Calculation by Standard Method 4500-CO2 D
100.0029	Ferrous Iron Analysis by Standard Method 3500-Fe D, Phenanthroline Method
100.0030	Phenols Analysis by EPA Method 420.1 and Standard Method 5530 B & D, Cleanup and Direct Photometric Method
100.0032	Total Volatile Solids for Solids by SM 2540 E, E160.4; Fixed and Volatile Solids Ignited at 550 C
100.0033	Total Cyanide by Auto-Colorimetric with Midi-Distillation by EPA Method 335.4
100.0053	ISM01.2 ICP-AES Analysis
100.0054	ISM01.2 ICP-MS Analysis
100.0055	Mercury Preparation and Analysis by ISM01.2
100.0056	Cyanide Preparation and Analysis by ISM01.2
100.0100	Sample Preparation of Soils by Acid Digestion for ICP/MS (3050B/6020A)
100.0103	AVS and SEM
100.0104	Sample Preparation of Soils by Acid Digestion for ICP/AES (3050B/6010C)
100.0106	Chemical Oxygen Demand Determination SM5220D
100.0110	Determination of Metals in Water and Wastes by Inductively Coupled Argon Plasma Mass Spectrometry by SW846 Method 6020A
100.0111	Determination of Metals in Water and Wastes by Inductively Coupled Argon Plasma Atomic Emission Spectrometry by SW846 Method 6010C
100.0112	pH in Soil Samples by SW846 9045C/OLM4.3/SOM1.2

**Spectrum Analytical, Inc. Rhode Island Division**  
**Standard Operating Procedures (SOPs)**  
**Master List**

SOP #	Title
100.0120	Specific Conductance EPA Method 120.1 and Salinity SM2520
100.0121	ICP Aqueous Preparation by ISM01.2
100.0122	Prep of Soil, Wipe/Air Filter for ICP Analysis by ISM01.2
100.0201	Ammonia Distillation & Determination by Manual Spectrophotometric Analysis(4500-NH3 B&C)
100.0208	Inorganic Analysis of Hexavalent Chromium in Soil Samples by SW846 Methods 3060A & 7196A
100.0209	Mercury Speciation...SW846 Method 3200
100.0308	Inorganic Analysis of Hexavalent Chromium in Aqueous Samples by SM 3500 Cr +6 D
100.0400	Inorganic Anions by IC EPA 300.0 and 9056A
100.0410	TOC in Soil by Lloyd-Kahn and SW-846 9060
100.0420	Volatile Fatty Acids by IC using EPA 300.0 (modified)
100.0430	Walkley Black TOC in Soil
100.0440	Total, Fixed and Volatile Solids in solid/semisolid samples by SM2540G
110.0006	Thermometer Calibration
110.0007	Balance Calibration
110.0008	Manual Integration of GC, IC and GC/MS Chromatograms
110.0012	Laboratory Security
110.0013	North Carolina Samples
110.0021	Bids and Proposals
110.0023	Project Management
110.0025	Toxicity Characteristic Leaching Procedure by SW846 Method 1311
110.0026	Handling of Evidentiary Materials
110.0027	Documentation Policy and Procedures
110.0028	Data Validation-Self Inspection Procedures
110.0029	Electronic Data Management

**Spectrum Analytical, Inc. Rhode Island Division**  
**Standard Operating Procedures (SOPs)**  
**Master List**

SOP #	Title
110.0031	Synthetic Precipitation Leaching Procedure by SW-846 Method 1312
110.0032	ASTM Leachate Procedure D3987-06
110.0034	Sample Data Control for Inorganic CLP (ILM/ISM)
110.0035	Sample Data Control for Organic CLP (SOM)
110.0038	Percent Solids Determination as Required for Various SW-846 and EPA Methods
110.0039	Sub-Sampling for Soil and Solid Samples
110.0040	Instrument Maintenance
110.0041	Multiple Extraction Procedure by SW846 EPA Method 1320
110.0043	Standard Elutriate Preparation
110.0060	Tissue Sample Preparation

## 12.0 LABORATORY QUALITY CONTROL CHECKS

Spectrum Analytical, Inc. RI Division's analytical procedures are based on sound quality control methodology, which derives from three primary sources:

1. Specific EPA and other approved analytical methods, and
2. "Handbook for Analytical Quality Control in Water and Wastewater Laboratories" (EPA 600/4-79-019).
3. Standards for Good Laboratory Practice.

In the application of established analytical procedures Spectrum Analytical, Inc. RI Division employs, at a minimum, the QC protocols described in the references found in the Analytical Methods section of this document. Specific projects may require additional quality control measures, due to such factors as difficult sample matrices or use of innovative techniques. For those projects Spectrum Analytical, Inc. RI Division will recommend and implement, subject to client approval, QC measures to produce data of known quality.

Each of the Spectrum Analytical, Inc. RI Division laboratory departments have an individual QC program, which includes, but is not limited to, the practices described below.

### 12.1 Method Detection Limit Determination/Verification:

Method Detection Limits are developed annually for certain inorganic and many organic analyses. Per NELAC Standards, MDLs are not required where target analytes are not reported below the lowest calibration standard concentration. For these analyses, results are only reported within the calibration range, and MDLs are not appropriate or needed. The reporting limit for these compounds is the concentration of the lowest standard in the calibration. For certain inorganic analyses and most organic analyses, Spectrum Analytical, Inc. RI Division typically reports analytes below the lowest level of the calibration range, but above the MDL, as estimated and are qualified with the "J" flag. Spectrum Analytical, Inc. RI Division reports estimated values below the calibration range for those analyses where results are able to be confirmed as in dual column confirmation, or by two concurrent determinative tests such as retention time and mass spectra as in GC/MS analyses. For these analyses MDLs are determined or verified annually, depending on program requirements.

MDLs are determined for all test methods where required by specific program or state regulations. Methods analyzed for the State of Massachusetts which do not detail MDL requirements within the published method, require preparation and analysis of the MDL samples over a minimum of three days. This is believed to

better mirror real world samples and day to day variability of preparatory and analytical steps.

In addition, to address special project requirements, MDLs can be determined for those tests which are not routinely reported below calibration range. If a client requests results to be reported below the calibration range without an MDL study, this is clearly identified in the workorder narrative.

MDL studies performed for the USEPA Contract Laboratory Program are performed on an instrument specific basis by method and matrix. Samples are reported with the determined detection limits reported based on which instrument they are analyzed on. Annually the MDL studies must be repeated or verified by the analysis of an MDL Verification sample.

Following an MDL study, the determined limits are verified by the analysis of an MDL Verification Standard. This standard is analyzed at approximately 2 to 3 times the calculated MDL for single analyte tests or 1-4 times the calculated MDL for tests with multiple analytes. This spike concentration is also referred to as the Limit of Detection in Department of Defense (DoD) QSM Version 4.1. DoD QSM requires quarterly verification of the LOD. For more details refer to SOP #80.0005 Determination of Method Detection Limits.

## 12.2 Personnel Training:

Chemists who begin their employment at Spectrum Analytical, Inc. RI Division are to be instructed under the lab's Safety Training Program within the first month. The Safety Training Program includes laboratory basics, safety video and testing, and MSDS instruction.

Before performing any analyses, a chemist is required to read the appropriate protocols and SOPs. The chemist is required to complete an SOP review form which lists all the SOPs he or she has read and understands.

The new analyst must become familiar with the laboratory equipment and the analytical methods, and begins a training period during which he or she works under strict supervision. Independent work is only permitted after the chemist successfully completes an accuracy and precision study.

The accuracy and precision study is also commonly referred to as a Demonstration of Capability exercise. Upon the successful completion of the Demonstration of Capability exercise, the QA Department issues a Demonstration of Capability Certificate (DOCC) which is signed by both the QA Director and Laboratory Director and is filed in the employee's personnel folder, which is stored in the QA Department.

Demonstration of Capability studies requires the acceptable mean recovery of 4 LCS samples for each matrix or the acceptable analysis of a blind spike sample

such as a Performance evaluation sample. Acceptance limits are established by the method. It is necessary to pass the study whether for extraction and/or analysis.

Annually thereafter the employee must perform an acceptable demonstration of capability study to document continued acceptable performance in his/her particular preparatory or analytical method specialty. This is referred to as the On-going DOC. All DOCC documentation is filed in the employee's personnel folder, which is stored in the QA Department. Refer to SOP #80.0016 Training Procedures and Tracking for additional information.

Initial and on-going personnel training include data integrity training. The 4 required elements of the data integrity system include: 1) data integrity training, 2) signed data integrity documentation, 3) in-depth, periodic monitoring of data integrity, and 4) data integrity procedure documentation.

Data integrity training topics will include the need for honesty and full disclosure in all analytical reporting, how and when to report integrity issues and what those issues could be. Employees will understand that infractions of data integrity procedures can result in an investigation that could lead to serious consequences which include immediate termination, and civil or criminal prosecution. At the start of employment all new employees read, discuss and sign a Confidentiality, Ethics and Data Integrity Agreement. Annually, an on-going integrity training session is held. An attendance sheet will be generated for every integrity session. These sheets are filed in the QA Office under "Training". Another option for the annual training session is having all staff review refresher materials online and document their having done so. This is done within the framework of the SOP database on the lab's intranet.

Data integrity procedures are reviewed and updated annually by senior management.

Training for the EPA Statement of Work occurs according to the above requirements. In addition, analysts are required to read the CLP Statement of Work as a part of the documentation training.

### 12.3 Control Charts:

For organic and inorganic analyses, the recoveries of analytes in the lab control samples are plotted on control charts. These charts are used to establish control and warning limits.

12.3.1 Control limits are calculated, compared, and/or updated at least annually from the LCS, MS/MSD, and Surrogate data points for each analyte and matrix using the following equations:

$$\text{Average}(\bar{x}) = \frac{\left[ \sum_{i=1}^n x_i \right]}{n}$$

$$SD = \sqrt{\frac{\sum_{i=1}^n (x_i - \bar{x})^2}{n - 1}}$$

In which:

SD = Standard Deviation

N = number of data points

Warning Limits = Average  $\pm$  2 \* SD

Control Limits = Average  $\pm$  3 \* SD

12.3.2 Control limits must be approved by the QA Director and by the Laboratory Director prior to adoption by the laboratory. In the event that limits are wider than method recommended limits, the method recommended limits may be adopted and the analytical procedure will be re-evaluated and/or re-determined to identify possible causes. Additionally, in the event that control limits are tighter than 15% from the average, the lab may adopt a control limit of  $\pm 15\%$  from the average. If in the experience of the laboratory, statistical control limits are unreasonably wide or narrow, alternative limits may be used until appropriate statistical limits are developed. Alternative limits are based on sources such as DoD QSM published guidelines, EPA limits from the specific test method or from similar methods, laboratory experience with the method or other sources.

12.3.3 Control charts are plotted in EXCEL using the Omega LIMS system.

Data from each laboratory is uploaded into the LIMS. The compounds, recoveries, and date analyzed for each test are recorded in the system. In order for LIMS generated control limits to be valid, all data, including data not meeting existing recovery criteria, must be uploaded. A control chart is then printed for review by the QA Director and by the Lab Supervisor. Out of control situations noted on the control chart are discussed with the Supervisor or Laboratory Director by the QA Director.

An example control chart is presented as Figure 12.3-1. LCS data must be reviewed and evaluated daily against the Control Limits to establish that the system is in control.

12.3.4 The following situations constitute an out of control situation on a control chart:

- One data point above or below the Control Limit line.
- Two consecutive data points above or below the Warning Limit line.
- Six or more consecutive data points above the Average Line or six or more consecutive data points below the Average Line. This situation suggests a trend and suggests the procedure has been changed in some way (for better or worse). The cause for this trend must be investigated.

