LABORATORY QUALITY ASSURANCE MANUAL

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- **Bold=one of the 23 elements required (Section 5.4 NELAC EPA600/R-04/003) for the quality manual and related quality documentation. Sections not in bold are relevant references for supplemental explanation of the requirements in the NELAC Standard.**

Section 2.0

Mission Statement

LABORATORY QUALITY ASSURANCE MANUAL

Our Mission is to be the leader in analytical services in terms of Quality, Service, and Value.

In order to achieve this goal, corporately and individually we must:

- Conduct our business to the highest ethical standards
- Operate within a Quality system that meets or exceeds the requirements of Regulators and our Customers
- Provide a complete, responsive, value added service that strives to exceed customer expectations.
- Deliver what we promise in terms of quality, turnaround time and performance to specifications.
- Work with our customers to solve their problems.
- Continuously improve our services based on customer feedback and internal review.
- Provide our employees with a challenging and rewarding work environment.
- Operate our business in a safe and environmentally friendly manner.

PURPOSE AND DESCRIPTION

The Southwest Analytical Quality Manual provides the guidance to laboratory personnel in fulfilling their responsibilities to produce quality data, and it serves as a statement of SA, Inc.'s commitment to quality. Implementation of the Program is management's responsibility. Management has the duty and authority to insist that these responsibilities are met, and that constant improvement be the goal. The procedures set forth herein must be followed to the extent possible. All deviations must be documented in each individual case and maintained as a matter of record.

To verify that the Quality System is successfully implemented, the Laboratory is audited regularly by various regulatory authorities, outside agencies and clients.

LABORATORY CAPABILITIES

Drinking water
Wastewater
Groundwater
Soils
Hazardous Wastes
Leachates (TCLP)
Microbiology

Section 3.0

3.0 MANAGEMENT REQUIREMENTS

Corporate Management

SA, Inc. operates a full-service laboratory facility. The laboratory is managed by a Laboratory Manager with full responsibility for the technical and financial performance of the facility. It is the goal of management to provide to clients a high-quality service in a timely manner. The corporate management and the Laboratory Manager are the entities which are held legally responsible to compliance with state and federal laws. It is management's responsibility to see that the lab carries out its testing and calibrations activities in such a way as to meet the requirements of the NELAC standard, to satisfy clients, and regulatory authorities overseeing the NELAC program. This plan applies to testing that is performed within the confines of the lab location, namely, 4208 Arcata Way, North Las Vegas, NV. Southwest Analytical is a full service lab that does undertake endeavors that would create a conflict of interest. An organizational chart is found in Appendix B.

4.0 ORGANIZATION AND MANAGEMENT

4.1.1 Laboratory Organization

SA, Inc. management will staff its laboratory with degreed chemists and highly trained technicians. These personnel will have strong technical backgrounds, high personal standards of service and quality, and experience in a variety of analytical specialties.

Laboratory Management will insure:

- That managerial and technical personnel will have the authority and resources needed to carry out their duties and to identify the occurrence of departures from the quality system or standard testing and calibration procedures and to initiate actions to prevent or minimize such departures,
- That processes exist to ensure management and personnel are free from any undue internal and external commercial, financial and other pressures and influences that may adversely affect the quality of their work.
- That policies and procedures exist to ensure the protection of clients' confidential information and rights, including electronic storage and transmission of results,
- That policies and procedures exist to avoid involvement in any activities that would diminish confidence in it competence, impartiality, judgment or operational integrity,
- That a definitive organizational and managerial structure so as to define the relationships between quality management, technical operations, and support services and also to specify the responsibilities, authority and interrelationships of

all personnel who manage, perform or verify work affecting the quality of test performed,

Section 4.0

- That adequate supervision will occur for all junior staff and trainees by senior, experienced staff knowledgeable in the methods and test procedures.
- That technical management will have the responsibility and means to provide resources needed to ensure the required quality of lab operations and that this technical director or lab manager will certify that only personnel with the appropriate education and or technical background perform tests for the laboratory is accredited and that such certification shall be documented.
- That the lab will participate in a proficiency test program for the purpose of qualifying and maintaining accreditation.
- That personnel will be appointed to key positions, i.e., technical director, quality manager etc., when the need arises,

4.1.2 QUALITY ASSURANCE ORGANIZATION

A Quality Assurance Manager shall be named with the key responsibilities and authority for ensuring that the quality system is implemented and followed at all times.

The QA Manager duties shall include:

- Serving as focal point for QA/QC and be responsible for the oversight and/or review of quality control data, reporting to the Lab Manager and Corporate Lab Management.
- Have functions independent of lab operations for which they have quality assurance oversight.
- To be able to evaluate data objectively and perform assessments without outside influence.
- Have documented training and experience in QA/QC procedures and be knowledgeable of NELAC requirements.
- Have general knowledge of the tests methods for which data review is performed.
- Arrange to conduct annual internal audits, and
- Notify lab management of deficiencies in the quality system and monitor corrective actions.
- Maintain the Quality Manual

In addition, each Laboratory Supervisor is responsible for assuring that the appropriate quality control measures are conducted with their group and that the continual updates made to the quality assurance program are responsibly carried through by all personnel in their group. Laboratory Supervisors are kept informed of

QA activities through department meetings, memos, and individual meetings with the QA Manager.

4.1.3 Confidentiality of Client Information

Analytical results will only be released to a third party when there is written authorization to do so. Clients who submit their samples to the lab through a consultant/client manager are assumed to have given their permission for the release of their results to that consultant.

4.1.4 Ethics Policies on Waste, Fraud, and Abuse

All employees will read and sign the Ethics Policy Addendum found on page 77.

4.2 QUALITY SYSTEM

4.2.1 QUALITY POLICY AND OBJECTIVES

QUALITY ASSURANCE, ASSESSMENT, AND QUALITY CONTROL

QUALITY ASSURANCE DEFINED

Quality Assurance (QA) is the integrated program for insuring data reliability.
A quality assurance program is a system for integrating the quality planning, quality assessment, and quality improvement efforts of various groups in an organization to enable operations to meet user requirements at a regulatory and economic level.

QUALITY ASSURANCE PROGRAM SUMMARY

Southwest Analytical's Quality Assurance Program encompasses the following items:

- Full commitment to producing high quality data.
- A full time Quality Assurance Manager to run the QA/QC Program
- Written SOP's for analyses, lab practices, and a review and revision process to same.
- PE samples for four different matrixes analyzed twice annually.
- All lab personnel are thoroughly trained before being permitted to analyze samples with only normal supervision.
- Keeping lab personnel informed concerning client needs.

Data is needed because of:

- Regulatory compliance
- Engineer design testing
- Risk assessment

- Enforcement and clean-up

Components Essential in Designing an Analytical Program:
- Knowledge of what compounds must be analyzed, equipment and skills needed.
- Essential detection limits in order to report to MCL or client requested levels.
- All NELAP required and Method QA/QC requirements.
- What the Analyst should ideally know before analyzing samples:
  - Background of the project
  - All objectives and especially the ultimate objective
  - Sample media
- Potential interferences and hazards
- Data package requirements

QUALITY ASSURANCE OBJECTIVES

- To provide a monitoring system to assure the reliability (accuracy and precision) of reported results are the best possible.
- To control the quality of all laboratory activities involved in sample handling, sample analysis, and data reporting.
- To verify that QC requirements are met whether they pertain to analytical methods, clients, contracts, or the agencies that certify Southwest Analytical, Inc.
- To provide a formalized yet flexible quality assurance program for each analytical test, thus precluding the need for analysts to originate individual QC efforts.
- To provide an easily retrievable documentation system for all quality control/quality assurance activities.
- To assure clients of SA, Inc, continual commitment to excellence in every aspect of analytical services.
- To have laboratory personnel responsible for the production of reliable and usable data.

QUALITY ASSESSMENT

Included in the program are quality assessments taken by personnel and the documentation of laboratory performance as specified in the Quality System. The program is an essential part of a sound analytical protocol for use by individuals and laboratories to detect and correct problems in the measurement process or to demonstrate that the analytical process is in a state of statistical control. The objective of a quality assurance program is to produce analytical results of acceptable or agreed-upon uncertainty. The following techniques are employed to assessment quality:

Internal Techniques:
- Repetitive Measurements

- Internal Test samples
- Control Charts
- Interchange of Operators
- Interchange of equipment
- Independent Measurements
- Audits
- Blind Sample Analysis
- Introspection based on a desire for excellence

External Techniques:
- Performance Evaluation Samples
- Round Robin Analysis
- External Reference Materials
- Standard Reference Materials
- Audits

QUALITY CONTROL DEFINED

Quality Control (QC) techniques include all practices and procedures that lead to statistical control and to the achievement of the accuracy requirements of the measurement process.

QC is the daily, specific actions taken within the laboratory to verify sample integrity, performance of analyses, data processing, and record maintenance. It is a system of inspections, testing, and remedial actions applied to processes or operations to estimate sample quality and to determine any changes necessary to maintain or achieve a required level of quality.

Basic Elements of Quality Control:

- Technical Competence of Staff
- Suitable Facilities and Equipment
- Good Laboratory Practices (GLPs)
- Good Measurement Practices
- Standard Operating Procedures (SOPs)
- Protocols for Specific Purposes, Project QA Plans or Statements of Work
- Inspections
- Documentation
- Training

Once the competence of the technical staff and the suitability of the facilities are assured, the focus of Quality Control is the practices of the staff. Quality Control then involves the routine application of all quality assessment procedures taken to

demonstrate that an analytical system is **in control**. Monitoring of that system and documentation to prove it, is the goal. Monitoring and documentation from sample collection through data reporting are required. Quality assessment data in written form is a must for an effective quality control program.

QUALITY ASSURANCE ORGANIZATION

The Director of Laboratory Operations assumes full responsibility for the reliability of all analytical results and for meeting laboratory QA objectives. The appointed QA Manager, however named, is the next person in the organization to coordinate, oversee, and implement the quality system. All supervisors are also responsible for assuring that all appropriate quality control measures are conducted within their groups or work cells. Continual upgrades need to be implemented. Each analyst, as well, is responsible to complete all Quality Assurance requirements of this manual.

ACCREDITATIONS, CERTIFICATIONS, AND METHODOLOGIES

ACCREDITATIONS AND CERTIFICATIONS

Nevada Department of Environmental Protection- Lab ID # NV0052
California Department of Health Services-Environmental Lab Accreditation Program- Cert. # 2002

METHODOLOGIES

ASTM-Annual Book of Standards (American Society for Testing and Materials)

EPA-Sampling and Analysis Procedures for Screening of Industrial Effluents for Priority Pollutants: Test methods for Evaluating Solid Waste ((i.e. SW846 and TCLP (40 CFR Part 261); 40 CFR Parts 50, 51.)

EPA-Methods for the Determination of Organic and Inorganic Compounds in Drinking Water, Vol. 1, and Supplement. III.

4.2.2 DOCUMENTATION OF THE QUALITY SYSTEM

The Quality Manual identifies the minimum policies, procedures, and methodologies adopted by all Southwest Analytical personnel to ensure that we consistently deliver the highest standard of quality and service to our clients. The policies and procedures

cited in this document are binding on all affected personnel and conform to NELAC requirements. This policy, in addition to day-to-day use, will be used by Southwest Analytical in staff training programs and will be used to meet requirements for lab accreditation.

This quality manual will be maintained current on an annual basis under the responsibility of the QA Manager.

4.3 DOCUMENT CONTROL

This manual does not include all the information necessary for complete implementation of the Quality System. Additional information can be found in various SOPs written for that distinct purpose.

These SOP's are retained and maintained up-to-date by the QA Manager and are readily available to all lab personnel in laboratory computer network.

A document control system is established which insures that all procedures, manuals, or documents clearly indicate the time period during which the procedures or document was in force. Additional information regarding procedures for document control can be found in *SOP 4103 present revision*, *Document Control and Record Disposition*.

4.4 REVIEW OF SOLICITATION, OFFER OR CONTRACT

Upon receipt of a Request for Proposal, Request for Quotation, etc., the Lab Manager, in conjunction with the Sales Staff, and Client Services would review and investigate the documents. A determination is then made as to scope of work, detection limits, time frames, and special QA/QC requirements. A determination would be made as to the appropriateness of the request and whether the lab could perform the project. Sales would pursue answers to any questions with the proposed client. Sales would complete the Request For Quotation following the gathering of the information needed for the Lab Manager final decision.

4.4.1 AMENDMENT TO CONTRACT

The Lab Manager would oversee any amendments to existing contracts and coordinate with group leaders and Sales staff.

4.5 SUBCONTRACTING OF TESTS

Commercial laboratories must subcontract certain test to qualified laboratories for various reasons throughout their daily activities in support of their customers.

Infrequently requested tests, niche market analyses, laboratory work loads, instrument failures all require a commercial laboratory to employ a subcontractor to fulfill obligations of service to a client.
All laboratories performing subcontracted service to SA, Inc., must have the required certifications to perform the testing under specific governmental driven programs if it is mandatory under the compliance program. A Quality Manual must be on hand and a copy of the certification. These must be updated annually and maintained by the QA Manager. The client must be aware that subcontracting will be necessary and will be informed at all times. Commitments are requested of subcontracting labs as to their expected delivery of data and final packages.

4.6 PURCHASING

When the lab purchases outside services and supplies, other than those referred to in this standard, in support of tests, the lab will require documentation to be provided from the appropriate governmental agency if applicable. This will provide assurance of the quality of the outside support services.

The lab maintains on file, the documentation in the form of subcontracted Chain-of-Custody for the outsourcing of tests not performed in the lab. For information regarding standards and reagents, traceability, labeling and documentation, see *SOP1002 current revision, Documentation of Standards and Reagent Preparation* and the appropriate sections of this Manual.

4.7 CLIENT SERVICES

Customer Service is an important and integral part of laboratory operation. From set-up to follow-up, the Customer Service Representative department has the greatest impact on the success of a client's analytical program. All business accounts are going to measure the responsiveness, effectiveness, and overall quality of the laboratory through their interaction with the customer service rep. They are the interface with the client and the key to any future relationship with them.

The Lab Manager is directly responsible for the Customer Service Group, including Sales, Customer Service Reps, Bottle department, log-in clerks, couriers, and Reporting.

4.7.1 PROJECT PLANNING

Tests and associated instrumentation are for commercial (volume) applications, and therefore, most of the work that is brought to the facility is to be completed following standardized EPA or ASTM testing procedures that have been incorporated in the facilities SOPs.

Sales and Customer Service are responsible for the type and quality of work that is accepted and promoted by SA, Inc. RFQ's (Requests for Quotations) and/or specific customer requests that are outside of standard products are reviewed by the laboratory management as to the viability of the request and the ability of the lab to successfully complete the objective of the scope. Sub-contracting, MDL studies for non-standard compounds, scheduling adjustments may be needed for the project's requirements. The objective of having prior notification of sample receipt or prior notification of any client expectation is to have as much **lead time** as possible to react the client's needs.

Customer Service assists in the review and formalization of the tests that performed, associated bottle ware for the field, labeling, test methodology to be requested, adequate and proper preservation reagents and the communication to the field. They also communicate with the lab regarding sample deliveries and holding times associated with the sample delivery groups. Customer Service is, by this point in the process, well aware of the scope and the necessary requirements within the project description.

As the client's samples are logged-in and processed, any discrepancies or issues will then be addressed as soon as possible through Customer Service, and work will not be initiated without the resolution of the outstanding concerns.

