QUALITY ASSURANCE MANUAL
VOLUME I

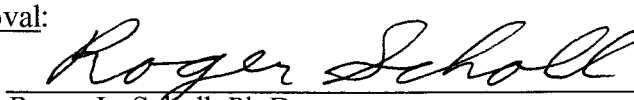
Section 1

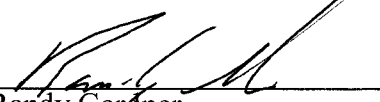
Identification Form

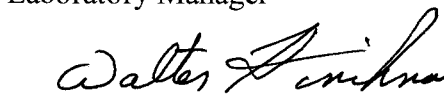
QUALITY ASSURANCE MANUAL IDENTIFICATION FORM

Document Title:	Quality Assurance Manual for Alpha Analytical Inc.
Organization Title:	Alpha Analytical, Inc.
Address:	Alpha Analytical, Inc. 255 Glendale Ave., Ste. 21 Sparks NV 89431
Responsible Officials:	Roger L. Scholl, Ph.D. Laboratory Director Phone No: (775)-355-1044 ext 127 Randy Gardner Laboratory Manager Phone No: (775)-355-1044 ext 157 Walter J. Hinchman Quality Assurance Officer Phone No. (775)-355-1044 ext 119
Prepared By:	Walter J. Hinchman Quality Assurance Officer Alpha Analytical, Inc. 255 Glendale Ave., Ste. 21 Sparks NV 89431
Document Coverage:	This is a document describing Alpha Analytical's Quality Assurance Manual (QAM). The QAM covers analytical chemistry data generated from samples submitted to Alpha for analysis.

Laboratory Approval:

(1) Signature:  Date: January, 2011
Name: Roger L. Scholl, Ph.D.
Title: Laboratory Director

(2) Signature:  Date: January, 2011
Name: Randy Gardner
Title: Laboratory Manager

(3) Signature:  Date: January, 2011
Name: Walter Hinchman
Title: Quality Control Officer

Note: January, 2011 also serves as the implementation and/or effective date for this version of the QAM.

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Section 3

Statement of Policies

ALPHA ANALYTICAL, INC.
QUALITY ASSURANCE MANUAL

3.0 INTRODUCTION

3.0.1 The quality assurance manual describes our quality systems incorporating laboratory activities to provide our clients with data of known quality with which to demonstrate regulatory compliance and for other decision-making purposes.

3.0.2 This system includes the description of analytical methods, how methods are continuously monitored and evaluated and how their performance is documented.

3.0.3 Our quality assurance program is designed to meet the requirements established in the use of Performance Based Measurements Systems (PBMS) in environmental testing and to provide the foundation for PBMS implementation of these standards.

3.0.4 References

The following references provide the foundation of this Quality Assurance Manual (QAM) to include the standardization and organization while providing guidance on the implementation of our quality systems.

3.0.4.1 National Environmental Laboratory Accreditation Conference's (NELAC) Chapter 5 Quality System, Approved June 5, 2003.

3.0.4.2 National Environmental Laboratory Accreditation Conference's (NELAC) Chapter 2 Proficiency Testing, Approved June 5, 2003.

3.0.4.3 Department of Defense, Quality Systems Manual (DoD QSM) for Environmental Laboratories, Department fo Defense, Version 4.2, October 25, 2010.

3.0.4.3.1 NELAC and DoD incorporated the requirements of ISO 9001, ISO 9002 and ISO/IEC 17025, relevant to the scope of environmental testing services into their quality systems documents.

3.0.4.3.2 Therefore, Alpha's quality systems have been established to comply with NELAC and DoD QSM as follows:

3.0.4.3.2.1 ISO 9001, used for the design/development of new methods, and/or to develop test programs combining standards and non-standard test and calibration methods,

3.0.4.3.2.2 ISO 9002, used for standard methods only,

3.0.4.3.2.3 ISO/IEC 17025, covers several technical competence requirements that are not covered by ISO 9001 and ISO 9002.

3.1 STATEMENT POLICY FROM MANAGEMENT

3.1.1 It is Alpha's policy and commitment from top management to perform all activities under good professional practices and to produce quality analytical data while servicing our clients under the guidelines of NELAC as described in this Quality Assurance Manual.

3.1.2 It is Alpha's policy and commitment from top management to define and document our quality assurance policies, systems, programs, procedures and instructions to the extent necessary to assure the quality of the test results.

3.1.3 It is Alpha's policy and commitment from top management to provide the necessary guidance and to require all personnel concerned with testing activities to throughly familiarize themselves with the quality system and to implement the policies and procedures in their work.

3.1.4 It is Alpha's policy and commitment from top management to ensure the quality systems are communicated to, understood by, available to and implemented by the appropriate personnel.

3.1.5 It is Alpha's policy and commitment from top management to continually work on improving the quality systems.

3.1.6 This commitment includes Alpha's CEO, President, Laboratory Director, Laboratory Manager and QA Officer.

3.2 PURPOSE

The purpose of the QA program is to:

a) Provide a consistent framework for the generation of analytical data in support of the programs enforced by various regulatory agencies;

b) Establish standard practices which permit inter-laboratory comparison of data; and,

c) Establish procedures for demonstrating that analytical systems are in control.

3.3 OBJECTIVES

More specifically, the objectives of the Quality Assurance program are to:

a) Provide a uniform basis for sample handling, instrument conditions, methods control, performance evaluations, and analytical data generation and reporting that will provide legally defensible results in a court of law;

b) Estimate the quality of each analytical system, which includes Precision, Accuracy, Representativeness, Comparability, and Completeness, (PARCC), that is sufficient for the needs of each project;

c) Assist in the early recognition of deficiencies which affect the quality of data;

d) Enable laboratory personnel with a system to identify and implement actions that are necessary to ensure the validity of laboratory data; and,

e) Require sufficient documentation to verify the quality of data submitted.

3.4 SCOPE

3.4.1 This document, the QA Manual, outlines the purpose, policies, organization, and operations of the quality systems established to support chemical analyses conducted for various programs and projects. All routine laboratory tasks have written Standard Operating Procedures (SOP's) to increase production uniformity, and are consistent with sound scientific principles. Advances in quality control procedures will be reflected in updated versions of this plan.

3.4.2 Implementation of the quality system described in the QA Manual will help ensure the validity of data and provide a reliable foundation on which to base decisions. It is a basic policy of Alpha Analytical to provide accurate, precise, complete, and representative determinations of chemical constituents in submitted samples, and to sufficiently document the analytical QC procedures.

3.4.3 In implementing the quality system, it is important to understand the difference between Quality Assurance (QA) and Quality Control (QC).

3.4.3.1 Quality Assurance refers to the system through which the organization provides assurance that monitoring of quality-related activities has occurred. Frequently, QA is interpreted as a record-keeping system to ensure documentation of all activities, including traceability, completeness, and security of documents.

3.4.3.2 Quality Control refers to specific actions taken to guarantee that

system performance is consistent with established limits regarding accuracy, precision, and comparability of results. Quality Control activities are conducted within a system of QA for proof that QC exists.

3.4.4 Implementation of the quality system is designed to assure data are collected under in-control conditions, rather than assuring documentation of poorly conducted analyses. The QA Manual is intended to establish a QA system and proper QC guidelines and procedures.

3.5 APPLICATION

3.5.1 The emphasis of this QA Program is on activities which generate analytical data, and includes those aspects of field sampling that may affect the integrity of samples. Alpha is not a sampling company; and therefore, have no ultimate control over the QA/QC procedures conducted in the field. Therefore, our role is advising field samplers as to the acceptable practices connected with field sampling.

3.5.2 The quality system provides the platform and organization for implementing the specific requirements necessary for sampling and chemical analysis of groundwater, surface water, soil, and sediment samples. In general, principles described herein are applicable to most field/laboratory activities.

3.5.3 This program has been written and designed specifically for Alpha Analytical and represents the system used by our staff during normal operating conditions. The QA plan may be superseded or appended with different or additional QA/QC activities related to a specific project or Statement of Work (SOW).

3.5.4 If a SOW or Quality Assurance Project Plan (QAPP) is used to supplement or append this QA/QC plan, then the Data Quality Objectives (DQO's) should be reflected in that particular QA/QC data package when requested.

3.6 HIGH PROFILE POLICIES

3.6.1 Ethics and Data Integrity Policy

The QA Manual and EPA methods are written as a set of policies and procedures that define what laboratory personnel are required to do; however, our ethics policies and our Laboratory Ethics Program, Appendix G, are written to ensure that employees are educated as to what they are not allowed to do.

Our data integrity training program includes discussions regarding the critical need for honesty, full disclosure in all analytical reporting and other related data integrity issues.

3.6.1.1 It is Alpha's policy to provide ethics and data integrity training to all new employees and provide this training to current employees on an annual basis.

3.6.1.2 Alpha Analytical has a zero tolerance policy regarding unethical situations, scientific misconduct and intentional lack of compliance with required procedures.

3.6.1.3 It is Alpha's policy to encourage laboratory personnel to come forward and report inappropriate activities.

3.6.1.4 It is managements philosophy and Alpha's policy to be proactive in the training, and education in the prevention of improper, unethical or illegal actions.

3.6.1.5 It is managements philosophy and Alpha's policy to clearly document how all analytical values were obtained and to supply the data user all data necessary to re-create and/or document final data results.

3.6.2 Manual Integration Policy

Manual integration is an important aspect of data integrity and is delineated to bring attention to and highlight the importance of this issue. Our manual integration training program includes discussion topics regarding the critical need for honesty, full disclosure in all analytical reporting and other related data integrity issues.

3.6.2.1 It is Alpha's policy to produce analytical data using automated and manual integration practices, in a manner that is non-arbitrary (meaning standards, control samples, and client samples are all integrated using consistent integration parameters).

3.6.2.2 It is Alpha's policy to produce analytical data using automated and manual integration practices, in a manner that is rational and can be backed up with the reason for a particular integration practice.

3.6.2.3 It is Alpha's policy to encourage laboratory personnel to come forward and report inappropriate activities.

3.6.3 Subcontract Laboratories

3.6.3.1 It is Alpha's policy to subcontract out analytical services not performed by Alpha, to laboratories that have been certified for those methods, to the best of our ability.

3.6.3.2 It is Alpha's policy, when subcontracting work because of unforeseen reasons (e.g., workload, need for further expertise or temporary incapacity, etc.), or on a continuing basis, this work is placed with:

a) a laboratory accredited under NELAP for the tests to be performed, or

b) with a laboratory that meets applicable statutory and regulatory requirements to perform the required analytical testing.

3.6.3.3 It is Alpha's policy to subcontract analytical services, not performed by Alpha, first to those laboratories that have been requested by the client and when possible, gain the approval of the client in writing.

3.6.3.4 If for any reason the subcontract laboratory is unable to perform the duties, then Alpha will procure an alternate laboratory of our choosing.

3.6.3.5 It is Alpha's policy to maintain a register of subcontract laboratories and a record or other evidence of appropriate certification.

3.6.3.6 It is Alpha's policy, to report all data issued by the subcontract laboratory on their official letter head and not retype and/or reissue hard copy data on Alpha's letter head. If this cannot be accomplished, the subcontract laboratory data must be clearly labeled as to the laboratory conducting the analysis.

3.6.3.7 It is the subcontract laboratories responsibility and business liability to ensure they have and maintain the appropriate State Certifications, method and compound certifications, program certifications and any other certifications or approvals necessary to perform the related tasks. It is the responsibility of all subcontract laboratories to ensure Alpha has been notified of those samples that cannot be analyzed due to:

- Inadequate sample / extraction holding time;
- Inadequate sample volume;
- Inappropriate sampling procedures;
- Inappropriate sample containers/preservatives;
- Loss of instruments or power failure;
- Loss of personnel;
- Loss of certifications or approvals to perform the requested task; or,

- Any other circumstances that would prevent the appropriate or adequate analysis of those affected samples.

3.6.3.8 It is Alpha's policy not to assume any liability of any subcontract laboratory data or the preponderance of the defenseability, documentation or data quality produced by any subcontract laboratory when the client or regulatory authority specifies which subcontractor laboratory to use. This is the responsibility of the subcontract laboratory and their respective certifying agents or authorities.

3.6.4 Training Policy

3.6.4.1 It is Alpha's policy to hire personnel which have appropriate education and/or On-the-Job-Training (OJT) adequate to perform their job duties.

3.6.4.2 It is Alpha's policy to conduct a training program that includes initial and continuing training of laboratory personnel.

3.6.4.3 It is Alpha's policy to ensure the competence of technical staff personnel, who operate analytical equipment, evaluate results, and sign test reports.

However, it is the responsibility of the trainee to ensure they have received adequate initial and continuing training and the documentation of that training to achieve and maintain skills commensurate with their responsibilities.

3.6.5 Policy for the Procurement of Supplies and Materials

3.6.5.1 It is Alpha's policy to evaluate and select supply vendors, chemicals, reagents and any other supplies which are critical to method performance to include additional testing to verify their quality before use.

3.6.5.2 It is Alpha's policy to purchase the item that meets or exceeds method specification, but not necessarily those brand name items specified in the method. In addition, it is Alpha's policy to determine what testing may be required to evaluate usability of supplies and materials.

3.6.5.3 It is Alpha's policy to purchase services and material supplies that affect the quality of environmental testing, at a level which will meet and/or exceed method/project criteria.

3.6.5.4 It is Alpha's policy to maintain purchasing documents for items affecting data quality.

3.6.5.5 It is Alpha's policy to maintain control over procured items such as containers, solvents, standards, etc. that have been recognized to be critical to environmental laboratories.

3.6.6 Computer Hardware/ Software Operation Policy

It is the policy of the laboratory, that the computer department has the responsibility for all software loading, upgrades, coding changes, debugging and has the responsibility for any hardware and/or software retirements. It is Alpha's policy to ensue that:

a) Policies and procedures are in place to protect the clients' electronic storage and transmission of results;

b) Only authorized and trained personnel are allowed to perform these functions;

c) Only authorized software is loaded on any company computer;

d) Only authorized, trained personnel are allowed to use company connections to the network or internet; and

e) No hardware, software, or raw data is removed from the laboratory without written authorization.

3.6.7 Policy for Testing of Proficiency Evaluation (PE) Samples

3.6.7.1 It is Alpha's policy to ensure adequate quality control procedures are in place for monitoring the validity of environmental testing through the participation of a Proficiency Testing (PT) program.

3.6.7.2 It is Alpha's policy to participate in two single-blind, single-concentrate PT studies, per year for each field of proficiency testing to maintain accreditation.

3.6.7.3 It is Alpha's policy to obtain PE samples from a Proficiency Testing Oversight Body (PTOB) / Proficiency Test Provider Accreditor (PTPA) approved PT provider.

3.6.7.4 It is Alpha's policy to analyze and submit PE sample results to the PT provider within their allotted time schedule as defined by the PT

provider.

3.6.7.5 It is Alpha's policy to analyze and treat PE samples in a manner consistent with real environmental samples using the same staff, analytical methods, procedure, equipment, facilities, etc. as used for routine analysis of that sample.

3.6.7.6 It is Alpha's policy that all staff members comply with the following restrictions on the transfer of PE samples and communication of PE sample results prior to the time results of the study are released:

a) No staff member shall send any PE sample to another laboratory for analysis for which accreditation is being requested;

b) No staff member shall knowingly receive any PE sample or portion of a PE sample from another laboratory for any analysis for which the sending laboratory is seeking accreditation;

c) No staff member shall communicate with any individual at another laboratory concerning the PE sample; and,

d) No staff member shall attempt to obtain assigned values of any PE sample from the PE provider.

3.6.8 Policy of Laboratory Organization and Staffing

It is Alpha's policy to have an organization that:

a) has sufficient managerial and technical personnel with the authority and resources needed to carry out their duties and to identify the occurrence of departures from the quality system or from the procedures for performing environmental tests, and to initiate actions to prevent or minimize such departures;

b) has processes to ensure that management and staff personnel are free from any undue internal and external commercial, financial and other pressures which might influence their technical judgement and adversely affect the quality of their work;

c) has policies and procedures to ensue the protection of client's confidential information and proprietary rights including procedures for protecting the electronic storage and transmission of results;

d) has policies and procedures to avoid involvement in any activities that may endanger or diminish the trust in Alpha's competence, impartiality, operational integrity or independence of judgement;

e) defines the organization and management structure of the laboratory, and the relationship between quality management, technical operations, and support services;

f) specifies the responsibility, authority and interrelationships of personnel who manage, perform or verify work affecting quality of the environmental tests;

g) provides adequate supervision of environmental testing staff, including trainees, by persons familiar with methods and procedures, purpose of each environmental test, and with the assessment of the environmental test results;

h) in-powers the technical management with the overall responsibility for the technical operations and for providing the resources needed to ensure the required quality of laboratory operations;

i) has a member of the technical staff appointed as the quality assurance officer who, irrespective of other duties and responsibilities, has the defined responsibility and authority for ensuring that the quality system is implemented and followed at all times. In addition, it is Alpha's policy to ensure the quality assurance officer has direct access to the highest level of management at which decisions are made on laboratory policies and resources;

j) appoints deputies for key managerial personnel, including the technical director and quality assurance officer; and

k) participates in a proficiency testing program as outlined by NELAC to qualify for and maintain laboratory environmental testing accreditation.

3.6.9 Waste Disposal Policy

A complete description of our Hazardous Waste SOP is found in Appendix C. There are two basic types of wastes within Alpha: 1) Hazardous, and 2) Non-hazardous. The important distinction between the two is that **all** hazardous waste generated at the 255 Glendale Avenue laboratory has to stay within that facility. **NO** hazardous waste can be generated, then moved to the Freeport facility behind the laboratory. It is Alpha's policy to have a waste disposal and pollution prevention program that:

a) has processes to ensure the safe handling and disposal of waste streams received or generated by Alpha;

b) has policies and procedures to ensue the safe protection of employees, employees health, clients, visitors and the general safe protection of our environment;

c) is throughly understood by the safety officer, waste disposal officer, employees, and anyone using, or contributing to the waste streams;

d) conducts continuous monitoring and training of employees regarding the proper and safe handling of generated wastes and their potential health concerns; and

e) has policies and procedures to ensure Alpha meets the requirements and documentation set forth by the Resource Conservation and Recovery Act.

3.6.10 Change in Ownership / Business Termination

For a change in ownership the following policies are to be met:

3.6.10.1 Alpha agrees to be accountable for any analyses, data and reports generated up to the time of legal transfer of ownership.

3.6.10.2 The buyer must agree in writing to be accountable for any analyses, data and reports generated after the legal transfer of ownership occurs.

3.6.10.3 It is Alpha's policy to ensure that analytical records are maintained or transferred according to the clients instructions, if either a change in ownership or business termination were to occur.

3.6.11 Service to Clients

The environmental testing business is a service oriented business, requiring a large amount of interaction with our clients. It is in our best interest, to emphasis the importance of conducting client communication in an environment that is professional, informational and confidential.

3.6.11.1 It is Alpha's policy to cooperate with our clients or their representatives to clarify the client's request and to monitor the analytical performance in relation to the work performed on their project, and to provide this service in a climate that ensures confidentiality to other clients.

3.6.11.2 Service to clients is a proactive engagement with our clients which requires staff to notify clients of problem situations such as:

a) incorrect, obsolete or improper method requests;

b) the need to optimize methods to ensure data quality objectives are met for difficult matrix or poor performing analytes;

c) lack of project guidance documents, such as the QAPP, or the need for clarification of requirements in the document; and

d) problems with sampling or analysis that may impact results (e.g., improper preservation of sample).

3.6.12 Customer Complaints

The documentation of customer complaints, the response to these complaints, and their resolution is useful information to improving the quality of our client service. This information, as part of our quality system, helps identify patterns of problems and is important in formulating a corrective response to those problems.

3.6.12.1 It is Alpha's policy to respond to complaints and/or problems in a reasonable time frame and in a courtesy manner that is both polite and professional to the customer.

3.7 GENERAL POLICIES

3.7.1 Equipment Purchase Policy - see section 10.02

3.7.2 Equipment Operation Policy - see section 10.1

3.7.3 Equipment Maintenance Policy - see section 10.5.1

3.7.4 Method Validation Policy - see section 8.1.2

3.7.5 Transcription and Calculation Error Policy - see section 12.3.4

3.7.6 Calibration and Reporting Policy - see section 9.1

3.7.7 Nonconforming Environmental Work - see section 13.0.1

3.7.8 The Use of New Sample Bottle Containers - see SOP E.4

3.7.9 Accommodation of Environmental Conditions Policy - see section 10.9.

3.7.10 Housekeeping and Workspace Policy - see section 10.10.

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Section 4

Organization and Responsibility

4.0 ORGANIZATION AND RESPONSIBILITY

4.1 INTRODUCTION

4.1.1 Alpha Analytical, Inc. is a business entity (environmental laboratory) incorporated in the state of Nevada that is legally responsible for all activities performed at this laboratory.

4.1.2 The guidelines described in this manual have been developed to ensure data quality is documented, controlled and data responsibilities are delegated and executed in such a way as to meet the requirements of NELAC and DoD QSM, while satisfying the needs of clients, the regulatory authorities and organizations providing recognition.

4.1.2.1 The Laboratory Director is ultimately responsible for the quality of data collected and reported in support of the various programs.

4.1.2.2 The Laboratory Manager is responsible for the implementation of the policies and procedures and for overseeing key operations within the laboratory.

4.1.2.3 The Quality Assurance Officer (QAO) is responsible for ensuring the quality system is implemented and followed at all times.

4.1.2.4 Staff members are responsible for assuming the accountability and reliability of the generated data.

4.1.3 All Alpha Analytical employees are trained and have a clear understanding of the laboratory's responsibility for producing data that are accurate, complete, documented, and meet the requirements of precision, representativeness, and comparability.

4.1.4 All employees have access to Alpha's Quality Assurance Manual and are trained on the specific portions that apply to their responsibilities.

4.1.5 All employees participate in on-going discussions of the lab's quality assurance procedures and are trained in the importance of the quality control data acquired during normal sample analysis.