12.4 General QC Protocols:

12.4.1. Organics Laboratory:

- Trip blanks and holding blanks, when applicable, are analyzed to detect contamination during sample shipping, handling and storage.
- Method blanks, at a minimum of one in every 20 samples, are analyzed to detect contamination during analysis.
- Volatile organic method blanks are analyzed once during each analytical sequence.
- One blank spike (Laboratory Control Sample or LCS) consisting of an analytical sample of laboratory water, anhydrous sodium sulfate, or Ottawa sand with every batch of 20 or fewer samples, is analyzed to determine accuracy.
- Sample spikes and spike duplicates, as requested, are analyzed to determine accuracy and the presence of matrix effects. The Relative Percent Difference (RPD) is also determined for matrix spike/matrix spike duplicates to measure precision. The criteria followed are stated in the individual methods. For batches without a sample duplicate (for example, if insufficient sample volume is provided), a duplicate blank spike (LCSD) is performed to provide for precision measurement.
- Performance evaluation samples from EPA and state agencies are analyzed to verify continuing compliance with EPA and NELAC QA/QC standards.

- Surrogate standards are added to samples and calculations of surrogate recoveries are performed to determine matrix effect and extraction efficiency.
- Internal standards for GC/MS analysis are added to sample extracts to account for sample-to-sample variation.
- Analysis of EPA traceable standards (ICV) to verify working standard accuracy and instrument performance.
- Initial multi-level calibrations are performed to establish calibration curves.
- Instrument calibration is established or verified with every analytical sequence.
- Tuning of GC/MS systems once every 12 hours for CLP and SW-846 methods or 24 hours for methods 624/625 to method specifications is implemented for consistency in data generation.
- Quarterly analysis of LOD and/or LOQ check samples to verify low level detection and reporting limits for Department of Defense QSM programs.
- Annual Verification of MDL for NELAC/TNI/USEPA CLP.

When QC limits are not met during an analytical run, the source of the problem must be investigated. Following an evaluation of the data, those samples affected must be re-analyzed after the problem has been solved. If QC limits continue to be out of control, the instrument must be checked and/or a service call made and/or further corrective action implemented.

#### 12.4.2. Inorganic Laboratory:

- Trip blanks are analyzed when applicable, to detect contamination during sample shipping, handling and storage.
- Method blanks are analyzed at a minimum of one every 20 samples, to detect contamination during analysis.
- One matrix spike of an analytical sample or laboratory water or soil is made and spike recoveries are calculated with every batch up to 20 samples to determine accuracy. Duplicate samples are analyzed and the RPD between the sample and duplicate is calculated for every

batch up to 20 samples. If insufficient volume of sample is received, a note is made in the appropriate preparation logbook.

- Performance evaluation samples from EPA and state agencies are analyzed to verify continuing compliance with EPA and NELAC QA/QC standards.
- Metals analysis instruments are calibrated for every analytical run.
- Analysis of EPA traceable standards (ICV) to verify working standard accuracy and instrument performance.
- QC/LCS checks samples are analyzed during every analytical batch of up to 20 samples in order to document accuracy.
- Quarterly analysis of LOD and LOQ check samples to verify low level detection and reporting limits for Department of Defense QSM programs.
- Annual Verification of MDL for NELAC/TNI/USEPA.

When QC limits are not met during an analytical run, the source of the problem must be investigated. Following an evaluation of the data, those samples affected must be re-analyzed after the problem has been solved. If QC limits continue to be out of control, the instrument must be checked and/or a service call made and/or further corrective action implemented.

#### 12.5. Lab Pure Water used for method blanks and dilutions:

Spectrum Analytical, Inc. RI Division uses several systems to generate analyte-free water for use in the laboratory. These systems generate high quality, analyte free water dedicated to the needs of specific analyses.

12.5.1. For inorganic analyses the wet chemistry and metals labs use a US Filter mixed-bed deionization system followed by particle and carbon filters. This is followed by a polishing system using Barnstead E-Pure cartridges optimized for removal of inorganic constituents. Purity is monitored using an on-line electrical resistivity meter.

12.5.2. For organic analyses, the extractable organics laboratory uses a Barnstead E-Pure system optimized for removal of organic constituents. As organic contaminants are not measured by a resistivity meter, this is not a relied-upon method to monitor the quality of organic analyte-free water. Instead, laboratory method blanks are used, typically several per working day, to monitor the acceptability of the water for its intended use. Any analyte detected above (half of) the reporting limit is investigated. If this can be

traced to the water purification system as its source, maintenance is performed on the water purification system. The volatile organics laboratory uses a Whirlpool Model WHER25 Reverse Osmosis Drinking water system to provide analyte free water.

Figure 12.3-1  
Example Control Chart

Date: 01-Jun-11

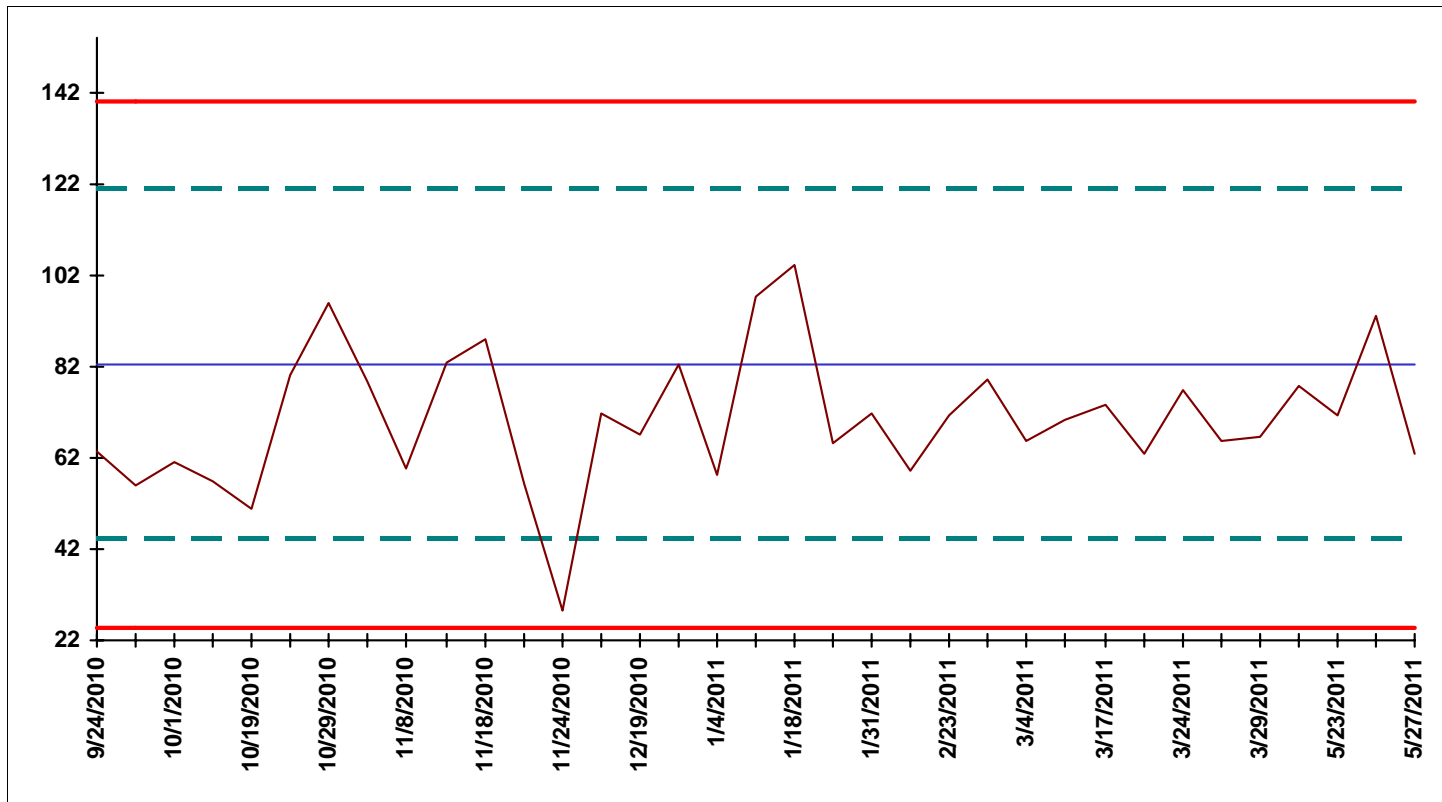
Test Code: SW8081\_W Analyte: ALDRIN

SampType	Sample ID	Analysis Date	Batch ID	Low Limit	High Limit	% Recovery
LCSD	LCSD-54152	9/24/2010	54152	25	140	63.3
LCS	LCS-54290	9/27/2010	54290	25	140	54.8
LCSD	LCSD-54290	9/27/2010	54290	25	140	57.1
LCS	LCS-54407	10/1/2010	54407	25	140	58.0
LCSD	LCSD-54407	10/1/2010	54407	25	140	53.1
LCS	LCS-54469	10/1/2010	54469	25	140	66.2
LCSD	LCSD-54469	10/1/2010	54469	25	140	67.4
LCS	LCS-54764	10/17/2010	54764	25	140	58.4
LCSD	LCSD-54764	10/17/2010	54764	25	140	55.1
LCS	LCS-54675	10/19/2010	54675	25	140	50.9
LCS	LCS-54936	10/21/2010	54936	25	140	77.4
LCSD	LCSD-54936	10/21/2010	54936	25	140	83.0
LCSD	LCSD-55058	10/29/2010	55058	25	140	95.5
LCS	LCS-55058	10/29/2010	55058	25	140	96.0
LCS	LCS-55202	11/3/2010	55202	25	140	73.7
LCSD	LCSD-55202	11/3/2010	55202	25	140	83.5
LCS	LCS-55349	11/8/2010	55349	25	140	59.8
LCSD	LCSD-55349	11/8/2010	55349	25	140	59.5
LCS	LCS-55315	11/9/2010	55315	25	140	81.7
LCSD	LCSD-55315	11/9/2010	55315	25	140	83.7
LCS	LCS-55547	11/18/2010	55547	25	140	88.2
LCSD	LCSD-55468	11/19/2010	55468	25	140	56.2
LCS	LCS-55468	11/19/2010	55468	25	140	56.2
LCS	LCS-55735	11/24/2010	55735	25	140	28.4
LCSD	LCSD-55796	11/29/2010	55796	25	140	69.6
LCS	LCS-55796	11/29/2010	55796	25	140	73.5
LCS	LCS-56305	12/19/2010	56305	25	140	67.0
LCSD	LCSD-56528	12/30/2010	56528	25	140	87.1
LCS	LCS-56528	12/30/2010	56528	25	140	77.3
LCS	LCS-56610	1/4/2011	56610	25	140	58.2
LCS	J0800-01B	1/17/2011	56848	25	140	100.8
LCS	J0800-02B	1/17/2011	56848	25	140	93.7
LCS	J0800-03B	1/18/2011	56848	25	140	104.3
LCS	J0800-04B	1/18/2011	56848	25	140	104.2
LCS	LCS-57043	1/26/2011	57043	25	140	62.8
LCSD	LCSD-57043	1/26/2011	57043	25	140	67.8
LCS	LCS-57163	1/31/2011	57163	25	140	71.9
LCS	LCS-57551	2/22/2011	57551	25	140	60.9
LCSD	LCSD-57551	2/22/2011	57551	25	140	57.0
LCS	LCS-57591	2/23/2011	57591	25	140	71.5
LCS	LCS-57639	2/25/2011	57639	25	140	79.0
LCSD	LCSD-57780	3/4/2011	57780	25	140	65.8
LCS	LCS-57780	3/7/2011	57780	25	140	70.4
LCSD	LCSD-58018	3/17/2011	58018	25	140	64.3
LCS	LCS-58018	3/17/2011	58018	25	140	83.1
LCS	LCS-58045	3/21/2011	58045	25	140	62.0
LCSD	LCSD-58045	3/21/2011	58045	25	140	64.0
LCS	LCS-58117	3/24/2011	58117	25	140	76.9
LCS	LCS-58189	3/28/2011	58189	25	140	62.6
LCS	LCS-58210	3/28/2011	58210	25	140	68.6
LCSD	LCSD-58210	3/29/2011	58210	25	140	66.5
LCS	LCS-58721	4/29/2011	58721	25	140	81.6
LCSD	LCSD-58721	4/29/2011	58721	25	140	74.1

Date: 01-Jun-11

Test Code: SW8081\_W Analyte: ALDRIN

SampType	Sample ID	Analysis Date	Batch ID	Low Limit	High Limit	% Recovery
LCS	LCS-59308	5/23/2011	59308	25	140	74.0
LCSD	LCSD-59308	5/23/2011	59308	25	140	68.6
LCS	LCS-59343	5/25/2011	59343	25	140	95.1
LCSD	LCSD-59343	5/25/2011	59343	25	140	91.2
LCS	LCS-59386	5/27/2011	59386	25	140	62.9



## 13.0 QUALITY ASSURANCE SYSTEMS AUDITS, PERFORMANCE AUDITS AND FREQUENCIES, PEER REVIEW

The Spectrum Analytical, Inc. RI Division Quality Assurance staff performs routine internal audits of the laboratory. The frequency of such audits depends on the workload in house but is done annually, at a minimum. These audits entail reviewing laboratory logbooks and all appropriate operations to ensure that all laboratory systems including sample control, analytical procedures, data generation and documentation meet contractual requirements and comply with good laboratory practices.