4.7.2 ORGANIZATIONAL AND TECHNICAL INTERFACE

The Customer Service Department has the ongoing responsibility to interface regularly with clients in the field and lab personnel. Any delays on either side of the project need immediate communication to the other to minimize problems. The lines of communication must be kept open at all times and remedies imposed if at all possible to keep the project on track. All **project deviations** should be brought to the attention of the Lab Manager, Group Leaders and the QA Manager. When a modification is necessary to employ methods that have not been established as standard, these will be subject to agreement with the client, be fully documented, and validated, and be available to the client and other recipients of the reports.

4.8 COMPLAINTS

Customer concerns are recognized as very important feedback within the lab organization. Services contracted by public companies, private organizations, municipalities, or by individuals bring different expectations and ideas. From time to time, an individual's expectation may not be met according to their measure, and a concern results from this shortfall. The Lab Manager and QA Manager monitor all concerns through the use of Customer Care Forms and when necessary Corrective Action Forms. When a concern is brought to the Customer Service Rep, a Customer Care form is initiated and given to the Lab Manager or the QA Manager. An investigation is initiated and if necessary a Corrective Action Report is performed.

The results are transmitted to the client as quickly and as completely as possible. If necessary, a corrected lab report will be issued and documented as a re-issued report. A letter should accompany the report explaining the corrective action procedures. These letters are signed by the Lab Manager or QA Manager.

4.9 CONTROL OF NONCONFORMING TESTING

If a client should question an analytical result, the analysis must be reviewed thoroughly in order to alleviate the client's concerns, as well as, to ensure that the lab is not suffering from a procedural problem.

A concern shall be documented with a Customer Care Form. This form will contain the general information of the data in question. Following the investigation by the Lab Manager and/or the QA Manager, the form and any other supporting documentation will be filed with the associated raw data and the client's final report. It will be reviewed by the QA Manager and the final report/letter will be signed by the Lab Manager.

The data inquiry may cause a Corrective Action to be initiated and that may lead to a correction of a final result, or a reanalysis of the sample.

4.10 CORRECTIVE ACTIONS ARISING FROM CUSTOMER CONCERNS

CORRECTIVE ACTION DOCUMENTATION

Corrective Actions are undertaken as soon as possible and the following are examples of when they should be performed:

- When an analyte on a Performance Evaluation is failed or when a "Check-for Error" on limits occurs.
- When a client questions a result and error was found on the part of the lab.
- When an analyst makes an error, or fails to document any result with QC issues and the results get reported to the client.
- When an error is found to be in lab notebooks and results have been reported to the client.
- When instruments are found to be malfunctioning and results are given to a client.

4.11 PREVENTIVE ACTION

There are preventive actions and long-term improvements the lab can make to prevent non-conformance and/or reoccurring non-conformance. The QA Manager observes instruments and analyst problems and brings these cases to the Lab

Manager for possible courses of action, to include replacement of equipment, re-training of the analyst involved, change in SOPs, other internal changes, or personnel replacement.

4.12 QUALITY RECORDS

4.12.1 RECORD KEEPING PRACTICES

RECORD KEEPING

See laboratory SOP 4103 current revision. SA, Inc., in its attempts to make results technically sound and legally defensible, has adopted the following guidelines for analysts.

All information about an analysis must be written down in a notebook or bench notebook, this includes, solution preparation, solution standardization, sample amounts, dilution factors, concentration factors, initial and final volumes, calibration information, QC data, corrective actions if QC results are out of spec, initials and signatures, which documents who performed the analysis and that the QC was acceptable and that the SOP was followed, notes on any changes in the procedure with approval from the Group Leader and QA Manager, comments, observations, and additional information might be used at a later date.

BENCH NOTEBOOKS-For routine and frequent analyses pre-printed report forms may be created and bound into bench notebooks for recording of data for a specific test or instrument. These are on the Lab network and are maintained by the Group Leaders and the analysts, with approvals by the Lab Manager and QA Manager. Each book should be used chronologically so that once it is started for a parameter all data of that type for that analysis or instrument should be recorded in it until it is full. Pages shall be numbered and the book will be logged into the Lab Master List on its creation. It will be annotated in the Lab Master List when the book is retired and where it is archived.

BOUND PARAMETER NOTEBOOKS-For routine but infrequent analyses, a bound notebook will be kept for recording all raw data. Raw data includes all information necessary for the calculation of the final result of that particular parameter. One notebook should be used exclusively for a given parameter and may be set up in chart or table form. These should be logged into the Lab Master List and used chronologically.

RULES FOR ALL DATA NOTEBOOKS

- No pages will be torn out of any bound logbook
- Pages shall be paginated

- Notebook entries must be legibly written and must be organized and clearly understandable to others. Other lab personnel must be able to recreate all calculations and understand how they were done.
- All calculations should be as described in the SOP and include units. OR if there are variances, explanations must be given.
- All entries must be in black indelible ink
- DO NOT erase or white out any entry in a notebook or similar document.
- Mistakes must be lined out and initialed and dated.
- When a page is not going to be filled to the bottom line or "Z" out the remainder, and sign across the diagonal line.
- Do not use ditto marks, but instead use a lined arrow coming down the page.
- Initials of analyst should appear somewhere on each page
- Initials of peer reviewer should appear somewhere on each page.
- The following should appear on each page of the notebook where applicable.
    - Lab Sample ID
    - Date and time of analysis
    - Instrument ID or reference to where this info is available
    - Operating conditions or where this info is available
    - Name of analysis
    - Example of the Calculation
    - Analyst's signature and date.

4.12.2 RECORDS MANAGEMENT SYSTEM

NUMBERING SYSTEM FOR DATA NOTEBOOKS

Bound notebooks are each assigned a sequential number when they are put into service. The identification number, purpose and date are logged into a bound notebook, the Lab Master List of Notebooks. This is retained in the QA Office and kept by the QA Manager.

ARCHIVING

While analysis is in progress, all project files are maintained at the Customer Service Desk in the active file. When analytical results are complete they are input by analysts into the LIMS system, (OMEGA), and QC'd by a peer or supervisor. Once completed, the Customer Service Coordinator, or Reporter issues a final letter, case narrative, result report pages, and QC pages for the Lab Manager or his designated reviewer to approve and sign the data package. At this time a Level IV data package, if needed, will be assembled in a timely fashion. In addition, EDD (Electronic Data Deliverables) may also be required in conjunction with the delivery of the hard copies. The Reporter prepares the EDDs and e-mails them to the client. Once signed, this project activity file is moved to the project completed file. These file cabinets are

rotated such that prior years are moved to bankers boxes and stored in storage for 7 years.

Notebooks, Bench Notebooks, and other books logged into the Lab Master List Log book, once filled, should be returned to the QA Manager for archival and re-issue of a new logbook.

All QA official documents, all log books from all departments, should be archived by the QA Officer and retained for 7 years.

Original raw data is kept in each specific department and periodically boxed and placed in the archival area. These are retained 7 years.
The following SOPs are offered at additional information concerning archival of records, record keeping, etc., *Data Reduction and Validation, SOP 4002, Data Review Process, SOP 4009, Document Control and Record Disposition, SOP 4103, current revision of each*.

Electronic Data Deliverables are generated using the OMEGA software. There are a number of EDD formats that are client specific. These are created, edited, reviewed, e-mailed to clients, and archived electronically on the LIMS Network system.

4.13 INTERNAL AUDITS

4.13.1 SYSTEM AND METHOD AUDITS

In-house quality audits are one way to evaluate the labs quality assurance practices.

INTERNAL SYSTEMS AUDITS

An annual audit to include each of the areas listed below is conducted by the QA Manager.

Organics-GC	Inorganics-Sample Preparation
Organics-GC/MS	Inorganics-General Chemistry
Physical Testing	Inorganics-Metals
Organic-Sample Preparation	Microbiology

- A sample is selected at random from the work area to be audited.
- The handling of the sample from receipt through reporting is tracked.

- All types of records associated with the sample are checked for completeness, adequacy, neatness, comprehensibility and ease of retrieval.
- Adequacy and completeness of records is determined by whether or not they follow the requirements of the referenced method, SOP and this QA Manual.
- Comments and recommendations are written into a formal audit letter and submitted to the Lab Manager and Department Supervisor.
- If Corrective Actions are deemed necessary or other actions, these follow-up procedures will be followed and monitored by the Lab Manager, QA Manager, and Group Leader and the records filed with the audit findings in the QA Office.

METHOD AUDITS

Method audits will be performed by the QA Manager, Group Leader or Laboratory Manager.

A method audit occurs when one specific method, SOP, analyst, instrumentation and all connected with the method are audited. On an annual basis, all methods should be audited, and an annual schedule of planned audits should be planned and published.

4.13.2 AUDIT REVIEW

All records associated with these audits and any Corrective Actions generated from method audits, are maintained by the QA Manager. All results are reported to the Lab Manager and archived by the QA Manager. If any client data or results are impacted from method audits, all such clients need informed for the impact on their data regardless of time passed. These formal letters need maintained and archived with the Method Audit findings as well as the clients files.

4.14 MANAGEMENT REVIEW

On an annual basis it is preferred that corporate management will conduct internal management system audits of the SA. Inc., facility. A review of the internal audit system, accounting practices, Client Services practices, Sales department practices, and all aspects of the lab operations, short of method and internal system audits performed by lab personnel and the QA Manager, should be visited for completeness, thoroughness and effectiveness in relation to client service and profitability. Client feedback, Client disputes and Corrective Actions should all be reviewed.

Section 5.0

5.0 TECHNICAL REQUIREMENTS

5.1 PERSONNEL

5.1.1 REQUIREMENTS

PERSONNEL / EXPERIENCE

A list of job descriptions for key personnel at Southwest Analytical, Inc. is in Appendix C.
Key personnel are defined as analysts or individuals key in the analyses of samples and the reporting of same to clients. Non-Key personnel have no role in the analysis of samples, but have duties of critical importance to the lab. The following are a list of key personnel titles:

- Laboratory Director
- Quality Assurance/Quality Control Analyst
- Laboratory Supervisor, Organics and Inorganics
- GC/MS Operator
- Analyst I, Analyst II, and Analyst III
- Laboratory Technician

Non-key personnel include:
- Client Service Representative
- Sample Receptionist
- Bottle Coordinator
- Courier
- Sales Representative
- Accounting Clerk
- Data Reporting Clerk
- Data Deliverable Coordinator
- IT Analyst

5.1.2 TRAINING

TRAINING AND RECORDING OF ANALYST PROFICIENCY

Thorough training is very important for both a safety and quality control point of view. Southwest Analytical, Inc. has implemented the following training program:

Each analyst will have a training file stored and maintained in the QC Office files containing the following:

- A training/acknowledgement form for every SOP required to fulfill the duties assigned the analyst. At a minimum this will include, ethics training, document handling and log books, reagent receiving, log books, bench notebooks, safety, chemical hygiene, and any specific SOP pertaining the analysts' job duties.
- An Initial Demonstration of Capability (IDC) or Demonstration of Capability (DOC) complete set of MDLs, a 4 point replicate series on the proper NELAP form, a blank and an internal blind sample result, or a Performance Evaluation performed and recorded as part of the lab ongoing certification process.
- For a newly hired analyst, the list above will be collected and sent to the state certifying officer for approval before samples are analyzed unsupervised for clients. If an analyst has personally passed an on-site state audit with the certifying officer this approval is not necessary, but the same documentation should be on record.
- A resume depicting past experience in lab work.
- Any training documents such as, certifications, training awards, transcripts.

SUPERVISORS RESPONSIBILITY, TRAINING AND DATA INTEGRITY

The proficiency of the analysts will be determined by the satisfactory analysis of QC samples and review of their performance of the method by their supervisor. The supervisor will enter the information on the analysts' Record of Training form and attach a copy of the QC sample results and the acceptance criteria. Each Group Leader or Department Supervisor is responsible for training and insuring that each analyst is familiar with all basic lab SOPs, as required by each analytical method they perform. This training is meant to insure that operator and laboratory error can be minimized if approved techniques are consistently practiced. Supervision means insuring the continued use of good technique, periodic review of those techniques and the pointing out of areas needing improvement. In addition to this, data integrity shall be taught formally from the Ethic Policy Statements, as well as other appropriate sources. This training will include, signed ethics statements, training records, close periodic monitoring and documentation on such monitoring.

5.2 ACCOMODATIONS AND ENVIRONMENT

LABORATORY FACILITIES

Southwest Analytical, Inc.

The Southwest Analytical, Inc. is a full service laboratory located at 4208 Arcata Way, North Las Vegas, NV. The lab occupies about 10,000 square feet of floor space in a one story building.

Lab facilities
The lab consists of the following work areas:

- Shipping and receiving, bottle orders, sample storage.
- Metals Sample prep lab
- Organic extraction/sample prep
- Inorganic instrument room,
- Wet Chemistry room
- Semi-volatile GC/MS and GC room
- Volatile GC/MS room
- HPLC room
- Microbiology room
- Receptionist area,
- QA Office
- Additional office support areas.
- Large storage areas/garages

Each area is separated from neighboring areas where activity is incompatible. Work area environments are maintained and monitored to reduce and eliminate contamination and to facilitate proper testing performance. Work areas are restricted to the appropriate laboratory personnel.

A diagram of the lab floor plan is in Appendix D.

GOOD LABORATORY PRACTICES

LABORATORY ETIQUETTE

Refill all solutions bottles for the next person.
Order needed items; don't take the last one of any item without checking on stocks or reordering.
Cleanup your own messes and work area.
Return borrowed or shared items to the proper place so they can be used by others.
Be considerate of fellow employees.

GLASSWARE PREPARATION

All glassware will be cleaned according to established laboratory SOPs which can specific to the analytical procedures.

REAGENT WATER

"Reagent water: is defined as water which an interactent is not observed at the method detection limit of the parameters of interest." USEPA SW846 3rd Edition.

Southwest Analytical employs an extensive tap water clean up procedure using reverse osmosis and a Nanopure purification system. This water is test semi-annually for all parameters tested at SA. Inc.

5.3 TEST METHODS AND SOPS

5.3.1 PROCEDURAL DOCUMENTATION

ANALYTICAL METHODS

Southwest Analytical, Inc. uses published ASTM methods, and established an approved
EPA methods. All of SA. Inc., analytical SOPs are written to reflect these methods.

These methods can be found in:

ASTM-ANNUAL BOOK OF STANDARDS, (AMERICAL SOCIETY FOR TESTING AND MATERIALS) 19th edition.

EPA-SAMPLING AND ANALYSIS PROCEDURES FOR SCREENING OF INDUSTRIAL EFFLUENTS ROR PRIORITY POLLUTANTS, TEST METHODS FOR EVALUATION SOLID WASTE, (SW846 AND TCLP (40 CFR PARTS 50-51)).

EPA-Methods for the Determination of Organic and Inorganic Compounds in Drinking Water, Vol. 1, and Supplement. III.

STANDARD OPERATING PROCEDURES

SA, Inc. has an *SOP 4030 current revision for the Reviewing and Revising of SOPs.* This SOP will be updated to include the 23 items required by the current NELAP manual, which include the following required topics (where applicable):

- Identification of the test method with signatures of authorizing lab personnel
- Applicable matrix or matrices
- Detection limits
- Scope and application

- Summary of the test method
- Definitions
- Interferences
- Safety
- Equipment and supplies
- Reagents and standards
- Sample collection, reservation, shipment and storage
- Quality control
- Calibration and standardization
- Procedure
- Calculations
- Method performance
- Pollution prevention
- Data assessment and acceptance criteria for QC measures
- Corrective actions for out-of-control data
- Contingencies for out-of-control data
- Waste management
- References
- Any tables, diagrams, flowcharts, and validation data

Every SOP has a training record attached to the back of each SOP for use in documenting training. An example of that training record is found in the Appendix B.