4.2 RESPONSIBILITIES AND AUTHORITY

It is Alpha's policy to ensue the laboratory has sufficient managerial and technical personnel to support the analytical process and to identify the occurrence of departures from the quality system, and to initiate actions to prevent or minimize such departures.

It is Alpha's policy to define and maintain current job descriptions for all personnel who manage, perform or verify work affecting the quality of the environmental tests. Personnel are assigned responsibilities delineating their specific jobs, i.e., clerical, analysis, and sample preparation.

The relationships between the organization and the responsibilities involved in our quality systems are outlined below. The following descriptions assign certain tasks to various levels of responsibility. The organizational chart is shown in Table 4-1. The resumes of employees designated as technical personnel are included in individual employee training documents.

4.2.1 Laboratory Director

The Laboratory Director is responsible for the overall collection and analysis of data produced and reported by Alpha Analytical. The following list describes some of the more important duties that are performed by the Laboratory Director. These duties include the following:

a) Ensures that all analytical activities are performed according to methods and protocols specified in the QA Manual;

b) Ensures that daily operations function smoothly within the operating conditions and guidelines established by Alpha Analytical;

c) Coordinates analytical work to ensure the completion of tasks are within established time frames;

d) Ensures the analytical data review process is functioning properly;

e) Oversees preventative maintenance activities;

f) Evaluates and implements changes in methods and quality control measures;

g) Identifies quality control problems and takes measures to correct or eliminate the problem source;

h) Assumes the responsibility for authorizing the resumption of work when nonconforming data has been identified.

i) Assumes the responsibility for determining the level of qualification, experience and skills necessary for staffing all positions in the laboratory to perform the technical duties with the required quality control;

j) Ensures that the training of each member of the technical staff is kept current;

k) Ensures that all technical laboratory staff members have demonstrated capability in the activities for which they are responsible, such as IDC and MDL studies;

l) Oversees personnel and evaluates their performance;

m) Ensures the laboratory is participating in a proficiency testing program and that corrective actions are implemented after testing and evaluating the effectiveness of the corrective actions; and

n) Notifies clients and discusses quality issues when data quality has impacted their sample results.

4.2.2 Laboratory Manager

The Laboratory Manager is responsible for the implementation of the policies and procedures as described by the QAM with the direction of the Laboratory Director. The Laboratory Manager is responsible for overseeing key operations within the laboratory to ensure the work flow is efficient, balanced and within the guidelines established in the QAM. The following list describes some of the more important duties that are performed by the Laboratory Manager. These duties include the following:

a) Ensures the Sample Custody Officer(s) has the appropriate staffing, training and experience necessary to carry out the responsibility for receiving and logging in samples as they arrive at the laboratory;

b) Ensures the Document Control Officer(s) has the appropriate staffing, training and experience necessary to carry out the responsibility for implementing the Document Control Program;

c) Ensures the Director of Client Services, Technical Representatives and Project Coordinators have the appropriate staffing, training, experience and/or education necessary to carry out their responsibilities and duties;

d) Ensures the Laboratory Information Management System (LIMS) Administrator has the appropriate staffing, training and experience necessary to carry out the responsibility of implementing the Software Quality Assurance Plan (SQAP);

e) Coordinates with the Laboratory Director and the QA Officer to assure all activities, both sampling and analysis are performed according to the specific QA Project Plans (QAPP), methods and protocols specified in this QA Plan;

f) Ensures the daily operations function smoothly within the operating conditions and guidelines established by the quality systems management;

g) Ensures the completion of all tasks are within the established time frames;

h) Ensures the laboratory is participating in a proficiency testing program and corrective actions are implemented after testing and evaluating the effectiveness of the corrective actions; and,

i) Responsible for the assessing, selecting and use of subcontract laboratories to meet project specific criteria.

4.2.3 Quality Assurance Officer

4.2.3.1 The Quality Assurance Officer (QAO) has the responsibility and authority for ensuring the quality system is implemented and followed at all times. The QA Officer:

a) serves as the focal point for QA/QC and is responsible for the oversight and/or review of quality control data;

b) is independent from laboratory operations;

c) objectively evaluates laboratory data and performs assessments without outside managerial influence;

d) has documented training and experience in QA/QC procedures and is knowledgeable in the quality system as defined under NELAC;

e) has a general knowledge of the analytical test methods for which data review is performed;

f) arranges for and/or conducts internal audits annually; and

g) notifies laboratory management of deficiencies in the quality system and monitors corrective actions.

h) ensures staff employees operate in accordance with the laboratory's documented ethics policies.

4.2.3.2 In addition, the QA Officer has the responsibility for reviewing, advising and improving on all aspects of the quality system, including the following:

a) assisting the data requester in specifying the QA/QC procedure to be used during the testing program;

b) makes recommendations to the data requester and Laboratory Director, if problems are detected, to ensure that appropriate corrective actions are taken;

c) oversees the review of quality control data to determine if test data is acceptable;

d) updates and supervises the updates of quality control markers, such as accuracy, precision, and method detection limits;

e) coordinates and oversees the preparation of quality assurance plans;

f) reviews new or proposed protocols to determine appropriate use;

g) reviews method validation data;

h) ensures continuous advancement of laboratory procedures by implementing, maintaining and improving the quality system;

i) ensures staff personnel understand their contributions to the quality system;

j) ensures communication takes place at all levels within the laboratory regarding the effectiveness of the quality system;

k) evaluates the effectiveness of training;

l) uses available tools, such as audit and surveillance results, control charts, proficiency testing results, data analysis, corrective and preventative actions, customer feedback, and management reviews in efforts to monitor trends and continually improve the quality system; and

m) ensures the QA manual is current.

4.2.4 Analysts

4.2.4.1 Analysts are primarily responsible for ensuring they are completely familiar with the quality systems documentation requirements and the implementation of the policies and procedures affecting their work. The following list

describes some of the more important responsibilities and duties that are performed by the analysts. These responsibilities include:

a) Analysts are responsible for ensuring they perform the required analyses according to test methods specified by rule, permit, QAPPS and/or SOPs;

b) Analysts are responsible for ensuring the instrument and related equipment is working to acceptable standards; and,

c) Analysts are responsible for ensuring the required supplies are available for their particular instrument.

4.2.4.2 Some of the various duties analysts perform include the following:

a) Ensures all analytical equipment has been properly calibrated before beginning analysis;

b) Ensures all identifying information, including sample identification numbers, have been accurately transcribed into records or computer databases;

c) Ensures all calculations are correct;

d) Ensures the appropriate confirmatory tests or procedures have been completed;

e) Identifies, documents, and begins corrective actions on any quality control problem that relates to the analytical test; and,

f) Maintains equipment in working condition and documents all preventative maintenance and repairs.

4.2.5 Extraction Technicians

Extraction Technicians are responsible for a large number of duties and activities which support personnel performing sample analysis. These activities are necessary to ensure quality extracts are prepared for instrumental analysis.

Extraction Technicians are responsible for ensuring they are completely familiar with the quality systems documentation and implementation of the policies and procedures affecting their work. The following list describes some of the more important responsibilities and duties that are performed by the extraction technician. These duties and responsibilities include the following:

a) Performs the required extraction, clean-up procedures, and final concentration steps according to the test methods used by Alpha Analytical;

b) Ensures all extraction equipment is properly maintained before and after use;

c) Ensures all analytical equipment, such as pH meters and balances, have been properly calibrated before use;

d) Ensures all identifying information, including sample identification numbers, have been accurately transcribed into the extraction logs and other pertinent areas;

e) Documents with meticulous accuracy all procedures performed on the sample and notes any irregularities observed during the sample extraction which may affect the analysis; and,

f) Communicates to analysts and all affected personnel irregularities or observations of the sample prior to analysis ensuring proper decisions are made regarding that particular sample.

4.2.6 Sample Custody Officer

The Sample Custody Officers (SCO) are responsible for implementing, updating, and maintaining Alpha Analytical's Sample Tracking Plan. The Sample Custody Officers (SCO) are primarily responsible for ensuring the proper handling and documentation of all samples are performed by the person who has legal custody of that sample during all phases of laboratory work. The SCO performs the following duties:

a) Assumes the responsibility for receiving and logging in samples as they arrive at the laboratory;

b) Obtains the documentation required to complete the chain-of-custody form for each specific sample;

c) Notes any irregularities of the sample and/or inquires of the client regarding these abnormalities;

d) Notes special project requirements with detailed information on the chain-of-custody;

e) Informs all personnel affected by special projects of the requirements and that the project is in-house; and,

f) Assumes responsibility for placing environmental samples in the proper storage area to prevent possible sample cross-contamination.

4.2.7 Document Control Officer

The Document Control Officer (DCO) is responsible for implementing, updating, and maintaining Alpha Analytical's Document Control Program. The Document Control Officer is primarily responsible for overseeing data assembly and documentation of client files. The DCO is responsible for the following areas:

a) Ensures that all documents concerning a client/sample file are accounted for when a project is completed;

b) Responsible for the organization and assembly of all documents related to a client sample file according to established SOP's;

c) Maintains control of confidential information;

d) Coordinates the Document Control Program; and,

e) Reports directly to the Laboratory Manager.

4.2.8 Laboratory Information Management System (LIMS) Administrator

The LIMS Administrator is responsible for implementing, updating and maintaining the Software Quality Assurance Plan (SQAP). When computers are used for the capture, processing, manipulation, recording, reporting, storage and retrieval of analytical data, the LIMS Administrator ensures the following:

a) All requirements of the SQAP are being met;

b) Computer software is documented and adequate for use;

c) Ensures procedures are established and implemented for protecting the integrity of data, such as data entry or capture, data storage, data transmission and data processing;

d) Ensures computer equipment is adequately maintained to function properly; and,

e) Establishes and implements appropriate procedures for the maintenance and security of data including the prevention of unauthorized access to, and the unauthorized amendment of computer records.

4.2.9 Client Services Manager (CSM)

The Client Services Manager is responsible for th procurement of new clients and projects and the maintenance of existing clients and ongoing work. The client services manager functions as the central clearing house when new work is being solicited or contracts are being reviewed for submission. When new work or contracts are being prepared for submission, the CSM ensures the following:

a) Are the requested methods of analysis adequately defined, documented and understood;

b) Does the laboratory have the appropriate facilities and resources to meet the contract requirements;

c) Can the requested methods of analyses be performed;

d) Reviews all contract documents;

e) Resolves contract differences;

f) Ensues contracts are properly documented; and

g) Resolves and/or follows up on contract disputes or contract amendments.

4.2.10 Training Coordinator

The Training Coordinator is responsible for the oversight of the training program and the maintenance of training and qualification records. This person coordinates the training program by updating Trainers and/or the Training Committee Members of when and what type of training is needed, and the overall recommendations to the Training Program.

4.3 WORK STOPPAGE

The following personnel have the authority to stop work or withhold test results in response to quality problems when nonconforming work is identified. They have the authority to approve or disapprove analytical and/or extraction batches and the authority to approve or disapprove final analyses.

a) Laboratory Director,

b) Laboratory Manager, and

c) Quality Assurance Officer

Alpha Analytical, Inc.

Organizational Chart

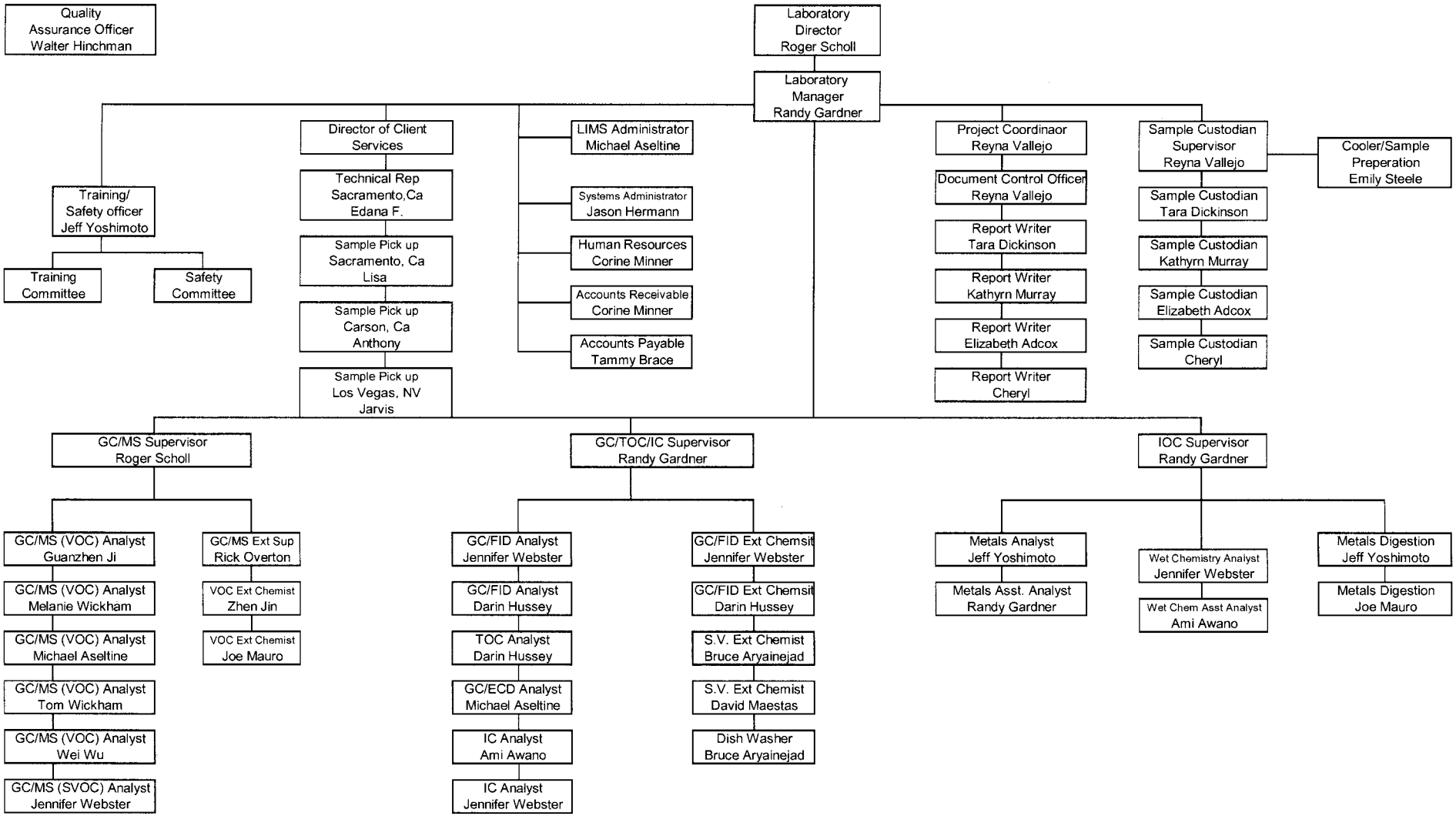

TABLE 4-1

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Quality Assurance Routines to Assess Precision, Accuracy and the Calculation of Method Detection Limits

5.0 QUALITY ASSURANCE ROUTINES TO ASSESS PRECISION, ACCURACY AND THE CALCULATION OF METHOD DETECTION LIMITS

5.1 GENERAL DATA QUALITY OBJECTIVES

5.1.1 Data Quality Objectives (DQOs) for each method and analyte are determined to ensure analytical and method-specific goals are met. DQOs for analytical measurements are commonly interpreted as Precision, Accuracy, Representativeness, Completeness and Comparability (PARCC). Precision and accuracy are the two most common criteria used to define DQOs.

5.1.2 Accuracy and precision are generally determined for each EPA method target analyte by data presented as either single laboratory or spooled data from multiple laboratories. This type of data, represents Method Derived Data Quality Objectives. As part of our QA program, Alpha statistically determines in-house Laboratory Derived Data Quality Objectives and ensures these DQOs are comparable to method derived DQOs.

5.1.3 The last three, representativeness, completeness and comparability, are DQOs that are laboratory and project specific. Therefore, these three elements do not have method specified DQOs. The determination of method detection limits is also a useful tool to evaluate project or analytical goals.

5.2 ESTIMATION OF THE UNCERTAINTY OF MEASUREMENTS

One of the primary objectives of the QA plan is to establish a framework that can estimate the quality of each analytical system, including precision, accuracy, representativeness, completeness, and comparability. Specific data quality objectives for accuracy, precision, and completeness are based on prior knowledge of the measurement system employed and method validation studies using duplicates, spikes, standards, recovery studies, etc. However, as a practical matter of laboratory responsibility, we can only estimate the portion of measurement uncertainty that is under our control.

Most well-recognized standardized test methods have been rigorously evaluated and validated through intra-laboratory method validation studies. In those cases, the method specifies the limits as statistical values of the major sources of uncertainty of measurement and specifies the form of presentation of calculated results. Therefore, for those environmental test methods which have published or referenced PARCC criteria, Alpha uses the method defined uncertainty criteria when evaluating test data.

In certain cases the nature of the test method may preclude the rigorous statistically valid calculation of uncertainty of the measurement. In these cases, the procedure is evaluated to identify the components of the method uncertainty and Alpha will attempt to make a reasonable estimation of this uncertainty and ensure that the form of reporting the data results does not give a wrong impression of the procedural uncertainty.

5.2.1 Precision

A distribution curve can be created from repetitive sample and parameter measurements in which the spread or dispersion about the mean can be calculated or expressed as precision. The calculations for precision where duplicate or replicate analyses have been performed are accomplished by the analysis of laboratory control samples and matrix spike samples.

Field duplicate samples and matrix spike duplicates are analyzed to assess field sampling precision. Laboratory control samples and laboratory control sample duplicates are analyzed to assess analytical precision. Precision measurements are determined using Relative Percent Difference (RPD) between the duplicate sample results and, for replicate analyses, the Relative Standard Deviation (RSD) is calculated and also used to determine precision.

5.2.2 Accuracy

The accuracy of an analytical measurement is defined as the amount of agreement between an experimental measurement of the concentration of a parameter and the known true concentration of that parameter. Accuracy is commonly expressed as a percent recovery of spiked target analytes. Analytical accuracy is assessed by comparing the percent recovery of analytes spiked into an LFB or LCS to a defined control limit. For organic methods of analysis, the surrogate compound recoveries are also used to assess accuracy and method performance for each sample analyzed.

Percent recovery, where applicable, is calculated using concentration limits, such as ug/Kg or ug/L, and converted to a percentage. Accuracy criteria are both compound and method specific.

5.2.3 Representativeness

The representativeness of samples describes the degree to which the sample represents an undisturbed matrix. Obtaining representative samples is a difficult task that requires site and project specific planing prior to field sampling. Matrix, target analytes, methods of analyses, sampling depths, sampling equipment, time of sampling, etc. are all contributing factors in obtaining a representative sample. In order to minimize random errors introduced by non-uniform sampling procedures, the SOP's cited in the FSP are suggested procedures to be followed for sample collection. The use of standard operating procedures will help to provide uniformity for the sample collection work.

5.2.4 Completeness

Data completeness is defined as the percentage of total tests conducted that are deemed satisfactory for a specific analysis or matrix. General criteria for data

completeness are determined and compared to project specific DQOs. Completeness is expressed as a percent of the overall data generated and is calculated as follows:

$C = V/T \times 100$

where,

C = Percent completeness;
V = Number of measurements judged satisfactory or validated and,
T = Total number of planned measurements.

Completeness goals are as follows:

a) 95% completeness for aqueous samples with a relatively clean background matrix (i.e., SDWA samples);

b) 90% completeness for methods which may contain a some degree of background interferences (i.e., CWA); and

c) 85% completeness for methods which more than likely will contain significant degrees of background contamination as well as matrix bias (i.e., RCRA type samples).

5.2.5 Comparability

Comparability of data is expressed as a measure of confidence where multiple sets of data may be used to evaluate a common analyte by a standard method of analysis.

Data comparability can be scrutinized by collecting independent samples in the same manner during different sampling episodes and by processing samples using the same procedures in the laboratory. Field sampling and laboratory analytical procedures for each parameter should remain as constant as possible at all times.

Alpha uses standard EPA methods to insure inter-laboratory comparability of data. In addition, generated data is expressed in units consistent with the data generated by other laboratories reporting similar analyses to allow comparability of data between organizations. SOPs are an important element in both inter- and intra-laboratory comparison of data. These procedures allow laboratory activities to become routine and standardized, which minimizes random errors.

5.3 CONTROL CHARTS

5.3.1 Shewhart Control Chart

The Shewhart control chart uses a population of data points to statistically calculate

the average percentage recovery and standard deviation for one or more variables mathematically describing the data set and is constructed from data representing the performance of a complete analytical method in order to monitor all activities associated with the analytical procedure.

Control charts are then prepared for target compounds such as surrogates and selected Data Qaulity Indicators (DQIs) chosen to represent the entire list of target analytes.

Recovery data is used and graphed on a Shewhart control chart to monitor variations in the accuracy of routine analysis and to detect possible trends in those variations. Although tabulations of precision and accuracy are used to evaluate if a datum point falls within the prescribed limits, trends are difficult to discern from tables. Therefore, control charts consist of a graphical portrayal of that information.

5.3.1.1 Control Limits

Several methods require laboratories to achieve a particular level of precision and accuracy, that are described and built into the methods. However, many of these methods establish precision and accuracy requirements from the results of a single laboratory study. Often these criteria are not indicative of the method nor do they accurately reflect how laboratories are being evaluated on a national scale. Therefore, in-house analytical data is used to determine, establish and evaluate the window of acceptability describing accuracy limits used in the Shewhart control chart trend analysis. In-house control limits are established that:

a) are statistically derived using scientifically valid and documented procedures;

b) meet the limits specified by the project or as stated in the method, if available;

c) are updated on the prescribed method/program frequency;

d) are based on at least 30 data points generated under the same analytical process; and

e) do not exclude failed LCS recovery data and statistical outliers from the calculation, unless there is a documented and scientifically valid reason (e.g., bad LCS standard, leaking purge vessel etc.).

If required, program specific windows of acceptability may be used (e.g., DoD) for the purpose of evaluating analytical QC data; however, this does not preclude the use of in-house developed control limits for monitoring trends.

A representative group of target analytes is chosen to chart trends for those methods which have a large number of target analytes.