### 13.1 System Audits:

The QA Director audits each individual laboratory annually in order to detect any sample flow, analytical or documentation problems and to ensure adherence to good laboratory practices as described in Spectrum Analytical, Inc. RI Division's Standard Operating Procedures and Quality Assurance Plan. A checklist used in an internal systems audit is presented in Figure 13.1-1.

Areas covered by the internal audit include logbook documentation and review, standard traceability, standard storage and expiration dates, method criteria adherence, instrument maintenance records, SOP review, and knowledge of the analysts. Often, deficiencies that have been noted during "outside" audits will also be reviewed.

Upon the completion of the internal audit, a formal audit report is presented to the laboratory supervisor who is given a specific timeframe to respond in writing to the deficiencies. The QA Department will do a follow up audit to check that at least the major deficiencies have been corrected. The follow-up audit occurs within 30-45 days from the date of the audit response. Refer to SOP #80.0006 Internal Audits for more detail.

### 13.2 Performance Audits:

Spectrum Analytical, Inc. RI Division participates in external Performance Test (PT) studies under the National Environmental Accreditation Program (NELAP) through the New Jersey Department of Environmental Protection (Primary Accreditation Authority). The QA department administers the Performance Evaluation Samples for Wastewater/Solid Waste (WW/SHW). Additionally, performance samples are administered for test methods not certified through the New Jersey program, such as specific state methods. PT samples are handled (i.e., managed, analyzed, and reported) in the same manner as real environmental samples utilizing the same staff, methods as used for routine analysis of that analyte, procedures, equipment, facilities, and frequency of analysis. When analyzing a PT sample, a laboratory shall employ the same calibration, laboratory quality control and acceptance criteria, sequence of analytical steps, number of

replicates and other procedures as used when analyzing routine samples. Analysts should refer to SOP #80.0050 Performance Testing Procedures when preparing PE samples for more details. PT samples are reported electronically via the vendor's website (ERA, RTC...), and results are sent directly to all applicable state or agency certification programs.

Clients also send performance evaluation samples (PES) to Spectrum Analytical, Inc. RI Division as part of their own quality control program. Spectrum Analytical, Inc. RI Division is blind to the true values of the PES. The USEPA CLP program provides quarterly blind (QB) studies for all tests and matrices. The lab is informed of their performance after the study has been graded through an Individual Laboratory Summary Report. When results in any section are less than 90.0%, the lab is required to complete a formal corrective action report to the EPA.

Spectrum Analytical, Inc. RI Division also participates in external electronic data QA monitoring audits and data package audits through the USEPA CLP program. On request, the Spectrum Analytical, Inc. RI Division CLP Project Manager submits instrument data tapes and all applicable documentation for tape audits, including a copy of the data package. All original documentation generated during sample analyses may be requested. The results of the tape audit are sent to Spectrum Analytical, Inc. RI Division in report format in the same manner as an on-site audit (see below). A formal response is required.

Several times a year outside agencies (federal, state, or private) may schedule an audit at Spectrum Analytical, Inc. RI Division in order to check the laboratory's processes. Most often these audits begin and end with a meeting between auditors and laboratory management. Each individual laboratory is examined. The QA Director and/or Senior Management Staff are most likely to remain with the auditors at all times during the audit.

Sometime after the audit, the lab receives a formal audit report to which it must respond. The audit report is initially reviewed by the QA Director who copies and distributes the report to each laboratory supervisor. The supervisors are required to respond in writing to the findings that pertain to his or her department. The QA Officer compiles the formal response that could be tweaked several times before the auditing authority accepts the results. A specific timeframe is set by the individual agency involved.

The QA Officer then sends a memo to each supervisor to detail what needs to be done in each department within a specific timeframe. The QA Department then follows up with the labs to ensure procedures have been modified and the corrective actions are in place.

Internally, performance is monitored on a daily basis at Spectrum Analytical, Inc. RI Division through the use of surrogate and internal standards, and LCS and

MS/MSD samples. Check samples from independent commercial sources are employed routinely in each of the Spectrum Analytical, Inc. RI Division laboratory departments and ensure continuing high-level performance. The QA Director may distribute internal blind PE samples to each laboratory department as needed. These blind PE samples can also be used to show on-going analyst proficiency in lieu of 4 LCS studies.

### 13.3. Peer Review:

Peer review is used as a vital quality control tool within all areas of the laboratory, and at all levels. Peer review allows defects in the acquisition, evaluation and reporting of sample data to be identified before moving on to the next step in the process of preparing and analyzing samples. Several steps of peer review are included at Spectrum Analytical, Inc. RI Division to prevent and catch mistakes, whether caused by human error or a system malfunction. As soon as samples enter the laboratory they are logged into the LIMS system and given unique sample identifiers that correspond to the client's IDs listed on the chain of custody. The individual jars or bottles are labeled and the technician employs a peer review of this labeling process. A project manager or peer technician visually inspects each jar or bottle for proper identification and matching lab/client IDs. Once the samples are sent into the labs for test preparation, they again undergo peer review as they are set up for extraction, digestion or distillation... This time the samples are inspected to confirm the samples at the bench match the identifications written into the lab preparation logbooks. Once the concentrated extract, digestate or distillate is ready for analysis and set up on the analytical instrument, an analyst will perform another peer review of the autosampler set up to avoid any misplacements of sample vials. In some lab areas this review may occur after instrument analysis, to verify all sample data were acquired electronically. Every analytical instrument sequence (GC/ECD, GC/FID, GC/MS, ICP/MS, ICP/AES, CVAA, FIA, IC) undergoes a technical peer review by a qualified analyst to verify positive and false positive results as well as manual integrations. Data reports are also reviewed at length according to the 5 level review processes described in Section 11 of the QAP as well as in SOP #110.0028 Data Validation/Self Inspection Procedures. At each point in the process, the peer review is documented.

Figure 13.1-1  
QA Systems Audit Checklist

Quality Assurance Department  
Spectrum Analytical, Inc. RI Division

Quality Review of Laboratory Department

Auditor:

Date:

**Purpose**

The Quality Review is a necessary tool to assess a department's quality and service functions. Each department will undergo a review of their process and procedures to evaluate their needs and areas of possible improvement. Each department will be tracked for quality, safety, compliance, reoccurring errors and process improvement.

**Process**

Each department will be broken down into several categories or areas of review. Each category will be reviewed and assessed for compliance. The categories will include at a minimum:

Personnel Training and Knowledge  
Equipment  
SOP Updates and Review  
Logbook Review and Control  
Chemicals/Standard Storage and Preparation  
Sample Procedures and Method Compliance  
QA/QC Procedures  
Corrective Actions in process

Each category will be reviewed and a listing of any deficiency or findings will be documented for response and correction. The department Supervisor (s) will be required to respond to each deficiency or finding within 30 days of receipt of this report. All deficiencies or findings must have its correction(s) documented. For example, logbook deficiencies will require a photocopy of the correction(s). All other responses will require a written response or adequate explanation. Deficiencies will be tracked for reoccurrence. All documentation should be forward to the QA department for evaluation. A follow up audit may be scheduled.

**Findings:**

**Personnel Training and Knowledge**

**Equipment**

**SOP Updates and Review**

**Logbook Review and Control**

**Chemicals/Standard Storage and Preparation**

**Sample Procedures and Method Compliance**

**QA/QC Procedures**

**Corrective Actions in process**

Items marked with an\* asterisk will require a written response by the lab supervisor or his designee to the QA Dept. This response must be submitted to the QA Department by *mm/dd/yyyy*. The response can be entered directly into this document in a different font color. Please note date that the CA was completed.

Auditor \_\_\_\_\_ Date \_\_\_\_\_

## 14.0 PREVENTIVE MAINTENANCE

Preventive maintenance is a routine practice at Spectrum Analytical, Inc. RI Division for all instrumentation. Scheduled preventive maintenance minimizes instrument downtime and subsequent interruption of analysis.

Only those equipment items meeting or exceeding applicable performance requirements are used for data collection. This includes items such as laboratory balances as well as major analytical instruments such as ICPs, ICP/MS, GCs and GC/MSs. All major instrumentation and equipment, as well as backup alternatives, are listed in Appendix A. Spectrum Analytical, Inc. RI Division SOP # 110.0040, Instrument Maintenance, describes routine maintenance in detail. Individual analytical standard operating procedures describe maintenance as well (See Figure 11.7-1 for SOP listing).

When new software is purchased or developed, it is loaded onto one workstation with copies of data that have been previously processed using older software, and known to be correct. The data is then reprocessed using the new software and then the new results are compared to the original results for defects. If the software was purchased and found to contain a defect, the vendor is contacted and a solution and/or patch are requested. If the software was developed in-house, the problems are identified and corrected. This process is applicable to all software including enhancements made to customize the LIMS and network servers. Refer to SOP #110.0029 for details on Electronic Data Management.