SOPs are written by the analysts and supervisors for all methods performed at SA, Inc. They are then reviewed by the QA Manager and Lab Director. When an SOP is revised or corrected and a new revision number will be assigned. All current SOPs are located in a read only file on the laboratory network and is available to all staff. Old SOPs will be removed from its current file placed into a retired file. The new, signed revision will be assigned a new revision number and dates of effectiveness and then it will be placed into the current file.
SOPS are assigned unique numbers based on the current system.
These include the following delineations.

General Lab Procedures-1000's
Sample Management-2000's
Microbiology-3000's
Quality Assurance-4000's
Physical Property Determinations-5000's
Metals-6000's
Inorganic-Non-Metals Determinations-7000's
Organic Determinations-8000's

The SOP Index and Table of Contents showing all current SOPs will be maintained by the QA Manager and kept on file in the same location as the final SOP versions.

LABORATORY SAFETY

General safety rules are enforced. Special safety rules as required by specific situations shall be established and followed. General safety rules include the following:

1. Safety glasses are worn at all times in the extraction laboratory and where posted.
2. Solvents are stored in vented fire-proof storage cabinets located in the storage room.
3. Hoods are turned on whenever an employee is using organic solvents or mineral acids in the extraction laboratory. A portion of the exhaust system remains on at all times to ensure that the vapors do not build up in the building.
4. Eye washes, safety showers, fire blankets and fire extinguishers are maintained in working order and each employee is instructed in their use.
5. A general laboratory safety kit is maintained in the extraction laboratory.
6. Material Safety Data Sheets (MSDS) are maintained in the break room and are accessible to employees at all times.

THE EMPLOYEE SAFETY COMMITTEE

An employee safety committee meets regularly and is responsible for formulating Safety policies and recommending them to laboratory management.

LABORATORY SAFETY GUIDES

SAFETY IS EVERYONES RESPONSIBILITY-THINK SAFE-WORK SAFE

The Do's:

- Know the safety limits of the chemicals you will use.
- Check your equipment before you start and again while in use
- Be sure you are using all mechanical safety devices, shields, hoods, etc. before you start and that they will handle any contingency.
- Protect eyes, face, hands and body. This means the safety glasses, or face shields, proper gloves, respirators, and protective clothing. Over-protect, rather that under-protect.
- Practice good house-keeping. Dispose of waste properly, clean up spills immediately.

- Know locations of eye baths, safety showers, fire extinguishers, and the exits.
- There is never any smoking allowed in the lab.
- Make safety a habit. Think safe

The Don'ts:

- Do not take chances
- Do not overlook toxicity
- Do not fail to ask for help or guidance
- Do not work alone in the lab with flammable, toxic, or corrosive materials if it can be avoided.
- Do not overlook the possibility of splashing hazards
- Do not wear loose clothing, jewelry or long hair
- Do not pipette with your mouth
- Do not block aisles
- Do no panic.

A GENERAL LABORATORY SAFETY CHECKLIST

Management and Safety Committee duties

- Education of personnel-management and supervisors
- Safe working conditions-all workers and management
- Training in first aid-Management
- Periodic inspections-management and safety committee.

Safety awareness

- Training of employees
- Safety meetings
- Safety bulletins
- Education and precautionary placards
- Periodic inspections

Laboratory Equipment and safety features

- Adequate exits, aisles, stairways, etc.
- Properly designed doors
- Exhaust hoods
- Ventilators
- Lighting
- Furniture arrangement
- Storage facilities
- Safety showers

- Fire extinguishers
- Personal protective gear
- Equipment setup and faculty power to code.

HAZARDOUS WASTE DISPOSAL

The hazardous waste disposal at Southwest Analytical, Inc. is the responsibility of the Lab Manager who maintains the necessary permits and interfaces with the appropriate local offices for inspections and permitting. All employees need to be familiar with the methods of handling and disposing of hazardous wastes generated from all of the procedures they perform. These are generally disposed of in waste storage barrels in the back of the facility. Each barrel is label as the type of waste that can be dumped into the barrel. These markings must be observed and heeded. Specific instructions for waste disposal are detailed in *SA, Inc. SOP 5030, Hazardous Waste Disposal.*

5.3.2 METHOD VALIDATION

DEMONSTRATON OF ANALYTICAL CAPABILTY

Prior to the certification of a test method by the regulating authorities, the lab must perform a Demonstration of Capability, (DOC) which shall confirm that it can properly operate the method before results can be reported. In addition, every key analyst shall have a DOC on file and submitted to regulatory authorities for approval, prior to analyzing samples. Any major changes in the method will require a repeat of the DOC for the lab, such as a new piece of equipment replacing an older model, change in primary personnel doing the testing, or in the method itself.

In all cases, the appropriate forms such as the NELAP Certification Statement (Appendix C) must be completed and retained by the laboratory to be made available upon request. Work Cells or analysts and those who are doing extractions sharing parts of the sample analysis procedure may share in the DOC and all must have forms documenting their parts in the analysis. If any part of the Work Cell is replaced or leaves, the DOC must be re-established with the new analysts.

For primary analyst, the following should be on file for their personal DOCs, an initial calibration of the instrument, a method blank, four midlevel calibration or Lab control spike replicates, an MDL study and the above Certification Statement, filled out, signed and filed.

Southwest Analytical, Inc. does not use non-standard methods.

DETECTION LIMITS

The Method detection limit (MDL) is defined as the minimum concentration of a substance that can be identified, measured, and reported with 99% confidence that the analyte is greater than zero and determined from the analysis of a sample in a given matrix containing the analyte.

The MDL may vary as a function of matrix type.
Procedure:

- Make an estimate of the detection limit. Many methods publish goals or examples of past MDL studies done by the developing scientists who wrote the method.
- Prepare in analyte free reagent water
- If the MDL is to be determined in reagent water, prepare a lab standard in reagent water 1 to 5 times the estimated MDL.
- Analyze a minimum of 7 aliquots, if a blank measurement is required to calculate the measured level, make a separate blank measurement for each aliquot.
- Calculate the standard deviation of the 7 results
- Compute the MDL as follows:

MDL= t (n-1)*s

Where: t (n-1) = the student t value at 99% confidence (see table below)
S= Standard deviation of the sample at (n-1)

Table of Student's t Values as 99% confidence level

Number of Replicates	Degrees of Freedom	t (n-1) at 99%
7	6	3.143
8	7	2.998
9	8	2.896
10	9	2.822
11	10	2.764
16	15	2.602
21	20	2.528

MDLs are evaluated to determine if they are acceptable in establishing the PQLs or the lab Reporting Limits, (RLs). PQLs are generally 3 to 5 times higher than the MDL and the PQL should be equal to or less than 50% of the MCL or Maximum Contaminant Level, as published for Drinking Water parameters and for a Profile One NDEP form 01. See Appendix D.
The calculated MDL should be reasonable. If the standard deviation of replicate measurements is too low, the calculated MDL value may be dubious. **An MDL of zero (0) is not realistic.** As a general rule, ten (10) times the MDL should be greater than the spike amount used to determine the MDL (10 X MDL > Spike Amount). If not, then the concentration of the MDL spike is too high. Conversely, if there is too much variation in the recovery of the MDL spikes, then the spike amount is too low. The appropriate spiking level for MDL determinations is method, analyst and instrument dependent. A trial and error,

process of elimination is the path used by most to identify the correct MDL spike level. In most cases there should be about 25-35% variability in MDL per cent spike recovery. However, greater variability does not invalidate the MDL.
Once established, MDLs can be on going. With each batch of samples analyzed, analyze a CCV or LFB at the same concentration used to calculate the established MDL. Compile the data and after seven analytical batches recalculate the MDL using the data acquired from the analysis of the low standard. The low standard can be used to bracket analysis and provide MDL information on a continuing basis. An acceptable procedure for evaluating the MDL can be found in 40 CFR Appendix B of part 136 (7). It describes an iterative procedure to address unreasonable MDLs. The following guidelines suggest a more "user friendly" approach to evaluate the MDL.

5.3.3 CONTROL OF DATA

MDLs are placed in the Omega system in each method as part of complete record keeping and for the use of data qualifiers when required. All analysts and their supervisors are responsible for the input of their current MDLs into the Omega software in a timely fashion. In addition, copies of current MDLs should be stored at the bench in three ring binders, and in the QA Office in a binder containing all MDLs. They can also be placed in personnel records as part of the DOC. MDLs should be performed annually, and filed accordingly.

5.4 EQUIPMENT

5.4.1 INSTRUMENT LIST

SA, Inc. MAJOR INSTRUMENTATION

Analysis	*Method(s)*	*Instrument Type*	*No. Of Instruments*
Volatile Organics	624/8260/524.2	HP 5890/5970/71 GC/MS	3
Semi-Volatile Organics	625/8270	HP 5890/5971 GC/MS	2
Semi-Volatile Organics	525.2	HP 6890/5973 GC/MS	1
TPH-Extractable 8151,	8015-M	HP 5890 GC, FID/FID	2
TPH-Gasoline/BTXE	8015/8021/602	HP 5890 GC, FID/PID	1
Pesticides/PCB's EDB/DPCA	608/8081/8082/508 504	HP 5890 GC, ECD/ECD	3
N & P Pesticides	507, 614, 8141	HP 6890 GC, NPD/FID	1
Metals-ICP	200.7/6010	THERMO IRIS Intrepid II X	1

Analysis	*Method(s)*	*Instrument Type*	*No. Of Instruments*
ICP-MS	200.8/6020	PE Sciex Elan 6000	1
Mercury	245.1/7470/7471	CETAC CVAA	1
Anions Perchlorate	300.0 314.0	Metrohm 733,762,732,709, Dionex 4000i	1 1
Inorganics	Misc.	Lachat Quick Chem 8000	1
TRPH	418.1	Miran 1FF IR/PE-297 Buck 404	1 1
GPC Cleanup	3040	ABC SP-1000	0
Accelerated Solvent Extraction	3545	Dionex ASE200	2

5.4.2 EQUIPMENT MAINTENANCE SPECIFICATIONS

All laboratory equipment is maintained in a reasonable and proper state of repair. No equipment is used until it is ascertained that it is in a safe and reliable operational state and then only by personnel who are thoroughly trained and duly qualified as operators. Instrument manuals are placed for ready access in convenient locations in the room that contains the instrument. Space is maintained according to rules of cleanliness, order and efficiency to facilitate the measurements and to protect the health and safety of the staff.

The laboratory has established the following preventive maintenance procedures:

1. Maintenance Logbooks are maintained which describes routine inspection, cleaning, maintenance, testing, calibration, and/or repair of equipment. Instrument operating manuals are maintained within easy access of the instrument.

2. Analysts using the instruments are trained operators and can trouble-shoot equipment problems to reduce equipment failure and to reduce dependence on outside service agencies. When necessary, however, outside service agencies will be used.

3. Written records are kept to document all equipment inspection, maintenance, trouble-shooting, calibration, or modifications. All equipment maintenance is documented in a logbook kept near the equipment as a means of monitoring the adequacy of maintenance schedules. The records contain the date (month, day, year), description of the maintenance done, the findings, the name of the maintenance person, and a statement of whether the maintenance operations were routine and if those operations followed the written SOP and/or the operating manual. The records shall also contain a description of the problem

and all steps taken to correct the problem and their result, whether or not they were effective.

4. Performance criteria are established for judging when data from instrument performance checks indicate the need to make equipment adjustments.

5. Tracking of internal standard areas, surrogate recoveries, etc., can serve to alert the analyst to trends that could be indicative of instrumental problems that can be corrected with preventative maintenance.

5.4.2.1 Chromatographic Instruments

The need for preventive maintenance can be ascertained from daily performance checks and continuing calibration checks. Parameters such as retention time and response factors are observed and back-checked with prior operational performance and QC criteria established in EPA methods, such as SPCC and CCC requirements in GCMS methods or peak shape, background noise, etc.

Other preventive maintenance includes:

- GC detectors are cleaned whenever performance degradation (i.e., calibration criteria are not met, retention time shifts, noisy baseline, etc.) is observed.
- Septa are replaced frequently.
- Incoming gas drying cartridges are replaced whenever a color change of the adsorbent is noticed; effluent adsorbent traps are changed every 6 months.
- Columns (GC and HPLC) are checked by performance and operating conditions when in use or prior to use.
- Check the kel-F seals, piston seals, piston flush seals, pistons, check valves and pump heads every six months on the HPLC systems. Clean all parts in methanol. Replace the seals, pistons and check valves when necessary.

5.4.2.2 Gas Chromatography/Mass Spectrometer (GC/MS)

The preventive maintenance includes:

Maintenance Requirements	Frequency
• Check pump oil level	Monthly
• Change mechanical pump oil	Every year or sooner if deemed necessary
• Clean source	When performance indicates
• Clean printer inside and outside	When needed
• General cleaning of instrument	Every 4 months
• Sensitivity analysis, BFB and DFTPP tune criteria	Every 12 hours/24hours

5.4.2.3 Atomic Absorption Spectrophotometers/ICP

Preventive Maintenance includes the following checks:

- Minimum 30-minute warm-up period.
- Alignment of hollow cathode tube/entrance slip for ICP to provide the maximum emitted light to the detector.
- For flameless AA, the inert gas flow inside the furnace is optimized to ensure maximum sensitivity and graphite tube checked for deterioration.
- Digital readout values obtained for the standard curve of each element are checked to ensure linearity.
- If readings are low prior to analysis, the operator checks the gas flows, burner or cell alignment·, wavelength, slit width, photo multiplier voltage, and lamp intensity.
- Burner heads, nebulizers, quartz cells, and reduction flasks are cleaned according to manufacturer instructions whenever excessive noise is apparent or whenever indicated by visual inspection.
- Tygon tubing is replaced when deterioration is apparent.
- Optical lenses are cleaned as needed.

5.4.2.4 General Laboratory Equipment

Balances

Analytical balances are calibrated annually by a licensed specialist who records an official verification of performance. Prior to use, calibration of balances is certified with standard ASTM Class 1 calibration weights that bracket the weight of interest. The resultant weights are checked to see if they meet acceptance criteria and are recorded in the balance logbook. The proper procedures for use and maintenance of balances are outlined in SOP 1020.

pH Meters

The pH/specific-ion meters are calibrated before use with a minimum of two standard solutions. All combination pH electrodes will be stored according to instruction manual. The proper use and maintenance of pH meters is outlined in SOP 7150.

Water

The DI water system is to be checked by a resistivity meter for conductivity and maintained at a purity of 1 megohm/cm. Weekly checks are recorded in a logbook to alert the technician to any trends, thus allowing proper preventative maintenance to occur. New deionizing and filtration cartridges will be installed if purity falls below 1 megohm/cm. The high purity water system is also checked and maintained at a

purity of 18 megohm/cm. Weekly checks are recorded in a logbook to alert the technician to any trends, thus allowing proper preventative maintenance to occur. New deionizing and filtration cartridges will be installed if purity falls below 16 megohm/cm.

The ASTM type 2 reagent water used for microbiological analyses is tested for the following parameters at the stated frequencies and must meet the listed acceptance criteria:

Parameter(s)	Acceptance Criteria	Test Frequency
Conductivity	< 2 micromhos/cm @ 25 ^{o}C	Monthly
Metals (Pb, Cd, Cr, Cu, Ni, Zn) Heavy Metals, Total	Not greater than 0.05 mg/L per contaminant, and no greater than 0.1 mg/L <= 0.1 mg/L	Annually Annually
Total chlorine residual Ammonia/organic Nitrogen	< 0.1 mg/L < 0.1 mg/L	Monthly Monthly
Heterotrophic plate count	< 1000 CFU/mL	Monthly

All microbiological reagent water checks are recorded in a QC logbook to alert the technician to any trends, thus allowing proper preventative maintenance to occur.