5.3.1.2 Upper and Lower Warning Limits

There are two sets of limiting lines that may be used in the construction of the Shewhart control chart. The inner window or warning limit is defined by the upper or lower warning limits (UWL) and (LWL), and is calculated as:

$$UWL = X + 2s$$
$$LWL = X - 2s,$$

where X is the mean and s is the standard deviation.

Statistically, one in twenty will fall outside the inner control limit, provided the data are statistically uniform (i.e., 95% confidence interval).

5.3.1.3 Upper and Lower Control Limits

The second window is defined by the Upper and Lower Control Limit (UCL) and (LCL) and is calculated as:

$$UCL = X + 3s$$
$$LCL = X - 3s$$

Statistically one in one-hundred will fall outside the outer control limits, provided the data are statistically uniform, (i.e., 99% confidence interval).

5.4 METHOD EVALUATION/VALIDATION

5.4.1 Environmental organic and inorganic analytical methods are evaluated for those parameters that adversely affect data quality. These parameters are things such as detection limit, reporting limit, accuracy and precision.

5.4.2 This includes non-standard methods, laboratory-designed/developed methods, standard test methods used outside of their published scope, and amplifications or modifications of standard methods to confirm that the methods are fit for its intended use.

5.4.3 Method validation is technically defined as the confirmation by examination and provision of objective evidence that the particular requirements for a specific intended use are fulfilled. In layman terms, method validation is as extensive as is necessary to meet the needs of the application. Method validation results and the procedure used for the validation are recorded. Records of these validation studies and their reviews are available for external assessment.

5.4.4 In general, these demonstration studies do not test the performance of the method using real world samples, but in a clean matrix sample (a matrix in which no target analytes or interferences are present at concentrations that may impact the results of a specific test method), e.g., drinking water. In addition, for analytes which do not lend themselves to spiking, the demonstration of capability may be performed using quality control samples.

5.4.5 Method Evaluation/Validation Studies

Method evaluation and validation studies are designed, conducted and assessed to meet the intended use and client's needs. This includes evaluating the range and accuracy of the values obtainable from validated methods (e.g. the uncertainty of results, detection limit, selectivity of the method, linearity, limit of repeatability and/or reproducibility, robustness against external influences and/or cross-sensitivity against interference from the matrix of the sample) to ensure they are assessed to meet the client requirements.

The following list briefly describes the minimum method parameters that are critically evaluated and documented prior to performing a new method.

5.4.5.1 Initial Demonstration of Capability (IDC) Study

A IDC study is performed prior to the analysis of samples or when a significant change in instrument, personnel, matrix or test method has taken place. Continuing Demonstrations of Capability (DOC) studies are performed annually.

This primary study is used to generate accuracy and precision data to demonstrate and document the analyst has the necessary skills and proficiency to perform the method by meeting the available prescribed method criteria.

When an IDC study is not feasible, the method is validated by a systematic assessment of factors that could influence the results; and/or an assessment of the precision and bias of the results based on the science of the method and practical experience.

5.4.5.2 Method Detection Limit (MDL) Study

A MDL study is conducted to determine the minimum amount of a substance that an analytical process can detect. Some agencies refer to this as a Limit of Detection (LOD) or Detection Limit (DL) study. Some methods state the MDLs and/or estimated MDLS that were achieved in multi-laboratory studies as a means to help the laboratory evaluate its' method data.

5.4.5.3 Limit of Quantitation (LOQ) Study

A LOQ study is conducted to determine and define the minimum level, concentration or quantity of a target analyte that can be reported with a specified degree of confidence. The LOQ was formerly known as the Practical Quantitation Limit (PQL), Minimum Reporting Limit (MRL) or simply the Reporting Limit (RL).

5.4.5.4 Calibrations

Initial calibration and calibration verification protocols are typically method specific and are found in individual method SOP's. The calibration procedure also includes the validation of reference material or standards by documenting their concentrations against a second source reference standard.

5.4.5.5 Proficiency Evaluation (PE) Samples

The results of PE sample analysis, if available, are used to evaluate the laboratory and analyst's ability to produce accurate data.

TABLE 5-1
STATISTICAL CALCULATIONS

STATISTIC	SYMBOL	FORMULA	DEFINITION	USES
Mean	$\bar{x}$	$\frac{\sum_{i=1}^{n} X_i}{n}$	Measure of central tendency	Determine average value of measurements
Standard Deviation	SD	$\left(\frac{\sum (x_i - \bar{X})^2}{(n-1)}\right)^{\frac{1}{2}}$	Measure of relative scatter of the data	Calculating variation of measurements
Relative Standard Deviation	RSD	$(\frac{s}{\bar{x}})\ x\ 100$	Relative standard deviation, adjusts for magnitude of observations	Assess precision for replicate results
Percent Difference	% D	$\frac{x_1 - x_2}{x_1}\ x\ 100$	Measure of the difference of 2 observations	Assess accuracy
Relative Percent Difference	RPD	$\left(\frac{(x_1 - x_2)}{(\frac{x_1 + x_2}{2})}\right)$ x 100	Measure of the variability that adjusts for the magnitude of observations	Assess total and analytical precision of duplicate measurements
Percent Recovery	% R	$\left(\frac{x_{measured}}{x_{true}}\right)\ x\ 100$	Recovery of spiked compound in pure matrix	Assess accuracy
Percent Recovery	% R	$\frac{\text{Value of Spiked Sample} - \text{Value of Unspiked Sample}}{\text{Value of Added Spike}}$ x 100	Recovery of spiked compound in sample matrix	Assess matrix effects and total precision

x = Observation (concentration)
n = Number of observations

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Section 6

Sampling Procedures

6.0 SAMPLING PROCEDURES

Alpha is not generally responsible for sample collection. When Alpha does collect samples for analysis, it follows the procedures outlined in this section, the Field Sampling Plan (FSP) found in appendix B or other project specific field sampling plan.

6.1 INTRODUCTION

It is Alpha's policy to ensure procedures are in place and being adhered to for the transportation, receipt, handling, protection, storage, retention and/or disposal, including provisions necessary to protect the integrity of the sample and to protect the interest of the laboratory and the client.

6.1.1 The most important aspect of field sampling is to obtain samples that are proper representations of the sampled matrix. Trace levels of contaminants from sources external to the sample must be eliminated through the use of good sampling techniques.

6.1.2 Once a sample has been collected, it should be properly stored and preserved to maintain the chemical and physical properties that it possessed at the time of collection. Sample containers are packaged for shipping in insulated containers, and constructed to ensure that sample bottles will arrive intact.

6.1.3 Samples must be expeditiously sent to the laboratory and as a general rule, storage at low temperature is the best way to preserve most samples; however, the length of time a sample can be held even at low temperature varies with the analyte and matrix.

6.1.4 When samples are received, the time lapse between sampling activity and analysis should not exceed the times shown in the sample holding times tables. Sample management and stringent documentation are the key factors as outlined in the FSP.

6.2 SAMPLE HANDLING

6.2.1 Samples are collected and handled in a manner that attempts to maintain sample integrity and preserves the potential contaminants being analyzed. Samples are collected in containers specific to the matrix and requested analysis.

6.2.2 Gloves should be used during the sampling and preparation procedures for protection against possible exposure to carcinogens and to minimize accidental contamination of samples by the collector. When wearing gloves, the person must be careful not to let the gloves come into contact with the sample, the interior of the container, or allow solvents to touch both the sample and any extract.

6.3 SAMPLE SHIPMENT

6.3.1 Samples requiring refrigeration are carefully placed in coolers with ice to maintain a temperature of < 6°C. Glass containers are securely packaged in ice chests using packing material, to avoid breakage in transit. Chain-of-custody forms should be completed at the sampling site and sent with the sample to maintain integrity at all times. Samples are then transported to the laboratory as soon as possible.

6.4 CONTAINERS

6.4.1 Sample containers are determined by the requested analysis. However, the following containers are generally used for environmental analysis:

- Standard 40 ml clear glass screw-cap Volatile Organic Analysis (VOA) vials with Teflon-faced silicone septum are used for volatile analysis;
- Narrow mouth, 1 L amber Boston round glass bottles with Teflon-lined lids are used for semi-volatile, analysis;
- Large mouth, 8 and 4oz glass bottles or brass tubes are typically used for soil and sediment samples; and,
- Narrow mouth, 125, 250, 500 ml and 1-L polyethylene bottles are typically used for the analysis of metals and other general inorganic parameters.

6.4.2 All sample containers are cleaned according to EPA established protocols. Factory cleaned sample containers require no further cleaning prior to sample collection, and are the containers of choice. Sample containers are not reused. Alpha maintains a sequestered supply of sample containers to eliminate the possibility of contamination of the sample from the container. Containers are sequestered by lot to track QA/QC procedures associated with that group of sample containers.

6.5 SAMPLE PRESERVATION

6.5.1 The purpose of sample preservation is to prevent or retard chemical degradation or sample modification during transit and storage; therefore, sample preservation maintains the chemical integrity of the sample. Most solid samples require cooling as the only preservation technique. Water sample are subject to a variety of specific preservation techniques, depending on the target analytes.

6.5.2 Samples requiring chemical preservation typically means that the pH or removal of residual chlorine of the sample should be performed and checked in the field and verified in the laboratory during sample preparation or analysis.

6.5.3 Samples are preserved according to analytical methods and programs by which the sample will be analyzed. Chemical preservation can be generally divided into two basic categories:

a) acids and bases added to control pH and minimize microbial degradation; and

b) dechlorination reagents, such as ascrobic acid or sodium thiosulfate, added to reduce the effect of residual chlorine or other oxidizers found in some samples.

6.5.4 Efforts to preserve the integrity of the samples are initiated at the time of sampling or immediately upon sample receipt at the laboratory. Sample preservation is typically accomplished in one of two ways:

a) send or take bottles to preserve at the sample site; or

b) add preservatives to sample containers prior to going to the field.

6.6 SAMPLE HOLDING

6.6.1 The holding time before analysis is of critical, practical and regulatory importance. Analytes will degrade and be lost from the sample over time, even when correctly preserved and stored. All analytes have required holding times, from immediate analysis for the determination of sample pH up to 6 months for heavy metals.

Note: Holding times were originally conceived with the idea of providing guidelines for performing analyses within a sensible time frame to minimize sample degradation. Now holding times have taken on a legal life of their own, often times with no connection to scientific reality. For instance the misconception of how a sample with a holding time of 7 days prior to extraction, is acceptable if the extraction is begun 6 days, 23 hours, 59 minutes and 59 seconds after the moment of sampling, but magically turns to garbage one second later if not in a separatory funnel!

6.6.2 The time that a preserved sample may be held between sampling and analysis is based on the specific analytical method and analytes of interest. Holding time limitations described in all standardized methods are intended to minimize chemical change in a sample before it is analyzed.

6.6.3 The holding time clock starts with the moment of sampling and ends with the beginning of the analytical procedure. In other words, holding times do not start from receipt of the sample at the laboratory.

6.6.4 Holding times, outlined in tables 6-1 through 6-8, are the maximum times allowable between sample collection, extraction and analysis. Allowable holding times apply to both solid and aqueous samples.

6.6.5 Samples analyzed after holding times have been exceeded are considered out-of-

control and analytical results are unacceptable to report unless requested or specified by the Client.

6.6.6 To expedite analysis and minimize the possibility of exceeding holding times, overnight courier service, such as Federal Express, UPS, etc., or other reliable methods of transportation are used.

6.6.7 Maintaining samples in cold storage is terminated only after all analysis has been finished and the minimum holding time requirements have been met.

6.7 FIELD SAMPLING

The sampler is ultimately responsible for collecting representative samples from the site to accurately reflect project site conditions. The client must specify the method of analysis, and the procedure to collect the sample that will represent the matrix of interest. The sampler should remove all items that are not integral components of the matrix of interest.

The client should develop a sampling plan with sample site locations, chosen to be representative of the area being investigated. These plans should be followed during the sampling excursions.

Compositing multiple samples into a single sample can be used as part of the initial sampling strategy to identify plumes of contamination and as a screening technique. Individual samples are subsequently collected and analyzed to describe the sampling points within that area of investigation.

6.7.1 Surface Water Sample Collection

6.7.1.1 Surface water samples from springs or other surface waters may be taken under many different site specific conditions. At the time of sampling, the client should designate the appropriate sampling techniques for the site-specific setting.

6.7.1.2 Before sampling, all equipment is rinsed downstream or away from the sampling point, taking care not to disturb sediments at the sampling point. After sampling each location, the equipment is rinsed with distilled water and decontaminated before further use.

6.7.1.3 All samples are placed in containers that have been pre-cleaned or have been cleaned according to established protocols. Organic samples are typically collected in glass containers with Teflon-lined lids and samples for inorganic chemical analyses are typically collected in separate glass or polyethylene containers.

6.7.1.4 Sample filtration is determined by the client prior to sampling.

Samples are then collected according to the QAPP or the sampling techniques described in the FSP and documented accordingly.

6.7.2 Ground Water Sample Collection

6.7.2.1 Groundwater sampling should occur only after wells have been completely developed. Well development disturbs natural groundwater systems and should remain undisturbed for several days to allow the groundwater system to return to chemical equilibrium.

6.7.2.2 All equipment used to measure and sample the groundwater system (e.g., bailer, pumps, tapes, ropes, etc.) should be cleaned before use to prevent cross-well contamination. When sediments adhere to sampling equipment, scrubbing is required in addition to the normal rinsing.

6.7.2.3 Samples are placed in containers that have been pre-cleaned or have been cleaned according to established protocols. Organic samples are typically collected in glass containers with Teflon-lined lids and samples for inorganic chemical analyses are typically collected in separate glass or polyethylene containers.

6.7.3 Soil Sampling

6.7.3.1 Sampling points are typically marked with a stake and labeled with the appropriate site identification information. Prior to sampling, surface vegetation, rocks, pebbles, leaves, twigs, and other debris should be cleared from the sample point to allow for the collection of a representative soil sample.

6.7.3.2 Background samples should be taken at distances outside the investigation area, but within a location that is geologically similar to the actual sampling site. These samples give the client additional information concerning concentration levels above the background (i.e. baseline concentration). The number and location of background surface samples should be specified in the QAPP.

6.7.3.3 Soil samples should be collected in containers cleaned according to established protocols of the appropriate size. Samples are then labeled and placed in a temperature controlled ice chest immediately after sampling and delivered to the laboratory as soon as possible.

6.7.3.4 After sampling each location, all equipment should be thoroughly cleaned to prevent cross contamination of samples. Equipment should be scrubbed and rinsed with distilled water.

SDWA TABLE OF SAMPLE PRESERVATION AND HOLDING TIME REQUIREMENTS
ORGANIC ANALYSIS
TABLE 6-1

METHOD	PARAMETERS	PRESERVATION	CONTAINER	HOLDING TIME
504.1*	EDB/DBCP	$Na_2S_2O_3$ 3mg/40ml	40ml, G, Cool, 4 °C	Extract and analyze within 14 days
505	Organohalide Pesticides and PCB's	$Na_2S_2O_3$ 3mg/40ml	40ml, G, Cool, 4 °C	Extract within 7 days / Analyze immediately after Extraction
507	Nitrogen / Phosphorus Pesticides	$Na_2S_2O_3$ 80mg/L	1L, G, Cool, 4 °C	Extract within 14 days / Analyze within 14 days of Extraction
508	Chlorinated Pesticides	$Na_2S_2O_3$ 80mg/L	1L, G, Cool, 4 °C	Extract within 7 days / Analyze within 14 days of Extraction
515.1	Acid Herbicides	$Na_2S_2O_3$ 80mg/L	1L, G, Cool, 4 °C	Extract within 14 days / Analyze within 28 days of Extraction
531.1	Carbamates	Monochloroacetic Acid Buffer pH-3 1.2ml/40ml	40ml, G, Cool, 4 °C	Analyze within 28 days
547	Glyphosate	$Na_2S_2O_3$ 4mg/40ml	40ml, G, Cool, 4 °C	Analyze within 14 days / 18 months if frozen
548.1	Endothall	$Na_2S_2O_3$ 80mg/L	500ml, G, Cool, 4 °C	Extract within 7 days / Analyze within 14 days of Extraction
549.2	Diquat	$Na_2S_2O_3$ 50mg/0.5L	500ml, P, Amber, Cool, 4 °C	Extract within 7 days / Analyze within 21 days of Extraction
525.2	SVOCs	Na_2SO_3 50mg/1L**	1L, G, Cool, 4 °C	Extract within 14 days / Analyze within 30 days of Collection
524.2*	VOCs	pH<2, 1:1 HCl + Ascorbic Acid 25mg/40ml	40ml, G, Cool, 4 °C	Analyze within 14 days
551.1*	Disinfectant Byproducts	Na_2HPO_4 2g + KH_2PO_4 198g + NH_4Cl 1.2g Buffer Salts	40ml, G, Cool, 4 °C	Extract within 14 days / Analyze within 14 days of Extraction / Store Extracts at -10 °C
551.1*	Chloral Hydrate only	Na_2HPO_4 2g + KH_2PO_4 198g + Na_2SO_3 1.2g Buffer Salts	40ml, G, Cool, 4 °C	Extract within 14 days / Analyze within 14 days of Extraction / Store Extracts at -10 °C
552.2	Haloacetic Acids	NH_4Cl 100mg/L	40ml, G, Cool, 4 °C	Extract within 14 days / Analyze within 7 days of Extraction when stored at 4 °C / Analyze within 14 days of Extraction when stored at -10 °C

Note (*) - Zero head space (no air bubbles) is required for these methods.
Note (**) - Sample pH is field adjusted <2 HCL if acid compounds like PCP are to be determined.
$Na_2S_2O_3$ - Sodium Thiosulfate is used for chlorinated source water only.
Na_2SO_3 - Sodium Thiosulfite is used for chlorinated source water only.
G - Glass P - Plastic

SDWA SAMPLE PRESERVATION AND HOLDING TIME TABLE
METHOD 524.2
TABLE 6-2

DESCRIPTION	SAMPLE VOLUME	DECHLORINATION	SAMPLE PRESERVATION	ANALYSIS HOLDING TIME
Full List Compounds	3 x 40mL	25mg ascorbic acid per 40mL sample	pH<2, 2 drops 1:1 HCL Field preserved, cool 4°C	14 days
Full List Compounds sample foams when HCL is added carbonaceous waters	3 x 40mL	25mg ascorbic acid per 40mL sample	No acid	Analyze within 24 hours
THM's only	3 x 40mL	25mg ascorbic acid per 40mL sample	pH<2, 2 drops 1:1 HCL Field preserved, cool 4°C	14 days
THM's only	3 x 40mL	3 mg sodium thiosulfate per 40 ml sample	No acid	14 days
THM's only sample foams when HCL is added carbonaceous waters	3 x 40mL	3 mg sodium thiosulfate per 40mL sample	No acid	14 days

SDWA TABLE OF SAMPLE PRESERVATION AND HOLDING TIME REQUIREMENTS
INORGANIC ANALYSIS
TABLE 6-3

METHOD	PARAMETER	PRESERVATION	CONTAINER	HOLDING TIME
EPA 120.1/SM2510B/9050A	Conductivity	Cool, ≤6°C, no head-space	0.1 - 0.2-L, G/P	Analyze within 28 days
EPA 150.1/SM4500H/9040C	pH	Cool, ≤6 , no head-space	0.1 - 0.2-L, G/P	15 minutes, field analyze if possible
SM2540C	TDS	Cool, ≤6 ^{O}C	1-L, G/P	Analyze within 7 days
EPA 180.1/SM2130B	Turbidity	Cool, ≤6°C	0.1-L, G/P	Analyze within 48 hours
EPA 300/9056	Anions	None ,if analyzed with 48 hrs, pH <2 H2SO4	0.1-L, G/P	Analyze within 28 days if preserved, 48 hours non-preserved
SM2320B	Alkalinity	Cool, ≤6^{O}C	0.2 - 1-L, G/P	Analyze within 14 days
EPA 314.0	Perchlorate	None	0.1-L, G/P	Analyze within 28 days
SM4500Cl G	Chlorine	Cool, ≤6 ^{O}C, no headspace, protect from light	0.1-L, Amber gls	15 minutes, field analyze if possible
SM4500 NH3 D	Ammonia	Cool, ≤6^{O}C, pH<2 H_2SO_4,	0.1 - 1-L, G/P	Analyze within 28 days if preserved, 24 hours non-preserved
SM3500Cr D	Cr^{+6}	Cool, ≤6 ^{O}C, filter 0.45 um, pH 9.3-9.7 NaOH	0.1-L, G/P	Analyze within 24 hours if preserved, 28 days non-preserved
200.8	Metals, ICP-MS	pH<2 HNO_3 Sample may be preserved in the laboratory. Preserve 24 hours prior to digestion.	0.2-L, G/P	Analyze within 6 months
SM2340B	Hardness (Calc)	Same as for 200.8	0.2-L, G/P	Analyze within 6 months
SM3500Fe D	Ferrous Iron	Cool, ≤6^{O}C, Field filter, then acidify pH <2 HCL,	0.1-L, G/P	72 hours to color develop and 72 hrs after color development
G-Glass, P-Plastic				