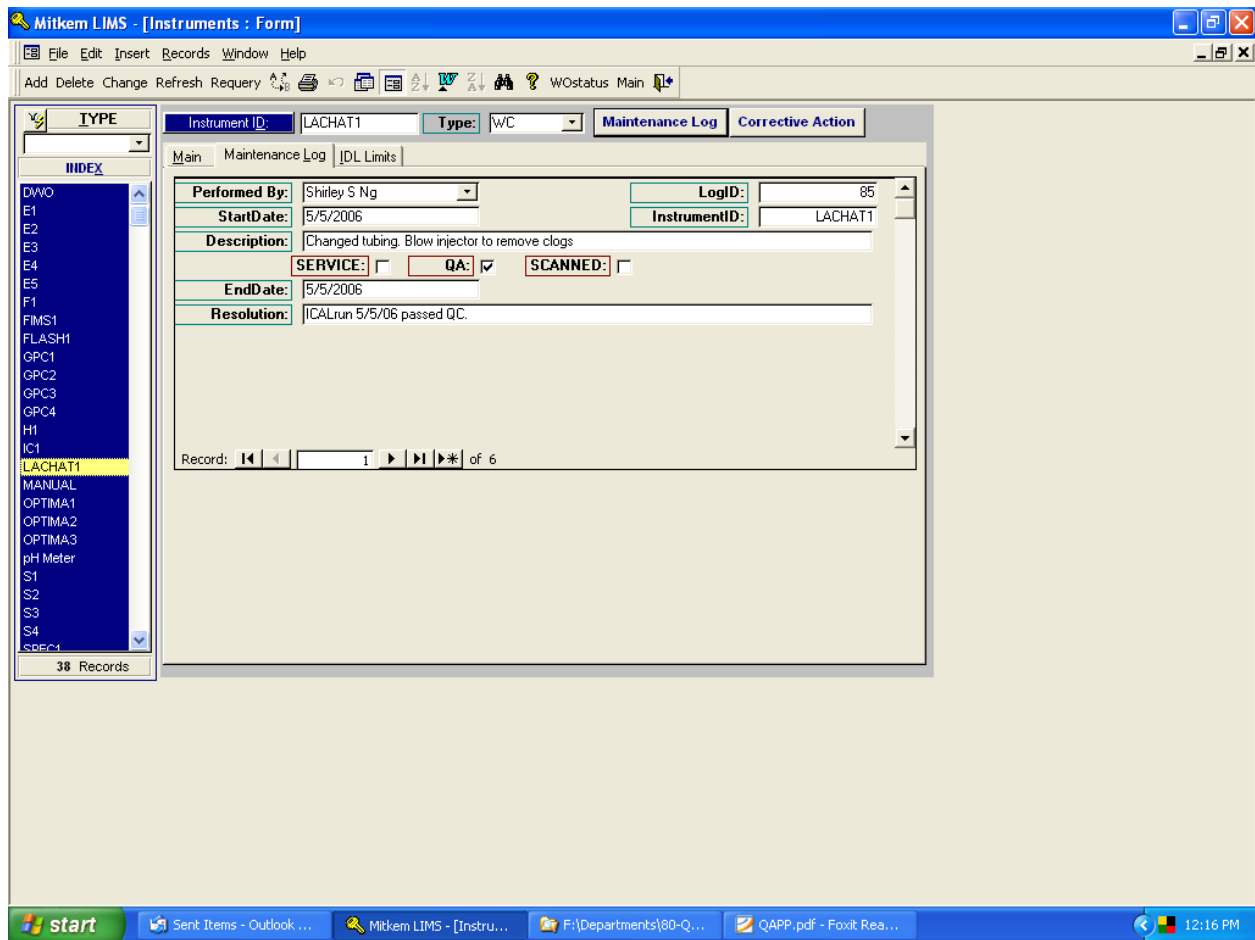
Spectrum Analytical, Inc. RI Division's laboratory personnel are familiar with the routine and non-routine maintenance requirements of the instruments they operate. This familiarity is based on education, hands-on experience and manufacturer's training courses. As needed, major equipment may under-go extensive maintenance or service by a contracted technician.

Instrument maintenance logs are kept for each instrument in the LIMS (figure 14-1). All employees have password protected access to the LIMS. The person performing the maintenance is required to provide the following information in the online log:

- Equipment identifier
- The inspection, maintenance, calibration or corrective action(s) performed.
- The trigger(s) for the maintenance action(s)
- The identity of the person(s) performing the maintenance
- The date on which the work was performed
- The need for a service call
- The condition of the equipment upon completion of the work (may include resolution of problems, date and type of ICAL run or other method of determining that the system is in good working order), and
- The presence of any scanned paperwork associated to the maintenance

Spectrum Analytical, Inc. RI Division maintains an inventory of replacement parts required for preventive maintenance and spare parts that often need replacement, such as filaments for GC/MS systems and the more mundane electrical fuses and GC column ferrules. To control cost, the appropriate supervisor shall decide the types and numbers of spare parts kept on hand for each equipment item.

Figure 14-1



Example of Instrument Maintenance Log

## 15.0 SPECIFIC ROUTINE PROCEDURES USED TO ASSESS DATA PRECISION, ACCURACY, COMPLETENESS, METHODS DETECTION LIMIT AND LINEAR DYNAMIC RANGE

These mathematical equations represent the means of calculating analytical figures of merit on a routine basis at Spectrum Analytical, Inc. RI Division. However, they may be supplanted with other calculations if requested by the client. Precision, accuracy and completeness are also discussed in Section 6.

### 15.1 Precision:

Precision is frequently determined by the comparison of replicates, where replicates result from an original sample that has been split for identical analyses. Standard deviations,  $s$ , of a sample are commonly used in estimating precision.

Sample standard deviation,  $s$ :

$$s = \sqrt{\frac{1}{n-1} \sum_{i=1}^n (x_i - \bar{x})^2}$$

where a quantity,  $x_i$  (e.g. a concentration), is measured  $n$  times with a mean,  $\bar{x}$ .

The relative standard deviation,  $RSD$  (or sample coefficient of variation,  $CV$ ), which expresses standard deviation as a percentage of the mean, is generally useful in the comparison of three or more replicates (although it may be applied in the case of  $n = 2$ ).

$$\%RSD = 100 (s / \bar{x})$$

or

$$CV = 100 (s / \bar{x})$$

In which:  $RSD$  = relative standard deviation, or

$CV$  = coefficient of variation

$s$  = standard deviation

$\bar{x}$  = mean

For duplicates (samples that result when an original sample have been split into two for identical analyses), the relative percent difference ( $RPD$ ) between the two samples may be used to estimate precision.

$$RPD = \frac{2(D_1 - D_2)}{(D_1 + D_2)} \times 100\%$$

In which:  $D_1$  = first sample value

$D_2$  = second sample value (duplicate)

### 15.2 Accuracy:

The determination of accuracy of a measurement requires knowledge of the true or accepted value for the signal being measured. Accuracy may be calculated in terms of bias as follows:

$$Bias = X - T$$

$$\% Bias = 100 \frac{(X - T)}{T}$$

In which:  $X$  = average observed value of measurement  
 $T$  = "true" value

Accuracy also may be calculated in terms of the recoveries of analytes in spiked samples:

$$\% Recovery(\% R) = 100 \times \frac{(SSR - SR)}{SA}$$

where:  $SSR$  = spikes sample result  
 $SR$  = sample result  
 $SA$  = spike added

### 15.3 Completeness:

Determine whether a database is complete or incomplete may be quite difficult. To be considered complete, the data set must contain all QC check analyses verifying precision and accuracy for the analytical protocol. Less obvious is whether the data are sufficient to achieve the goals of the project. All data are reviewed in terms of goals in order to determine if the data set is sufficient.

Where possible, the percent completeness for each set of samples is calculated as follows:

$$\% Completeness = \frac{\text{valid data obtained}}{\text{total data planned}} \times 100$$

### 15.4 Method Detection Limit:

The method detection limit (MDL) is the minimum concentration of a substance that can be measured and reported with 99% confidence that the analyte

concentration is not zero. It is computed as follows from data obtained by repeatedly determining an analyte in a given sample matrix:

1. Analyze at least seven samples of a homogeneous matrix spike that contains the analyte(s) of interest at concentrations of three to five times the expected MDL. The entire sample preparation and analysis protocol must be applied in each analysis; simply preparing one sample and repeating a measurement three or more times on the sample is not acceptable.
2. Upload the acceptable data into LIMS.
3. The LIMS will compute the standard deviation of the results for each analyte using the following equation:

$$\text{MDL} = t_{(n-1, \alpha=0.99)} (s)$$

Where  $t$  is the one-sided student's  $t$  value appropriate for the number of samples analyzed,  $n$ ;  $\alpha$  is the statistical confidence level; and  $s$  is the standard deviation.

The one-sided  $t$ -values are presented below:

<u>Number of samples</u>	<u><math>t</math>-value</u>
7	3.14
8	2.996
9	2.90
10	2.82

4. The MDL is then checked against 40CFR136 requirements by the QA Department. If the MDL is acceptable then it is uploaded into the LIMS by either the QA Department or LIMS Administrator.
5. Immediately following the determination of the MDL, MDL check samples are analyzed at a concentration approximately equal to 2-3 x the new MDL for SW846 tests. The analyte of interest must be detected at this concentration, or the raising the MDL may be required. Once the MDL check is acceptable, the detection limit (DL) has been established.
6. An elevated MDL can be uploaded if necessary into the LIMS as long as documentation is available to show that the applicable method can produce an MDL at least that low. This can commonly occur for ICP analysis in which extremely low MDLs can cause method compliance issues. When appropriate, the MDL study may be prepared and analyzed over several days to increase the variability of the preparation and/or analytical steps.

7. More detail on MDLs can be found in SOP# 80.0005 Method Detection Limit Determination.

#### 15.5 Linear Dynamic Range:

The linear dynamic range is the concentration range over which the instrument response is linear. It is determined by analyzing a series of standard solutions that extends beyond the non-linear calibration region at both the low and high extremes, and selecting that range of standards which demonstrates a linear relationship between instrument response and concentration.

For ICP analysis, the linear dynamic range is determined by analyzing each metal at 3 different concentrations. The concentration which produces results within a 10% error is determined to be the linear dynamic range. This procedure must be performed per individual method requirements.

ILM5.4 requires the analysis of the linear dynamic range be determined quarterly, with a 5 % error.

## 16.0 CORRECTIVE ACTION

An essential element of the QA Program, Corrective Action provides systematic, active measures taken in the resolution of problems and the restoration of analytical systems to their proper functioning.

Corrective actions for laboratory problems are described in Spectrum Analytical, Inc. RI Division's laboratory standard operating procedures (SOP). Personal experience often is most valuable in alerting the bench scientist to questionable results or the malfunctioning of equipment. Specific QC procedures are designed to help the analyst determine the need for corrective actions (see Section 11, Data Reduction, Validation and Reporting). Corrective actions taken by scientists in the laboratory help avoid the collection of poor quality data. The lab's corrective action program divides these issues into routine and non-routine corrective actions as described below.

Routine Corrective Action – A routine corrective action is taken when the out-of-control event encountered is one that is detected at the appropriate level in the QA process. Routine corrective actions are defined in the analytical SOP with specific steps to be taken as corrective action (i.e., low surrogate recovery, continuing calibration verifications, project specific protocols that do not meet acceptance criteria, etc.) Routine corrective actions must be documented as described in the analytical SOP, but do not require further documentation in the corrective action logbook. Examples of routine corrective action situations: surrogate/surrogates out, LCS out, CCV out, ICV out, IS area/areas out, typographical errors, random blank contamination, or false positive hit/spectral ID match corrected during data review.

Non-Routine Corrective Action – A non-routine corrective action is taken when the out-of-control event encountered is not typical for the method. For example, QC failures that pass through the final review to the client, procedural errors – not following the SOP, or a situation not being detected by normal QA procedures that could adversely impact the accuracy, precision, etc. of a result. Non-routine corrective actions must be documented in the Corrective Action Request (CAR) system, located within the LIMS. The analyst, using his/her own judgement, may deem any corrective action situation non-routine and formally document it in a CAR. When in doubt about a corrective action, the analysts are instructed to err on the side of formal CAR documentation. Examples of non-routine corrective action situations include: bad standard, expired standard mix being used, incorrect equation, “client-detected” problems, not following SOP protocols, using bad or contaminated lot of chemical/reagent/solvent, deciding to release data not conforming to SOP requirements, compound retention time outside of range, or improper library spectrum that leads to re-occurring mis-identification of compounds.

The essential steps in Spectrum Analytical, Inc. RI Division's corrective action system are:

1. Identify and define the problem.
2. Assign responsibility for investigating the problem. Usually this individual is the department supervisor.
3. Investigate and determine the root cause of the problem.
4. Determine a corrective action to eliminate the problem and prevent recurrence. Any changes that result from the corrective action investigation must be documented.
5. Assign and accept responsibility for implementing the corrective action.
6. Establish effectiveness of the corrective action and implement it.
7. Verify that the corrective action has eliminated the problem.
8. Both the laboratory and the QA Department need to monitor the corrective action to ensure it is effective.
9. Any corrective actions that cast doubt on the laboratory's compliance with its own policies and procedures may require an internal audit by the QA Department.

This scheme is generally accomplished through the use of Corrective Action Report Forms available to each of the laboratory areas within the LIMS system. Use of this report notifies the QA Department of a potential problem as described in SOP # 80.0007. The QA Director initiates the corrective action by relating the problem to the appropriate laboratory managers and/or project managers who then investigate or assign responsibility for investigating the problem and determine its cause. Once determined, the QA Director will approve appropriate corrective action. Its implementation is later verified through an internal laboratory audit. Once the QA Director feels the system has returned to control, s/he will finalize the CAR using a password protected QA step.