5.4.3 Contingency Plan For Equipment Failure

SA, Inc. wherever practical, maintains all of its instruments on a routine schedule as outlined above. This ensures all instruments are running at optimal efficiency and decreases instrument down time. SA, Inc. keeps an ample supply of common replacement parts. In the event of equipment failure, the instrument can be rapidly repaired for most common failures. However, equipment failure is unavoidable and for that reason we have developed a contingency plan for dealing with this eventuality.

SA, Inc. maintains redundant instrumentation to ensure we have backup equipment in the event of an equipment failure. SA, Inc. also requests additional volumes of samples from the clients so that an equipment failure does not consume the client sample.

1. Upon failure, the analyst notifies the Area Supervisor and QA Manager of the problem.

2. The Area Supervisor and QA Manager determine the best course of action to take with the samples. The options are
   A) Hold the sample until the instrument is repaired and is demonstrated to be operating under control
   B) Ship to an approved sub-contract laboratory for analysis.

   Note: Option B requires the laboratory to contact the client for prior approval.
3. Document the equipment failure, corrective action taken to correct the problem, and contact with client regarding sample analysis.
4. All information that may affect the data integrity needs to be included in the case narrative on the final report.

5.4.4 MAINTENANCE AND DOCUMENTATION

When quality assessment data or other instrument criteria indicate maintenance is needed or scheduled preventive maintenance is due, the analyst should perform the needed operation or call for service techs as soon as possible. If service is required, the analyst will be required to schedule the needed maintenance by an outside contractor who specializes in that particular instrument; upon completion of service a certificate listing specific repairs/maintenance will be kept in the maintenance log. Equipment manuals are located near to instrument and are readily available to all analysts or repair technicians. Maintenance logs are kept for all analytical equipment for recording routine or major repairs.

5.4.6 OUT OF SERVICE TAGGING OF EQUIPMENT

Out-of-service equipment or instruments in need of service must be clearly marked with a sign so that it cannot be used for analysis until repaired or calibrated.

5.5 MEASUREMENT TRACEABILITY

5.5.1 TRACEABILITY OF MEASUREMENTS

The laboratory has established programs for the calibration and verification of its measuring and testing equipment. This includes balances, thermometers and reference standards.

Calibration of laboratory balances is performed annually and contracted to National Calibration Co. 3611 W. Tompkins Ave. Las Vegas, NV, 89103. or other approved vendor.

Calibration of Class 1 weights is performed annually and contracted with Quality Control Services 2340 11th Ave., Portland Oregon 92714 (800-843-1237) Calibration of all thermometers is performed annually against an NIST certified thermometer. The calibration of the NIST certified thermometer is performed annually by Quality Control Services or other approved vendor.

All certifications of calibration are kept on file in the QA Office.

All reference standards are NIST traceable. If this is not possible the laboratory purchases standards from two different manufacturers or QC Standards from ERA for confirmation purposes. All certificates are retained in the lab or in the QC Office.

5.5.2 STANDARDS, REAGENTS, AND REFERENCE MATERIAL

CHEMICALS AND REAGENTS

"Analytical reagent grade " chemicals will be used for most analyses. For each parameter the published method may specify a level of purity for each reagent used in the analysis. Reagents at least as pure as that directed by the method shall be used. The suitability of each lot or batch of reagents is monitored for use in any given method. It is possible that a reagent chemical of sufficient purity called for by the method may contain interferences. In this case, a reagent chemical of sufficient purity to perform the analysis successfully shall be used and documented in the parameter/method SOP.

STANDARDS

Stock and working standards shall be prepared from chemicals documented as being of sufficient purity to satisfy the published method requirements or be purchased from a commercial supplier with documentation of concentration traceability to NIST (NBS) or EPA standards. The documentation received with the standards must be kept on file and accessible.

All calibration curves are checked against a second source calibration mix, either used as Laboratory Spike mix or an Initial Calibration Verification check.

Stock and working standard solutions shall be checked regularly for signs of decomposition, including but limited to, discoloration, and formation of precipitate as well as concentration change due to evaporation.
All solutions shall be properly labeled with identification of the compound, concentration, date, and analyst initials who prepared the solution.
All standards used for ICP and ICP-MS shall be of high purity.
All chemicals, solutions, and standards, shall be dated and initialed upon receipt by the analyst in charge of the chemical or storing it.

Special purity compressed gases, solvents and reagents many be required for specific organic analysis.
When any standard or reagent is received in the laboratory the receiving personnel will assign a unique ID# and log in the receipt of the item into the Standard/Reagent Log book

RECORD KEEPING

Documentation and traceability of solutions and standards prepared in the laboratory.

The analyst must follow the following steps to document the traceability of all solutions and standards.

All solutions and standards must be prepared as described in the relevant SOP.
At a minimum the following information must be written in the solution or preparation log book.

- The name and description of the solution
- The date of preparation
- The names of the chemicals used or the manufacture's designation for the mix
- The amount of the chemical or mix used
- The manufacturer's lot number and/or the original number assign when received
- The name and amount of the solvent used
- The name of the person preparing the solution
- The concentration of the solution
- The expiration date of the batch or solution. This date may be based on either a recommended length of stability for the solution or the closest expiration date of any component.

Label the vial or bottle of the solution with the name, concentration, date, and the initials of maker for ease in traceability. This same information should follow this solution or mix onto every logbook page where it is used, as well as all GC-GC/MS logbooks or quant pages.

For small bottles were room is a premium, the minimum of name or description, date of prep, logbook page, and preparer's initials shall be affixed on a small label in such a way as not to fall off or be lost.

Removal of expired chemical and standards from use.

- Each laboratory group manages in own chemical storage inventory for expiration date. Any chemical, reagent, solution, and standard, which is past its expiration

date, is removed from service and placed in the lab's internal waste management system.
- Expired standards or chemical must never be used for analyzing samples.

5.5.3 SUPPORT EQUIPMENT

MEASUREMENT DEVICES

Thermometers

Temperature readings for refrigerators, freezers incubators, water baths, and ovens are documented as defined by the current SOP. The temperatures are recorded in temperature logs and compared to acceptance criteria recorded on the log. Temperature adjustments are made for out of limit temperatures and the temperature is monitored until stable. In the event the temperature, does not come down within acceptance limits within a reasonable amount of time (before sample integrity could be affected), samples and standards will be moved and the equipment services.

Thermometers are calibrated annually against an NIST traceable thermometer, which is checked and certified annually. Corrections factors are recorded in the thermometer calibration log book and written on a small label and affixed to the top of the thermometer. In addition, these correction factors are logged into the front of the temperature log record book.

Glassware

Glassware for analytical measurements will be Class A.

Pipettes and Syringes

Pipettes and syringes must meet accuracy limits in the methods they are used for and are calibrated and/or checked annually by each department.

Balances

Analytical balances must meet and be operated in accordance with the following specifications;

- Mounted on a stable surface
- The balance is level and checked and adjusted daily
- Not located near heavy foot traffic
- Equilibrated to room temperature

- Special precautions are taken to prevent spillage or corrosive chemicals on the pan or inside the balance case. The inside of the case must be kept clean at all times
- Balances are calibrated daily, when used, with Class S (annually certified to NIST) weights. These calibration checks are recorded in the log book for the balance.

Balances are cleaned and calibrated annually by a contractor.

5.5.4 INSTRUMENT CALIBRATION

CALIBRATION-STANDARD CURVES-CORRELATION COEFFICIENTS

INSTRUMENT CALIBRATION

Any equipment or methodology used to provide numerical data that influences a measured value is calibrated to the accuracy required for its use. Records are kept of all calibrations whether made externally or within the laboratory. Calibration schedules are established for all aspects of physical and chemical measurements and are strictly observed.
The purposes of calibration are many fold:

- To determine and set up the first standard curve.
- To diagnose any problems within the recoveries or linearity of the curves
- To determine the accuracy of the method
- To set-up the instrument to meet the criteria of the method
- To determine the linearity of the curves or lack thereof,
- To determine detection limits (MDLs) for the analytes and PQLs
- To determine the sensitivity of the method and instrument

Instrument initial calibrations are performed when the following occurs:

- The first time the instrument is set up and Initial Demonstration of Capabilities are done.
- A daily Calibration Verification (CCV) criteria are not met
- Major instrument maintenance have been performed
- Major instrument parts have been replaced
- New analyst performing Initial Demonstration of Ability (DOC)
- Change in standard supplier or standard purchased from new vendor
- When instruments have been moved to a different location
- On any occasion that the analyst desires a new calibration or the SOP/Method requires it

Physical calibration of instruments is carried out with standards or measuring devices traceable to NIST standards when available.
Chemical calibrations and standardizations are made using secondary standards traceable to primary standards. The primary standards, which include multi-component mixes, are made using materials of the highest obtainable purity prepared by analysts trained in the proper methods. The materials used to prepare the primary standards are traceable to the National Institute of Standards and Technology (NIST) or the Environmental Protection Agency (EPA) whenever possible.

Primary standards obtained commercially adhere to the requirements of internally prepared primary standards. Whenever possible NIST or EPA traceable standards are to be obtained. Suppliers must certify compliance and provide evidence of the quality of services and materials upon request.

Secondary standards used for calibration and standardization are validated by comparison to standards prepared from another, second source and/or EPA/NIST traceable reference materials.

SIGNIFICANT FIGURES

The number of significant figures to be reported should be based on the precision and accuracy of the method as determined from control charts. Example:

- For analysis “A” the control chart of a control sample indicates that the 95% confidence interval for the analysis on this matrix is +/-0.14 mg/L, the uncertainty in a sample will be the same if the matrix is the same. Thus if the sample result is calculated to be 12.437 mg/L, the actual result is between 12.297 and 12.577, (95% of the time). The reported value should include the left most uncertain digit (the 1 in 0.14), i.e. 12.4 mg/L is the correct answer.

- If a dilution is involved, the uncertainty should be multiplied by the dilution factor, and then applied to the data.

PREPARATION AND VERIFICATION OF CALIBRATION CURVES

Calibration is performed to determine the response of the method to the concentration of the parameter being measured. For details see the SOP for the parameter measured. For titrimetric methods, this would correspond to the volume of tritrant required to react with an amount of an Analyte being measured. For gravimetric methods, it would correspond with the weight of an analyte after reaction and compared to a previous weight. The most common calibration is for

spectrophotometric methods. This calibration consists of determining the relationship between analyte concentration and absorbency.

Generally, a calibration consists of 3 to 8 standard points containing the analyte or analytes of interest and covers the widest possible concentration range of interest. Omitting all sample pretreatment steps, such as digestion, distillation, etc. perform the method as described in the approved literature. Read the output of the respective instrument (usually absorbance) and plot the absorbance versus the concentrations on the y and x axes and calculated the best fit straight line though the points. The slope is a measure of the sensitivity of the method.

For methods requiring sample pretreatment, such as filtration, distillation etc., repeat the above procedure including the sample pretreatment. The slope and intercept of this curve should compare closely with that above.

If the slope and intercepts do not match, be sure the same concentration and volume of reagents were added in all variations of the same method. Also check all reagents for possible contamination. Identify the source of disagreement before proceeding to analyze samples. If the disagreement is consistently small throughout the concentration range of interest, use the calibration curve developed that includes all the sample pretreatment steps.

If it is impractical to develop a new standard curve every time samples are analyzed, and if the same volume and concentrations of reagents are used each time the analysis is performed, the calibration curve should remain the same. One does not assume the cal curve never changes. This is verified each time samples are analyzed by running at least one and preferably three calibration check standards and a blank with each set of samples. The blank should compare statistically with the historical blanks and the standards should fall close to the established calibration curves. Any significant variation from the norm should be investigated and corrected before proceeding.

The approved methodologies discuss common interferences for each parameter. Interferences such as sample color, turbidity, ionic content, or any other one of a number of specific chemicals can make the analysis of real samples much more difficult than standards. The proper analysis of standards is no guarantee that real samples can be properly analyzed. The significance of interference depends upon the relative concentrations of the parameters of interest and the interference and sensitivity of the method. When a method has adequate sensitivity, the interference can be diluted out.

For certain GC and GC/MS methods, a calibration curve may be determined and checked for acceptable %RSD against method limits. Once an acceptable curve is established, it must be checked each day or every 12 hours by the analysis of a mid-

range standard and the % difference from the predicted value must fall within the method specified units.

For many instrument methods, a computer calculates and evaluates the calibration. Such calibrations should meet method requirements and the manufacturer's specifications before samples are analyzed.

If typical methods are rendered ineffective by matrix interferences or if analytical parameters or detection limits, precision, specificity, etc., would require method variance, the Laboratory Director or his representative will notify the client of the method modification. A copy of the variance will be sent to the client for approval. The modification request must show that the conditions for the laboratory variance are similar to the expected conditions (i.e., sampling and handling techniques, environmental matrix concentration range, interferences, etc.) in the EPA-approved methods. Changes in operations prior to instrumental analysis (e.g., sample preparation and storage) must be documented.

For a listing of all compliance testing parameters and approved methods performed by SA, Inc., see Appendix F the current state certification notice.

WET BENCH PARAMETERS

PARAMETERS	SOLID WASTE METHOD	WATER/WASTE WATER METHOD
ACIDITY	NA	2310B
ALKALINITY	NA	2320B
AMMONIA	NA	4500-NH_3
BIOCHEMICAL OXYGEN DEMAND (BOD)	NA	5210 B, 405.1
BROMIDE	NA	300.0
CHEMICAL OXYGEN DEMAND (COD)	NA	5220 D, 410.4
CHLORIDE	9056	300.0
CHROMIUM VI	7196,7197	218.4,218.5,3500-Cr
CYANIDE	9010B	4500-CN
FLUORIDE	NA	300.0, 4500-F
Kjeldahl Nitrogen (TKN)	NA	4500-N_{org}
HARDNESS	NA	2340C
HYDROGEN ION (pH)	9040,9045	150.1
NITRATE	9200	300.0,4500- NO_3
NITRITE	NA	300.0, 4500-NO_2
NITRATE/NITRITE (TOTAL)	NA	300.0, 4500-NO_3
OIL AND GREASE	9070,9071	413.1
Perchlorate	NA	300.0
PETROLEUM HYDROCARBONS (TRPH)	NA	418.1
PHENOLICS	NA	420.1
PHOSPHORUS, HYDROLYZABLE	NA	4500-P

PARAMETERS	SOLID WASTE METHOD	WATER/WASTE WATER METHOD
ACIDITY	NA	2310B
PHOSPHORUS, ORTHO-, DISSOLVED	NA	300.0, 4500-P
PHOSPHORUS, TOTAL	NA	4500-P
PHOSPHORUS, TOTAL DISSOLVED	NA	4500-P
RESIDUE, FILTERABLE (TDS)	NA	2540C
RESIDUE, NON-FILTERABLE (TSS)	NA	2540D
RESIDUE, SETTLEABLE (SS)	NA	2540 F
SILICA	NA	SM 2130
SPECIFIC CONDUCTANCE	NA	2510B
SULFATE	9056	300.0
SULFIDE	9030	4500-S^{2-}
SURFACTANTS (MBAS)	NA	5540C
TURBIDITY	NA	SM2130B
HAZARDOUS WASTE IGNITABILITY	1010	NA

VOLATILE PARAMETERS

PARAMETERS	SOLID WASTE METHOD	WASTE-WATER METHOD
PURGEABLE HALOCARBONS	8021B,8260 B	601,624
PURGEABLE AROMATIC HYDROCARBONS	8021B, 8260B	602,624
NONHALOGENATED VOLATILE ORGANICS	8015M,8260 B	NA
VOLATILE ORGANICS (VOC/VOA)	8260B	624