CWA TABLE OF SAMPLE PRESERVATION AND HOLDING TIME REQUIREMENTS
ORGANIC ANALYSIS
TABLE 6-4

METHOD	PARAMETERS	PRESERVATION	CONTAINER	HOLDING TIME
601*	Purgeable Hydrocarbons	$Na_2S_2O_3$ 10mg/40ml	40ml, G, Cool, 4 °C	Analyze within 14 days
602*	Purgeable Aromatics	pH<2, 1:1 HCl, $Na_2S_2O_3$ 10mg/40ml	40ml, G, Cool, 4 °C	Analyze within 14 days
603*	Acrolein / Acrylonitrile	$Na_2S_2O_3$ 10mg/40ml, pH 4-5 (HCL/NaOH)	40ml, G, Cool, 4 °C	Analyze within 14 days
604	Phenols	$Na_2S_2O_3$ 80mg/L	1L, G, Cool, 4 °C	Extract within 7 days / Analyze within 40 days of Extraction
605	Benzidine	$Na_2S_2O_3$ 80mg/L, pH 2-7 H_2SO_4	1L, G, Cool, 4 °C	Extract within 7 days / Analyze within 7 days of Extraction
606	Phthalate Esters	No Preservation	1L, G, Cool, 4 °C	Extract within 7 days / Analyze within 40 days of Extraction
607	Nitrosamines	$Na_2S_2O_3$ 80mg/L, pH 7-10 (H_2SO_4/NaOH)	1L, G, Cool, 4 °C	Extract within 7 days / Analyze within 40 days of Extraction
608	Organochlorine Pesticides/PCB's	$Na_2S_2O_3$ 80mg/L, pH 5-9 (H_2SO_4/NaOH)	1L, G, Cool, 4 °C	Extract within 7 days / Analyze within 40 days of Extraction
609	Isophrone	No Preservation	1L, G, Cool, 4 °C	Extract within 7 days / Analyze within 40 days of Extraction
610	Polynuclear Aromatic Hydrocarbons	Na_2SO_3 80mg/1L	1L, G, Cool, 4 °C	Extract within 7 days / Analyze within 40 days of Extraction
611	Haloethers	Na_2SO_3 80mg/1L	1L, G, Cool, 4 °C	Extract within 7 days / Analyze within 40 days of Extraction
612	Chlorinated Hydrocarbons	No Preservatives	1L, G, Cool, 4 °C	Extract within 7 days / Analyze within 40 days of Extraction
613	Dioxin	Na_2SO_3 80mg/1L	1L, G, Cool, 4 °C	Extract within 7 days / Analyze within 40 days of Extraction
614	Organophosphorus Pesticides	pH 6-8 (H_2SO_4/NaOH)	1L, G, Cool, 4 °C	Extract within 7 days / Analyze within 40 days of Extraction
615	Chlorinated Herbicides	No Preservatives	1L, G, Cool, 4 °C	Extract within 7 days / Analyze within 40 days of Extraction
619	Triazine Pesticides	Not Specified	1L, G, Cool, 4 °C	Extract within 7 days / Analyze within 40 days of Extraction
622	Nitrogen / Phosphorus Pesticides	pH 6-8 (H_2SO_4/NaOH)	1L, G, Cool, 4 °C	Extract within 7 days / Analyze within 40 days of Extraction
624*	Purgeables	pH<2, 1:1 HCl, $Na_2S_2O_3$ 10mg/40ml	40ml, G, Cool, 4 °C	Analyze within 14 days
625	Base Neutral Acids	$Na_2S_2O_3$ 80mg/L	1L, G, Cool, 4 °C	Extract within 7 days / Analyze within 40 days of Extraction
632	Carbamate Pesticides	$Na_2S_2O_3$ 80mg/L	1L, G, Cool, 4 °C	Extract within 7 days / Analyze within 28 days of Extraction

Note (*) - Zero head space (no air bubbles) is required for these methods $Na_2S_2O_3$ - Sodium Thiosulfate G - Glass P - Plastic

CWA/RCRA TABLE OF SAMPLE PRESERVATION AND HOLDING TIME REQUIREMENTS
INORGANIC ANALYSIS
TABLE 6-5

METHOD	PARAMETER	PRESERVATION	CONTAINER	HOLDING TIME
EPA 120.1/SM2510B/9050A	Conductivity	Cool, ≤6°C	0.1 - 0.2-L, G/P	Analyze within 28 days
EPA 150.1/SM4500H/9040C	pH	Cool, ≤6 , no head-space	0.1 - 0.2-L, G/P	15 minutes, field analyze if possible
SM2540B	TS	Cool, ≤6°C	1-L, G/P	Analyze within 7 days
SM2540C	TDS	Cool, ≤6 °C	1-L, G/P	Analyze within 7 days
SM2540D	TSS	Cool, ≤6 °C	1-L, G/P	Analyze within 7 days
EPA 180.1/SM2130B	Turbidity	Cool, ≤6°C	0.1-L, G/P	Analyze within 48 hours
EPA 300/9056	Anions	None ,if analyzed with 48 hrs, pH <2 H2SO4	0.1-L, G/P	Analyze within 28 days if preserved, 48 hours non-preserved
SM2310B	Acidity	Cool, ≤6 °C	0.2 - 1-L, G/P	Analyze within 14 days
SM2320B	Alkalinity	Cool, ≤6°C	0.2 - 1-L, G/P	Analyze within 14 days
EPA 314.0	Perchlorate	None (Nevada requires samples to be field filtered 0.2 um)	0.1-L, G/P	Analyze within 28 days
SM4500Cl G	Chlorine	Cool, ≤6 °C, no headspace, protect from light	0.1-L, Amber gls	15 minutes, field analyze if possible
SM/4500NH3/SM4500 NH3 D	Ammonia	Cool, ≤6°C, pH<2 H_2SO_4,	0.1 - 1-L, G/P	Analyze within 28 days if preserved, 24 hours non-preserved
SM4500Norg/SM4500NH3 D	Total Kjeldahl -N	Cool, ≤6 °C, pH<2 H_2SO_4,	0.1 - 1-L, G/P	Analyze within 28 days if preserved, 24 hours non-preserved
EPA 365.3/SM4500P E	Total Phosphorus	Cool, ≤6 °C, pH<2 H_2SO_4,	0.2-L, G/P	Analyze within 28 days
SM4500S D	Sulfide	Cool, ≤6 °C, 0.2 mL 2N zinc acetate, 0.2 ml 6 N NaoH, pH >9 per 0.1-L	0.2-L, Clear glass	Analyze within 7 days
SM4500S D	Sulfide, dissolved	Cool, ≤6 °C, 0.2 ml 6 N NaoH, pH >9 per 0.1-L, no head-space	0.2-L, Clear glass	Analyze within 7 days
SM4500SO3 B	Sulfite	None	0.5-L, G/P	15 minutes, field analyze if possible
EPA 410.4/SM5520D	COD	Cool, ≤6 °C, pH<2 H_2SO_4,	0.1-L, G/P	Analyze within 28 days
SM 5210B	BOD	Cool, ≤6 °C	1-L, Plastic	Analyze within 48 hours
SM5540C	MBAS/Surfactants	Cool, ≤6 °C	2-L, Clear Glass	Analyze within 48 hours
SM5310C/9060	TOC	Cool, ≤6°C, pH<2 H_2SO_4, protect from sunlight	0.1-L, G/P	Analyze within 28 days
EPA 245.1/7470	Mercury	Cool, ≤6 °C, pH<2 HNO_3	0.2-L, G/P	Analyze within 28 days
200.8/6020	Metals, ICP-MS	pH<2 HNO_3 (Field filter prior to pH adjustment for dissolved metals)	0.2-L, G/P	Analyze within 6 months
SM3500Cr D/7196A	Cr^{+6}	Cool, ≤6 °C, filter 0.45 um, pH 9.3-9.7 NaOH	0.1-L, G/P	Analyze within 24 hours if preserved, 28 days non-preserved
SM3500Fe D	Ferrous Iron	Cool, ≤6°C, Field filter, then acidify pH <2 HCL,	0.1-L, G/P	72 hours to color develop and 72 hrs after color development
1664A/9070A	Oil and Grease	Cool, ≤6°C, pH<2 HCL or H2SO4,	1-L Glass only	Analyze within 28 days

RCRA TABLE OF AQUEOUS SAMPLE PRESERVATION AND HOLDING TIME REQUIREMENTS
ORGANIC ANALYSIS
TABLE 6-6

METHOD	PARAMETERS	PRESERVATION	CONTAINER	HOLDING TIME
8010*	Halogenated Volatiles	$Na_2S_2O_3$ .008%	40ml, G, Cool, 4 °C	Analyze within 14 days
8011*	EDB/DBCP	$Na_2S_2O_3$ 3mg/40ml	40ml, G, Cool, 4 °C	Analyze within 14 days
8021B*	Aromatic Volatiles	pH<2, 1:1 HCL, $Na_2S_2O_3$ .008%	40ml, G, Cool, 4 °C	Analyze within 14 days
8041	Phenols	$Na_2S_2O_3$ .008%	1L, G, Cool, 4 °C	Extract within 7 days / Analyze within 40 days of Extraction
8081A	Organochlorine Pesticides	pH 5-9 (H_2SO_4/NaOH) $Na_2S_2O_3$ .008%	1L, G, Cool, 4 °C	Extract within 7 days / Analyze within 40 days of Extraction
8082	Polychlorinated Biphenyls (PCBs)	pH 5-9 (H_2SO_4/NaOH) $Na_2S_2O_3$ .008%	1L, G, Cool, 4 °C	Extract within 7 days / Analyze within 40 days of Extraction
8141A	Organophosphorus Pesticides	pH 5-9 (H_2SO_4/NaOH)	1L, G, Cool, 4 °C	Extract within 7 days / Analyze within 40 days of Extraction
8151A	Chlorinated Herbicides	$Na_2S_2O_3$ .008%	1L, G, Cool, 4 °C	Extract within 7 days / Analyze within 40 days of Extraction
8260*	Volatile Organics	pH<2, 1:1 HCl, $Na_2S_2O_3$ .008%	40ml, G, Cool, 4 °C	Analyze within 14 days
8270	BNAs	$Na_2S_2O_3$ .008%	1L, G, Cool, 4 °C	Extract within 7 days / Analyze within 40 days of Extraction
8310	Polynumclear Aromatics	$Na_2S_2O_3$ .008%	1L, G, Cool, 4 °C	Extract within 7 days / Analyze within 40 days of Extraction
8318	N-methyl carbamates	Monochloroacetic Acid Buffer pH 4-5; 1.2mL per 40mL	40ml, G, Cool, 4 °C	Extract within 7 days / Analyze within 40 days of Extraction
8015B/Purgeable*	TPH/GRO	pH<2, 1:1 HCl	40ml, G, Cool, 4 °C	Analyze within 14 days
8015B/Extractable	TPH/DRO	pH<2, 1:1 HCl	40ml, G, Cool, 4 °C	Extract within 7 days / Analyze within 40 days of Extraction
8015B	Nonhalogenated VOC's	pH<2, 1:1 HCl, $Na_2S_2O_3$ .008%	40mL, G, Cool, 4 °C	Analyze within 14 days

Note(*) - Zero head space (no air bubbles) is required for these methods
$Na_2S_2O_3$ - Sodium Thiosulfate
G - Glass P - Plastic

RCRA TABLE OF SOIL / WASTE SAMPLE PRESERVATION AND HOLDING TIME REQUIREMENTS
ORGANIC ANALYSIS
TABLE 6-7

METHOD	PARAMETERS	PRESERVATION	CONTAINER	HOLDING TIME
8081A	Organochlorine Pesticides and PCBs	Soil - Cool to 4 ^{o}C Waste - None	4 to 8 oz., G, Cool, 4 ^{o}C	Extract within 14 days / Analyze within 40 days of Extraction
8082	PCB's (Aroclor)	Soil - Cool to 4 ^{o}C Waste - None	4 to 8 oz., G, Cool, 4 ^{o}C	Extract within 14 days / Analyze within 40 days of Extraction
8141A	Organophosphorus Pesticides	Soil - Cool to 4 ^{o}C Waste - None	4 to 8 oz., G, Cool, 4 ^{o}C	Extract within 14 days / Analyze within 40 days of Extraction
8151A	Chlorinated Herbicides	Soil - Cool to 4 ^{o}C Waste - None	4 to 8 oz., G, Cool, 4 ^{o}C	Extract within 14 days / Analyze within 40 days of Extraction
8260*	Volatile Organics	Soil - Cool to 4 ^{o}C Waste - None	4 to 8 oz., G, Cool, 4 ^{o}C	Analyze within 14 days
8270	BNAs	Soil - Cool to 4 ^{o}C Waste - None	4 to 8 oz., G, Cool, 4 ^{o}C	Extract within 14 days / Analyze within 40 days of Extraction
6020	Metals	Soil - Cool to 4 ^{o}C Waste - None	4 to 8 oz., G, Cool, 4 ^{o}C	Digest and analyze within 6 months.

Note (*) - Zero Head-space is required for these methods
G - Glass P - Plastic

TABLE OF TCLP/SPLP PRESERVATION AND HOLDING TIME REQUIREMENTS
ORGANIC AND INORGANIC ANALYSIS
TABLE 6-8

Method	Parameters	Preservation	Container	Holding Time
1311/1312	VOCs	No Preservation	2L/300g, G, Cool, 4° C	See Table Below
1311/1312	SVOCs	No Preservation	2L/300g, G, Cool, 4° C	See Table Below
1311/1312	Metals	No Preservation	2L/300g, G/P, Cool, 4° C	See Table Below
G - glass P - Plastic				

Method	Parameters	From Field Collection to TCLP/SPLP Extraction	From TCLP/SPLP Extraction to Preparative Extraction	From Preparative Extraction to Determinative Analysis	Total Elapsed Time
1311/1312	VOCs	14 days	NA	14 days	28 days
1311/1312	SVOCs	14 days	7 days	40 days	61 days
1311/1312	Metals (except Hg)	180 days	NA	180 days	360 days
1311/1312	Mercury	28 days	NA	28 days	56 days

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Section 7

Sample Custody

7.0 SAMPLE CUSTODY

7.1 SAMPLE CUSTODY PROCEDURES

7.1.1 Traditionally, record keeping is the primary emphasis of a QA program and this is the case in sample custody. Without a rigid and formal record keeping system, the QC procedures would not be documented appropriately.

7.1.2 All samples, from receipt through analysis, are handled under our Sample Tracking Plan. This portion of the QA program helps ensure the maintenance of sample integrity. It also helps to ensure all test procedures are performed in a timely and efficient manner.

7.1.3 The Sample Tracking Plan (STP, Appendix C) is a set of SOPs written with the maintenance of custody as a primary objective interwoven through all of the SOPs. Alpha is responsible for sample tracking and the maintenance of custody once it arrives and continues through sample analysis.

7.1.4 The Sample Custody Officer (SCO) is responsible for sample custody and for the overall implementation of the STP. This responsibility includes assuring proper handling and that the documentation of all samples are performed according to the described SOPs.

7.2 SAMPLE DOCUMENTATION PROCEDURES

7.2.1 Sample documentation, identification and chain-of-custody procedures are designed to assure accountability and control of all samples. Analytical records are kept and maintained in sufficient detail to track and recreate all analytical activities to ensure sample integrity. Project and client communication is also an extremely important aspect of the sample documentation procedures. SOP topics associated with the sample custody and documentation procedures are as follows:

- Sample Identification Procedure,
- Labeling Field Samples,
- Sample Receiving and Project Communication,
- Sample Containers, Preservatives, Holding Times and General Sample Receipt Protocols,
- Sample Acceptance Policy,
- Manual Chain-of-Custody Procedure,
- LIMS Generated Chain-of-Custody Procedure,
- Internal Chain-of-Custody Procedure,
- Sample Log-In Ledger,
- Sample Storage Procedure,
- Sample Custody, and
- Sample Tracking Procedure.

7.2.2 Chain-of-Custody (COC)

Samples are documented on a chain-of-custody form and signed by both the client and laboratory. This document formalizes the sample transaction and is critical to the maintenance of sample custody. The chain-of-custody is generally regarded as a legal document and should be completely filled out as legibly and as error free as possible. Information required on this document is described in the following SOPs found in Appendix C:

- Manual Chain-of-Custody Procedures,
- LIMS Generated Chain-of-Custody Procedures, and
- Internal Chain-of-Custody Procedure.

7.2.3 Sample Log-in Ledger

Upon arrival at the laboratory, samples are logged into the LIM system with the pertinent information by the Sample Custodian. A long term ledger is maintained as a permanent record of samples logged into our LIM system. Approximately every 2 weeks the LIM system is queried to print all samples logged-in during that period. A computer program or macro is activated to parse-out the relevant information from each chain-of-custody produced by the laboratory. This information is printed and stored in a 3-ring binder labeled "Log-In". Sample information contained in the "Log-In" book includes:

- Initials,
- Date of sample receipt,
- Laboratory's sample identification,
- Client's sample identification,
- Matrix type,
- Analysis requested,
- Turn-around-time (TAT),
- Date sampled, and
- Work order comments.

This document is used primarily by the sample custodian as a quick source to check historical information on the chain-of-custody records.

7.2.4 Sample Scheduling

The Laboratory Supervisors coordinate sample scheduling to maintain an even production flow while ensuring samples are extracted and analyzed within their prescribed holding times.

Scheduling is coordinated with the appropriate personnel to maximize production

according to the numbers and types of analyses to be performed during the analytical or extraction batch. Scheduling requires a working knowledge of instruments, personnel and lab activities. This balance is often shifted by the presence of "rush" analysis. A rush analysis takes precedence in the scheduling of samples. All other normal analyses are pushed back by these requests, and are scheduled according to their sampling date.

Large sample projects are coordinated with the Laboratory Manager or Laboratory Director before samples arrival. This preplanning eases potential workload difficulties while ensuring the appropriate level of QA/QC is maintained. All discrepancy reporting is handled by the Laboratory Director to ensure the problem is expressed to the client and is properly documented.

7.3 LABORATORY LOGBOOK POLICY

Bound logbooks are the preferred method of record keeping. However, certain laboratory functions are formalized enough to use standard forms (e.g., Sample Preparation Log). These activities are documented using a spiral bound or loose leaf three-ring binders to record the relevant information. In this case, pages are dated in chronological order which helps reference data.

Each analyst, instrument or specific laboratory function has its own logbook to track and document lab activities, dates and times more effectively. When more than one analyst shares a common logbook, they delineate their data insertions by initialing and dating data entries.

All logbook entries are made in ink. Corrections are made by drawing one line through the incorrect entry, and then entering the correct information, initialing and dating this change. Complete information is entered so that during an examination it can be decided what was done, by whom, when and what the results were. All logbook entries are signed by the analyst or technician recording those entries.

7.3.1 Instrument Sequence Logbook

Associated with each instrument is a sequence logbook in which all tuning, calibration and analytical activities conducted on that instrument are recorded. Analytical schedules are the preferred method of tracking analytical instrument activities. This logbook is instrument-specific, not person-specific.

At the end of each day or upon completion of an analytical batch, each analyst must sign and date the first page that contains data entries for that day.

Appropriate information contained in the standard preparation logbook may be annotated in the instrument logbook. This has the advantage of correlating standards, QC checks, Lot numbers, etcetera, to the appropriate analytical batch without additional searching of records.

7.3.2 Analytical Data Record Keeping System

The need for a single, yet efficient, procedure for analytical data record keeping is paramount in reconstructing historical analytical records. Therefore, the analytical data record keeping system is designed for this procedure.

For each analytical instrument there is an associated record keeping system. Every analytical run made by an instrument is partially or completely documented by this system.

7.4 PHYSICAL SECURITY AND DOCUMENT CONFIDENTIALITY

Data may be compromised in many ways other than QA/QC measures normally associated with the validation of generated data. An important aspect of data integrity is answering the question and eliminating the possibility that data may have or could have been compromised by the lack of or inappropriate security measures. Physical security, security measures, document confidentiality and employee policies regarding ethics, waste, fraud and abuse are addressed and continuously monitored in order to generate data of the highest quality that will stand a legal challenge. Physical security, security measures and document confidentiality are generally of the following type:

- Building security,
- Perimeter - door security,
- Visitor security,
- Document confidentiality, and,
- Sample security.

7.4.1 Building Security

Alpha Analytical, Inc. has installed a Z1100 security system through ADT Security Systems. The Z1100 is a digital communicator system monitored 24 hours a day by a central station. When the monitoring station receives an alarm from the laboratory, it immediately contacts the correct response agency (ambulance, police or fire).

The Z1100 is an arming system programmed with a pre-alarm and automatic bell cutoff. Alpha has door contacts at all entrances. Passive and infrared motion detectors are strategically located throughout the laboratory to detect intruders in sensitive areas. Alpha has smoke and heat detectors in all areas where flammable chemicals or heat-generating equipment is located. Alpha also has glass breakage detectors on all high travel perimeter glass windows, where intruder entrance through a window could take place.

Alpha continuously updates the security codes to prevent unwanted entry by former employees or others who may have had access to security codes.

7.4.2 Perimeter Door Security

The normal layout of Alpha's laboratory includes a series of perimeter access doors which would not intentionally be designed in the physical plant of a laboratory such as ours. However, since our laboratory is situated in an office building complex, there are a large number of perimeter access doors which necessitate additional perimeter security. The types of perimeter access doors have been identified and are as follows:

- Perimeter doors which will remain open at all times during our normal laboratory hours (e.g., the two main entrance doors);
- Perimeter doors not in a direct or associated travel pattern of our employees, and which otherwise have no need to be opened for normal business practice; and,
- Perimeter doors in a direct line with the travel path of most employees, and need to be secured in a way which allows unfettered access to employees while also being a deterrent from outside intrusions.

The majority of perimeter doors are one-way locking doors such that an employee may exit a perimeter door without a key. However, to gain access to the building would require a security code.

7.4.3 Visitor Security

Unwanted physical intrusions may occur at any time unless a number of security measures are implemented. Once the premise has been secured, attention is now addressed to non-employees who may try to obtain access to our facility.

7.4.3.1 Visitors or non-employees are given a visitor badge upon entrance to our facility.

Note: Clients logging in samples or visitors constrained to the main reception area are not required to sign the visitor logbook.

7.4.3.2 Visitors are required to sign-in upon arrival at the front desk, and answer the following questions:

- Date/Time of arrival,
- Company or institute they represent,
- Personal signature,
- Badge or ID number,
- Person whom they are visiting, and
- Date/Time of departure.

7.4.3.3 Visitors are required to place this badge on their garment in a position to be highly visible at all times.

7.4.3.4 Visitors are required to be escorted to their area of interest and accompanied while on premises.

7.4.3.5 Visitors without a badge will be escorted immediately to the front area and asked to remain there until their party arrives.

7.4.3.6 Visitors without badges will be questioned to obtain the seriousness and extent of this breach of security and the possibility of data invalidation.

7.4.3.7 Visitors are required to return the badge and sign out at the front desk upon leaving our facility.

Visitor Log-In

Page:_____

Badge ID	Printed Name	Signed Name	Company you represent	Person Visiting	Date/Time Arrival	Date/Time Departure

7.4.4 Sample Security

7.4.4.1 Sample security is the responsibility of the person who has custody of the sample at any particular time. The overall responsibility resides with the SCO to ensure sample custody practices and procedures are being followed.