Information contained on corrective action reports is kept confidential within Spectrum Analytical, Inc. RI Division and is generally limited to the individuals involved. Severe problems and difficulties may warrant special reports to the President of Spectrum Analytical Inc., who will ensure that the appropriate corrective actions are taken.

Nonconformance:

Any breach of standard protocols is a nonconformance item that is documented on the Corrective Action Request Form and management informed immediately. The following are example nonconformance items:

1. Sample holding time exceeded.
2. Hoods, Class "1" weights, NIST Thermometers, balances, automatic pipettes, being used but not certified.
3. Expired standards being used.
4. Manual integration being misrepresented.

#### 16.1 Client Complaints:

Spectrum Analytical, Inc. RI Division ensures client complaints are dealt with quickly and completely. The policies are stated in the laboratory Standard Operating procedure (SOP # 80.0002 Client Complaints).

Figure 16-1

Quality Assurance Corrective Action Request Form

## 17.0 QUALITY ASSURANCE REPORTS TO MANAGEMENT

The Spectrum Analytical, Inc. RI Division Quality Assurance Director submits a QA report annually to upper management. The report should be completed and submitted no later than the 15<sup>th</sup> of July in any calendar year.

The report contains detailed laboratory information and QA activities during the previous twelve months. Items to include are the status of internal and external audits, client complaints, quality control activities, resources and staffing. See the following pages for the report format.

Management will review the QA report and respond to outstanding issues. Management will add a review of the suitability of policies and procedures, and any other relevant issues. The response report is due within 30 days of the QA Report receipt.

A copy of the report is kept on file in the QA department.

In case of a severe problem or difficulty, a special report is prepared by the QA Director and submitted immediately to management.



5. Proficiency Testing.

6. Changes in volume and type of work undertaken.

7. Client Feedback.

8. Reports from management and supervisory personnel.

## 18.0 SAFETY

Spectrum Analytical, Inc. RI Division maintains safety through a program managed by the Safety Officer and the Safety Committee. Responsibilities include many activities needed to comply with the Right-to-Know Laws.

- Training seminars with information on OSHA safety instruction for new employees.
- Introductory training to include location of fire extinguishers, first aid supplies, etc.
- Health and Safety manual review when hired.
- Annual Health and Safety Manual review and revision as needed.
- Monthly Safety Committee meetings.
- Centralized MSDS information.
- Maps with safety equipment and all exits noted.
- Posted safety rules.

If a chemical spill occurs, proper actions are described in Spectrum Analytical, Inc. RI Division's Contingency Plan. Additionally, the local fire department (Warwick) and hospital (Kent County) also have a copy in case a need arises. Each new hire is required to read the Contingency Plan and sign off on this. An annual meeting is held as a refresher for all employees. A copy of the Contingency Plan is located on the company Intranet and is available to all personnel.

Emergency equipment, such as spill control kits, fire extinguishers and fire blankets are located throughout the laboratory areas. The Contingency Plan has instructions for evacuation, notification of emergency authorities and regulatory personnel in the event of a chemical accident.

## 19.0 WASTE MANAGEMENT

### 19.1 Pollution Prevention

The waste management option of choice is to prevent pollution by minimizing the amount or types of chemical wastes that are generated. Spectrum Analytical, Inc. RI Division's ability to minimize waste generation is limited by the chemical analysis techniques that are required by the EPA or other authors of test methods. As new test methods are utilized in the laboratory, the type and volume of chemical waste generated by the new test is considered. Analysts and Supervisors are encouraged to look for ways to reduce the amount of chemical waste, or the type of chemical waste generated during the testing process; HOWEVER, no method is allowed to be modified without discussion among the Laboratory and/or Technical Director, QA Director and other management personnel to determine the affect of the change on the resulting data.

### 19.2. Waste Management

Spectrum Analytical, Inc. RI Division has identified and routinely disposes of chemical wastes in several hazardous waste streams. In general these are acids, caustics, solvent wastes and various laboratory waste solids. No laboratory chemical waste is disposed in the trash or dumped down the drain. All remaining sample volume following testing, and after contract-required disposal date has past, are disposed in one of these waste streams. These wastes are fully described in Spectrum Analytical Inc., RI Division's Contingency/Waste Management Plan and in the lab's Profile Log. New England Disposal Technologies is Spectrum Analytical, Inc. RI Division's waste hauler. Other hazardous wastes are identified and properly disposed according to these documents.

Continued compliance is monitored monthly by an outside consultant to ensure all RI DEM regulations are met. Key personnel attend an annual RCRA Facility Training, which focuses on the requirements for hazardous waste disposal and its proper documentation.

## **20.0 DEFINITIONS, ACRONYMS, ABBREVIATIONS:**

**ACCURACY:** The closeness of agreement between an observed value and an accepted reference value.

**ALIQOT:** A measured portion of a field sample, standard, or solution taken for sample preparation and/or analysis.

**ANALYTICAL SERVICES BRANCH (ASB):** The division of United States Environmental Protection Agency's (USEPA) Office of Superfund Remediation and Technology Innovation (OSRTI) responsible for the overall management of the Contract Laboratory Program (CLP).

**ASTM:** American Society for Testing and Materials, a developer and provider of voluntary consensus standards.

**BATCH:** A group of samples of the same matrix that are processed as a unit at the same time in the same location using the same method.. Unless defined differently by a specific analytical method (such as Oil & Grease by Method 1664), the maximum batch size is 20 samples.

**BIAS:** The deviation due to analytical or matrix effects of the measured value from a known spiked amount.

**BLANK:** A "clean" matrix analysis. Such as: Equipment Blank, Method Blank, and Trip Blank.

**BREAKDOWN:** A measure of the decomposition of certain analytes (DDT and Endrin) into by-products.

**CAS:** Chemical Abstracts Service, a registry where chemicals are assigned identification numbers.

**CCB:** Continuing Calibration Blank

**CCV:** Continuing Calibration Verification standard.

**CLP:** Contract Laboratory Program. A contract used by EPA to purchase analytical services. Also refers to the test protocols described in that contract. The CLP analyses can be used for EPA or for other clients. CLP-format data reports are arranged as described in the EPA CLP contract, including specified data report pages and all raw data.

**CONTROL SAMPLE:** A QC sample introduced into a process to monitor the performance of the system.

**DL:** Dilution, not used when the initial analysis is performed at dilution, but is used for a secondary dilution.

**DUPLICATE:** See Matrix Duplicate, Field Duplicate, and Matrix Spike Duplicate.

**EQUIPMENT BLANK:** A sample of analyte-free water that has been used during sample collection to measure any contamination introduced during sample collection.

**ICB:** Initial Calibration Blank

**ICV:** Initial Calibration Verification standard

**IDL:** Instrument Detection Limit. Statistical value similar to MDL, but with analyses performed on standards that have not been through the sample preparation process.

**FIELD DUPLICATES:** Independent samples that are collected as close as possible to the same point in space and time. They are two separate samples taken from the same source, stored in separate containers, and analyzed independently. These duplicates are useful in documenting the precision of the sampling process.

**LAB CONTROL SAMPLE (LCS):** A blank spiked with compound(s) representative of the target analytes. This is used to document laboratory performance in a “clean” matrix.

**MATRIX:** The component or substrate (e.g., water, soil, air, and oil) which contains the analyte of interest.

**MATRIX DUP (DUP):** A sample split by the laboratory that is used to document the precision of a method in a given sample matrix.

**MATRIX SPIKE (MS):** An aliquot of sample spiked with a known concentration of target analyte(s). The spiking occurs prior to sample preparation and analysis. A matrix spike is used to document the bias of a method in a given sample matrix.

**MATRIX SPIKE DUP (MSD):** Laboratory split samples spiked with identical concentrations of target analyte(s). The spiking occurs prior to sample preparation and analysis. They are used to document the precision and bias of a method in a given sample matrix.

**METHOD** An analyte-free matrix to which all reagents are added in the same  
**BLANK(MB):** volumes or proportions as used in sample processing. The method blank should be carried through the complete sample preparation and analytical procedure. The method blank is used to document contamination resulting from the analytical process.

**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 and is determined from analysis of a sample in a given matrix type containing the analyte. For operational purposes, when it is necessary to determine the MDL in the matrix, the MDL should be determined by multiplying the appropriate one-sided 99% t-statistic by the standard deviation obtained from a minimum of seven analyses of a matrix spike containing the analyte of interest at a concentration estimated to be three to five times the MDL, where the t-statistic is obtained from standard references.

**MSA:** Method of Standard Additions

**ND:** Not Detected. Used in conjunction with the reporting limit.

**ORGANIC-FREE REAGENT WATER:** For volatiles, all references to water in the methods refer to water in which an interferent is not observed at the reporting limit of the compounds of interest. Organic-free reagent water can be generated by passing tap water through a carbon filter bed containing about 1 pound of activated carbon. A water purification system may be used to generate organic-free deionized water. For semivolatiles, all references to water in the methods refer to water in which an interferent is not observed at the reporting limit of the compounds of interest. Organic-free reagent water can be generated by passing tap water through a carbon filter bed containing about 1 pound of activated carbon. A water purification system may be used to generate organic-free deionized water.

**PPB:** Parts Per Billion, ug/L, ug/Kg

**PPM:** Parts Per Million, mg/L, mg/Kg

**PQL:** Practical Quantitation Limit, Equivalent to Reporting Limit.

**PRECISION:** The agreement among a set of replicate analyses.

**PS:** Post Spike. Spike added at the analysis level (as opposed to at the beginning of sample preparation) to determine interferences.

**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 generally 5 to 10 times the MDL. However, it may be nominally chosen other than these guidelines to simplify data reporting. For many analytes the RL concentration is selected as the lowest non-zero standard in the calibration curve. Sample RLs are matrix-dependent, and are adjusted by the amount of sample analyzed, dilution, and percent moisture.

RE: Re-extraction or Re-analysis

RPD: Relative Percent Difference, used to determine precision.

RRF: Relative Response Factor. Used for quantification with the internal standard procedure.

RT: Retention Time for a chromatographic peak, as calculated from the time of injection.

SAMPLE: A portion of material to be analyzed that is contained in single or multiple containers and identified by a unique sample number.

SAMPLE DELIVERY GROUP (SDG): A unit within a sample Case that is used to identify a group of samples for delivery.

SERIAL DILUTION (SD): A five-fold dilution of a sample. When corrected by the dilution factor, the diluted sample must agree with the original undiluted sample within specified limits. Serial dilution may reflect the influence of an interferent.

SAMPLE MANAGEMENT OFFICE (SMO) - A Contractor-operated facility operated under the SMO contract, awarded and administered by USEPA.

SOP: Standard Operating Procedure.

STANDARD ADDITION: The practice of adding a known amount of an analyte to a sample immediately prior to analysis. It is typically used to evaluate interferences.

STANDARD CURVE: A plot of concentrations of known analyte standards versus the instrument response to the analyte. Calibration standards are prepared by successively diluting a standard solution to produce working standards which cover the working range of the instrument. Standards should be prepared at the frequency specified in the appropriate method. The calibration standards should be prepared using the same type of acid or solvent and at the same concentration as will result in the samples following sample preparation. This is applicable to organic and inorganic chemical analyses.

**SURROGATE:** An organic compound that is similar to the target analyte(s) in chemical composition and behavior in the analytical process, but which is not normally found in environmental samples.

**TRIP BLANK:** A sample of analyte-free media taken from the laboratory to the sampling site and returned to the laboratory unopened. A trip blank is used to document contamination attributable to shipping and field handling procedures. This type of blank is useful in documenting contamination of volatile organics samples.

From EPA SW-846, Revision 4, and other sources.