SEMI-VOLATILE PARAMETERS

PARAMETERS	SOLID WASTE METHOD	WATER/WASTE-WATER METHOD
PHENOLS	8270C	625
BENZIDINES	8270C	625
PHTHALATE ESTERS	8270C	625
NITROSAMINES	8270C	625
PCB'S	8081A	608
PESTICIDES, CHLORINATED	8082	608
NITROAROMATICS & ISOPHORONE	8270C	625
POLYNUCLEAR AROMATIC HYDROCARBONS (PAH/PNA)	8270C,8310	610, 625
HALOETHERS	8270C	625
CHLORINATED HYDROCARBONS	8270C	625
CHLORINATED HERBICIDES	8151	615
SEMI-VOLATILE ORGANICS	8270C	625
TCLP EXTRACTION (HAZ. WASTE TOXICITY)	1311	NA
TOTAL PETROLEUM HYDROCARBONS	M8015	NA

Drinking Water Parameters

PARAMETERS	DRINKING WATER METHODS
VOLATILE HALOGENATED ORG. CMPDS.	524.2
EDB/DBCP	504.1
N & P CONTAINING PESTICIDES	507
CHLORINATED PESTICIDES/ PCB's	508
CHLORINATED ACIDS (HERBICIDES)	515.3
PURGEABLE ORGANIC COMPOUNDS	524.2
ORGANIC COMPOUNDS (EXTRACTABLE)	525.2
TOTAL COLIFORM/E.coli (Colilert)	SM9223B

METALS PARAMETERS

PARAMETERS	DRINKING WATER METHODS	SOLID WASTE METHOD	WATER/WASTE WATER METHOD
HEXAVALENT CHROMIUM	NA	7196A	3500-Cr D
METALS(EXCEPT MERCURY AND CHROMIUM VI)	200.7, 200.8, 3113B	6010B, 6020	200.7, 200.8, 3113B
MERCURY	245.1	7470A, 7471A	245.1, 7470A, 7471A

5.6 SAMPLING

SAMPLE PRESERVATION

Degradation of a sample may start immediately after collection for some chemical species. Any delay in analysis can lead to error in the reported data. The shorter the time that elapses between collection of a sample and its analysis, the more reliable the results will be.

However, many times the analysis cannot be started immediately. This is due to the complexity of the analysis, a heavy workload, or due to remoteness of the laboratory. Preservation methods lengthening the period of sample stability have been established.

A “holding time” starts the time a sample is collected. The preservation should be added at the time the sample is collected. Complete and unequivocal preservation of samples, either domestic sewage, industrial wastes or natural waters is a practical impossibility, Regardless of the nature of the sample, compete stability for every constituent can never be achieved. At best, preservation techniques can only retard the chemical and biological changes that inevitably continue after the sample is removed from the parent source. In the case of chemical changes, certain changes occur in the chemical structure of the constituents that are a function of physical conditions. Metal cations may precipitate as hydroxides or form complexes with other constituents; cations or anions may change valence states under certain deducing or oxidizing conditions; other constituents may dissolve or volatilize with the passage of time. Metal cations may also absorb onto surfaces of glass, plastic, quartz, etc., such as iron and lead. Biological changes taking place in a sample may change the valence of an element a radical into a different valence. Soluble constituents may be converted to organically bound materials in cell structures, or cell lysis may result in release of cellular material into solution. The well-known nitrogen and phosphorous cycles are examples of biological influences on sample decomposition. This is especially true when the analyte concentration is expected to be in the low ppb range.

Methods of preservations are relatively limited and are intended generally to (1) Retard biological action, (2) Retard hydrolysis of chemical compounds and complexes, (3) Reduce volatility of constituents, and (4) Reduce absorption effects. Preservation methods are generally limited to pH control, chemical addition, refrigeration, and freezing.

Many regulatory programs, such as SDWA, NPDES, RCRA, etc., mandate the type of sample container, the method of preservation, and the length of time allowed for a holding time. A summary table of sample methods, preservation needed and bottles appear in Appendix E.

5.7 SAMPLE HANDLING AND TRANSPORTATION

5.7.1 SAMPLE TRACKING AND HANDLING

Southwest Analytical, Inc. collects environmental samples for client's quarterly or on an as needed basis. The type of material to be collected and the analytical method to be used depends upon the physical locations of the site, site history, known and unknown conditions and contaminants, and detection levels. SA, Inc.'s sampling processes are performed in a manner that produces accurate and legally defensible data. Detailed procedure for sampling of environmental sample could be found in *SOP 1112. Sampling Procedure for Environmental Samples*.

A critical aspect of sound sample collection and analysis protocols is the maintenance of strict chain-of-custody procedures. Chain-of-custody procedures include inventory and documentation for transporting and/or, shipment and laboratory processing. A sample is considered to be in an individual's custody if the sample is:

1) in the physical possession or view of the responsible person,
2) secured by that person to prevent tampering, or
3) Secured by that person in a restricted area.

Sample receipt, login, preservation and storage are outlined in SOP 2101-"Sample Receipt and Login Procedure and Bottle Preparation SOP 2102, SOP 2102 also outlines the use of the proper sample containers. The Appendix preservation guide applies to aqueous samples. All soil samples are stored at 4°C.

NOTE: **ALL SAMPLES MUST BE CHECKED FOR SAMPLING DATE VERSUS REQUESTED TURNAROUND TIME. IF A SAMPLE WILL REACH HOLDING TIME PRIOR TO THE EXPECTED DUE DATE THE SAMPLE MUST BE PROCESSED WITHIN HOLDING TIME. IF THIS CAUSES THE SAMPLE TO BE ANALYZED IN A RUSH CAPACITY, THE CLIENT MUST BE NOTIFIED ABOUT APPLICABLE SURCHARGE.**

5.7.2. SAMPLE HANDLING

Sample Labels applied in the field

A label should be attached to all sample containers at the time of collection, and completed in waterproof ink. All samples must be labeled before the laboratory will accept custody. The label must contain the following:
•Sample number/description
•Date and time collected (client specific, optional)
•Required analysis (client specific, optional)
•Source/location and location of the sample (client specific, optional)

Chain-of-Custody Record

Sample custody begins with the detailed records kept by the client's field sampling personnel. The COC record details the documentation and control necessary to identify and trace a sample from sample collection to final analysis. It should include field sample labeling (to prevent mix-up), custody seals (to prevent sample tampering), securing custody, and records of support information (for potential litigation).

COC forms are used to document the integrity of all samples. To maintain a record of sample collection, transfer between personnel, shipment, and receipt by the laboratory, each sampling set must be accompanied by a COC. The COC form will contain the following information:

- sample identification (for each sample in shipment),
- collection date and time(for each sample),
- number of containers of each sample,
- client name, address, phone/fax number
- client project manager, project name, project number
- sample type (environmental matrix, preservative present and expected concentration),
- analyses to be performed
- status of the custody seal
- Temperature and pH/preservation of samples received (where applicable) {this may be recorded in a separate book.}
- Custody transfer signature blocks.
- **A statement will be affixed to the COC, acknowledged by sampler signature, that no attempt has been made to alter or tamper with the name, locations, dates and times of samples. Such tampering could constitute fraud.**

The individual in charge of shipping samples to the laboratory is also responsible for completing the COC form and inspecting their form for completeness and accuracy. Any changes made to the COC form shall be crossed out with one line, initialed and dated by the person making the change.

Transfer of Custody and Shipment

Samples must be accompanied by an approved COC record. When the possession of samples is transferred, the individual relinquishing the samples signs and records the date and time on the COC document. The individual receiving the samples repeats the procedure. This record represents the official documentation for all transfers of the sample custody until the samples arrive at the laboratory.

If samples are to be split with another laboratory facility or government agency, a separate COC record is prepared for those samples. This COC record indicates with whom the samples have been split and is appropriately signed and dated.

Receipt and Logging of Samples

This section describes the laboratory custody procedures associated with sample receipt, storage, preparation, analysis and security.

Laboratory COC procedures include sample inventory and record maintenance during sample collection, shipment and laboratory processing. The Sample Custodian manages and tracks the storage and distribution of samples after their arrival.

The sample tracking and COC procedure presented includes the following components:

1. A Chain-of-Custody accompanies the samples from collection.
2. All samples are inspected; anomalies are noted in the COC form, and the client and analysts are alerted.
3. Each sample is assigned a unique laboratory identification number, which is cross-coded with the client's identification number. Sample identification information is entered into a Sample Log Book, and the assigned number is used to track sample location and status throughout the analytical process.

   The following sample information is recorded onto the sample log book:
   - Date of receipt
   - Laboratory identification
   - Client name
   - Turnaround time, date due
   - Initials of laboratory personnel logging in the samples

4. The COC document is completed, Signed by necessary parties and copies are returned to the appropriate party(s).
5. While within the laboratory, sample integrity is maintained in storage areas where samples remain except during analysis
6. When the analysis is concluded, any remaining samples are either archived in secured storage areas or are properly disposed.

Project Initialization

The Sample Custodian for the laboratory has duties and responsibilities that include but are not limited to:

- The sample custodian shall assign laboratory project/sample numbers in sequential numeric order by date of receipt. These sample numbers will be listed on laboratory documents to cross-reference with the client number, and sample tag number.
- A file folder shall be prepared for each project number by the sample custodian prior to or at the time of sample receipt. The project file shall be placed in the "in-process" file or in the current project file (arranged in sequence.
- Inspect sample shipping containers for presence, and condition of: custody seals, locks, "evidence tapes", etc., container breakage, leakage, headspace, and/or container integrity
- Record the condition of shipping and sample containers on the COC form;
- Record sample temperatures upon receipt; recording sample pH upon receipt, where appropriate.
- Sign appropriate documents shipped with samples, such as, air bills, freight bills, COC records, verifying and recording agreement or non-agreement of information on sample documents on appropriate forms.
- If there is a variance from contract requirements, the lab project manager and customer are notified immediately.
- Notify project personnel and analysts of sample or paperwork problems.
- Initiate the paperwork for sample analyses on appropriate laboratory documents.
- Check collection dates against receipt dates to determine that the sample holding time requirements have not been exceeded.
- Label samples with laboratory sample numbers and cross-referencing laboratory numbers with client numbers and sample tags.
- Place samples, sample extracts, and spent samples into appropriate storage and/or secure areas.
- Control access to samples in storage and assuring that laboratory SOPs are followed during sample movement.
- Returning shipping containers to sampling teams or clients.

Sample Security

Samples are placed in storage areas except during laboratory analysis. All laboratory personnel who receive samples are responsible for the care and custody of samples from the time they take custody until samples are returned to the storage areas. Tracking of all samples within the laboratory is accomplished by Omega with the use of an internal COC system. All subsets (extraction, digests, etc.) of the samples shall be kept in secured storage which is controlled by the appropriate Department Supervisor.

The laboratory employs the following security measures:

- Doors to the laboratory are closed and secured during non-business hours
- During business hours, only authorized personnel and visitors under escort shall have access to the chemistry lab.
- All laboratory personnel are aware of the need to question and determine legitimacy of a stranger's presence in the laboratory.
- Sample storage areas are monitored at night by motion sensitive alarm systems.

Sample Storage and Disposal

Once the samples have been logged, the analysts shall be responsible for the following:

- Upon completion of the analysis, any remaining sample will be placed into long-term storage.
- When sample analysis and all QC checks have been completed and a final report has been issued, the unused sample portion shall be stored by the analyst for a period of not less than 30 days after the report has been issued.
- The Client Services Person shall be responsible for returning to the client all unused bottles, shipping containers, packing materials blue ice packs, and if requested, the unused sample portions.
- Any remaining sample may be properly disposed of after a 30-day period, unless further instructions are received from the client.

Sample Log-In Document Storage

There is one repository for documents associated with a project. This is the **project file**. This file will contain all documents concerning the receipt, transfer and reporting of the samples. Raw data need not be kept in the **project file**, but all other correspondence will be filed here.

The project file also contains a copy of the final project report, QC report, and other documents related to the project management.

Data Control will keep all files together under secured storage in a file cabinet or in the lab archives. Computer data backups will be stored in a secure, waterproof, fireproof environment.

Corrections to Documentation

When it becomes necessary to make corrections to any form of laboratory documentation the obsolete information is crossed out with a single line and the

changes are made and initialed/dated by the person making the change. This procedure is detailed in *SOP 1003 - Entering of data into logbooks.*

5.8 ASSURING TEST QUALITY

5.8.1 FIELD QC SAMPLES

Client should periodically prepare quality control (QC) samples in the field and submit for analysis with regular samples. These QC samples should consist of field blanks, travel blanks and replicate samples. Other blanks be made and designated, such as, equipment blanks, bailer blanks, cooler blanks, etc. The frequency of QC samples should be as follows:

- One field travel blank is prepared in the field for every sampling event using laboratory-grade organic free water. The field travel blank is poured into a bottle at one of the sampling sites, which is noted on the field sampling form. The field travel blank is analyzed for the complete set of organic parameters requested for the regular samples.

- One replicate sample is collected for every sampling event and submitted for analysis. The replicate is analyzed for the complete set of parameters requested for the regular sample.

5.8.2 TYPES OF LABORATORY QC ASSESSMENT SAMPLES

5.8.2.1 Method Blanks; Frequency: daily, with each batch.

Method blanks, also known as reagent blanks, are analyzed for each matrix and each batch of sample analyses (about 1 per group of up to 20 samples) prepared at the same time. An aliquot equal in volume or weight to the sample is used for method blank analyses. The method blank is taken through the whole analytical process. The method blank must be free of any substances being analyzed, or interferences, as defined in method-specific SOPs, or corrective actions will be taken. For volatiles analyses, a method blank is run after the standard or standards and before any samples during every batch to demonstrate that the system is performing properly and there is no carryover.

5.8.2.2 Matrix Spike/Matrix Spike Duplicate (MS/MSD); Frequency: daily, with each batch.

MS/MSD is used to check for precision and accuracy. These are replicate samples spiked with a known spike concentration that are taken through the whole sample preparation and analysis process. MS/MSD analyses are performed on one sample

in each group of 20 samples (5%) on each type of sample matrix. An MS must be performed on one sample in each group of 10 samples (10%) for drinking water analyses.

The sample analysis process and the spike sample process differ in the adding of known amounts of the substances to be analyzed to the aliquot of the replicate sample. The amount of spike added varies according to the working range of the analytical instrument.

Should the sample also have the native analyte present, the concentration of that analyte in the sample is subtracted from the value of the spiked sample and the percent (%) recovery of the spike is calculated using the following equation:

$$\text{Recovery} = \frac{(\text{Spike Sample Result} - \text{Sample Result})}{\text{Spike Added}} \times 100\%$$

$$\text{Post Digestion Spike} = \frac{(\text{Spike Sample Result-df} \times \text{Sample Result})}{\text{Spike Value}} \times 100\%$$

At times the sample value is outside the operating range of the analytical instrument. In such cases, it is impossible to know the concentration of the analytes before the spike is added. Occasionally, the sample and the spiked sample require dilutions to allow analysis within the linear range of the instrument. No MS/MSDs will be diluted, but comments in the case narrative will explain that certain labeled analytes of interest were over the analytical range of the instrument. The sample itself will be diluted and the diluted result reported. The data should be reported with the appropriate data qualifier.