7.4.4.2 Sample storage refrigerators are not locked for most routine samples; however, occasionally a higher level of sample security and custody is required by a sensitive project. Alpha Analytical is prepared and has the resources to use locked and secured storage facilities for this purpose.

7.4.4.3 Samples that require separately locked storage facilities will be the responsibility of the SCO. Staff members associated with that particular project will be assigned access and sample custody will remain with the designated project-specific personnel during laboratory activities.

7.4.5 Document Confidentiality

7.4.5.1 All samples and project documents are considered to be confidential. Standard Business Records Confidentiality practices apply to all documents, materials and relevant information.

7.4.5.2 Specific procedures that are followed to maintain legal confidentiality include the following:

7.4.5.2.1 All documents and files are secured in locked file cabinets or equally secured areas, i.e., secured building, during other than normal working hours, unless the files are personally attended by someone authorized to have access to such files;

7.4.5.2.2 Employees other than management, DCO or SCO are not allowed immediate or direct access to confidential files or documents without approval of the Laboratory Director; and,

7.4.5.2.3 All sample documents and any verbal information will only be released to the client who requested sample analysis.

Note: Persons or organizations, other than the client, requesting such information may only receive the information upon approval to release the data.

If there are any doubts concerning the identity of the organization or authority, then they must show proof of identification before Alpha will release information.

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Section 8

Analytical Procedures

8.0 ANALYTICAL PROCEDURES

8.1 INTRODUCTION

8.1.1 Standardized analytical methods used by Alpha are generally published methods from recognized federal agencies (i.e., SW-846, Standard Methods, EPA's Methods for the Determination of Organic Compounds in Drinking Water etc.).

8.1.2 Most standardized methods require an initial demonstration of capability study which generates precision and accuracy data establishing a baseline typical of routine analysis. The initial demonstration of capability studies are used primarily to preclude a laboratory from analyzing unknown samples via a new, unfamiliar method prior to obtaining some experience with it.

8.1.3 It is Alpha's policy to demonstrates the ability to perform that method of analysis with the prescribed degree of precision and accuracy before using an analytical method to analyze environmental samples and, where appropriate, to estimate the measurement uncertainty as well as statistical techniques for the analysis of environmental test data.

8.1.4 It is Alpha's policy to have instructions on the use and operation of all relevant equipment, and on the handling and preparation of samples where the absence of such instructions could jeopardize the results of the environmental tests. It is a general policy to keep and maintain all instructions, standards, manuals and reference data relevant to the work on the laboratory up-to-date and is readily available to all laboratory personnel. Deviations from environmental test methods are acceptable for use only if the deviation has been documented, technically justified, authorized and accepted.

8.2 ANALYTICAL METHODS

Standardized analytical methods are described by a set of written procedures defining the techniques used to process a sample and obtain analytical results. Descriptions of analytes, sample matrix, sample preparation, types and quantities of reagents, instrumental calibration and measurement parameters, and computations are all integral parts of a complete method.

8.2.1 Selection of Methods

Analytical environmental testing methods, including those procedures for sampling, handling, transportation, storage and sample preparation used to meet the needs of our clients and methods which are typically cited in regulation as the appropriate methods for the specific regulatory programs are selected by our laboratory for certification and which we carry on our scope of approved methods.

8.2.2 Sources of Standardized Methods

8.2.2.1 Alpha uses standardized methods for commonly encountered analytes

to provide a common point of reference, and to establish standard practices that allow inter-laboratory comparison of data. Alpha uses methods that are program specific and are typically referenced in the regulatory literature.

8.2.2.2 In addition to specifying sample preparation and analytical procedures, most methods specify calibration procedures, acceptance criteria, methods of preparing standards, and QC samples.

8.2.2.3 The latest valid edition of these referenced methods are used unless it is not appropriate or impossible to do.

8.2.2.4 Requested methods of analysis are used unless it is not appropriate, or if we are not certified for the requested method and an equivalent certified method exists, then that method will be used.

8.2.2.5 When the client does not specify a method of analysis, Alpha only uses methods that have been fully documented and validated and the client informed of the chosen method.

8.2.2.6 When a client proposes a method that is considered to be inappropriate or out of date, the client is informed as to the problem and instructed as to the most appropriate method.

8.2.3 Procedure Manual

Analytical methods used in our laboratory have an associated technical in-house analytical SOP and in total comprise our Procedure Manual. These SOPs are written, reviewed, approved, and distributed according to the procedures outlined in Appendix D. When the referenced method is ambiguous or does not provide sufficient information, our in-house analytical SOP clarifies those issues detailed in Clarification Boxes.

8.2.4 Laboratory Developed Methods

The introduction of a laboratory developed or non-standardized test method typically requires an enormous amount of planning and is assigned to a member of the technical staff who is equipped with the adequate resources to carry out the duties of method development and final evaluation. As method development proceeds, the modifications, changes, experiments, etc. are communicated to all involved personnel, in order to keep them abreast of those changes.

8.2.5 Non-Standard Methods

In the event that analyses must be conducted for compounds for which no reliable

method exists, or when it is necessary to use methods not covered by standardized method procedures, the method goals are discussed and agreed upon with the client in order to fulfill the client's requirements. Both laboratory developed and non-standardized methods should be appropriately validated before use.

As part of the method development process and to ensure continuous quality of data, QC criteria are proposed and established that is consistent with similar methods or technology. At a minimum, the method development process must address these QC requirements:

- Calibration;
- Interference/Contamination;
- Analyte identification;
- Selectivity;
- Sensitivity;
- Precision; and
- Accuracy.

When testing of the analytical procedure has been successfully completed, the method is evaluated for scientific and technical soundness and is documented in the standardized format.

8.3 SUMMARY OF ANALYTICAL PROCEDURES

The analytical and extraction procedures presented in the following sections are methods currently used at Alpha Analytical in support of the various environmental regulatory programs. A brief description of the methods are in the subsections following the tables.

8.4 ANALYTICAL METHODS IN SUPPORT OF THE SAFE DRINKING WATER ACT (SDWA)

A series of inorganic methods of analysis are found in Methods for Chemical Analysis of Water and Wastes. These are a series of wet chemistry and various metals methods used in support of the Safe Drinking Water Act (SDWA). Standard Methods for the Examination of Water and Wastewater is also used in support of the SDWA; however, this reference contains inorganic wet chemistry, metals and organic methods of analysis. An additional series of methods written by the EPA covering all methods required under the SDWA is found in Methods for the Determination of Organic and Inorganic Compounds in Drinking Water and associated supplements. Methods of analysis Alpha Analytical uses in support of the SDWA is as follows:

SDWA Methods of Analysis
Table 8-1

EPA METHODS	STANDARD METHODS	PARAMETERS
Inorganic		
120.1	SM2510B	Conductivity
150.1	SM4500H B	pH
	SM2540C	TDS
180.1	SM2130B	Turbidity
200.8		Metals
300.0		Anions
	SM2320B	Alkalinity
314.0		Perchlorate
	SM4500Cl G	Free Residual Chlorine
	SM3500Cr D	Chromium VI
	SM2340B	Hardness (calculated)
Organic		
524.2		Volatile Organic Compounds

8.4.1 Conductivity - EPA Method 120.1/Standard Method 2510B

Conductivity is the ability of a solution to pass a current. The amount of current a solution may conduct is proportional to the number of ions present in the sample. Therefore, conductivity is a measure of the total ionic concentration in a sample. Specific conductance of a sample is determined by the use of a self-contained conductivity meter at 25°C, thus standardizing the measurement by compensating for cell geometry and temperature.

8.4.2 pH - EPA Method 150.1/Standard Method 4500H B

The pH of a sample is determined electrometrically using a combination electrode. The pH meter is calibrated using a series of standard pH buffers at a known pH.

8.4.3 Total Dissolved Solids (TDS) - Standard Method 2540C

A sample is filtered through a standard glass-fiber filter, and the filtrate is evaporated to dryness in a pre-weighed crucible and dried to a constant weight in an oven at a final fixed temperature of 180°C. The increase in weight over that of the empty crucible represents the total dissolved solids.

8.4.4 Turbidity - EPA Method 180.1/Standard Method 2130B

Turbidity measurement is based upon a comparison of the intensity of light scattered by the sample under defined conditions with the intensity of light scattered by a standard reference suspension.

8.4.5 Metals - EPA Method 200.8

This procedure is a multi-elemental procedure for the determination of analytes by ICP-MS in environmental samples. Elements in solution are introduced by pneumatic nebulization and the resulting aerosol is transported by argon gas into a radio frequency plasma where the energy transfer process causes desolvation of the elements followed by atomization and ionization. The ions produced by high temperatures are entrained in the plasma gas and introduced, by means of a vacuum interface, into a mass spectrometer. The ions produced are sorted according to their mass-to-charge ratios by a quadrapole mass spectrometer and detected with the assistance of a electron multiplier. Isobaric elemental interferences and interferences from polyatomic ions derived from the plasma gas, reagents and sample matrix are corrected by the data acquisition software.

8.4.5.1 For the determination of total recoverable metals, analytes are solubilized by gentle refluxing with nitric and hydrochloric acids i.e., block digestion. After cooling, the sample is brought back to its original volume, mixed and centrifuged or allowed to settle overnight prior to filtration and sample analysis.

8.4.5.2 For the determination of dissolved metals in a filtered sample, or the direct analysis of analytes in drinking water samples where the sample turbidity is <1 NTU, the sample is made ready for analysis by the addition of nitric acid prior to sample analysis.

8.4.6 Anions - EPA Method 300.0

A small volume of sample is introduced into an ion chromatograph to flush and fill a fixed volume sample loop. The sample is then injected into a mobile phase eluent of carbonate-bicarbonate. The anions are separated and measured using an Ion Chromatograph (IC) comprised of a guard column, an analytical column, a suppressor device and the conductivity detector. The suppressor device reduces the amount of background conductivity of the carbonate-bicarbonate eluent to a negligible level. Anions are identified based on their retention times compared to known standards.

8.4.7 Alkalinity - Standard Method 2320B

An unaltered sample is titrated to an electrometrically determined end point of pH 4.5

for total alkalinity and to a second endpoint of 8.3 if the speciation of alkalinity is required. The sample is not filtered, diluted, concentrated, or altered in any way.

For samples of low alkalinity (less than 20 mg $CaCO_3$/L) an extrapolation technique is used to determine the equivalence point. The amount of standard acid required to reduce the pH exactly 0.30 pH units beyond the normal end point of 4.5 corresponds to an exact doubling of the hydrogen ion concentration.

8.4.7.1 Chemical Reactions

Sulfuric acid (hydrochloric acid may also be used) reacts with three forms of alkalinity converting them to water or carbonic acid. If hydroxide is present, it reacts to form water:

$$2OH^- + H_2SO_4 \rightleftarrows 2H_2O + SO_4^{2-}$$

This conversion usually is complete at a pH of approximately 10. Phenolphthalein alkalinity is determined by titration to an end point of pH 8.3, which corresponds to the conversion of carbonate to bicarbonate.

$$2CO_3^{2-} + H_2SO_4 \rightleftarrows 2HCO_3^- + SO_4^{2-}$$

If hydroxide is present, titration to pH 8.3 will indicate the alkalinity due to all of the hydroxide plus one-half of the carbonate. Continued titration to pH 4.5 completes the conversion of carbonate plus any bicarbonate present to carbonic acid. This value is termed total alkalinity.

$$2HCO_3^- + H_2SO_4 \rightleftarrows 2H_2CO_3 + SO_4^{2-}$$

8.4.8 Perchlorate - EPA Method 314.0

A volume of sample is introduced into an ion chromatograph to flush and fill a fixed volume sample loop. The sample is then injected into a mobile phase eluent of 70 mM KOH. The perchlorate anion is separated and measured using an IC comprised of a guard column, an analytical column, a suppresser device and the conductivity detector. The suppressor device reduces the amount of background conductivity of the KOH eluent to yield a baseline with no more than 4-5 nanosiemen (nS) noise/drift per minute. Perchlorate is identified based on its retention time compared to known standards. Quantitation is accomplished by measuring peak area and comparing it to a calibration curve generated from known standards.

8.4.9 Free Residual Chlorine - Standard Method 4500Cl G

Free residual chlorine also known as free available chlorine exists in most waters as hypochlorous acid (HOCl) or hypochlorite ion (OCl^-). These analytes react immediately with DPD (N,N-diethyl-p-phenylenediamine) indicator to form a pink color. The intensity of the pink color is proportional to the chlorine concentration. Chlorine is measured at a wavelength of 530nm.

8.4.10 Chromium VI - Standard Method 3500Cr D

Hexavalent chromium is determined colorimetrically by a reaction with diphenylcarbazide in an acid solution. A purple color will appear if hexavalent chromium is present in the absence of interfering analytes. The concentration of hexavalent chromium is determined by its absorbance measured photometrically at a wavelength of 540 nm.

8.4.11 Hardness (Calculated) - Standard Method 2340B

Total hardness is defined as the sum of calcium and magnesium concentrations, both expressed as calcium carbonate, in milligrams per liter. Although hardness can be determined in a number of ways, the preferred procedure is to compute hardness from the results of separate determinations of calcium and magnesium as a ratio to calcium carbonate. Therefore, the calculation of "Hardness (calc), mg equivalent $CaCO_3$ = 2.497 [Ca, mg/L] + 4.118 [Mg, mg/L].

8.4.12 Volatile Organic Compounds - EPA Method 524.2

Helium is sparged through a 25ml water sample contained in a purge-and-trap chamber at ambient temperature. The purgeable organics are transferred from the aqueous phase to the vapor phase. The vapor is swept through a sorbent trap where the purgeables are absorbed. After purging is completed, the trap is heated to thermally desorb the purgeables onto a gas chromatographic column. The GC is temperature programmed to separate the purgeables which are then detected with a mass spectrometer. Analytes are quantitated using an internal standard procedural calibration process.

8.5 ANALYTICAL PROCEDURES IN SUPPORT OF THE CLEAN WATER ACT (CWA)

The Clean Water Act includes a promulgated series of methods enacted to satisfy the analytical requirements of a facility holding an NPDES discharge permit. The 600 series organic methods of analysis are found in 40 CFR, Appendix A, Part 136, and Methods for Organic Analysis of Municipal and Industrial Waste Water. These are a series of GC, GC/MS and HPLC methods for the determination of compounds that may be found in municipal and industrial discharges.

The inorganic methods of analysis are found in Methods for Chemical Analysis of Water and Wastes. These are a series of wet chemistry and various metals methods used in support of the CWA. These are the same procedures as used in support of the SDWA. Standard Methods for the Examination of Water and Wastewater is also used in support of the CWA. Methods of analysis Alpha Analytical uses in support of the CWA is as follows:

CWA Methods of Analysis
Table 8-2

EPA METHODS	STANDARD METHODS	PARAMETERS
Inorganic		
120.1	SM2510B	Conductivity
150.1	SM4500H B	pH
	SM2540C	TDS
	SM2540D	TSS
	SM2540B	TS
180.1	SM2130B	Turbidity
200.8		Metals
300.0		Anions
	SM2310B	Acidity
	SM2320B	Alkalinity
314.0		Perchlorate
	SM4500Cl G	Total and Free Residual Chlorine
	SM4500NH_3 D (NH_3 B-distillation)	Ammonia
	SM4500NH_3 D (Norg C-digestion)	Total Kjeldahl-N
365.3	SM4500P E (B5 - digestion)	Total Phosphorus
	SM4500S D	Sulfide
	SM5210B	BOD5
410.4		COD
	SM5310C	TOC
	SM2340B	Hardness (calculated)
	SM3500Cr D	Chromium VI
	SM3500Fe D	Ferrous Iron
1664A		n-Hexane Extractable Material (Oil and Grease)
Organic		

608		Organochlorine Pesticides and PCBs
624		Purgeables
625		Semivolatile Base/Neutal and Acids

8.5.1 Total Suspended Solids (TSS) - Standard Method 2540D

A well-mixed sample is filtered through a pre-weighed glass-fiber filter and the residue retained on the filter is dried to a constant weight at a final fixed temperature of 103-105°C. The increase in weight of the filter represents the total suspended solids.

8.5.2 Total Solids (TS) - Standard Method 2540B

A well-mixed sample is evaporated in a pre-weighed crucible and dried to a constant weight in an oven at a final fixed temperature of 103 to 105°C. The increase in weight over that of the empty crucible represents the total solids.

8.5.3 Acidity - Standard Method 2310B

The sample pH is determined and a measured amount of standard acid is added to lower the pH to 4 or less. If the initial sample pH is less than 4.0, the addition of acid is not required. This is an arbitrary inflection point, because accurate identification of inflection points may be difficult or impossible in buffered or complex mixtures.

The sample is oxidized with hydrogen peroxide because samples of industrial wastes, acid mine drainage, or other solutions that contain appreciable amounts of hydrolyzable metal ions such as iron, aluminum or manganese may exist in other reduced forms of the polyvalent cations. The sample is subsequently boiled to hasten hydrolysis.

The sample is cooled and titrated electrometrically with standard alkali to a pH of 8.3. The titration to an end point of 8.3 corresponds to the stoichiometric neutralization of carbonic acid to bicarbonate and is reported as total acidity (pH 8.3). This end point is generally accepted as the standard of total acidity, including CO_2 and most weak acids. However, for more complex mixtures or buffered solutions such as waste waters or grossly polluted waters, use two end points, 3.7 and 8.3 for standard acidity determinations where simple carbonate equilibria cannot be assumed. In this case acidity is reported as "methyl orange acidity" (pH 3.7) and total acidity (pH 8.3).

8.5.4 Total Residual Chlorine - Standard Method SM4500-Cl G

8.5.4.1 Free Residual Chlorine (A fraction)

Free residual chlorine also known as free available chlorine exists in most waters as hypochlorous acid (HOCl) or hypochlorite ion (OCl^-). These

analytes react immediately with DPD (N,N-diethyl-p-phenylenediamine) indicator to form a pink color. The intensity of the pink color is proportional to the chlorine concentration. Chlorine is measured at a wavelength of 530nm (A reading).

8.5.4.2 Combined Chlorine (B-A fraction) + (C-A fraction)

Chloroanimes analyzed and reported as combined chlorine may be estimated with an additional sample preparation procedure.

Monochloramine (B-A fraction)

The monochloramine (ClH_2N) fraction (B-A) is estimated by the addition of 0.1mg of KI per 10 mL sample volume using the same sample as used in the free residual analysis followed by immediate sample analysis (B reading).

Dichloramine (C-A fraction)

The dichloramine (Cl_2HN) fraction may further be estimated by the addition of 0.2g KI per 10 mL sample volume using this same sample followed by immediate analysis.

A simplified approach for determining monochloramine and dichloramine together as combined chlorine would be to add 0.2g KI per 10 mL sample volume and not fractionate the combined chlorine subfractions (total residual chlorine). To determine the concentration of the combined chlorine, run a free residual chlorine test. Subtract the results of the free chlorine test from the total chlorine test to obtain the combined chlorine concentration.

8.5.4.3 Total Chlorine (A fraction) + (B-A fraction) + (C-A fraction)

Total chlorine may be determined as the addition of free residual and combined chlorine. This is accomplished in a single spectophotometric reading by adding the full amount of KI, 0.2g per 10 mL sample volume, at the start of the analysis along with the DPD indicator.

Total chlorine analysis is actually a determination of the iodine present in a sample. Iodine is produced in a stoichiometric relationship with combined chlorine from the addition of KI. The combined chlorine oxidizes iodide in the reagent to iodine. The iodine and free chlorine react with DPD to form a pink color which is proportional to the total chlorine concentration.

8.5.5 Ammonia - Standard Method SM4500NH_3 D

8.5.5.1 Distillation - Standard Method SM4500NH_3 B

Samples are buffered to a pH of 9.5 with a borate buffer solution to decrease hydrolysis of cyanates and organic nitrogen compounds. Samples are then distilled into a weak sulfuric acid solution to trap the ammonia and analyzed by an ammonia-selective electrode.

8.5.5.2 Analysis

The ammonia concentration of a sample is determined potentiometrically using an ion selective gas-sensing combination ammonia electrode. Dissolved ammonia ($NH_{3(aq)}$ and NH_{4+}) are converted to $NH_{3(aq)}$ by raising the pH to above 11 with a strong base. The gas sensing electrode responds to dissolved ammonia gas in solution. The dissolved ammonia gas diffuses across a hydrophobic gas-permeable membrane into a small volume of (ammonium chloride) buffer solution, specific to the ammonia electrode. Reaction of the gas with the buffer causes a pH change sensed by an internal pH electrode. The fixed level of chloride in the internal fill solution is sensed by a chloride ion-selective electrode that serves as the reference electrode. Because the reference electrode is built-in, a separate reference electrode is not necessary.

This same procedure using a gas-sensing electrode can also be used to measure ammonium ions after conversion to ammonia, or organic nitrogen after Kjeldahl digestion of the sample.

8.5.6 Total Kjeldahl Nitrogen (TKN)- Standard Method SM4500NH3 D

8.5.6.1 Digestion - Standard Method $SM4500NH3_{org}$ C

Prior to the distillation or analysis of ammonia as described below, the sample is heated in the presence of concentrated sulfuric acid, potassium sulfate and copper sulfate until the solution becomes colorless or pale blue-green. The ammonia is subsequently distilled from the sample and determined potentiometrically.

8.5.6.2 Analysis

See ammonia analysis above.

8.5.7 Total Phosphorus - EPA Method 365.3/Standard Method SM4500P E

The determination of phosphorus can generally be summed up into two procedural steps: a) conversion of the various forms of phosphorous to ortho-phosphate and b) colorimetric determination of ortho-phosphate.

8.5.7.1 Digestion - Standard Method SM4500B5

Total phosphorus procedure converts organic and inorganic phosphorous to the ortho-phosphate form by a persulfate digestion.