**SPECTRUM ANALYTICAL, INC. RI DIVISION**  
**MAJOR INSTRUMENTATION and EQUIPMENT LIST**  
**APPENDIX A**

**Spectrum Analytical, Inc. RI Division  
Equipment List**

**Department: Inorganics : Metals & Wet Chemistry**

Equipment	Manufacturer	Serial #	Date Received	Date in Service	Condition New/Used	Equipment ID	Location
ICP/OES	Perkin Elmer	077N3102302	Nov-03	Nov-03	New	Optima3	Metals
ICP/AES	Perkin Elmer	069N8060801	Nov-98	Nov-98	New	Optima2	Metals
ICP/MS	ThermoScientific	SN01407C	Oct-08	Dec-09	New	X1	Metals
Mercury Analyzer	Perkin Elmer	1131	Mar-00	Mar-00	Used	FIMS1	Metals
Mercury Analyzer	Perkin Elmer	10157071002	Jan-11	Feb-11	Used	FIMS2	Metals
GPR Centrifuge	Beckman Instruments	7M149	Apr-02	Apr-02	Used	Centrifuge	Unit 3
Total Organic Carbon Analyzer	Tekmar/Dohrmann	US03035002	Apr-03	Apr-03	Demo	TOC1	Unit 3
Flow Injection Analyzer	Lachat Instruments	A83000-1020	Apr-96	Apr-96	New	Lachat1	Unit 3
Ion Chromatograph	Dionex	95030498E980802	May-03	May-03	New	IC1	Unit 3
Spectrophotometer	Spectronic Instruments	3SGD332010	Apr-02	Apr-02	New	SPEC2	Wetchem
Spectrophotometer	Milton Roy Company	3310004028	Mar-06	Mar-06	New	SPEC3	Wetchem
Pensky Marten	Koehler 16200	5539	June-95	June-95	New	FLASH1	Wetchem
Dessicator	Sanplatec Corp	none	June-06	June-06	New	DryKeeper	Unit 3
COD Reactor	Hach Company	990900019429	Nov-03	Nov-03	New	COD1	Wetchem

COD Reactor	Hach Company	950200012193	Apr-02	Apr-02	New	COD2	Wetchem
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**Spectrum Analytical, Inc. RI Division  
Balance List**

<b>Equipment</b>	<b>Manufacturer</b>	<b>Serial #</b>	<b>Date Received</b>	<b>Date in Service</b>	<b>Condition New/Used</b>	<b>Equipment ID</b>
TOP-LOADING Balance	OHAUS	1121230069	2000	2000	New	TL10
Analytical Balance	Denver A-250	0070742	2010	2010	Used	AB-3
TOP-LOADING Balance	OHAUS Voyager	F2921120391055	2001	2001	New	TL9
TOP-LOADING Balance	Denver	0079896	2000	2000	New	TL1
TOP-LOADING Balance	OHAUS Precision Std.	C22427176	2002	2007	New	TL6
TOP-LOADING Balance	OHAUS Navigator	1121122373	2002	2002	New	TL11
TOP-LOADING Balance	OHAUS	CD8910	2000	2000	New	TL4
TOP-LOADING Balance	OHAUS Navigator	1122173423	2003	2003	New	TL12
TOP-LOADING Balance	OHAUS Scout Pro	7126212230	2007	2007	New	TL13

**Spectrum Analytical, Inc. RI Division  
Equipment List**

Department: Organic Prep

Equipment	Manufacturer	Serial #	Date Received	Date in Service	Condition new/used	Equipment ID
TurboVap II	Caliper	TV0845N14899	Jan-09	Jan-09	New	TV-4
TurboVap II	Caliper	TV0902N15012	Jan-09	Jan-09	New	TV-3
TurboVap II	Caliper	4364	Mar-08	Mar-08	Used	TV-2
TurboVap II	Caliper	Unable to view	Mar-08	Mar-08	Used	TV-1
Shaker	Glas-Col	412383	Mar-08	Mar-08	New	N/A
Water Bath	Precision Scientific	9508-005	Dec-95	Jan-96	Used	N/A
Nitrogen Concentrator Bath	Organomations	16526	Jun-97	Jun-97	New	NVAP1
Deionized Water Generator	Barnstead Thermodyne	582941018789	Jun-95	Jun-95	New	DI1
Accelerated Solvent Extractor ASE-350	Dionex	10031445	Apr-10	Apr-10	New	ASE1
Gel Permeation Chromatograph	J2/AccuPrep	P26D031	Aug-05	Aug-05	New	GPC3
Gel Permeation Chromatograph	J2/AccuPrep	Q25D003	Jul-06	Jul-06	New	GPC4
Gel Permeation Chromatograph	J2/AccuPrep	Q28C041			New	GPC5
Misonex Ultrasonic Disruptor	Sonic Dismembrator Fisher Model 550	Unable to view			New	OPH1
Misonex Ultrasonic Disruptor	Sonic Dismembrator Fisher Model 550	Unable to view			New	OPH2

Misonex Ultrasonic Disruptor	Sonic Dismembrator Fisher Model 500	Unable to view			New	OPH3
Misonex Ultrasonic Disruptor	Sonic Dismembrator Fisher Model 500	Unable to view			New	OPH4
Ultrasonic Cleaner FS30H	Fisher Scientific	RTB030721702	Apr-07	Apr-07	New	N/A
Centrifuge Centra CL-2	International Equipment Company	42606943			Used	N/A
Neslab Chiller CFT-150	Neslab	Unable to view			Used	N/A

**Spectrum Analytical, Inc. RI Division  
Equipment List**

**Department: Pest/PCB**

Equipment	Manufacturer	Serial #	Date Received	Date in Service	Condition New/Used	Equipment ID	Location
GC/ECD	Hewlett Packard	3336A55650	Oct-94	Oct-94	New	E1	Pest/PCB
GC/ECD	Hewlett Packard	3336A59890	Oct-94	Oct-94	New	E2	Pest/PCB
GC/ECD	Hewlett Packard	3235A45554				E3	Pest/PCB
GC/ECD	Hewlett Packard	US00032017				E4	Pest/PCB
GC/ECD	Agilent	US00037060				E5	Pest/PCB
GC/FID	Hewlett Packard	US00001898				F1	Pest/PCB

**Spectrum Analytical, Inc. RI Division  
Equipment List**

Department: Receiving

Equipment	Manufacturer	Serial #	Date Received	Date in Service	Condition New/Used	Equipment ID	Location
Dry Weight Oven	Thello	600011006			used	DWO	Unit 3
Walk in Cooler		Not Applicable			used	R1	Receiving
Cyrotary Shaker table	New Brunswick Sci. Co.	unable to read			used	n/a	Unit 3
pH meter	Eutech Instruments	1446253			new	WC-02	Unit 3
Kin model TNF24-3	Paragon Touch n Fire	324341				n/a	Unit 3
Stoppering tray dryer	FTS Systems Dura-Stop M	TD-12-90-133				n/a	Unit 3
Freeze Dryer	FTS Systems Dura-Dry MF	unable to see				n/a	Unit 3



**Spectrum Analytical, Inc. RI Division  
Equipment List**

**Department: VOA**

Equipment	Manufacturer	Serial #	Date Received	Date in Service	Condition New/Used	Equipment ID	Location
GC/MS	Hewlett Packard	3336A55963				V1	VOA
Auto sampler	OI	13193				V1	VOA
Concentrator	OI	J651460769				V1	VOA
GC/MS	Hewlett Packard	3336A58222				V2	VOA
Auto sampler	OI	13091				V2	VOA
Concentrator	OI	H340460074				V2	VOA
GC	Hewlett Packard	3336A56504				V3	VOA
Auto sampler	OI	C508411868				V3	VOA
Concentrator	OI	J430460188				V3	VOA
GC/FID/PID	Hewlett Packard	2843A21041				V4	VOA
Auto sampler	Tekmar/Dohrmann	90312004				V4	VOA
Concentrator	Tekmar/Dohrmann	88341012				V4	VOA

**Spectrum Analytical, Inc. RI Division  
Equipment List**

**Department : VOA**

Equipment	Manufacturer	Serial #	Date Received	Date in Service	Condition New/Used	Equipment ID	Location
GC/MS	Hewlett Packard	US00007055				V5	VOA
Auto sampler	OI	13462				V5	VOA
Concentrator	OI	J651460769				V5	VOA
GC/MS	Hewlett Packard	US00031343				V6	VOA
Auto sampler	OI	B03745A407				V6	VOA
Concentrator	OI	J651460769				V6	VOA
GC	Hewlett Packard	3140A37463				V7	VOA
Auto sampler	Tekmar/Dohrmann	US01170015				V7	VOA
GC/MS	Hewlett Packard	CN10411124	Oct-10	Nov-10	NEW	V10	VOA
Auto sampler	Tekmar/Dohrmann	US01157003	Oct-10	Nov-10	USED	V10	VOA
Concentrator	Tekmar/Dohrmann	US02021003	Oct-10	Nov-10	NEW	V10	VOA

## **Weight Sets**

### **Laboratory weights for daily calibration use:**

1. WT1-Organic Prep Weight Set
2. WT2-Organic Prep 100g
3. WT3-Organic Prep 300g
4. WT4-Organic Prep 1kg
5. WT5-Inorganics Weight Set
6. WT6-VOA Weight Set
7. WT7-Unit 3 Weight Set

### **NIST Weight sets used for monthly verification:**

1. W-01 Denver Instrument set: Serial number 98-121303 Class 1
2. W-03 Troemner set: Serial number 7283 Class 1

## Laboratory Information System Equipment

### 1. Data Collection:

1.1. Seventeen- Hewlett Packard (HP) chem station software for collecting GC and GC/MS data (below) and one Perkin Elmer (PE) Total Chrom for collecting data from the GC-TCD/SCD.

- 5 GC-ECD (GCSEMI)
- 1 GC-FID (GCSEMI)
- 6 GC-MS (MSSEMI)
- 5 GC-MS (MSVOA)
- 1 GC-Hall/PID (GCVOA)
- 1 GC-FID/NPD (GCVOA)

1.2. Hardware varies but is x86 compatible

1.3. OS is Windows, Various Versions (9x, NT, 2000,Xp)

### 2. Data Storage:

2.1. Dell Poweredge servers (Windows 2003 server)

2.1.1. Bernoulli (primary file server, non-organic instrument data)

- Dual core Xeon processor
- 4 GB RAM
- 1 TB storage
- Symantec Backup Exec 12.5
- Tape drive Quantum DLT-S4 (800-1600 GB)

2.1.2. Avogadro (organic instrument data)

- Dual P IV Xeon processors
- 2 GB RAM
- 105 GB storage
- Tape drive Seagate LTO-1 (100-200 GB)

2.1.3. Planck (database server)

- Dual P IV Xeon processors
- 2 GB RAM
- 450 GB storage
- Tape drive Seagate LTO-1 (100-200 GB) not currently used

2.2. Tapes are for daily backup, long term archiving and data restoration

### 3. Compound Identification:

3.1. Fourteen - Target 4.14 chromatographic software

3.2. Hardware is Intel based for Target 4.14

3.3. OS is Windows Xp

4. **Forms Generation:**

4.1. In-house forms generation LIMS modules for SW-846, ILM and ISM metals

4.2. In-house forms generation LIMS modules for SW-846, OLC, OLM/ASP and SOM organics

4.3. Hardware varies but is x86 compatible

4.4. OS is Windows, Various Versions (2000 and Xp)

**Spectrum Analytical, Inc. Rhode Island Division**

**CONFIDENTIALITY, ETHICS, and DATA INTEGRITY AGREEMENT**

**APPENDIX B**

## CONFIDENTIALITY, ETHICS, AND DATA INTEGRITY

The confidentiality, ethics, and data integrity agreement attached must be signed and dated by all new personnel associated with the data generated by Spectrum Analytical, Inc. Rhode Island Division. All said personnel will complete a training course and understand the information stated in the agreement. The course must include the ethical and legal responsibilities including the potential punishments and penalties for improper, unethical, or illegal actions. In addition, personnel are instructed on the importance of data confidentiality in both hard copy and digital forms. All personnel must fully understand this information before signing the agreement. A separate form is used for subcontractors and external auditors that request data for review.