In the event that a matrix spike(s) recovery is outside the established control limits, the sample and the matrix spike must be sent through the entire sample preparation and analysis procedure a second time if there is not a documented matrix effect. Many metals analysis methods call for an external spike to be performed on the extract if the matrix spike(s) recovery is outside the established control limits. If the reanalysis of the sample and the matrix spike(s) are acceptable, the results of the reanalysis will be reported without a data qualifier. If the reanalysis of the sample and the matrix spike(s) are outside the established control limits again, the results of the original analysis will be reported with the appropriate data qualifiers.

The calculated percent recoveries are then used to assess data precision expressed as relative percent deviation (RPD). It is calculated using the following equation:

$$\text{RPD} = \frac{(\text{MS Result} - \text{MSD Result})}{\text{Mean of MS and MSD Results}} \times 100$$

Percent recoveries and RPD values outside of contract, method, or laboratory-determined control limits require addressing in the narrative and a corrective action may be required based on the problem.

5.8.2.3 VOA Refrigerator Blank; Frequency: When samples are in process.

A refrigerator blank is an aliquot of VOA-free laboratory reagent water contained in a 40-mL septum-sealed VOA vial free of headspace. It is prepared and carried through the holding period of a batch of sample shipments received and is used to monitor sample storage cross-contamination for volatile organics. Presence of volatile organics in the refrigerator blank indicates possible diffusion of volatile organics (particularly fluorocarbons and methylene chloride) past the septum seal of the contaminated sample during storage and handling. Corrective action should be taken to remedy the storage problem.

5.8.2.4 Calibration Blank/System Blank (i.e. metals) Frequency: daily, with each batch.

A calibration blank/system blank is prepared by analyzing the same matrix used for the preparation of the calibration standards. It is used to establish the analytical curve by taking into account background responses during the calibration process. It is also used to check for carry-over contamination after a standard run or after a contaminated sample run.

5.8.2.5 Laboratory Control Samples Frequency: daily, with each batch.

The Laboratory Control Sample (LCS) is a standard of known concentration spiked into a consistent (homogeneous) matrix of soil or water, which is prepared and analyzed along with a batch of samples. The spiked materials are representative of the materials to be quantitated by the analysis.

LCS spiking solutions are obtained from a second source supplier or a different lot number of the analyte from the same supplier. USEPA Quality Assurance Branch, Environmental Monitoring and Support Laboratory (EMSL), by the National Institute of Standards and Technology (NIST), or they are prepared internally by the laboratory. The quality control samples are prepared for analysis in strict accordance with the procedures provided with the materials. LCS are prepared and analyzed with each sample batch to check accuracy of analysis and to monitor standard degradation. LCS is also used to check efficiency of the digestion, extraction and the instrumental analysis. For metals, commercially obtained/prepared LCS soils are used to check EPA 3050B digestion efficiency.

Results from LCS must be within the target range values determined by the specific methods.

5.8.2.6 Surrogate Spike Analyses: Frequency: every sample

For GC/MS, GC and HPLC methods, the analytical process includes the addition, subsequent detection, and recovery calculation of surrogate spiking compounds.

- Surrogate compounds are analyte compound substitutes, (i.e., compounds not specifically requested to be determined as analytes) that do not occur naturally.
- Surrogate compounds are added after sample aliquots have been measured out, prior to beginning the sample preparation process.
- Surrogate compounds must not interfere with the determination of the analytes of interest. Surrogates also must be chemically similar to the analytes of interest and capable of emulating the analyte responses.
- All samples, blanks and quality control samples are spiked with surrogate spiking compounds before purging or extraction in order to monitor preparation and analysis of samples.
- Percent recovery of the surrogates is determined and checked against laboratory established control limits.

Laboratory established control limits are used when method-specific or contract-specific criteria are not provided or when the lab can demonstrate tighter control of a system than given in the method. These results are plotted by the analyst to alert them to any trends that may suggest a problem. If a trend suggests a problem in the system, preventative action is taken to remedy the situation before a problem arises. If a surrogate fails the established control limits, the sample preparation and/or analysis must be repeated. A second surrogate failure indicates the presence of sample-based matrix interference and the original result is reported. VOAs may have one of the three surrogates fail, but two failures will necessitate that the sample be reanalyzed.

5.8.2.7 Method of Standard Addition (Metals); Frequency: daily, with each batch.

The method of standard additions is used to check the accuracy of the analysis method under optimum conditions and to provide an estimate of concentrations in the presence of chemical interferences from a sample matrix.

5.8.2.8 ICP Interference Check Sample Analysis; Frequency: daily, with each batch.

An ICP interference check sample verifies inter-element and background correction factors of the ICP instrument.

5.8.2.9 NIST Traceable and/or Commercial Reference Standards : Frequency: at least quarterly.

These standards are analyzed routinely for the parameter of interest. Commercial standards such as Environmental Resource Associates, Performance Evaluation Samples are analyzed quarterly for each category of certified field of testing, i.e., one for Drinking water (WS), one for soil and hazardous wastes (SOIL), one for Underground Storage Tank(UST), and one for Waste Water (WP). Additionally, secondary source standards from commercial sources are used to check the accuracy of the primary commercial standard and the laboratory preparation of the standards in use. Secondary source standards are also used in all Initial Calibration Verifications, MS, MSD and LCSs to confirm the accuracy of the standard.

5.8.2.10 Internal Standards Analysis; Frequency: with each sample.

For some methods, internal standard areas are monitored as a measure of instrument performance. Internal standard determinations are made on all samples and blanks to monitor instrument efficiency, and they are used as a reference retention-time indicator, to check retention-time shifts of peaks of interest. A known amount of internal standard is added to a sample extract prior to analysis. Like the surrogate standard, it must not interfere with the determination of the analytes of interest. It also must be chemically similar to the analytes of interest and capable of emulating the analyte response. Often these are isotopically-labeled materials, which can be distinguished by the mass spectrometer from native analytes. Pass/ fail is monitored and defined in the specific method.

5.8.2.11 Initial Calibration Standards; Frequency: every six months (for inorganic), or as needed.

Calibration standards are used to quantitate the concentrations of analytes present in the sample. These are analyzed prior to any sample analyses.

5.8.2.12 ICP, GFAA, and CVAA multi-point; Frequency: daily, with each batch.

Initial calibration verification and continuing calibration verification used for metals analysis at $\leq$ 10% ICV is second vendor.

5.8.2.13 Sample Bottle Sterility Check; Frequency: daily, with each batch.

The sterility of each lot of sample containers or bags is confirmed by adding 25 mL of a sterile non-selective broth to at least one container, incubating at 35 $\pm$ 0.5 ^{o}C for 24 hours and checking for growth.

5.8.2.14 Controlled Organism Laboratory Control Samples; Frequency: monthly

Certified Total Coliform and Fecal Coliform positive and negative known organisms will be analyzed on a monthly basis to measure the accuracy of the testing process.

5.9 STATISTICAL TECHNIQUES

Quality assessment data generated from all instruments and all analyses are subjected to appropriate statistical analyses. Control Charting is a type of statistical analysis which may be appropriate for each type of QA data listed below.

For Surrogate recoveries: chart semi-annually from OMEGA, taking into account as least 40 previous points for each matrix and using the Grub's test. Calculate new limits and installing them into Omega to be actively used.

For MS/MSD, LCS/LCSD pairs: chart like surrogates above, but for each constituent or compound or element of interest.

For Regression curves, calibration curves, or Initial calibrations use Average Response Factors for most GC/MS calibration curves. In most other methods, Linear Regression Curves, non-weighted and not forced through zero, with a Correlation Coefficients of .990 or better, (and in metals, .995 or better), are also allowed. For additional curve types and specifics, see SW 846 Method 8000.

5.9.1 QUALITY CONTROL CHARTS

Control charts are recognized as a prime means to document the statistical control of the measurement process and to describe measurement proficiency. It is a matter of policy that suitable control charts for all critical operations and sub-operations are developed. When the use of control charts and specific control samples are specified in a given SOP, they are maintained in as close to real time as possible and are the basis for corrective actions when indicated. The control charts are either generated by the LIMS system or generated in a spreadsheet program by the LIMS system.

Use of Control Charts

The performance of a measurement system can be demonstrated by the measurement of homogeneous and stable control samples in planned repetitive process. The data generated are plotted as a control chart to indicate whether the system is providing adequate process control (i.e., staying within performance specifications). It alerts the laboratory to possible deviation from 95% confidence level by identifying systematic errors, drifts, or other types of problems.

The control charts:

- Provide graphic assessment of accuracy and precision for the analysis of each analyte and detection of erroneous data.

- Allow efficient observation of recovery trends for a particular analyte, and they provide a long-term mechanism for self-evaluation of analytical output.

- Provide assessment of the analytical capability of the staff chemists, with regard to the Output of valid analytical data.

A system must be verified as being in control, in order to be maintained in control. A system is not in control if it is observed to produce unexpected data more than once every 20-25 runs or a trend of seven points on one side of the mean. Control limits usually become tighter once a process is under a controlled protocol (i.e., the original limits were based on data produced during uncontrolled operation).

Types of Control Charts Used

Control charts are used to monitor method performance:

- Accuracy Control Charts (e.g., Surrogate Recovery, LCS/LCSD Recovery, MS/MSD Recovery)

- Precision Control Charts (e.g., MS/MSD Relative percent difference, LCS/LCSD Relative percent difference)

Control Chart Preparation

In methods for which quality control acceptance limits and corrective actions are not specifically established (e.g., non-CLP), control charts are used to evaluate lab performance. As a minimum for other methods, the Laboratory shall prepare a control chart for each type of analysis and sample matrix. Each control chart shall consist of a centerline, two warning limits, and two control limits, as described on page 42 of the HAZWRAP DOE/HWP-65/R1 and EPA Handbook on Analytical Quality Control in Water and Wastewater Laboratories. Until the Laboratory has 20 points to use in setting control chart limits, the recommended EPA recoveries for the methods shall be used.

Control Chart Interpretation

For each method, representative concentrations of material described in reference methods and lab SOPs are spiked into a selected percentage of samples. A data base of percent recovery (%R) for QC reference samples or spiked samples is

collected. The mean (x) and standard deviation (σ) of this set (usually at least 20 points are necessary) are calculated. From this information, warning and control limits for the method are determined. These are defined as:

- Warning Limits are defined as $x \pm 2\sigma$

- Control Limits are defined as $x \pm 3\sigma$. The %R of each QC sample or spike sample is plotted on a control chart and compared with the control limits.

Data precision (RPD) is evaluated based on the results of spiked samples analyzed in duplicate. Calculations for warning and control limits are the same as above for spiked samples.

Interpretation of control charts for out of control situations and need for corrective action:

- One or more points outside the control limit (3σ)
- A run of two or more consecutive points outside warning limits (2σ)
- A run of seven or more consecutive points on the same side of the mean (x indicating trends or shifts
- Cycles or non-random patterns in the data
- A run of six or more consecutive points in the same direction

Corrective actions taken for out-of-control points are described in Nonconformance and Corrective Action.

5.10 NONCONFORMANCES AND CORRECTIVE ACTION

Definitions

An out-of-control event is defined as any occurrence failing to meet pre-established criteria. Non-conformance is a deficiency in documentation, or procedure sufficient to make the quality indeterminate or unacceptable. An out-of-control event is a subcategory of non-conformance. When either situation is identified, it will be categorized as:

A deficiency: recognition of a specific requirement (e.g., program, process or procedure) that has been violated. (method deviation)
An observation: recognition of an activity or action that might be improved but is not in violation of a specific requirement. Left alone, the activity or action may develop into a deficiency. (reporting error)

Criteria Used for Determination of an Out-of-Control Event

Factors that affect data quality (failure to meet calibration criteria, inadequate record keeping, improper storage or preservation of sample) require investigation and corrective actions.

Some factors can be easily assessed through the use of control charts. Control charts can reveal shifts, trends, biases and conditions where parts of the analytical system are out of control.

The detection of one of these conditions is an indication that the analytical system is out of control. The out-of-control value(s) is placed on the control chart, circled and documented in a corrective action form according to SOP 4003. The Area Supervisor is notified and both the analyst and Area Supervisor investigate and determine whether the condition indicates a procedure that is truly out of control or which reflects a possible random error. The Area Supervisor shall document corrective actions taken (i.e., whether the sample run was repeated or whether the data were reviewed and released for reporting to the client) on the corrective action form and submit to the QA Manager for placing in the permanent records.

The charts which follow are lists of QC check standards, their pass/fail criteria and the corrective action for each.

SUMMARY OF QUALITY CONTROL PROCEDURES FOR METALS BY ICP

QC ELEMENT	FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
Method Blank (MB)	1 per batch, not to exceed 20 samples (non-drinking water samples) 1 per batch, not to exceed 10 samples (drinking water samples)	< RL < 0.5*RL for all USACE projects RL = laboratory reporting limit	1) Check calculations. 2) Investigate contamination source 3) Take and document appropriate corrective action. 4) Reanalyze blank. If passes, report. 5) If still out, re-extract/reanalyze all samples. 6) Flag sample result associated with contaminated method blank.
Matrix Spike/Matrix	1 per batch, not to	Project specific	1) Check calculations.

QC ELEMENT	FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
Spike Duplicate (MS/MSD)	exceed 20 samples (non-drinking water samples) 1 per batch, not to exceed 10 samples (drinking water samples)	requirements. If there are not project specific requirements use method specific requirements until in-house limits can be determined.	2) Run external spike and external spike duplicate on original sample. 3) If still out, reanalyze external spike and external spike duplicate on original sample at a dilution. 4) If still out report with suspected matrix interference.
Laboratory Control Sample (LCS)	1 per batch, not to exceed 20 samples (non-drinking water samples) 1 per batch, not to exceed 10 samples (drinking water samples)	Project specific requirements. If there are not project specific requirements use method requirements until in-house limits can be determined.	1) Check calculations. 2) Identify correct and document problem. 3) Reanalyze LCS, if passes report. 4) If still out, re-extract/reanalyze LCS and associated samples.
Serial Dilution	1 per batch, not to exceed 20 samples (non-drinking water samples) 1 per batch, not to exceed 10 samples (drinking water samples)	$\pm$ 10% of original concentration	1) Evaluate data for interference.
Interference Check Sample	Initial calibration	$\pm$ 20%	1) Recalculate inter-element correction factors and reanalyze.
Reporting Limit Verification	Beginning of each run	$\pm$ 100% of the true value	1) Reanalyze a new aliquot of RLV solution. 2) If still out, reanalyzed calibration blank. 3) Reprint correlation coefficient.

QC ELEMENT	FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
			4) Reanalyze instrument detection limit (IDL).
Continuing Calibration Verification Sample	1 per every 10 samples	Method requirements.	1) Reanalyze, if passes continue with analyses. 2) If still out, recalibrate.

SUMMARY OF QUALITY CONTROL PROCEDURES FOR METALS BY ICP/MS

QC ELEMENT	FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
Method Blank (MB)	1 per batch, not to exceed 20 samples (non-drinking water samples) 1 per batch, not to exceed 10 samples (drinking water samples)	< RL < 0.5*RL for all USACE projects RL = laboratory reporting limit	1) Check calculations. 2) Investigate contamination source 3) Take and document appropriate corrective action. 4) Reanalyze blank. If passes, report. 5) If still out, re-extract/reanalyze all samples. 6) Flag sample result associated with contaminated method blank.
Matrix Spike/Matrix Spike Duplicate (MS/MSD)	1 per batch, not to exceed 20 samples (non-drinking water samples) 1 per batch, not to exceed 10 samples (drinking water samples)	Project specific requirements. If there are not project specific requirements use method specific requirements until in-house limits can be determined.	1) Check calculations. 2) Run external spike and external spike duplicate on original sample. 3) If still out, reanalyze external spike and external spike duplicate on original sample at a dilution. 4) If still out report with suspected matrix interference.
Laboratory Control Sample (LCS)	1 per batch, not to exceed 20 samples (non-drinking water samples) 1 per batch, not to exceed 10 samples (drinking water samples)	Project specific requirements. If there are not project specific requirements use method requirements until in-house limits can be determined.	1) Check calculations. 2) Identify correct and document problem. 3) Reanalyze LCS, if passes report. 4) If still out, re-extract/reanalyze LCS and associated samples.
Serial Dilution	1 per batch, not to exceed 20 samples (non-drinking water samples)	± 10% of original concentration	1) Evaluate data for interference.