Acid-hydrolyzable phosphorous or polyphosphates forms of phosphorus are converted to the orthophosphate form by sulfuric acid hydrolysis. This form of phosphorus will contain free orthophosphate plus a small amount of organically bound phosphorous. Therefore acid-hydrolyzable phosphorous is reported as the difference between the results obtained using the hydrolysis procedure and the results of ortho-phosphate analyzed directly without acid hydrolysis.

8.5.7.2 Analysis

These preparatory procedures are then followed by the analysis of orthophosphate. Orthophosphate reacts with molybdate in an acid medium to produce a phosphomolybdate complex. This complex is reduced to an intensely blue-colored complex by ascorbic acid. This colorimetric procedure is based on reactions that are specific for the orthophosphate ion. The color is proportional to the phosphorus concentration.

8.5.8 BOD_5

The BOD method is used to determine the oxygen requirements of a water source, in particular sewage treatment plants or polluted water bodies. The BOD test takes 5 days to complete and is performed using a dissolved oxygen probe. A sample is placed into a specially designed BOD bottle and the dissolved oxygen concentration is determined. This value is reported as the initial DO value. The sample is then incubated in the dark for 5-days at 20° C. At the end of the incubation period the DO concentration in the sample is again measured. The difference in the DO concentrations measured during the 5-day incubation period corrected for sample dilution represents the amount of oxygen required for the decomposition of any organic material in the sample and is reported as the BOD concentration.

8.5.9 Sulfide - Standard Method SM4500S D

The methylene blue method is based on the reaction of sulfide, ferric chloride, and N,N-dimethyl-p-phenylenediamine in an acidic solution to form the dye methylene blue. The excess color due to ferric chloride is removed by the addition of diammonium hydrogen phosphate.

8.5.9.1 Total Sulfide

Hydrogen sulfide and acid-soluble metal sulfides are collected and preserved with zinc acetate which forms the insoluble ZnS, and further basified with

sodium hydroxide. This preservation and extraction treatment limits the loss (volatilization) of potential sulfide prior to sample analysis. Interferences are removed from the sample (and the sample concentrated) by carefully withdrawing the supernatant liquid from the ZnS precipitate, and either replacing the removed water with deionized water or leaving at the lesser volume for sample concentration. Sulfide is then color developed by the reaction with N,N-dimethyl-p-phenylenediamine sulfate to form methylene blue. The intensity of the blue color is proportional to the sulfide concentration. Sulfide is measured at a wavelength of 665 nm.

8.5.9.2 Dissolved Sulfide

Dissolved sulfide may be determined after the suspended solids have been removed by flocculation and settling prior to color development and sample analysis.

8.5.10 Chemical Oxygen Demand (COD) - EPA Method 410.4

The mg/L COD results are defined as the mg of O_2 consumed per liter of sample under conditions outlined in this procedure. Samples are prepared by a closed-reflux digestion procedure with sulfuric acid along with an excess of potassium dichromate ($K_2Cr_2O_7$) followed by sample analysis. The COD reagents also contain silver and mercury ions. Silver sulfate ($AgSO_4$) serves as a catalyst to assist oxidation of straight-chain hydrocarbons such as diesel fuel and motor oil and mercury is used to control the chloride interferences. The sample is digested at a temperature of 150°C for two hours to drive the reaction to completion. During digestion the samples' carbon bearing compounds are oxidized by the acid and potassium dichromate, reducing the dichromate ion (Cr_2O_7) to green chromic ion (Cr_3+) while the organic matter and inorganic carbon compounds are oxidized to CO_2 and H_2O. Colorimetric analysis is suitable for COD because the two chromium ions absorb at different wavelengths in the visible range Cr_3+ at 600nm and Cr_6+ at 420 nm.

8.5.11 Total Organic Carbon (TOC) - Standard Method 5310C

Inorganic carbon in most water samples is the main fraction of carbon and is generally many times greater than the TOC fraction. TOC is the main focus of this procedure, therefore, the IC fraction is first eliminated by acidification of the sample to convert the inorganic carbon to CO_2. Subsequent purging of the sample strips the sample of CO_2; however, sample purging also removes POC so that the organic carbon measurement made after eliminating the IC fraction is actually a NPOC determination.

Note: In most surface and ground waters the POC fraction is negligible. Therefore, in practice, the NPOC determination is substituted for TOC.

Organic carbon in the sample is then converted to carbon dioxide (CO_2) by persulfate

oxidation and UV irradiation. The CO_2 formed is stripped from the sample with a stream of gas and measured directly by a non-dispersive infrared (NDIR) detector. The amount of CO_2 is directly proportional to the concentration of organic carbon material in the sample.

8.5.12 Ferrous and Ferric Iron - Standard Method SM3500Fe D

8.5.12.1 Ferrous Iron (Fe^{2+})

The 1,10-phenanthroline indicator in the Ferrous Iron Reagent pillow reacts with ferrous iron in the sample to form an orange color in proportion to the iron concentration. Ferric iron does not react.

8.5.12.2 Ferric Iron (Fe^{3+})

The ferric iron concentration can be determined by subtracting the ferrous iron concentration from the results of a total iron test.

8.5.13 Hexane Extractable Material (HEM) - EPA Method 1664A

This method is a performance based procedure applicable to aqueous matrices that requires the use of n-hexane as the extraction solvent and gravimetry as the determinative technique.

8.5.13.1 A 1-L sample is acidified to a pH <2 and extacted with n-hexane. The n–hexane is reduced to dryness, desiccated and weighed. The residual weight gain is calculated and reported as HEM.

8.5.13.2 The HEM may be further speciated into Silica Gel Treated (SGT-HEM) by re-dissolving the dried extract with n–hexane and silica gel. The silica gel adsorbs the polar fraction leaving the non-polar fraction. This fraction is reduced to dryness, desiccated and weighed. The residual weight gain is calculated and reported as SGT-HEM.

8.5.14 Pesticides and PCBs - EPA Method 608

This procedure is a GC method used to determine organochlorine pesticides and PCBs. A measured volume of water sample is extracted with solvent. The extract is separated by a GC equipped with an Electron Capture Detector for the identification of the target compounds.

8.5.15 Purgeables - EPA Method 624

Method 624 is a GC/MS method used in the determination of a number of volatile organics in industrial and municipal wastewater. Helium is bubbled through a 25 ml

water sample contained in a specially designed purging chamber at ambient temperature. The purgeables are efficiently transferred from the aqueous phase to the vapor phase. The vapor is swept through a sorbent trap where the purgeables are trapped. After purging is completed, the trap is heated and back-flushed to desorb the purgeables onto the GC. The gas chromatograph is temperature programmed to separate the purgeables which are then detected with a mass spectrometer.

8.5.16 Base/Neutrals and Acids - EPA Method 625

Method 625 is a GC/MS procedure used in the determination of a number of organic compounds that are partitioned into an organic solvent and are amenable to gas chromatography. A measured sample volume is extracted with methylene chloride at a pH greater than eleven and again at a pH less than two using a separatory funnel or by mechanical tumbling or shaking. The methylene chloride extract is dried, concentrated and analyzed by GC/MS.

8.6 ANALYTICAL PROCEDURES IN SUPPORT OF THE RESOURCE CONSERVATION AND RECLAMATION ACT (RCRA)

Several of the hazardous waste regulations under Subtitle C of RCRA require that specific test methods described in Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, Final Update III (SW-846) be employed for certain applications. Specific requirements are found in 40 CFR, part 140 through 290.

SW-846 provides the test procedures and guidelines for field and laboratory quality control, sampling, determining hazardous constituents in waste, determining the hazardous characteristics of waste (toxicity, ignitability, reactivity, and corrosivity) and for determining the physical properties of waste. Methods of analysis Alpha Analytical uses in support of RCRA is as follows:

RCRA Methods of Analysis
Table 8-3

EPA METHODS	OTHER METHODS	PARAMETERS
Inorganic		
SW6020/6020A		Metals
SW7196A		Chromium (VI)
SW9040C/9045D		Corrosivity
SW9050A		Conductivity
SW9056		Anions
SW9060A		TOC

Organic		
SW8015B/C/D-DRO	NWTPH-dx	Total Petroleum Hydrocarbons (Diesel Range)
SW8015B/C/D-GRO	NWTPH-gx	Total Petroleum Hydrocarbons (Gasoline Range)
SW8081A/B		Organochlorine Pesticides
SW8082/SW8082A		Polychlorinated Biphenyls (PCBs)
SW8260B		Volatile Organics
SW8270C		Semi-volatile Organics

8.6.1 Metals - EPA Method SW6020/6020A

An aliquot of a well mixed, aqueous or solid sample is weighed or measured for sample processing. For total metals analysis, analytes are solubilized by acid digestion. After cooling, the sample is made up to volume prior to analysis.

This procedure is a multi-elemental procedure for the determination of analytes by ICP-MS in environmental samples. This method measures ions produced by a radio frequency inductively coupled plasma. Analyte species originating in a liquid are nebulized and the resulting aerosol is transported by argon gas into the plasma torch. The ions produced by high temperatures are entrained in the plasma gas and introduced, by means of an interface, into a mass spectrometer. The ions produced in the plasma are sorted according to their mass-to-charge ratios and quantified.

8.6.2 Hexavalent Chrome - EPA Method 7196A

Hexavalent chromium is determined colorimetrically by a reaction with diphenylcarbazide in an acid solution. A purple color will appear if hexavalent chromium is present in the absence of interfering analytes. The concentration of hexavalent chromium is determined by its absorbance measured photometrically at a wavelength of 540 nm.
An alkaline digestion procedure using a 0.28M Na_2CO_3/0.5M NaOH solution and heating the digestate to 90-95°C for 60 minutes is required to extract hexavalent chromium in soils prior to color development and spectrophotometric analysis. The pH of the sample digest must be carefully adjusted during the digestion procedure and temperature monitored to ensure the complete dissolution of Cr(VI) and stabilize it against reduction to Cr(III).

8.6.3 Conductivity - EPA Method 9050A

Conductivity is the ability of a solution to pass a current. The amount of current a solution may conduct is proportional to the number of ions present in the sample. Therefore, conductivity is a measure of the total ionic concentration in a sample. Specific conductance of a sample is determined by the use of a self-contained

conductivity meter at 25°C, thus standardizing the measurement by compensating for cell geometry and temperature.

8.6.4 Anions - EPA Method SW9056

A small volume of sample is introduced into an ion chromatograph to flush and fill a fixed volume sample loop. The sample is then injected into a mobile phase eluent of carbonate-bicarbonate. The anions are separated and measured using an Ion Chromatograph (IC) comprised of a guard column, an analytical column, a suppressor device and the conductivity detector. The suppressor device reduces the amount of background conductivity of the carbonate-bicarbonate eluent to a negligible level. Anions are identified based on their retention times compared to known standards. An extraction procedure is performed on soil and/or solid samples prior to sample analysis.

8.6.5 Total Organic Carbon (TOC) - EPA Method SW9060A

Inorganic Carbon (IC), carbonate and bicarbonate is removed by acidification and purging. Sample purging also removes Purgeable Organic Carbon (POC) so that the organic carbon measurement made after eliminating IC interferences is actually a Non Purgeable Organic Carbon (NPOC) determination. Therefore, in practice, the NPOC determination is substituted for TOC. Organic carbon in the sample is then converted to carbon dioxide (CO_2) by persulfate oxidation. The CO_2 formed is measured directly by a non-dispersive infrared detector. The amount of CO_2 is directly proportional to the concentration of carbonaceous material in the sample.

8.6.6 Total Petroleum Hydrocarbons (TPH) - EPA Method 8015B/C/D-DRO

This method is applicable to the analysis of semi-volatile petroleum hydrocarbons, commonly referred to as Diesel Range Organics (DRO). DROs typically correspond to the range of petroleum compounds from C_{13} to C_{22}; however, this range may be changed as required. Diesel fuel is used as the default standard for quantitation of petroleum hydrocarbons identified in this C range. Samples are solvent extracted and analyzed by GC/FID.

8.6.7 Total Petroleum Hydrocarbons (TPH) - EPA Method 8015B/C/D-GRO

This method is applicable to the analysis of volatile petroleum hydrocarbons, commonly referred to as Gasoline Range Organics (GRO). The SW846 Method 8015B/D-GRO specifies a C range of C_6 to C_{10} using 2-methylpentane and 1,2,4-trimethylbenzene as the C range markers. Our policy is to define GROs as those hydrocarbons which correspond to the range of alkanes from C_4 to C_{13}; however, this may be changed as required. Gasoline is used as the default standard for quantitation and includes compounds from C_4 to C_{13}. Samples are analyzed by the GC/MS purge-and-trap procedure.

8.6.8 Organochlorine Pesticides - EPA Method SW8081A/B

Method 8081A is used to determine the concentration of various organochlorine pesticides in extracts from solid and liquid matrices. A measured volume or weight of sample is extracted using the appropriate sample extraction technique. After the sample has been extracted and dried it is exchanged into hexane for final concentration. The extract is injected into a GC equipped with an Electron Capture Detector for separation and quantitation. All compounds identified tentatively in the primary analysis are confirmed on a dissimilar GC column.

8.6.9 Polychlorinated Biphenyls (PCBs) - EPA Method SW8082/SW8082A

Method 8082 is used to determine the concentration of the several common PCBs as Aroclors or as individual PCB congeners in extracts for solid and liquid matrices. A measured volume or weight of sample is extracted using the appropriate sample extraction technique. After the sample has been extracted and dried, it is exchanged into hexane for final concentration. The extract is injected into a GC equipped with an Electron Capture Detector for separation and quantitation. All compounds identified tentatively in the primary analysis are confirmed on a dissimilar GC column.

8.6.10 Volatile Organics - EPA Method SW8260B

Volatile (or purgeable) organics in water and soil samples are analyzed using method SW8260B. This method uses a gas chromatography mass spectrometry technique. Volatile compounds are introduced into the GC by purge and trap (SW5030C). Helium gas is bubbled through the sample to transfer the purgeable organic compounds from the liquid to vapor phase. Soil samples are extracted with methanol before purging or are directly sparged with a special purge and trap device. The vapor is then swept through a sorbent trap where the purgeable organics are trapped. The trap is back- flushed and heated to desorb the purgeable organics onto a capillary GC column where they are separated and then detected with a mass spectrometer. The Internal Standard (IS) procedure is used for the quantitation of analytes of interest. For quantitation, RFs are calculated from the base ion peak of a specific IS that is added to each calibration standard, blank, QC sample, and sample.

8.6.11 Semi-volatiles - EPA Method SW8270C

Semi-volatile organics (also known as base/neutral and acid extractables) in water and soil samples are analyzed using method SW8270C. This technique quantitatively determines the concentration of a number of SVOCs. Samples are extracted and both base/neutral and acid extracts are then combined. Compounds of interest are separated and quantified using a capillary column GC/MS. The IS procedure is used for quantitation of target analytes. For quantitation, RFs are calculated from the base ion peak of a specific IS that is added to each calibration standard, blank, QC sample, and sample.

8.7 SAMPLE EXTRACTION

8.7.1 Water Sample Preparation

The need to filter water samples depends on whether total or dissolved contaminants are of interest. The client will determine this prior to sample collection for each specific site/project.

Samples which are analyzed only for dissolved analytes such as metals must be filtered prior to chemical preservation. Analysis for volatile organic compounds and oil/grease are the only two universal exceptions to this guideline; they are never filtered. Samples may be filtered in the laboratory prior to extraction if requested by the client. The filter material used by Alpha is chosen on the basis of compatibility factors between the filter paper and the analytes of interest. Compatibility is defined in the following way:

- The sample being filtered is not changed by the filter material; and,
- The filter paper does not absorb or leach out the chemical analytes of interest.

Generally, particulate matter is not considered to be a natural component of groundwater, and normally will be filtered through a 0.45 micron filter prior to analysis, especially if the particulate matter is suspected of interfering with sample workup except for VOCs and Oil and Grease analyses. Filtration of drinking water or tap water will occur if specified by the method of analysis (e.g. all HPLC methods require filtration prior to analysis).

Organic samples for RCRA analysis are prepared using the EPA 3500 series methods when appropriate. In particular, water samples are prepared according to Method 3510C, separatory funnel, Method 3511, micro-extraction, Method 3520C continuous liquid-liquid extractions, or Method SW3535 Solid Phase Extraction (SPE). These preparation methods are used specifically with the 8000 series methods of analysis.

Inorganic RCRA samples are generally prepared by one of two procedures, EPA method 3010, block digestion or method 3015, microwave digestion. These two acid digestion procedures are used with analytical method 6020 as well as various other analytical metals procedures.

Methods of analysis in support of the SDWA and CWA have their extraction or digestion procedures written into the analytical procedure. Even though they do not have an extraction method assigned to them; they are essentially the same procedures as outlined in the equivalent RCRA procedures. Method specific extraction procedures can be found in the various analytical SOPs.

8.7.2 Soil/Sediment Sample Preparation

Soil and sediment samples are complex mixtures, even within a single sample site. Therefore, surrogate and analyte recovery depends on many factors, including organic content, mineral content, particle size and moisture content of the soil. Soil and sediment samples are analyzed in the condition they are received. Soil samples are generally prepared for extraction or digestion as follows:

The sample is mixed as thoroughly as possible in the original wide-mouth glass bottle by shaking or stirring. Glass rods are used for stirring. If samples have analyses for both volatile organic compounds and other analyses, the VOC sample preparation activity takes precedence before any other sample work-up and subsequent sample homogenization.

Generally, samples are quantitated on a "wet-weight" basis. When necessary, samples are quantitated on a "dry-weight" basis and the percent dry weight content of the sample is determined.

For each soil sample, an aliquot of the sample is dried according to the procedure established in Standard Operating Procedure for Percent Dry Weight and Percent Moisture. Soils are weighed and dried at 105°C for no less than four hours. The calculated % dry weight for each sample is determined and used in final analytical determinations.

The determination of % dry weight is calculated as follows:

$$\% \text{ dry weight} = \frac{\text{Sample Weight (Dry)}}{\text{Sample Weight (Wet)}} \times 100$$

Composite samples are proportioned according to the number of samples and % dry weight content is determined.

Organic sample extractions for soil are prepared according to EPA Methods 3540C, soxhlet extraction; Method 3545, Pressurized Fluid Extraction (PFE), Method 3550A, sonication or Method 3570, micro-extraction. Exceptions to these techniques are method specific as outlined in the SOPs found in Appendix E.

Inorganic soil sample digestion procedures for the analysis of metals are generally prepared according to EPA method 3051, micro-wave digestion.

8.7.3 Sample Batch

Samples are routinely analyzed by a batch system. Alpha uses two types of batch systems: 1) an extraction batch; and 2) an analytical batch. An extraction batch consists of a maximum of 20 samples, that can be extracted together. An analytical

batch is any number of samples that can be analyzed during an 8 or 12 hr GC/MS period which is associated with a MS tune.

GC methods usually require calibration verification standards analyzed at a specific sample frequency; however, this does not preclude the analyses of additional standards interspersed with samples. The rate of sample collection or shipment does not determine maximum batch size, although it may limit the number of samples available for analysis at a given time. A batch may consist of samples from more than one client. However, all samples in one batch must be completely processed through any given step in the same time period.

8.8 Extraction Test Procedures for Hazardous Waste

There are two primary extraction tests using buffered reagents to simulate particular environmental conditions in order to determine if a solid waste exhibits the characteristic of toxicity. These procedures are referred to as: 1) Toxicity Characteristic Leaching Procedure (TCLP); or 2) Synthetic Precipitation Leaching Procedure (SPLP). These procedures are used if the total concentration in the waste equals or exceeds the Maximum Concentration of Contaminants for the Toxicity Characteristic (TC) Limits.

8.8.1 Toxicity Characteristic Leaching Procedure (TCLP) - EPA Method SW1311

Method SW1311 is an extraction procedure, using a buffer system similar to acid rain used for the determination of the concentration of organic (volatile and semi-volatile) and inorganic analytes that are leachable from waste or other materials.

8.8.2 Synthetic Precipitation Leaching Procedure (SPLP) - EPA Method SW1312

Method SW1312 is designed to determine the mobility of both organic and inorganic analytes present in liquids, solids, and wastes. This procedure is exactly like the TCLP procedure with the exception of a different buffering medium.

8.9 GENERAL LABORATORY OPERATIONS

There are numerous activities required by our laboratory to be executed with minimal mistakes on a routine basis. Many of these activities are critical in the overall production of analytical methods and in some way influence the QA and QC of the laboratory. Many of these procedures have SOPs not only because they are routine activities, but also because they are regulated by law or by a regulatory agency. Activities other than analytical methods and extraction procedures which have written SOPs are as follows:

- Dish Washing and Steam Scrubber Operations, Appendix E,
- Manual Glassware Cleaning, Appendix E,

- Sample Container Cleaning Procedure, Appendix E,
- Prevention of Sample Contamination, Appendix E,
- Standards Preparation, Appendix E,
- Storage Blank Procedures, Appendix E,
- A Practical Application Guide for Performing a Demonstration of Capabilities (DOC) and Method Detection Limit (MDL) Study, Appendix E,
- Manual and Automated Integration Procedures, Appendix E,
- Waste Disposal, Appendix C,
- Preparation of Reagent Grade Water, Appendix E,
- Sample Compositing and Sub-sampling Procedure, Appendix E, and
- A Practical Application Guide to Performing Initial Calibration, Calibration Model Determination and Calibration Verification, Appendix E,

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Section 9

Calibration Procedures, Reference Standards, Reagents and the Procurement of Supplies and Materials

9.0 CALIBRATION PROCEDURES, REFERENCE STANDARDS, REAGENTS AND THE PROCUREMENT OF SUPPLIES AND MATERIALS

9.1 Instrument Calibration

The following section specifies the essential elements that define the procedures and documentation for initial instrument calibration and continuing instrument calibration verification to ensure that the data is of a known quality and is appropriate for a given regulation or decision.

This section does not specify the detailed procedural steps ("how to") for calibration, but establishes the essential elements for selection of the appropriate technique(s). This approach allows flexibility and permits the employment of a wide variety of analytical method prescribed procedures and statistical approaches currently applicable for calibration.

If more stringent standards or requirements are included in a mandated test method or by regulation, those procedures will take precedence to ensure those requirements are met. If it is not apparent which standard is more stringent, then the requirements of the regulation or mandated test method are followed.