Data Integrity training will be done on an annual basis. All employees are required to attend a training session or read a refresher document and sign off in hardcopy or through the digital SOP Database. All hard copy documents are stored in the employee's personnel file located in the QA Department.

All upper management personnel are required to sign a Non-disclosure Agreement which covers protecting confidentiality and proprietary rights. This Agreement is kept on file at the Spectrum Analytical, Inc., main offices in Agawam, Massachusetts.

**SPECTRUM ANALYTICAL, INC.**  
**FEATURING HANIBAL TECHNOLOGY**  
**Rhode Island Division**

**CONFIDENTIALITY, ETHICS AND DATA INTEGRITY AGREEMENT**

- I. I, \_\_\_\_\_ (*Name*), state that I understand the standards of confidentiality, ethics and data integrity required of me with regard to the duties I perform and the data I report in connection with my employment at Spectrum Analytical, Inc. Rhode Island Division.
- II. I agree that in the performance of my duties at Spectrum Analytical, Inc. Rhode Island Division.
- :
- A. I shall not improperly use manual integrations to meet calibration or method QC criteria, such as peak shaving or peak enhancement.
  - B. I shall not intentionally misrepresent the date or time of analysis by resetting computer or instrument date/time.
  - C. I shall not falsify analytical results.
  - D. I shall not report analytical results without proper analysis documentation to support the results; dry-labbing.
  - E. I shall not selectively exclude data to meet QC criteria, such as calibration points, without technical or statistical justification.
  - F. I shall not misrepresent laboratory performance by presenting calibration data or QC limits within data reports that are not linked to the data set reported.
  - G. I shall not represent matrix interference as basis for exceeding acceptance criteria in interference-free matrices, such as method blanks and Laboratory Control Standards (LCS).
  - H. I shall not manipulate computer software for improper background subtraction or chromatographic baseline manipulations.
  - I. I shall not alter analytical conditions such as EM voltage, GC temperature program, etc. from standards analysis to sample analysis.
  - J. I shall not misrepresent QC samples such as adding surrogates after sample extraction, omitting sample preparation steps, or over-spiking/under-spiking.
  - K. I shall not report analytical results from the analysis of one sample for those of another.
  - L. I shall not intentionally represent another individual's work as my own.

- III. I agree to report immediately any accidental or intentional reporting of non-authentic data by myself. Such report must be made to any member of Spectrum Analytical, Inc. Rhode Island Division Management or the QA Director (Hanibal Tayeh, Yihai Ding, Edward Lawler, Cinde Gomes, Sharyn Lawler) both orally and in writing.
  
- IV. I agree to report immediately any accidental or intentional reporting of non-authentic data by other employees. Such report must be made to any member of Spectrum Analytical, Inc. Rhode Island Division Management or the QA Director (Hanibal Tayeh, Yihai Ding, Edward Lawler, Cinde Gomes, Sharyn Lawler) both orally and in writing.
  
- V. Questions pertaining to confidentiality, ethics, and integrity may be posed to any of the above individuals.
  
- VI. I agree not to divulge any pertinent confidential information including but not limited to data and any other information about a project to outside sources without the prior consent from the client.

I understand that failure to comply with the above confidentiality, ethics and data integrity agreement can result in my immediate dismissal from Spectrum Analytical, Inc. Rhode Island Division.

\_\_\_\_\_  
(Signature)

\_\_\_\_\_  
(Date)

\_\_\_\_\_  
(Print Name)



## SUBCONTRACTORS

### CONFIDENTIALITY, ETHICS AND DATA INTEGRITY AGREEMENT

- I. I, \_\_\_\_\_ (*Name*), authorized representative of \_\_\_\_\_ (*Subcontractor*) state that I understand the standards of integrity required of me and the Subcontractor with regard to the duties performed and the data reported in connection with the analysis/analyses contracted by Spectrum Analytical, Inc. Rhode Island Division.
- II. Subcontractor agrees that in the performance of analysis for Spectrum Analytical, Inc. Rhode Island Division:
- A. Subcontractor shall not intentionally report data values or results that are not the actual values measured or observed;
  - C. Subcontractor shall not modify data values unless the modification can be technically justified through a measurable analytical process;
  - D. Subcontractor shall not intentionally report the dates and times of data analyses that are not the true and actual dates and times of analyses; and
  - D. Subcontractor shall not intentionally represent another's work as its own.
- III. Subcontractor agrees to report immediately any accidental or intentional reporting of non-authentic data to Spectrum Analytical, Inc. Rhode Island Division.
- IV. Subcontractor agrees not to divulge any pertinent information including but not limited to data and information about any Spectrum Analytical, Inc. Rhode Island Division projects to outside sources without the prior consent from Spectrum or its clients.

I understand that failure to comply with the above ethics and data integrity agreement can result in immediate termination of the subcontract relationship with Spectrum Analytical, Inc. Rhode Island Division.

\_\_\_\_\_  
(*Signature*)

\_\_\_\_\_  
(*Date*)

\_\_\_\_\_  
(*Name*)

\_\_\_\_\_  
(*Title*)

## Confidentiality Agreement for External Audits

During the course of the laboratory audit/assessment certain information may become available to the auditor/assessor that is confidential.

All sample-related and project-related information at Spectrum Analytical, Inc. Rhode Island Division is confidential between Spectrum Analytical, Inc. Rhode Island Division and its client.

Any information obtained during the course of this audit/assessment may be used for audit/assessment purposes only.

No information obtained during the course of this audit/assessment may be disclosed by the auditor/assessor to any outside party, regardless of affiliation with the auditor/assessor.

Auditor/Assessor (signature): \_\_\_\_\_

(Print name): \_\_\_\_\_

(Date): \_\_\_\_\_

Company/organization name: \_\_\_\_\_

QAF.0014

**Spectrum Analytical, Inc. RI Division  
Resumes of Key Personnel**

**APPENDIX C**

# **YIHAI DING**

## **Laboratory Director**

Mr. Ding has experience in a wide variety of analytical chemistry techniques, including GC, GC/MS, HPLC and FTIR. His expertise includes the operation, calibration and maintenance of sophisticated analytical instrumentation, and the efficient operation of state of the art environmental service laboratories.

Mr. Ding's responsibilities as Laboratory Director at Spectrum Analytical, Inc. Featuring Hanibal Technology Rhode Island Division, involves the daily coordination of all laboratory sections to insure the production of high quality data meeting customer's technical and schedule requirements. His duties in this role include overseeing department supervisors and analysts in the daily calibration, maintenance and troubleshooting of analytical instruments, monitoring schedules and holding times, analysis of samples, review of sample and QC data. He also is involved with the implementation of Standard Operating Procedures, documentation of instrument and method QC criteria and development of new methods and implementation of new analytical technology.

Mr. Ding's prior experience includes research into the mechanisms and kinetics of various biochemical processes. A large portion of this research has required the analysis of reactants and products using state-of-the-art chemical instrumentation. Mr. Ding has also taught chemistry and biochemistry laboratory courses at the university level.

### **EDUCATION**

#### **MIDDLE TENNESSEE STATE UNIVERSITY**

Murfreesbro, Tennessee

- Chemistry, MS

#### **JILIN UNIVERSITY**

Changchun, China

- Biochemistry, BS

### **RELATED EXPERIENCE**

2005-present

#### **Spectrum Analytical, Inc. Featuring Hanibal Technology Rhode Island Division (formerly Mitkem)**

Warwick, Rhode Island

- Laboratory Director

2005

#### **STL LABORATORIES**

Savannah, Georgia

- Supervisor of Semi-Volatile GC and GC/MS
- GC/MS Analyst

- GC/ECD Analyst

1998-2005

**MITKEM CORPORATION**

Warwick, Rhode Island

- GCMS Supervisor for both Volatile Organics and Semi-Volatile Organics Laboratories
- GC/MS Analyst

1994-1998

**MIDDLE TENNESSEE STATE UNIVERSITY**

Murfreesboro, Tennessee

- Researcher
- Laboratory Instructor, chemistry and biochemistry

1993-1994

**NATIONAL ENZYME ENGINEERING LAB**

Changchun, China

- Researcher



-Proposal/Contract Manager  
-Director of Project Management

1983-1989

**CAMBRIDGE ANALYTICAL ASSOCIATES, INC.**  
Boston, Massachusetts  
-Project Manager  
-Volatile Organic Laboratory Manager

1978-1983

**ENERGY RESOURCES COMPANY, INC. - ERCO**  
Cambridge, Massachusetts  
-Volatile Organics (GC) Manager  
-Analytical Chemist-Volatile Organics Lab  
-Analytical Chemist-Organic Preparation Lab

1978

**LAPUCK LABORATORIES, INC.**  
Watertown, Massachusetts  
-Analytical Chemist-Wet Chemistry & Metals  
-Microbiologist

# **SHARYN B. LAWLER**

## **Quality Assurance Director**

Ms. Lawler has over twenty years of experience in the environmental laboratory industry. She has experience in implementation, operation and management of QA systems operating under USEPA, US Army Corps of Engineers and NELAC programs.

Ms. Lawler's responsibilities as Quality Assurance Director include development and implementation of the Quality Assurance Plan and Standard Operating Procedures. Her duties include interacting with federal and state regulatory officials in the acquisition and maintenance of laboratory certifications. She is also responsible for managing Spectrum Analytical, Inc. Rhode Island Division's document control program. Ms. Lawler performs both internal and external audits as well as overseeing the corrective action system, training program and evaluating QC check samples.

Previously Ms. Lawler was a senior data reviewer, where she was responsible for final QA/QC review of organic, metals and wet chemistry data. She insured final data met all method and in-house QC criteria prior to release to the customer, and that any issues were documented and described for inclusion in case narratives. A significant portion of this work involved review of full CLP-format data deliverables packages, both for standard as well as non-routine analyses. Prior to Spectrum Analytical Inc., Ms. Lawler worked for two CLP laboratories where she held positions including senior data review specialist, CLP Organics Task Manager and analyst in several laboratory sections.

### **EDUCATION:**

#### **UNIVERSITY OF MASSACHUSETTS**

Amherst, Massachusetts

Independent Conc., Coastal Plant Ecology, BS

### **RELATED EXPERIENCE:**

1997-Present

#### **Spectrum Analytical Inc., Featuring Hanibal Technology RI Division (formerly Mitkem)**

Warwick, Rhode Island

- QA Director

- Senior Data Reviewer

1988-1997

#### **NATIONAL ENVIRONMENTAL TESTING**

Bedford, Massachusetts

- Senior Data Reviewer
- CLP Organic Task Manager

1983-1988

**CAMBRIDGE ANALYTICAL ASSOCIATES**

Boston, Massachusetts

- CLP Organic Task Manager
- Semivolatiles Analyst
- Preparation Laboratory Analyst

# SCOTT P. HUNTLEY

## IT Manager

Mr. Huntley has over twenty years experience in the environmental testing field. He has considerable experience in computer sciences and had been involved, throughout his career, in the setup and implementation of several Laboratory Information Management Systems (LIMS) and automated data reduction systems. Mr. Huntley's responsibilities include the set-up and validation of automated data transfer, reduction, storage, evaluation and reporting programs within Spectrum Analytical, Inc. RI Division's LIMS. He also is responsible for set-up of the electronic data delivery capabilities as well as the control charting capabilities of this system.

Previously Mr. Huntley has held several supervisory positions in environmental laboratories focusing on CLP and other DOD analytical programs. He has a wide range of experience in routine and state of the art analytical programs and methods. Mr. Huntley is experienced in the use of automated data transfer and reduction systems and laboratory automation techniques.

### EDUCATION:

#### RHODE ISLAND COLLEGE

Providence, Rhode Island

Chemistry, BS

Computer Science, BS

### RELATED EXPERIENCE:

1999-Present

#### Spectrum Analytical, Inc. Featuring Hanibal Technology RI Division (formerly Mitkem)

Warwick, RI

- MIS Senior Systems Analyst

1996-1999

#### MITKEM CORPORATION

Warwick, RI

- Senior Chemist

- Organic Lab Manager

1991-1996

#### EA LABORATORIES

Sparks, MD

- Supervisor of Organic Chemists

1989-1991

#### CEIMIC CORPORATION

Narragansett, RI

- Night shift supervisor

1986-1989

**RI ANALYTICAL LABORATORIES**

Providence, RI

- GC Chemist

# **AGNES R. HUNTLEY**

## **Project Manager**

Ms. Huntley has gained extensive and diversified experience in environmental laboratories using U.S. EPA CLP and SW846 methodologies, as well as participating in US Navy and Army analytical services programs.