	1 per batch, not to exceed 10 samples (drinking water samples)		
Interference Check Sample	Initial calibration	± 20%	1) Recalculate inter-element correction factors and reanalyze.
Reporting Limit Verification	Beginning of each run	± 100% of the true value	1) Reanalyze a new aliquot of RLV solution. 2) If still out, reanalyzed calibration blank. 3) Reprint correlation coefficient. 4) Reanalyze instrument detection limit (IDL).
Internal Standard (IS)	Every sample including QC	Method requirements	1) Check calculations. 2) Dilute the digest. Repeat if necessary.
Continuing Calibration Verification Sample	1 per every 10 samples	Method requirements	1) Reanalyze, if passes continue with analyses. 2) If still out, recalibrate.

SUMMARY OF QUALITY CONTROL PROCEDURES FOR METALS BY CVAA

QC ELEMENT	FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
Method Blank (MB)	1 per batch, not to exceed 20 samples (non-drinking water samples) 1 per batch, not to exceed 10 samples (drinking water samples)	< RL < 0.5*RL for all USACE projects RL = laboratory reporting limit	1) Check calculations. 2) Investigate contamination source 3) Take and document appropriate corrective action. 4) Reanalyze blank. If passes, report. 5) If still out, re-extract/reanalyze all samples. 6) Flag sample result associated with contaminated method blank.
Matrix Spike/Matrix Spike Duplicate (MS/MSD)	1 per batch, not to exceed 20 samples (non-drinking water samples) 1 per batch, not to exceed 10 samples (drinking water samples)	Project specific requirements. If there are not project specific requirements use method specific requirements until in-house limits can be determined.	1) Check calculations. 2) Run external spike and external spike duplicate on original sample. 3) If still out, reanalyze external spike and external spike duplicate on original sample at a dilution. 4) If still out report with suspected matrix interference.
Laboratory Control Sample (LCS)	1 per batch, not to exceed 20 samples (non-drinking water samples) 1 per batch, not to exceed 10 samples (drinking water samples)	Project specific requirements. If there are not project specific requirements use method requirements until in-house limits can be determined.	1) Check calculations. 2) Identify correct and document problem. 3) Reanalyze LCS, if passes report. 4) If still out, re-extract/reanalyze LCS and associated samples.
Continuing Calibration Verification Sample	1 per every 10 samples	Method requirements	1) Reanalyze, if passes continue with

QC ELEMENT	FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
			analyses. 2) If still out, recalibrate.

SUMMARY OF QUALITY CONTROL PROCEDURES FOR ORGANICS BY GC

QC ELEMENT	FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
Method Blank (MB)	1 per batch, not to exceed 20 samples (non-drinking water samples) 1 per batch, not to exceed 10 samples (drinking water samples)	< RL < 0.5*RL for all USACE projects RL = laboratory reporting limit	1) Check calculations. 2) Investigate contamination source 3) Take and document appropriate corrective action. 4) Reanalyze blank. If passes, report. 5) If still out, re-extract/reanalyze all samples. 6) Flag sample result associated with contaminated method blank.
Matrix Spike/Matrix Spike Duplicate (MS/MSD)	1 per batch, not to exceed 20 samples (non-drinking water samples) 1 per batch, not to exceed 10 samples (drinking water samples)	Project specific requirements. If there are not project specific requirements use method specific requirements until in-house limits can be determined.	1) Check calculations. 2) Reanalyze MS/MSD and sample. 3) If still out, re-extract/reanalyze MS/MSD and sample. 4) If still out report with suspected matrix interference.
Laboratory Control Sample (LCS)	1 per batch, not to exceed 20 samples (non-drinking water samples) 1 per batch, not to exceed 10 samples	Project specific requirements. If there are not project specific requirements use method requirements until in-house limits can be	1) Check calculations. 2) Identify correct and document problem. 3) Reanalyze LCS, if passes report. 4) If still out, re-extract/reanalyze LCS

QC ELEMENT	FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
	(drinking water samples)	determined.	and associated samples.
Surrogate spike	Every sample including QC	Method requirements until in-house limits can be established.	1) Check Calculations. 2) Evaluate batch for trends. 3) Reanalyze sample, if acceptable report. 4) If not acceptable, re-extract/reanalyze sample.
Continuing Calibration Verification Sample	1 per every 10 samples	Method requirements	1) Reanalyze, if passes continue with analyses. 2) If still out, recalibrate.

SUMMARY OF QUALITY CONTROL PROCEDURES FOR ORGANICS BY GC/MS

QC ELEMENT	FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
Method Blank (MB)	1 per batch, not to exceed 20 samples (non-drinking water samples) 1 per batch, not to exceed 10 samples (drinking water samples)	< RL < 0.5*RL for all USACE projects RL = laboratory reporting limit	1) Check calculations. 2) Investigate contamination source 3) Take and document appropriate corrective action. 4) Reanalyze blank. If passes, report. 5) If still out, re-extract/reanalyze all samples. 6) Flag sample result associated with contaminated method blank.
Matrix Spike/Matrix Spike Duplicate (MS/MSD)	1 per batch, not to exceed 20 samples (non-drinking water samples) 1 per batch, not to exceed 10 samples (drinking water samples)	Project specific requirements. If there are not project specific requirements use method specific requirements until in-house limits can be determined.	1) Check calculations. 2) Reanalyze MS/MSD and sample. 3) If still out, re-extract/reanalyze MS/MSD and sample. 4) If still out report with suspected matrix interference.
Laboratory Control Sample (LCS)	1 per batch, not to exceed 20 samples (non-drinking water samples) 1 per batch, not to exceed 10 samples (drinking water samples)	Project specific requirements. If there are not project specific requirements use method requirements until in-house limits can be determined.	1) Check calculations. 2) Identify correct and document problem. 3) Reanalyze LCS, if passes report. 4) If still out, re-extract/reanalyze LCS and associated samples.
Surrogate spike	Every sample including QC	Method requirements until in-house limits can be established.	1) Check Calculations. 2) Evaluate batch for trends. 3) Reanalyze sample, if acceptable report. 4) If not acceptable,

QC ELEMENT	FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
			re-extract/reanalyze sample.
Internal Standard spike (IS)	Every sample including QC	IS area 50-200% of the IS area in the CCV	1) Check sensitivity of instrument and sample. 2) Reanalyze sample or standard. 3) If still out and the failure is sample specific report data with suspected matrix interference.
MS Tune	Every 12 hours	Method abundance criteria.	1) Rerun tune. 2) If still out take and document appropriate corrective action.
Continuing Calibration Verification Sample	1 per every 12 hours or at the start of each 12 hour run/batch	Method requirements	1) Reanalyze, if passes continue with analyses. 2) If still out, recalibrate.

SUMMARY OF QUALITY CONTROL PROCEDURES FOR ORGANICS BY HPLC

QC ELEMENT	FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
Method Blank (MB)	1 per batch, not to exceed 20 samples (non-drinking water samples) 1 per batch, not to	< RL < 0.5*RL for all USACE projects RL = laboratory	1) Check calculations. 2) Investigate contamination source 3) Take and document appropriate corrective action.

QC ELEMENT	FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
	exceed 10 samples (drinking water samples)	reporting limit	4) Reanalyze blank. If passes, report. 5) If still out, re-extract/reanalyze all samples. 6) Flag sample result associated with contaminated method blank.
Matrix Spike/Matrix Spike Duplicate (MS/MSD)	1 per batch, not to exceed 20 samples (non-drinking water samples) 1 per batch, not to exceed 10 samples (drinking water samples)	Project specific requirements. If there are not project specific requirements use method specific requirements until in-house limits can be determined.	1) Check calculations. 2) Reanalyze MS/MSD and sample. 3) If still out, re-extract/reanalyze MS/MSD and sample. 4) If still out report with suspected matrix interference.
Laboratory Control Sample (LCS)	1 per batch, not to exceed 20 samples (non-drinking water samples) 1 per batch, not to exceed 10 samples (drinking water samples)	Project specific requirements. If there are not project specific requirements use method requirements until in-house limits can be determined.	1) Check calculations. 2) Identify correct and document problem. 3) Reanalyze LCS, if passes report. 4) If still out, re-extract/reanalyze LCS and associated samples.
Surrogate spike	Every sample including QC	Method requirements until in-house limits can be established.	1) Check Calculations. 2) Evaluate batch for trends. 3) Reanalyze sample, if acceptable report. 4) If not acceptable, re-extract/reanalyze sample.
Continuing Calibration Verification Sample	1 per every 10 samples	Method requirements	1) Reanalyze, if passes continue with analyses. 2) If still out, recalibrate.

SUMMARY OF QUALITY CONTROL PROCEDURES FOR IR & UV/VIS SPECTROSCOPIC METHODS

QC ELEMENT	FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
Method Blank (MB)	1 per batch, not to exceed 20 samples (non-drinking water samples) 1 per batch, not to exceed 10 samples (drinking water samples)	< RL < 0.5*RL for all USACE projects RL = laboratory reporting limit	1) Check calculations. 2) Investigate contamination source 3) Take and document appropriate corrective action. 4) Reanalyze blank. If passes, report. 5) If still out, re-extract/reanalyze all samples. 6) Flag sample result associated with contaminated method blank.
Matrix Spike/Matrix Spike Duplicate (MS/MSD) OR Matrix Spike and Sample duplicate	1 per batch, not to exceed 20 samples (non-drinking water samples) 1 per batch, not to exceed 10 samples (drinking water samples)	Project specific requirements. If there are not project specific requirements use method specific requirements until in-house limits can be determined.	1) Check calculations. 2) Reanalyze MS/MSD and sample. 3) If still out, re-extract/reanalyze MS/MSD and sample. 4) If still out report with suspected matrix interference.
Laboratory Control Sample (LCS) and LCSD (Duplicate)	1 per batch, not to exceed 20 samples (non-drinking water samples) 1 per batch, not to exceed 10 samples (drinking water samples)	Project specific requirements. If there are not project specific requirements use method requirements until in-house limits can be determined.	1) Check calculations. 2) Identify correct and document problem. 3) Reanalyze LCS, if passes report. 4) If still out, re-extract/reanalyze LCS and associated samples.
Continuing Calibration Verification Sample	2 per every 10 samples	Method requirements	1) Reanalyze, if passes continue with analyses. 2) If still out, recalibrate.

Batches are analyzed as follows: CCV, BLK, LCS, Samples, MS/MSD, LCSD, CCVD

SUMMARY OF QUALITY CONTROL PROCEDURES FOR IC ANALYSES

QC ELEMENT	FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
Method Blank (MB)	2 per batch, not to exceed 10 samples	**<** RL < 0.5*RL for all USACE projects RL = laboratory reporting limit	1) Check calculations. 2) Investigate contamination source 3) Take and document appropriate corrective action. 4) Reanalyze blank. If passes, report. 5) If still out, re-extract/reanalyze all samples. 6) Flag sample result associated with contaminated method blank.
Matrix Spike/Matrix Spike Duplicate (MS/MSD) OR Matrix Spike and Sample duplicate	1 per batch, not to exceed 10 samples	Use method specific requirements, +/-20%.	1) Check calculations. 2) Reanalyze MS/MSD and sample. 3) If still out, re-extract/reanalyze MS/MSD and sample. 4) If still out report with suspected matrix interference.
Laboratory Control Sample (LCS) and LCS Duplicate	1 per batch, not to exceed 20 samples (non-drinking water samples)	Use method requirements of +/-10%	1) Check calculations. 2) Identify correct and document problem. 3) Reanalyze LCS, if passes report. 4) If still out, re-extract/reanalyze LCS and associated samples.

QC ELEMENT	FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
Continuing Calibration Verification Sample	2 per every 10 samples	Method requirements	1) Reanalyze, if passes continue with analyses. 2) If still out, recalibrate.
Detection Limit Standard	Once per day	50-150% Recovery	Repeat analysis, if still out re-calibrate, if new calibration does not correct, new MDL study is in order.

Run order is as follows, CCV, LCS, LCSD, Det Lim STD, Blk, samples 1-10,Dup,MS,CCV,BLK,samples 11-20, Dup, MS, CCV, BLK

SUMMARY OF QUALITY CONTROL PROCEDURES FOR TITRATIONS

QC ELEMENT	FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
Method Blank (MB)	1 per batch, not to exceed 20 samples (non-drinking water samples) 1 per batch, not to exceed 10 samples (drinking water samples)	< RL < 0.5*RL for all USACE projects RL = laboratory reporting limit	1) Check calculations. 2) Investigate contamination source 3) Take and document appropriate corrective action. 4) Reanalyze blank. If passes, report. 5) If still out, re-extract/reanalyze all samples. 6) Flag sample result associated with contaminated method blank.
Sample Duplicate	1 per batch, not to exceed 20 samples (non-drinking water samples) 1 per batch, not to exceed 10 samples (drinking water samples)	Method requirements until in-house limits can be determined.	1) Reanalyze sample in duplicate. 2) If still report with suspected matrix interference.
Laboratory Control Sample (LCS)	1 per batch, not to exceed 20 samples (non-drinking water samples) 1 per batch, not to exceed 10 samples (drinking water samples)	Method requirements until in-house limits can be determined.	1) Check calculations. 2) Identify correct and document problem. 3) Reanalyze LCS, if passes report. 4) If still out, re-extract/reanalyze LCS and associated samples.

SUMMARY OF QUALITY CONTROL PROCEDURES FOR BIOCHEMICAL OXYGEN DEMAND (BOD)

QC ELEMENT	FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
Unseeded Feed Water Blank	2 per batch, not to exceed 20 samples	< 0.2 mg/L	1) Check calculations. 2) Investigate contamination source 3) Take and document appropriate corrective action. Correction for feed water contribution is not satisfactory. 4) Sample results are suspect. Resample and reanalyze.
Seed Control Blank	2 per batch, not to exceed 20 samples	0.6 mg/L $\geq$ DO $\leq$ 1.0mg/L	1) Check calculations. 2) Investigate the source of the nonconformance. 3) Take and document appropriate corrective action. 4) Sample results are suspect. Resample and reanalyze.
Acceptable Concentration Range	All customer samples	The final DO > 1 mg/L with a minimum depletion of 2.0 mg/L in DO	1) Determine if the sample has a very low BOD level. If it does this criterion may not be attainable. If it does not have a low BOD level the sample needs to be reanalyzed if no replicate has a final DO > 1mg/L.
Replicates	Each sample must be analyzed at	For the results which meet the acceptable	1) Check calculations. 2) Resample and re-

QC ELEMENT	FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
	minimum of 3 dilutions.	concentration range requirement, the relative standard deviation must be less than 10%.	analyze samples which exhibit greater variability or which only have one dilution that meets the acceptable concentration range criterion.
Glucose-Glutamic Acid Standard LCS/LCSD	2 per batch, not to exceed 20 sample batch, one after blank and seed controls and second, after last sample	198 mg/L +/- 30.5 mg/L	Identify correctly and document problem Resample and reanalyze

SUMMARY OF QUALITY CONTROL PROCEDURES FOR MICROBIOLOGICAL TESTING

QC ELEMENT	FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
Sample Bottle Sterility Check	Must be performed on every new lot number of sample bottles	Absence	1) Analyze a second bottle from lot. 2) Second failure, reject sample bottle lot
Media Check (autofluoresence)	Must be performed on every new lot number of medium	Absence of fluorescence	1) Medium which exhibits faint fluorescence must discarded
* Control Organisms Laboratory Control Sample (COLCS)	Analyze Total Coliform and Fecal Coliform known positive and negative controls.	Positive known – presence Negative known - absence	1) Reanalyze COLCS known, if passes report. 2) Identify, correct and document problem.
**Control Organisms Blank (COB)	Analyze Total Coliform and Fecal Coliform known blank controls	Absence	1) Reanalyze known blank, if passes report. 2) Identify, correct and document problem.
Autoclave sterility check	Monthly	Passes, OK, Fails	Call for Autoclave service.