9.1.1 Initial Instrument Calibration

The following items are defined as essential elements of initial instrument calibration:

a) The details of the initial instrument calibration procedures including calculations, integrations, acceptance criteria and associated statistics is included and/or referenced in the test method SOPs (Procedural Manual).

b) Raw data records are retained to permit the reconstruction of the initial calibration. Raw data records include such things as: calibration date, test method, instrument, analysis date, each analyte name, analyst's initials or signature, concentration and response, calibration curve or response factor, or the unique equation or coefficient used to reduce the instrument response to concentration.

c) Sample results are quantitated from the initial instrument calibration and may not be quanitated from any continuing instrument calibration verification, unless otherwise required by regulation, method or program.

d) Second Source Standard for Initial Calibration Verification (ICV)

All initial instrument calibrations are verified with a standard obtained from a second source manufacturer or a lot different from the source used for the initial calibration. If the same source is used for both it must be demonstrated, the lot was prepared form the manufacturer independently from other lots. If

available, commercially-purchased standards are traceable to a national standard and documented with the Certificate of Analysis (C of A).

The following guidelines are used when method guidance does not exist.

Note 1: The use of standards from a second lot is acceptable when only one manufacturer of the calibration exists.

Note 2: The requirement for a second source standard for the initial calibration is waived if a second source standard is used for the calibration verification.

Note 3: The date of preparation of each second source standard is considered when evaluating its suitability for use. This consideration includes an assessment of the stability of the standard solution, as well as its natural degradation rate.

Note 4: The second source standard is prepared at a concentration at or near the middle of the calibration range. Since most methods do not require the analysis of this standard and no method criteria exists, the criteria of acceptance is generally established using the calibration verification criteria.

e) The initial calibration criteria established and documented in the method SOPs are derived directly from the referenced method, if available. If the referenced method does not provide calibration criteria, then the in-house criteria detailed in the method SOP is established to be appropriate to the calibration technique employed.

f) The limit of quantitation (LOQ) and/or the reporting limit, can not be established lower then the lowest calibration standard used in the initial calibration.

Note: Data reported below the limit of quantitation, or the lowest initial calibration point is considered to have an increased uncertainty and is only reported using data flags or footnotes.

g) The highest calibration standard is the highest concentration for which quantitative data is reported.

Note: Data reported above the highest calibration point is considered to have an increased quantitative uncertainty and is only reported using data flags or footnotes.

h) Analyte concentrations reported outside the established working calibration range, are reported as having less certainty and are reported using data flags or footnotes. The lowest calibration standard is above the established limit of detection.

Noted NELAP exceptions if a method or instrument technology (such as ICP/MS) employs a single point calibration strategy, then the following are required:

1) Prior to the analysis of samples a zero point (i.e., calibration blank) and a single point calibration must be analyzed and the linear calibration range of the instrument must be established by analyzing a series of standards, one which is at the lowest quantitation limit. Sample results reported within the established linear calibration range do not require data flags.

2) Zero points, (calibration blanks) and single point calibration standards must be analyzed with each analytical batch.

3) A standard corresponding to the limit of quantitation must be analyzed with each analytical batch and must meet established acceptance criteria.

4) The linearity is verified at a frequency established by the method and/or the manufacturer.

Note: See the ICP/MS method SOP for details.

i) If the initial instrument calibration results are outside of the established acceptance criteria, corrective actions are performed and all associated samples reanalyzed. If reanalysis of the samples is not possible, data associated with an unacceptable initial calibration is reported with the appropriate data flags or footnotes.

j) If the referenced analytical method does not specify the number of initial calibration points, then the minimum number is two (one of which must be at the limit of quantitation), not including blanks, or zero standards, with the exception of methods which only require a single point calibration. The minimum number of calibration points are established and referenced in each of the analytical SOPs.

Note: It is Alpha's policy to establish the minimum number of contiguous calibration points as 3 for inorganic analysis and 5 for organic analysis. All reported single response target analytes are included in the initial calibration with the noted exceptions for some multi-component

analytes, such as PCBs, toxaphene and technical chlordane which may require a separate initial calibration.

9.1.2 Initial Calibration Verification

See section 9.1.1 d above for details

9.1.3 Calibration Verification

When an initial instrument calibration is not performed on the day of analysis, the validity of the initial calibration is verified prior to sample analyses by analyzing an acceptable continuing calibration verification standard at the method specified frequency. As long as the continuing calibration verification is acceptable, a new initial instrument calibration is not necessary.

Note: This is applicable only when method-specific guidance does not exist.

The following items are essential elements of continuing instrument calibration verification:

a) The details of the continuing instrument calibration procedure, calculations and associated statistics are included or referenced in the analytical method SOP.

b) Calibration is verified for each discrete (single response) analyte or element except for multi-component analytes such as Aroclors, Total Petroleum Hydrocarbons, toxaphe or technical chlordane where a single response standard mix is used.

c) Instrument calibration verification is performed as follows:

c1) Frequency

1) at the beginning and end of each analytical batch (except, if an internal standard is used, only one verification standards needs to performed at the beginning of the analytical batch);

2) whenever it is expected that the analytical system may be out of calibration or might not meet the verification acceptance criteria;

3) if the time period for calibration or the most previous calibration verification has expired; or

4) if the method specifies a calibration verification requirement.

Note 1: When the method specifies that the CVs be analyzed at a specific sample interval (for example, every 10 or 20 samples), the count of these samples generally includes field samples only.

Note 2: QC samples must be analyzed with their associated batches. The grouping of QC samples from a variety of batches is unacceptable.

Note 3: If the method does not specify an interval for periodic calibration verification standards, then every analytical batch, using an external calibration procedure, should bracket at least every 20 field samples.

c2) Concentration

1) Methods which employ an internal standard calibration procedure should analyze CVs at or just below the middle of the calibration range.

2) Methods which employ an external standard calibration procedure should alternate the concentration of CVs to cover both the low and high end of the initial calibration range.

c3) Standard Source

1) The source of the CV standard can be the same standard used for the initial calibration. As noted above, the requirement for a second source standard for the initial calibration verification is waived if a second source standard is used for the calibration verification.

d) Raw data records are retained to permit the reconstruction of the continuing instrument calibration verification, e.g., test method, instrument, analysis date, each analyte name, concentration and response, calibration curve or response factor, or unique equations, or coefficients used to convert instrument responses into concentrations. Continuing calibration verification records and supporting data are maintained in a manner to explicitly connect the continuing calibration to the initial instrument calibration.

e) The criteria for the acceptance of a continuing instrument calibration verification is established and detailed in the individual method SOPs.

e1) Corrective Action

1) If the continuing calibration verification results obtained are outside of the established acceptance criteria, corrective action is generally required.

2) If routine corrective action procedures fail to produce a second consecutive (immediate) calibration verification within acceptance criteria, then either, after corrective action, the next two consecutive calibration verification standards have to be acceptable or a new initial instrument calibration should be performed.

e2) Reporting non-perfect CV Data

If the calibration has not been verified, sample analyses cannot occur until the analytical system is calibrated or the calibration is verified. However, if samples are analyzed on an instrument that has failed the continuing instrument calibration, then the results are flagged for the failing analytes. Data associated with an unacceptable calibration verification may be fully useable under the following special conditions:

1) when the acceptance criteria for the continuing instrument calibration verification is exceeded high, (i.e., high bias), and there are associated samples that are non-detects, then those non-detects may be reported. Otherwise the samples affected by the unacceptable calibration verification should be reanalyzed after corrective action and an acceptable calibration verification or a new calibration curve has been established, evaluated and accepted.

2) when the acceptance criteria for the continuing instrument calibration verification is exceeded low, (i.e. low bias), those samples may be reported if they exceed a maximum regulatory limit/decision level. Otherwise the samples affected by the unacceptable calibration verification should be reanalyzed after corrective action and an acceptable calibration verification or a new calibration curve has been established, evaluated and accepted.

9.2 Standards, Reagents and Reference Materials

9.2.1 Policy

9.2.1.1 It is Alpha's policy to purchase calibration and/or verification and

validation standards, to include S class weights, and thermometers, that are traceable to national standards and measurements when possible. These are typically documented with a Certificate of Analysis.

9.2.1.2 It is Alpha's policy to participate in an inter-laboratory comparison program such as a proficiency testing program in an effort to provide additional evidence of correlation of sample results.

9.2.1.3 It is Alpha's policy to properly label and store all bottles, flasks, beakers or vials that contain samples, sample extracts or standard solutions.

9.2.2 Standards and Reference Material

9.2.2.1 During standard calibration and sample analysis, solutions containing known target compounds at known concentrations are prepared. These standards are used to calibrate instruments and quantitate analytical data. Alpha Analytical uses the following types of reference material:

a) commercial standards;

b) commercially prepared custom standards; and

c) custom-made standards prepared from reagent grade or neat chemical.

9.2.2.2 Commercial Standards

Commercial standards are the primary source of reference material used in the determination of analytical data. Commercial standards are compared with a secondary source (e.g., ICV standards) of commercial standards to verify standard concentrations and analyte purity. Individual and standard mix solutions procured from commercial vendors are purchased for specific methods of analysis.

9.2.2.3 Custom Standards

Commercial vendors are used to prepare custom standards, that are not easily prepared in the laboratory or are not typically prepared by the vendor on a normal basis. Typically custom standards are prepared with specific analytes and concentrations that are different than their catalogue products.

9.2.2.4 Neat Standards

Neat standards are purchased to prepare in-house custom-made

standards. This type of standard is an additional source of reference standard material used in the determination of analytical data. Reference materials used in the preparation of custom-made standards are typically purchased as ACS reagent grade which is typically certified at a purity of greater then 98 %.

9.2.3 Reference Material

See section 9.3 below for details.

9.2.4 Transport and Storage

9.2.4.1 Standards and Reference Material

The Standard Preparation SOP, found in Appendix E, describes our procedure for the safe handling, transportation, storage and use of reference material (i.e., instrument calibration standards) in order to prevent contamination or deterioration and in order to maintain and protect its integrity.

Semi-volatile reference materials are generally stored in a refrigerator at <6°C and volatile reference materials are generally stored in a freezer at <-10°C. Most inorganic reference material is either stored at <6°C or at room temperature.

9.2.4.2 Reagents

Laboratory reagents and chemicals must be stored according to method guidelines and the manufacturer's instructions. All solvents used for VOC analyses (i.e., methanol) must be isolated and stored separately from solvents which may be target analytes.

Reagents are stored and segregated according to compatibility groups (e.g. flammable solvents and non flammable solvents). Storage of all chemicals and solvents follow all OSHA requirements.

9.2.5 Documentation and Labeling of Standards, Reagents and Reference Material

9.2.5.1 Reference Material

A record of all standards, reagents reference materials and chemicals are maintained in the Standards Preparation Logbook. The documentation of this material is a critical component of our QA program; therefore, all purchased standards are the finest quality available.

In addition, to ensure the proper quantification of sample analytes and to help maintain data integrity records are retained for all standards, reagents, and reference material and documented as follows:

- the manufacturer/vendor,
- the manufacturer's Certificate of Analysis or purity,
- the lot number,
- the date of receipt,
- the recommended storage conditions, and
- the expiration date.

Note: Standards may not be used past their expiration date unless its reliability is verified.

If standards are prepared, the following additional information is required:

- the preparation date,
- the amounts and concentration of all source reagents and compounds used, and
- the signature and initials of preparer.

Upon receipt of the standards or reagents, the technician or chemist responsible for the preparation and traceability of that standard, dates and initials all certificates. The certificates are then placed in a 3-ring binder for historical reference.

9.2.5.2 Original containers (such as those provided by the manufacturer or vendor) are labeled with an expiration date.

9.2.5.3 The standard preparation logbook is designed to maintain records on standards, reagents and reference material preparation. These records document the traceability of purchased stock and neat standard materials by referencing the method of preparation to include the lot number, the expiration date and the preparer's initials.

9.2.5.4 All containers of prepared standards and reference material are labeled with a unique identification and expiration date that unequivocally links that container with its associated standard and reference material documentation.

9.2.5.5 The standard preparation logbook contains method requirements to ensure the standards meet the test method criteria. Method specified reagent preparation criteria are specified in the individual analytical SOPs.

9.2.5.6 Containers holding prepared standards and reagents are labeled with their identification, preparation data and expiration date.

Note: The standard or reagent preparation date is included in its identification and there is no need to duplicate this information.

9.3 Procurement of Supplies and Materials

9.3.1 Vendor Evaluation and Supply Policy

9.3.1.1 It is Alpha's policy to evaluate and select supply vendors, chemicals, reagents and any other supplies which are critical to method performance to include additional testing to verify their quality before use.

9.3.1.2 Supply vendors are typically required to provide information to substantiate their ability to provide suitable quality, e.g., Certificates of Analysis (C of A), and their mechanism to assure consistent delivery.

9.3.2 Service and Supplies Purchasing Policy

9.3.2.1 If a method specifies a particular brand name, it is Alpha's policy to purchase the item that meets or exceeds method specification, but not necessarily those brand name items specified in the method.

9.3.2.2 If no industry standard is available which specifies the appropriate quality grade, then it is Alpha's policy to determine what testing may be required to evaluate usability of supplies and materials.

9.3.2.3 It is Alpha's policy to purchase services and material supplies that affect the quality of environmental testing, at a level which will meet and/or exceed method/project criteria. These services and supplies are typically thought of as:

- Instruments,
- Solvents,
- Reagents,
- Gasses,
- Reference material,
- Sample containers, and
- Other laboratory supplies.

9.3.3 Sample Containers

9.3.3.1 All sample containers used by Alpha are pre-cleaned according to EPA established protocols and require no further cleaning prior to sample collection.

9.3.3.2 Alpha does not clean or reuse sample containers.

9.3.3.3 Alpha maintains a sequestered supply of sample containers to eliminate the possibility of contamination of the sample from the container and to track sample containers by lot numbers.

9.3.3.4 Containers are purchased and sequestered by lot in order to minimize the tracking associated with a group of sample containers.

9.3.3.5 VOC containers are checked by Alpha to insure that the container is not contaminated and is not adding low level analytes to the sample.

1) Clarification: DoD requires sample containers used for the collection of DoD samples to be checked for contamination. Sample containers are analyzed on a lot-by-lot basis for the method and target analytes used for their particular samples to one-half of the project specified reporting limit. This also includes other supplies and materials used for DoD samples.

9.3.4 Once chemical purity, grade or quality has been established, it is Alpha's policy to repurchase these same items and document their continuing quality with the associated certificate of analysis for historical records.

9.3.5 Materials and supplies which have an associated certificate of analysis will be inspected upon receipt. If the quality of the supplies or materials is suspect, certain actions will be required on the part of the analyst or extraction chemist.

i. The analyst/extraction chemist will first verify the lot number and C of A of the material in question and assure that it has met vendor/supplier specification.

ii. Secondly, the analyst/extraction chemist will document the quality issue and inform the Laboratory Director, QA Officer and Purchasing Agent of the problem and any additional correction taken on his/her part.

iii. Thirdly, the Laboratory Director or QA Officer, will take the necessary actions to rectify and document the problem and the solution to the problem.

9.3.6 Service and Supply Documentation Policy

9.3.6.1 It is Alpha's policy to maintain purchasing documents for items affecting data quality. These items are reviewed and approved for technical content by the purchasing agent prior to release. Such items are as follows:

a) Materials such as standards, reagents, solvents, chemicals, etc. are documented with:

i. date of receipt,
ii. expiration date (if applicable),
iii. identification of vendor or manufacturer,
iv. lot number, and
V. Certificate of Analysis (if applicable.)

See the Preparation of Standards SOP for additional details.

b) Services such as balance calibration are documented by the vendors certificates as described in the Analytical Balance Logbook SOP.

c) The documentation of accuracy and precision for S class weights is described in the Analytical Balance Log Book SOP.

d) The documentation of accuracy and precision for Class A glassware and manual volumetric dispensing devices is described in the Manual Volumetric Dispensing Device SOP.

e) The documentation of accuracy and precision for thermometers is described in the Annual Thermometer Calibration SOP.

9.3.7 Policy for the Control of Procured Items

Alpha Analytical has contracted with major environmental suppliers for the procurement of critical and non-critical items.

9.3.7.1 Items purchased on a regular basis that have an impact on the final data results are sequestered by lot to maintain quality uniformity and controlled conformity. Items such as containers, solvents, standards, etc. have been recognized by the industry to be critical to environmental laboratories; therefore, suppliers have specialized in producing these items to meet and exceed method specification.

9.3.7.2 These items are controlled by our procurement process by repeatedly

buying the same product. If an item does not meet the required specification, our procurement process will have the documentation to pull these items from our laboratory and return them to the supplier.

9.3.7.3 All critical items are constantly checked by their daily use, i.e., methanol is checked by analyzing daily method blank and standards are checked against the response factors of independent standards. These activities occur daily and are documented in various areas of the laboratory such as the analysts' logbooks and the instrument document control books.

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Section 10

Equipment and Instrument Maintenance

10.0 EQUIPMENT AND INSTRUMENT MAINTENANCE

10.0.1 Alpha Analytical, Inc. provides a full range of environmental analyses for contaminants in soil, water, industrial waste and other matrices. Alpha has analytical capabilities including sophisticated instrumentation for the detection of metals, inorganic analytes, industrial solvents, and other analytes encompassing the range of Hazardous Substances, Priority Pollutants, and the identification of thousands of organic compounds by Gas Chromatography/Mass Spectrometry (GC/MS).

10.0.2 It is Alpha's policy to purchase equipment capable of achieving the accuracy, precision, sensitivity, and selectivity required for the intended use of the analytical test methods.

10.1 EQUIPMENT OPERATION POLICY

10.1.1 Analytical test instruments are only operated by authorized personnel. Instrument manuals on the use and maintenance of this equipment is available for use by the analysts.

10.2 EQUIPMENT IDENTIFICATION

10.2.1 Analytical test instruments and their associated software are uniquely identified. This identification is used on all documents to reference that particular instrument.

10.2.2 Instrument identification documents are maintained to record the following information:

a) the identity of the item of equipment and its software;

b) the manufacturer's name, type of equipment, and serial an/or model number;

c) date received and date placed in service (if available);

d) equipment location; and

e) if available, condition when received (e.g., new, used, reconditioned).

10.3 MAJOR EQUIPMENT

Alpha Analytical is equipped with modern instrumentation required for the correct performance of the environmental testing specified by the regulatory agencies and to provide redundancy for all major systems.

10.3.1 Gas Chromatograph/Mass Spectrometers for Volatile Organic Analysis

Alpha is equipped with Hewlett-Packard (HP) 5890 and 5890 series II GCs attached to HP 5970 and 5972 MSDs and Agilent Technolgies (AT)6890 GC's attached to AT 5973 MSD's. The MSDs are quadruple mass analyzers and are equipped with turbo molecular pumps. Mass spectral data is acquired using windows based PCs and the HP/AT ChemStation and Enviroquant software.

Water samples are introduced into the GC's using Tekmar and OI Liquid Sample Concentrators (LSC) and Automatic Liquid Samplers (ALS). These(GC/MS) systems are dedicated to the analysis of Volatile Organic Compounds (VOCs) by methods 624, 8260B, 524.2 and total petroleum hydrocarbons..

10.3.2 Gas Chromatograph/Mass Spectrometer for Semi-volatile Analysis

Alpha's semi-volatile instrument uses the same hardware as described for the volatiles GC/MS systems excluding the purging devices. The instruments are configured for automatic sample injection using the AT 7683 and a AT 7693 auto-tower. Data acquisition and reduction are acquired through the use of a windows-based PC, AT ChemStation and Enviroquant Software. This instrument is dedicated to the analyses of semi-volatile organic compounds by methods 625 and 8270C.

10.3.3 Gas Chromatograph with Flame Ionization Detectors

Alpha is equipped with HP 5890 GC's and AT 6890 each with Flame Ionization Detectors (FID). Several of these systems are configured with the ProSep® large volume injection inlets and capillary columns for Method 8015-DRO, Total Petroleum Hydrocarbon (TPH) analysis. One system is configured with a headspace analyzer for the analysis of dissolved gases, and the other for sreening SVOCs.

10.3.4 Gas Chromatograph with Electron Capture Detectors

Alpha is equipped with a AT 7890 GC configured with a low thermal mass MACH system to enhance the thermal cycle frequency. This system employs Gerstel large volume injectors and is configured with dual micro electron capture detectors. This instrument is dedicated to the analyses of pesticides and PCBs.

10.3.5 High Pressure Liquid Chromatograph (HPLC) with UV and Fluorescence Detectors

Alpha has a HP1050 (TI) HPLC quaternary pump suitable for both gradient and isocratic instrument conditions. This system is configured with either/or Ultra Violent (UV) and fluorescence detectors. Data is acquired on a PC through an A/D converter box using AI-450 software.

10.3.6 Ion Chromatograph (IC) with Conductivity Detector

Ions are determined by the use of a Dionex DX 500 and DX 600 Ion Chromatography system. The DX500 is a modular system consisting of the GP40 pump, CD-20 conductivity detector, LSC 25 column heater department and AS40 auto-sampler.

The DX600, IC is configured essentially the same as above; however this instrument has a GP 50, a CD25 detector. Both systems use the Chromeleon software for data acquisition.

Perchlorates are determined by the use of a Dionex ICS2000 Ion Chromatography system. This is a non-modular system where the pump, detector and column heater are built into a single instrument. The AS-40 auto-sampler is modular and is the same auto-sampler as used for the other two systems. This instrument also uses the Chromellian software for data acquisition.

10.3.7 Inductively Coupled Plasma / Mass Spectrometer (ICP-MS)

Most metals analysis is performed on an AT7500i ICP-MS. This system is configured with the Cetax ASX510AS autosampler and a Neslab NSLCF-100 chiller. Data acquisition is accomplished with the Agilent ChemStation software. This instrument is dedicated to metals analysis by methods 200.8 and 6020.

10.3.8 Accelerated Solvent Extractor (ASE 200)

The ASE 200 is used by method SW3545 for accelerated solvent extraction of base/neutral and acids (BNAs). The ASE 200 accelerates the extraction of solid matrices by using solvents at elevated temperature and pressure. Increased temperature accelerates the extraction kinetics, while elevated pressure keeps the solvent below the boiling point, thus enabling safe and rapid extraction.