Ms. Huntley's responsibilities involve the management of Spectrum Analytical Inc. Rhode Island Division's EPA Contract Laboratory Program (CLP) analytical services contracts. This includes the daily oversight of incoming samples, maintenance of chain of custody documentation and communication records and resolution of any discrepancies or other issues involving CLP sample assignments. Her responsibilities also include ongoing communication with EPA, sampler and CSC personnel, as well as monitoring data delivery schedules, writing project narratives and finalizing case communication. Ms. Huntley has managed four contracts with the EPA, which included one Organics Low Concentration (OLC), two Organics Low/Medium Concentration (OLM) and one Inorganics Low/Medium Concentration (ILM) analytical services contracts. At present Ms. Huntley manages the Organics Multi-Media, Multi-Concentration (SOM01.2) Analytical Services Contract.

Previously, Ms. Huntley held the position of QA/QC Manager where her responsibilities included the development and implementation of Standard Operating Procedures, documentation of instrument and method performance using Method Detection Limit studies, and routine review of final laboratory data reports, review of analyst training and performance data and management of the corrective action system. Her duties also included interaction with federal and state regulatory officials in the acquisition and maintenance of laboratory certifications.

Prior experience includes management of the daily operations of the Organic Preparation Laboratory. Duties in this position included monitoring sample backlog, holding times, process work flow, and delivery due dates. Ms. Huntley also developed and implemented new test methods, trained laboratory staff, performed instrument maintenance and reviewed sample and QC data. Prior to joining Spectrum Analytical Inc. Ms. Huntley worked as an analytical chemist at NET Cambridge Division performing analyses under a wide variety of programs including Army COE, Navy NEESA, DOE HAZWRAP and EPA CLP.

### **EDUCATION**

#### **SIMMONS COLLEGE**

Boston, Massachusetts

- Chemistry, BS
- Mathematics, BS

### **RELATED EXPERIENCE**

- 1997-Present
- Spectrum Analytical Inc. Featuring Hanibal  
Technology Rhode Island Division (formerly Mitkem)**  
Warwick, Rhode Island
- Project Manager, SOM Contract manager
  - Supervisor, Sample Receiving Department
- 1997-2008
- MITKEM CORPORATION**  
Warwick, Rhode Island
- CLP Project Manager
  - QA/QC Manager
  - Manager, Sample Preparation Laboratory
- 1995-1997
- NATIONAL ENVIRONMENTAL TESTING**  
Bedford, Massachusetts
- Chemist, Organic Preparation
- 1992-1995
- SIMMONS COLLEGE CHEMISTRY DEPT.**  
Boston, Massachusetts
- Teaching Assistant, Chemistry Department

# **HUIYAN HEATHER ZHAO-ANDERSON**

## **Inorganics Department Manager**

Ms. Zhao-Anderson is employed as the Manager in Spectrum Analytical Inc. Rhode Island Division's Inorganics Department. Ms. Zhao-Anderson's responsibilities involve the coordination of metals and wet chemistry analyses using ICP/MS, ICP/AES and a variety of other instrumentation following both US EPA CLP and SW846 protocols. Her duties in this role include supervising analysts in the daily calibration, maintenance and troubleshooting of analytical instruments, monitoring schedules and holding times, analysis of samples, review of sample and QC data. She is involved with the implementation of Standard Operating Procedures, documentation of instrument and method QC criteria and development of new methods and implementation of new analytical technology. Ms. Zhao-Anderson also insures the production of inorganics organic data is coordinated with other laboratory sections. Prior to managing the inorganic department, Ms Zhao-Anderson was the department manager of our volatile organics laboratory for several years.

### **EDUCATION**

**Yale University**  
New Haven, CT  
School of Forestry and  
Environmental Study, MS 2005

**Peking University**  
Beijing, China  
Environmental Science and Economics  
BS 2002

### **RELATED EXPERIENCE**

09/2005 -Present

**Spectrum Analytical Inc. Featuring  
Hanibal Technology Rhode Island  
Division (formerly Mitkem)**  
Warwick, RI

- Manager, Inorganic Department
- Manager, VOA Department
- GC/MS Chemist, VOA Laboratory

# Catherine L. Mosher

## Semivolatiles (SVOA) Department Manager

Ms. Mosher has experience in a wide variety of analytical chemistry techniques, including GC/FID and GC/MS. Her expertise includes the operation, calibration and maintenance of sophisticated, computer controlled instrumentation. Her expertise also includes analyses and QA review of forensics extended alkylated PAH and Biomarker analyses.

Ms. Mosher is employed as the Organics Department Manager in Spectrum Analytical Inc. Rhode Island Division, and oversees both the Volatile and Semivolatile departments. Ms. Mosher's responsibilities involve the coordination of organics analyses using GC/MS and GC instrumentation following both US EPA CLP and SW846 protocols. Her duties in this role include supervising analysts in the daily calibration, maintenance and troubleshooting of analytical instruments, monitoring schedules and holding times, analysis of samples, review of sample and QC data. She is involved with the implementation of Standard Operating Procedures, documentation of instrument and method QC criteria and development of new methods and implementation of new analytical technology. Ms. Mosher also insures the production of organic data is coordinated with other laboratory sections.

### EDUCATION

**Community College of Rhode Island**  
Warwick, Rhode Island  
Certificate of Chemical Technology - 1991

### RELATED EXPERIENCE

02/2007-Present

**Spectrum Analytical Inc. Featuring  
Hanibal Technology Rhode Island  
Division (formerly Mitkem)**

Warwick, RI  
- Manager, SVOA Department  
- Senior Scientist, SVOA Laboratory

05/2005 – 12/2006

**Alpha Woods Hole Laboratories**

Rahnham, MA  
- Development of Volatile Air Laboratory  
- Supervisor for Organics analyses  
including GC/MS VOA and SVOA,  
ECD's and FIDs  
- Forensic Team Leader

03/1997 – 05/2005

**Woods Hole Group Laboratories**

Raynham, MA

- Forensic Team Leader
- GC/MS Group Leader

04/1996 – 03/1997

**Inchcape Testing**

New Bedford and Raynham, MA

- Semivolatile analyst
- Volatile analyst

09/1991 – 04/1996

**Energy and Environmental Engineering Inc.**

Somerville, MA

- Semivolatile GC/MS Supervisor
- GC-Pesticide/PCB analyst

01/1989 – 09/1991

**New England Testing Laboratory**

North Providence, RI

- Senior Chemical Technician - including  
Organic, Inorganic, Metals, and  
Microbiology analyses

10/1987 – 09/1988

**Rhode Island Analytical Laboratory**

Warwick, RI

- Chemical Technician

# **DAWNE SMART**

## **Data Reviewer, Project Manager**

Ms. Smart's responsibilities as project manager involve the management of Spectrum Analytical Inc. Rhode Island Division's EPA Contract Laboratory Program (CLP) analytical services contract for ISM. This includes the daily oversight of incoming samples, maintenance of chain of custody documentation and communication records and resolution of any discrepancies or other issues involving CLP ISM sample assignments. Her responsibilities also include ongoing communication with EPA, sampler and CSC personnel, as well as monitoring data delivery schedules, writing project narratives and finalizing case communication. Ms Smart also reviews sample and QC data, and completed CLP data packages for both organic and inorganic programs.

Ms. Smart was previously the Manager of the Inorganics Department. In this role, she had oversight of analysts in the daily calibration, maintenance and troubleshooting of analytical instruments and the analysis of samples. Dawne supervised technicians responsible for sample preparation for metals and mercury analysis and the analyses of soil and water samples for nutrients, minerals and demand as well as cyanide. She was also responsible for monitoring schedules and holding times.

Ms. Smart has extensive experience in Data Review as well as Quality Assurance. A significant portion of her previous employment included management of the Data Review department as well as the on-site QA Specialist for a major specialized laboratory.

### **EDUCATION**

#### **COMMUNITY COLLEGE of RHODE ISLAND**

Warwick, Rhode Island

Certificate of Chemical Technology - 1991

Associate in Applied Science - 1997

### **RELATED EXPERIENCE**

2007-Present

#### **Spectrum Analytical Inc. Featuring Hanibal Technology Rhode Island Division (formerly Mitkem)**

Warwick, RI

- ISM Contract manager

-Manager, Metals Department

-Supervisor, Inorganic Department

1999 – 2007

#### **ALPHA WOODS HOLE LABORATORIES**

Raynham, Massachusetts  
-QA Specialist  
-Manager, Data Review Department

1996 – 1999

**ANALYTICAL BALANCE COMPANY**  
Middleboro, Massachusetts  
- Department Head, Metals Analysis

1995 – 1996

**FOXBORO COMPANY**  
West Bridgewater, Massachusetts  
- Chemist

1988 – 1995

**NEW ENGLAND TESTING  
LABORATORY**  
North Providence, RI  
- Senior Laboratory Technician  
- Laboratory Technician

1987 – 1988

**RHODE ISLAND ANALYTICAL  
LABORATORIES**  
Warwick, RI  
- Metals Preparation Technician  
- Laboratory Assistant  
-

# THOMAS E. SAWYER

## Metals Analyst

Mr. Sawyer has experience in a wide variety of analytical chemistry techniques, including ICP/AES, ICP/MS, HPLC and Ion Chromatography using EPA MCAWW, SW-846, ILM as well as the new ISM methodologies. His expertise includes the operation, calibration and maintenance of various forms of chemical analysis instrumentation. Prior to his employment at Spectrum Analytical Inc. Rhode Island Division, Mr. Sawyer worked for another environmental testing laboratory, Ceimic, which held EPA CLP contracts in inorganic and organic analyses. Mr. Sawyer was the metals laboratory supervisor and lead analyst for ICP/MS on Ceimic's EPA CLP ILM contracts.

Mr. Sawyer's responsibilities at Spectrum Analytical Inc. Rhode Island Division include the daily calibration, maintenance and troubleshooting of analytical instruments, preparation of calibration and spiking standards, analysis of sample digestates, and review of sample and QC data. He also is involved with the development of new methods and implementation of new analytical technology.

Mr. Sawyer's prior experience included extensive research positions within the Department of Environmental and Molecular Toxicology and the Department of Microbiology at Oregon State University. Mr. Sawyer has extensive publication credits in the fields of environmental microbiology and biochemistry.

## EDUCATION

### University of Maine

Orono, Maine

- Microbiology, MS-1990

### Colby College

Waterville, Maine

- Environmental Science, BS-1987

## RELATED EXPERIENCE

2006-present

### Spectrum Analytical Inc., Featuring Hanibal Technology Rhode Island Division (formerly Mitkem)

Warwick, Rhode Island

- Metals (ICP/MS, ICP/AES)Analyst
- IC Analyst
- HPLC Analyst

1996-2006

### CEIMIC CORPORATION

Narragansett, Rhode Island

- Supervisor of Metals Department
- ICP/MS Analyst
- IC Analyst
- HPLC Analyst

1993-1996

**OREGON STATE UNIVERSITY**

Corvallis, Oregon

- Faculty Research Assistant in Department of Environmental and Molecular Toxicology
- Faculty Research Assistant in Department of Microbiology

1987-1992

**UNIVERSITY OF MAINE**

Darling Marine Center Walpole, Maine

- Researcher Technician II
- Graduate Research Assistant

QAP Revision Page:

**06/01/2011:** Full Revision with laboratory name change and edits from EPA SOM QAP Audit

**07/27/2011:** minor edit to section 9 (missing ICP/MS calibration information). Footer has revision "A" added to date.