*Test each lot of media for presence/absence, each batch for membrane filtration and MPN, as well as, and time confirmation of colonies or MPNs are done. Always observe manufacturers expiration date.
**Test each lot of media for presence/absence, and each batch for membrane filtration

5.10.1 Responding to Nonconformances; Roles & Responsibilities

When a nonconformance is recognized, each individual involved with the analysis in question has the following interactive role responsibilities:

- The Analyst: must be able to recognize nonconformance and immediately notify the area supervisor or QA Manager as to the corrective action taken to solve the problem. Each analyst is responsible for documenting and correcting problems that might affect quality.

- The Data Reviewer: must review all analytical and QC data for reasonableness, accuracy, and clerical errors; he is also responsible for monitoring QC charts (in terms of control limits). In an out-of-control event, the area supervisor works with the analyst and QA Manager or Area Supervisor to solve the problem and prevent the reporting of suspect data by stopping work on the analysis. All results that are suspect are repeated, if possible, after the source of the error is determined and remedied.

- Area Supervisor: In the event that an out-of-control situation occurs that is unnoticed at the bench or review level, (i.e., performance failure on a QC sample), the Laboratory Director will notify the analyst, help identify and solve the problem where applicable, ensure the work is stopped on the analysis, and verify that no suspect data are reported. The Area Supervisor must review and approve all corrective action reports and submit them to the QA Manager for review. The QA Manager is responsible for reviewing nonconformance report forms, recommending or approving proposed corrective actions, maintaining an up-to-date nonconformance log, verifying that corrective actions have been completed, distributing and filing nonconformance report forms, and assisting in resolving disagreements.

5.10.2 Procedures for Stopping Analysis

Whenever the analytical system is out of control, all concerned personnel initiate investigation-correction efforts.

If the problem is instrumental or specific only to preparation of a sample batch, any samples prepared after the out-of-control event are reanalyzed after the instrument has been repaired and recalibrated, provided holding times have not been exceeded.

If a sample batch is still out of control after reanalysis, all method-related activities shall stop immediately. A detailed laboratory-wide investigation shall be conducted to isolate and correct faulty operations. Sample security, integrity of standards, reagents, glassware, laboratory notebooks, instrument performance, and adherence to the methods shall be included in the investigation.

All actions taken shall be documented and placed in their respective case/contract file.

5.10.3 Corrective Action

The need for corrective action comes from several sources: equipment malfunction, failure of internal QA/QC checks, failure of follow-up on performance or system audit findings, and noncompliance with QA requirements. When measurement equipment or analytical methods fail QA/QC requirements that can not be solved by the analyst, the problems will immediately be brought to the attention of the Area Supervisor. Corrective measures will depend on the type of analysis, the extent of the error, and whether the error is determinant or not. The corrective action will be determined by the Area Supervisor, the analyst, and the QA Manager or by all of them in conference, if necessary.

A corrective action can be as extensive as replacing a complete lot of contaminated extraction solvent and re-extracting and reanalyzing a complete batch of samples due to reagent blank contamination; or it can be as simple as recalculating a series of results because an incorrect dilution factor was applied. Furthermore, the "right" corrective action must be determined on a case-by-case basis.

If failure is due to equipment malfunction, the equipment will be controlled by segregation or tagging until its repair; precision and accuracy will be reassessed, and the analysis will be rerun. All attempts will be made to reanalyze all affected parts of the analysis so that in the end, the product is not affected by failure of QC requirements.

When a result in a performance audit is unacceptable, the laboratory will identify the problems and implement corrective actions immediately. A step-by-step analysis and investigation to determine the cause of the problem shall take place as part of the corrective action program. If the problem cannot be controlled, the laboratory will analyze the impact on the data.

When a system audit reveals an unacceptable performance, work shall be suspended until corrective action has been implemented and performance can be verified as acceptable.

If the external audit (system or performance) report identifies deficiencies that require corrective action, the QA Manager shall notify the Laboratory Director and log the pertinent information, including the date a response is due, in the nonconformance log.

The QA Manager and the Laboratory Director shall assure that corrective action is taken. The QA Manager shall verify that the problem has been corrected. With the responsible supervisor, the QA Manager shall prepare a formal written response to the external organization and shall transmit the response to the external organization, with copies to other managers, as deemed necessary.

All incidents of QA failure and the corrective actions will be documented. Those reports will be placed in the appropriate case/contract file. Corrective action will be taken promptly for any deficiencies noted during the spot-check of raw data. When corrective actions are implemented, evidence of correction of deficiencies will be presented. Corrective action documentation will be forwarded to the Laboratory Director and QA Manager for evaluation and approval. Corrective Actions are discussed in *SOP 4003, Corrective Action Reporting*.

5.11 REPORTING

5.11.1 LABORATORY DATA REVIEW

All bench chemists document sample preparation activities in bound laboratory notebooks or pre-numbered, bound bench sheets. These serve as the primary record for subsequent data reduction. The data for GC/MS and GC analyses are generated by stand-alone computers and integrators, respectively. The data for atomic absorption analysis are collected using the instrument recorder to measure absorbance readings and strip charts to record absorbance expressed in peak height units. Results of each analysis are transferred onto analytical results through different translation formats uniquely designed for each particular analysis. QC samples and verifications are checked for accuracy and precision at the bench by the instrument operator/ analyst. The validity of instrument generated data shall be supported by the maintenance and inspection of the daily run check sheets, designed for each test method, and filled out each day by the analyst. The daily report follows the data to the Peer Review process. Once peer reviewed, the data is “QA” in OMEGA and this allows the Data Reporting personnel to produce a full report ready for the Lab Manager or his designee to give final review and management signature.

DATA VALIDATION

The goal is to have a systematic procedure of reviewing a body of data against set criteria to provide assurance of its validity prior to its intended use. Validation is accomplished through close scrutiny of the data collected and the procedures used to analyze the data including checks on the QC sample results. It is imperative that the analyst and reviewer know the methods completely and that the checklists are used.

The laboratory shall certify in writing that the data have been validated in accordance with the defined process.

DATA REVIEW

The review of data quality involves several levels of evaluation. In general, the analysts and the data reviewer are responsible for reviewing the data relative to instrument calibration, standard preparation, method blanks, raw data, calculations

and transcriptions. The analyst normally reviews 100% of the raw analytical data generated, including the calibration data and all calculations. Upon completion of the analyst review, the peer reviewer or Area Supervisor who is generally responsible for a review of 100% of the data generated performs a second level of review of the raw data. The emphasis is on the data acceptability relative to the data quality indicators and on the accuracy of the final data summaries. The Laboratory Director, if not already involved in the review process, performs the subsequent review, where 10-25% of the data quality indicators (such as method blanks, replicate analyses and spike recovery determinations) are compared to the acceptance criteria described in the analytical procedures. The QA Manager randomly audits 10% of raw data and data quality indicators to ensure the quality of the overall system.

All analytical problems encountered during sample analysis are properly addressed to provide explanations for data users.

DATA REPORTS

The format and content of a data report shall be dependent upon project needs such as whether explanatory text is required, client or contract requirements, and government agency reporting formats. Reports are prepared in a format suited to the end use. Each report however will contain the following information:

- A letter to the client, naming the number of samples and introducing the case narrative,
- A case narrative, including any QC qualifying statements
- Complete description of samples (client and SA, Inc. ID, and other information submitted by the client, such as the client sample name and location, date and time of sampling and date and time of analysis.
- A reference to each method analyzed
- Sample results including the parameter name and reporting limit
- Summary of QC data if requested by the client

Utmost care will be exercised in the confidentiality of client information and will only be released to third parties with expressed written permission.

Written results include only those analytes requested by the client. A list of personnel authorized to sign final reports is maintained by the Laboratory Director. Signatories attest that data and associated information in the report are believed to be correct. Oral preliminary release of data is prohibited. Written preliminary data is reviewed and reported although all of the requested test may not be done. It is understood that the remaining tests must be completed as soon as possible.

The content and format of data reports are approved by the Laboratory Director and the QA Manager. Once a format is agreed upon, a report may not be issued that deviate from the format without the approval of the Laboratory Director and the QA Manager. The Lab Manager and his reporting staff report some data as Electronic Data Deliverables. These formats are built into Omega and are built as needed and transmitted electronically.

DATA ARCHIVE

Client confidentiality and data integrity are of a high priority at SA, Inc. All active client files and those files that are less than six months old are maintained by the Customer Service Representative. All raw data less than six months old are maintained by the analysts in daily files. These daily files contain all information necessary to reconstruct all analyses performed on that instrument in a day.

All data and original observations that are greater than six months old or existing in a completed logbook are maintained in the SA, Inc. archives. All data backup tapes are also maintained in the archives. Access to the archives is limited to read only unless you have system administrator rights.

All SA, Inc. facilities that contain data are kept under tight security. The facilities are equipped with fire alarm systems and motion sensitive burglary systems. All data are maintained for a period of five years, unless other arrangements are made with the client.

5.12 REFERENCES

E.P.A. Requirements for Quality Management Plan, EPA/240/B-01/002, March 2001

E.P.A. Requirements for Quality Assurance Project Plans, EPA/240/B-01/003, March 2001

E.P.A. Guidance for Developing Quality Systems for Environmental Programs, EPA/240/R-02/008, November 2002

National Environmental Laboratory Accreditation Conference, Standard 5, Quality Systems, Revision 16, July 12, 2002

HAZWRAP DOE/HWP-65/R1

EPA Handbook on Analytical Quality Control in Water and Wastewater Laboratories.

ANALYTICAL METHOD REFERENCES

SA, Inc. Laboratories maintains a current reference library for the analyses performed. The current documentation includes the following:

Annual Book of ASTM Standards, Water and Environmental Technology, American Society for Testing and Materials, 1993.

E.P.A., Statement of Work for Contract Lab Program for Organic Analysis, OLM 4.2, May 1999

E.P.A. Statement of Work for Contract Lab Program for Inorganic Analysis, ILM05.2, December 2001

Federal Insecticide, Fungicide and Rodenticide Act (FIFRA); Good Laboratory Practice Standards; Final Rule 40 CFR Part 160, July 1, 1995.

"Guidelines Establishing Test Procedures for the Analysis of Pollutants Under the Clean Water Act; Final Rule and Interim Final Rule and Proposed Rule." Federal Register 40 CFR Part 136 Par VII, July 1, 1995.

Leaking Underground Fuel Tank Field Manual: Guidelines for Site Assessment, Cleanup, and Underground Storage Tank Closure, State Water Resources Control Board, 1989.

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

Standard Methods for the Determination of Water and Wastewater, 19th Edition, Edited by Lenore S. Cerceri, Arnold E. Greenbery, R. Rhodes, American Public Health, 1995.

SW-846 Test Methods for Evaluating Solid Waste, United States Environmental Protection Agency, Volume 1-4, Third Update, May 1997.

ETHICS POLICY ADDENDUM

SA Inc. Management will take action to implement training and personnel policies concerning improper manipulation of data and/or falsification of data or data reports to include immediate dismissal of the employee(s) involved. Misuse of standards, extraction methods, samples, computer software or data processing equipment to alter the reported data in a way not supported by Good Laboratory Practices will not be tolerated (USEPA 910/9-92/032). Any and all deviations from contractual requirements or the Quality Manual will be handled on an individual basis and may result in as little action as qualification of data to as severe an action as immediate dismissal of the employee(s) involved. All reports containing fraudulent data must be reported to the Quality Assurance Manager and the Laboratory Director.

When samples are analyzed which are related to enforcement sensitive, chain of custody, proprietary, on-going criminal investigations or other limiting projects, all information submitted to the Lab that is written, verbal, or implied must be considered private and confidential. Project information including, but not limited to, analytical results, data interpretation, responsible parties or project manager(s), must not be discussed or speculated upon with any entities other than the Lab Staff and the designated project manager(s). To discuss enforcement sensitive issues or projects outside the lab is considered abuse of the position and a violation of professional ethics and will be treated accordingly.

NAC445A/0642 states that the lab can not knowingly employ anyone, directly or indirectly, convicted of laboratory fraud or who has participated in unethical or inappropriate practices.

LABORATORY QUALITY ASSURANCE MANUAL

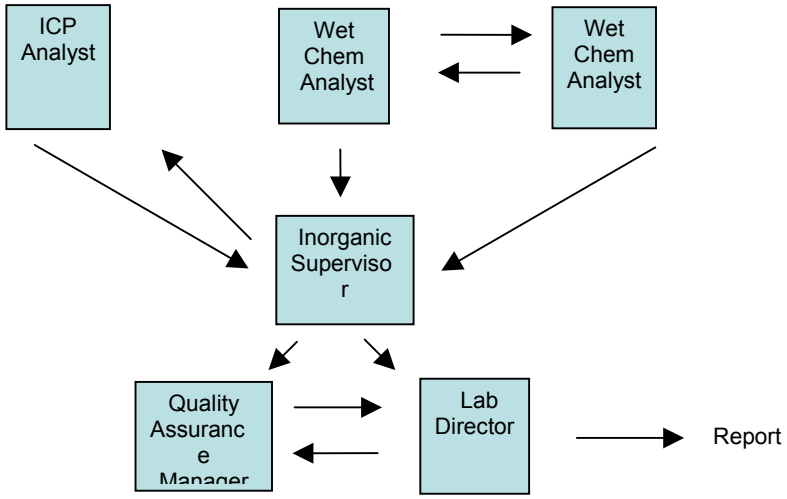
Appendix A

Peer Review Flow Chart

PEER REVIEW

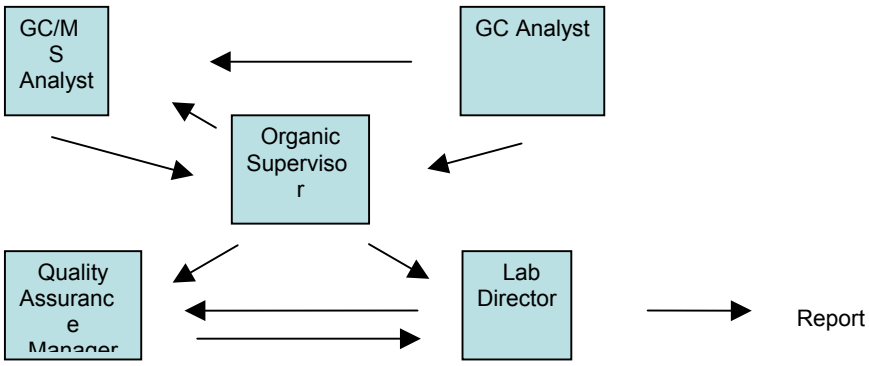
PROCESS

Inorganic



PEER REVIEW PROCESS

Organic



LABORATORY QUALITY ASSURANCE MANUAL

Appendix B

Current Certifications