10.3.9 Microwave for the Digestion of Metals

The digestion of RCRA samples for metals analysis is performed using a Milestone Ethos Plus microwave digestion instrument. This microwave digestion instrument is designed with a complete temperature and pressure control system, thus preventing over-pressurization. The instrument is controlled with a PC using the EasyWAVE software for automatic parameter control.

ANALYTICAL INSTRUMENT LIST
TABLE 10-1

INSTRUMENT ID	MODEL	DETECTOR	AUTO-SAMPLER	DATA ACQUISITION SYSTEM	METHODS OF ANALYSIS
GC/MSD # 1	HP5890	HP5970B	LSC-3000 Aqua Tek-70	HP ChemStation	VOC Screens
GC/MSD # 2	HP5890	HP5970B	HP7673A ProSep 800 plus	HP ChemStation	MeOH, EtOH
GC/MSD # 3	HP5890 (Series II)	HP5970	LSC-2000 ALS-2050	HP ChemStation	524.2
GC/MSD # 4	HP5890 (Series II)	HP5972A	LSC-2000 ALS-2050	HP ChemStation	VOC Screens
GC/MSD # 6	HP5890 (Series II)	HP5972	Velocity XPT Aqua Tek-70	HP ChemStation	VOC Screens
GC/MSD # 7	AT6890	AT5973	LSC 3000 Aqua Tek-70	AT Chem Station	624, 8260, TPH-G
GC/MSD # 8	AT6890	AT5973	OI Eclipse OI 4554-A	AT ChemStation	624, 8260, TPH-G
GC/MSD # 9	AT6890N	AT5973N	LSC-3000 Aqua Tek-70	AT ChemStation	624, 8260, TPH-G
GC/MSD # 10	AT6890	AT5973	LSC-3000 Aqua Tek-70	AT ChemStation	624, 8260, TPH-G
GC/MSD # 11	HP5890	HP5970	HP7673A ProSep 800 plus	HP ChemStation	MeOH, EtOH
GC/MSD # 12	AT6890N	AT5973N	LSC-3100 Aqua Tek-70	AT ChemStation	624, 8260, TPH-G
GC/MSD # 14	AT6890N	AT5973Inert	AT7683	AT ChemStation	625, 8270C, PNA SIMs
GC/MSD # 15	AT6890N	AT5973Inert	Velocity XPT Aqua Tek-70	AT Chem Station	624, 8260, TPH-G
GC/MSD # 16	AT7890A	AT5975C	AT7693	AT Chem Station	625, 8270C, PNA SIMs

ANALYTICAL INSTRUMENT LIST
Continued
TABLE 10-1

INSTRUMENT ID	MODEL	DETECTOR	AUTO-SAMPLER	DATA ACQUISITION SYSTEM	METHODS OF ANALYSIS
GC/FID #1	AT 6890	FID	AT 7683B ProSep 800 Plus	AT ChemStation	TPH-D
GC/FID #2	AT 6890	FID	AT 7683B ProSep 800 Plus	AT ChemStation	TPH-D
GC/FID #5	HP 5890 Series II	FID	HP 7673A	HP Chem Station	SV Screen
GC/FID #6	AT 6890N	FID	AT G1888 Headspace Sampler	AT Chem Station	Dissolved Gases
GC/FID #7	AT 6890	FID	AT 7683B ProSep 800 Plus	AT ChemStation	TPH-D
GC/ECD #1	AT 7890A	ECD	AT 7683B	AT ChemStation	Pesticides/PCBs
HPLC # 1	HP1050 (TI)	HP1046A HP 1050UV	HP7673/1050	DIONEX AI-450	Organic Acids Neutraceuticals
HPLC # 3	HP1050 (TI)	HP1046A HP1050UV	HP7673/1050	DIONEX AI-450	Organic acids Neutraceuticals
IC #1	DX500	CD-20	AS-40	Chromeleon	300.0
IC #2	DX600	CD-25	AS-40	Chromeleon	300.0
IC #3	ICS2000	D-56	AS-40	Chromeleon	314.0
TOC	Dohrmann Phoenix 8000	UV	Dohrman STS 8000	TOC Talk	SM5310C / 9060
ICP-MS	Agilent 7500i	MS	Cetax ASX510AS	ChemStation	200.8, 6020/6020A

EQUIPMENT IDENTIFICATION FORM
TABLE 10-2

Equipment Identification Form			
Instrument ID:			
1) Date Reviewed:			Location:
3)	GC	Equipment Manufacturer	
		Model Number	
		Serial Number	
		Purchase Date	
		Date Placed in Service	
4)	Detector	Equipment Manufacturer	
		Model Number	
		Serial Number	
		Purchase Date	
		Date Placed in Service	
5)	Liquid Sample Concentrator	Equipment Manufacturer	
		Model Number	
		Serial Number	
		Purchase Date	
		Date Placed in Service	
6)	Auto sampler	Equipment Manufacturer	
		Model Number	
		Serial Number	
		Purchase Date	
		Date Placed in Service	
7)	Computer	Equipment Manufacturer	
		Model Number	
		Serial Number	
		Purchase Date	
		Date Placed in Service	
8)	Software	Software Name	
		Software Revision	

10.4 SUPPORT EQUIPMENT

10.4.1 Support equipment is defined as those devices that may not be the actual test instrument, but are necessary to support general laboratory operations. This equipment includes such things as: balances, ovens, refrigerators, freezers, incubators, water baths, temperature measuring devices (including thermometers and thermisters), thermal and pressure sample preparation devices, and volumetric dispensing devices (such as pippetors and automatic dispensing devices) or other equipment where the quantitative results are dependent on their accuracy, as in standard preparation and dispensing or diluting into a specified volume.

10.4.2 QA Manual, Volume II, Appendix D is a collection of support equipment SOP's. These support equipment SOP's describe the basic operation necessary to carry out the duties which support the major laboratory operations such as sample extraction/digestion and analysis. These operations include the following:

a) All support equipment is maintained in proper working order and a record of all repair and maintenance activities including service calls are maintained in their respective maintenance logbooks.

b) All support equipment is calibrated or verified at least annually using NIST traceable references when available, over the entire range of use. The results of these calibrations or verifications are maintained to ensure the equipment is within the specification required of the application for which the equipment is used. If the equipment does not meet these specifications then:

1) the equipment is removed from service until repaired; or

2) correction factors are established to correct all measurements and those correction factor records are maintained.

c) Raw data records are retained to document equipment performance.

d) Prior to use on each working day, balances, ovens, refrigerators, freezers, and water baths are checked in the expected range with NIST traceable references where commercially available. The acceptability of use or continued use is established according to the needs of the analysis or application for which the equipment is being used.

e) Mechanical volumetric dispensing devices (MVDD) (excluding Class A glassware) is checked for accuracy on a quarterly basis. Glass microliter syringes are considered the same as Class A glassware since the manufacturer (the Hamiltion Company) provides a certificate of accuracy document attesting the to accuracy of the syringes they produce.

10.5 INSTRUMENT MAINTENANCE

10.5.1 Policy

It is Alpha's policy to properly maintain, inspect and clean all equipment, and to document maintenance procedures in the maintenance log book.

10.5.2 Purpose

Alpha's Preventative Maintenance Program (PMP) establishes the basic procedures for the safe handling, transportation, storage, use and planned maintenance of analytical test and measurement equipment used to conduct sample analyses. These procedures are established to ensure laboratory equipment function properly and in order to prevent contamination or deterioration.

10.5.3 Responsibility

Preventative maintenance is a critical element of the quality assurance program at Alpha Analytical. Responsibility for preventative maintenance lies with the analyst and their direct supervisor in charge of monitoring equipment. Our analytical staff is dedicated to the implementation of the preventative maintenance program and are always watchful for signs that there is a need for maintenance activities.

10.5.3.1 In-house Maintenance

Analysts perform routine preventive maintenance such as the replacement of parts, cleaning of components, and changing of pump oil. Analytical instruments such as GCs, GC-MS and ICP-MS systems are serviced and maintained by in-house personnel.

10.5.3.2 Outsourced Maintenance

Occasionally there are instances when service personnel are contracted to replace or service instruments that cannot be serviced by our personnel. Other instruments such as analytical balances are serviced on a routine basis by a contracted company. All instrument maintenance is recorded in an instrument maintenance logbook specifically associated with that particular instrument.

10.5.4 Monitoring

10.5.4.1

Instruments are constantly monitored by the use of daily standards, sensitivity, and response checks to determine if maintenance is required. In the event that an instrument does fail, every effort is made to meet obligations to the client's holding times and due dates.

10.5.4.2 Instruments that have been subjected to overloading or mishandling, gives suspect results, or has been shown to be defective or outside specified limits are taken out of service.

Note: NELAC suggests the instrument to be isolated to prevent its use or to clearly label or mark the instrument as being out of service, until it has been repaired and shown by calibration or test to perform correctly

10.5.4.3 If a piece of equipment or an instrument is sent in for bench repair, the instrument is re-calibrated and/or checked to ensure the instrument is functioning satisfactorily before it is returned to analytical service.

10.5.4.4 Laboratory support equipment such as refrigerators and ovens are also monitored and serviced regularly. The laboratory quality assurance program is designed to reduce data loss by monitoring and recording the functioning of these systems, allowing rapid correction of any malfunction before data loss can occur.

10.5.4.5 Alpha Analytical's Preventative Maintenance Program concentrates on four primary areas of concern and they are as follows:

1) A suggested PM schedule. See Table 10-3;

2) Documentation of all maintenance and repairs;

3) Vendor/manufacturing operation and maintenance manuals available for all instruments; and,

4) Alpha Analytical's Analytical Contingency Plan.

10.5.5 Maintenance Schedule (Plan)

10.5.5.1 Equipment Purchase

When Alpha Analytical was established in 1987, one of the primary goals in the start-up operation was to buy the finest equipment available to reduce and minimize down time.

10.5.5.2 Suggested Maintenance Schedules

Maintenance that is performed on a regular schedule consists of changing pump oil, changing septum and injection inserts, cleaning syringe barrels on automatic sample injectors, etc. Most other types of maintenance are those that cannot be prevented by regular

servicing, such as electronic board failure, filament burnout, detection degradation, etc.

Table 10-3 identifies suggested preventative maintenance activities by instrument type and recommended frequencies. It should be noted that it may be necessary to perform activities more or less frequently depending on the workload, sample types analyzed, and/or instrument performance. Frequency of instrument maintenance activities incorporates both laboratory experience and instrument manufacturer's recommended PM frequency.

Note: The suggested maintenance schedule is only suggested and does not require the frequency and/or suggested activity need take place. For instance: the GC instrument maintenance suggests to change septa, check cylinder gas pressure and moisture and oxygen traps on a daily basis. This does not mean that a check list and/or annotation in the maintenance log that these were checked on a daily basis need to be documented. In fact, many days the instrument may have not even been run. These are suggested activities and frequencies, and should only be documented when they have occurred.

10.6 MAINTENANCE LOGBOOK

10.6.1 An individual instrument maintenance logbook is assigned to each instrument. This logbook is used to record preventive maintenance checks and services in addition to emergency maintenance procedures. The maintenance logbook contains descriptions of instrument problems, solutions, replacement parts, and maintenance personnel. The maintenance logbook contains the following information:

a) the identification of the item of equipment and its software;

b) the manufacturer's name, type, serial number or other unique identification;

c) checks that the equipments complies with the specifications;

d) the current location;

e) the manufacturer's instructions, if available,

f) dates, results and copies of reports and certificates of all calibrations, adjustments, acceptance criteria, and the due dates of next calibration;

Note: This information is maintained in a number of logbooks and not necessarily the maintenance logbook.

g) maintenance plan, and the maintenance carried out to date; documenting all routine and non-routine maintenance activities and reference material activities;

h) any damage, malfunction, modification or repair to the instrument;

i) date received and date placed in service; and

j) if available, condition when received (e.g., new, used, reconditioned).

10.7 MAINTENANCE MANUALS

10.7.1 As stated earlier in this section, Alpha Analytical's instrumentation consists primarily of Hewlett-Packard/Agilent Technologies GCs, GC-MSs, ICP-MSs and HPLCs. Agilent generally publishes three to four separate service books associated with each analytical instrument and they are as follows:

1) Operators Handbook - A description of the use and operation of a particular instrument;

2) Installation and Maintenance Guide - A handbook that describes how to install, troubleshoot, and maintain each instrument;

3) Getting It All Together - A handbook that describes how to connect different modules to make a complete system; and,

4) Parts Manual - This handbook displays blow-ups of various sub-components of a system while giving part numbers and electronic schematics.

Alpha Analytical maintains a library of maintenance manuals for all Hewlett-Packard and Agilent equipment and PM manuals for all other equipment Alpha Analytical uses on a regular basis.

10.8 CONTINGENCY PLANS

10.8.1 Instrument Capacity/Back up Instruments

For most methods, we have the instrument capacity to perform analysis on multiple instruments. This capability changes over time based on sample work load and the availability of its backup.

10.8.2 Spare Parts Inventory

In the event of complete instrument failure, a number of decisions need to be quickly

made, in order to prevent invalidating the current sample work load. All priorities and effort will be addressed to resolve the instrument problem. Alpha Analytical maintains a sizeable inventory of spare parts for this scenario. However, it is impractical and impossible to predict and maintain an inventory of all possible spare parts. Parts can be delivered overnight for repair the next day.

10.8.3 Service Calls

The second decision which is made at the onset of instrument failure is if our in-house personnel are able to repair the problem at hand. If not, then a service call is made to determine the logistical requirements of getting a service person to the laboratory at the earliest possible date. Service calls also are useful in talking or resolving an instrument problem over the phone. The conversation usually entails a series of diagnostic activities, which will guide the analyst or person in charge of that instrument to a reason for the instrument failure. This activity is the first plan of attack when an instrument fails for non-obvious reasons. If the problem can still not be resolved, then the service call goes one step further and a repair person is dispatched to the laboratory.

Alpha Analytical's service priorities are as follows:

1) Service our clients by performing the work within their contract or specified turn-around time;

2) Perform this work under the guidance of a certification program; and,

3) Provide quality analytical data.

10.8.4 Sub-contracting Laboratories

In the event of instrument failure, it is Alpha's goal to accomplish and perform all testing and analysis in-house. When it becomes apparent that our laboratory cannot meet these obligations, Alpha Analytical will then sub-contract this work to one of several laboratories that are available.

10.9 ACCOMMODATION AND ENVIRONMENTAL CONDITIONS

10.9.1 It is Alpha's policy to ensure that the environmental conditions do not invalidate the test results or adversely affect the required quality of any measurement. These types of environmental conditions include but are not limited to energy sources, lighting, heating, cooling humidity and ventilation.

10.9.2 The technical requirements for accommodation and environmental conditions, when required by the method or where they may influence the quality of the test results are monitored and documented. In the event environmental conditions have deteriorated

to a point when environmental conditions may jeopardize the results of environmental tests, then testing should be stopped until these issues are resolved.

10.9.3 Neighboring work areas in which there are incompatible activities are physically separated as a precaution to prevent cross-contamination. Physically separating work areas is a key component in producing accurate data results and includes such activities as volatile organic handling areas and semivolatile sample preparation.

10.10 HOUSEKEEPING AND WORKSPACE

10.10.1 It is Alpha's policy to ensure good housekeeping in the laboratory and to make accommodations for special procedures when necessary.

10.10.2 In is Alpha's policy to ensure work areas are unencumbered to include:

a) access and entryways to the laboratory;

b) sample receipt areas;

c) sample storage areas;

d) chemical and waste storage areas; and

e) data handling and storage areas.

TABLE 10-3
SUGGESTED PREVENTATIVE MAINTENANCE ACTIVITIES

Note: The suggested maintenance schedule is only suggested and does not require the frequency and/or suggested activity need take place. For instance: the GC instrument maintenance suggests to check the septa, cylinder gas pressure and moisture and oxygen traps on a daily basis. This does not mean that a check list and/or annotation in the maintenance log that these were checked on a daily basis needs to be documented. In fact, many days the instrument may have not even been run. Common sense needs to be applied; therefore, maintenance which affects data quality should only be documented.

INSTRUMENT	ACTIVITY		FREQUENCY
Gas Chromatographs	General	Check septa, cylinder gas pressure, and oxygen, moisture traps	D
		Bake out injection body	2
		Check electronics (voltages, waveforms, etc.)	3, 4
	Columns	Change glass inserts, shorten ends of columns, change glass wool plugs, replace ferrules, and check for leaks	3, 5
	Electron Capture Detector	Wipe tests	A
		Return detector to factory for cleaning and refoiling	2,3, 4
	Flame Ionization Detector	Clean	Q
		Replace flare tip	A
		Replace flare ignition	1
	Nitrogen Phosphorous Detector	Clean	Q
		Replace bead	1, 3, 4
Mass Spectrometers	General	Replace vacuum pump oil	A (1)
		Check ion source and analyzer (dismantle and clean, replace parts as needed)	A (1, 2, 3, 4, and 5)
		Check mechanical (vacuum pump, gas pressures, and flows)	Q
	Purge and Trap	Bake vessels	2
		Change trap	A
		Bake trap	2
		Check purge flow	M
		Check for leaks	M

Key to Frequencies: (1) Replace as necessary; (2) High background; (3) Loss of sensitivity or failing resolution; (4) Erratic response; (5) QC failure; (A) Annually; (D) Daily; (M) Monthly; (Q) Quarterly; (BA) Bi-annually; (SA) Semi-annually; and (W) Weekly.

TABLE 10-4
SUGGESTED PREVENTATIVE MAINTENANCE ACTIVITIES

Note: The suggested maintenance schedule is only suggested and does not require the frequency and/or suggested activity need take place. For instance: the HPLC instrument maintenance suggests checking the gas lines for leaks on a daily basis. This does not mean that a check list and/or annotation in the maintenance log that these were checked on a daily basis needs to be documented. In fact, many days the instrument may have not even been run. Common sense needs to be applied; therefore, maintenance which affects data quality should only be documented.

INSTRUMENT	ACTIVITY		FREQUENCY
High Pressure Liquid Chromatographs	General	Gas Lines Checked for leaks	D
		Clean mobile phase flow system with 10% nitric acid	BA (3,4)
		Clean detector flow cells	BA(3,4)
		Clean injection valve (replace rotors and seals)	A(1)
		Check solvent filters	W
		Check pump seals and check valve assemblies (clean and replace as pressures and flows of mobile phase indicate)	D(1)
		Lubricate post column reagent pumps	M
		Check valves and replace post column restrictors and frits or clean restrictors with 10% nitric acid	M(1,3,4)
Ion Chromatographs	General	Replace or quick start suppressor	1,2,3,4,5
		Clean detector flow cell	2,3,4
		Clean injection valve (replace rotors and seals)	A(1)
		Check pump seals (clean and replace as pressures and flows of mobile phase indicate)	D(1)
ICP-MS	General	Check cylinder gas pressure	D(1)
	ICP	Clean sample and skimmer cones	2,3,4,5
		Clean torch and spray chamber assemblies	2,3,4,5
		Replace liquid sample lines	M(2,4,5)
	MS	Check turbo pump	Q
		Replace vacuum pump oil	A(1)
		Check ion source and analyzer	A(1,2,3,4,5)
Auto Samplers	General	Check needles/syringes	D(1)
		Replace motors, belts, carriage assemblies, and sensors	1,4
		Clean	Q
Data Systems	General	Clean computers, check battery backup, and check ventilation fans	A

Key to Frequencies: (1) Replace as necessary; (2) High background; (3) Loss of sensitivity or failing resolution; (4) Erratic response; (5) QC failure; (A) Annually; (D) Daily; (M) Monthly; (Q) Quarterly; (BA) Bi-annually; (SA) Semi-annually; and (W) Weekly.

TABLE 10-5
SUGGESTED PREVENTATIVE MAINTENANCE ACTIVITIES

Note: The suggested maintenance schedule is only suggested and does not require the frequency and/or suggested activity need take place. Therefore this does not mean that a check list and/or annotation in the maintenance log that these were checked on the suggested frequency be documented. In fact, many days the instrument may have not even been run. Common sense needs to be applied; therefore, maintenance which affects data quality should only be documented.

INSTRUMENT	ACTIVITY		FREQUENCY
Refrigerators/Ovens	General	Clean interiors	SA
		Check thermometer temperatures against NBS certified thermometer	A
Analytical Balances	General	Clean pan and compartment	D
		Check with S class weights	D
pH and Ion Selective Electrodes	Probes	Check probe for cracks and proper levels of filling solution; check reference junction; clean electrode	D(1)
	Meter	Check electronics or batteries for loose connections and cracked leads	D(1)
Spectrophotometers	General	Clean sample compartment interior	SA
		Check wave-length calibration with holmium-oxide filter	A
TOC Instrument	General	Check carrier gas, and reagents	D
		Clean UV reactor and IC sparger	W(2,3,4)
		Inspect chorine scrubber	M(1,2,3,4,5)
		Inspect permangenate dryer	M(1,2,3,4,5)
Thermometers	General	Check for cracks in the glass and gaps in the fluid	D(1)

Key to Frequencies: (1) Replace as necessary; (2) High background; (3) Loss of sensitivity or failing resolution; (4) Erratic response; (5) QC failure; (A) Annually; (D) Daily; (M) Monthly; (Q) Quarterly; (BA) Bi-annually; (SA) Semi-annually; and (W) Weekly.

QUALITY ASSURANCE MANUAL
VOLUME I

Section 11

Quality Control Procedures to Assess Laboratory Data

11.0 QUALITY CONTROL PROCEDURES TO ASSESS LABORATORY DATA

11.1 ELEMENTS OF QUALITY CONTROL CHECKS

This section presents QC requirements relevant to analysis of environmental samples that are followed during all analytical activities. The purpose of this QC program is to provide quantitative evidence that the entire analytical method is performed within the specified criteria. Data generated from control samples are used to monitor day-to-day variations in routine analysis, which provides a mechanism for ongoing control and evaluation of data quality measurements through the use of QC material.

11.2 INTRA LABORATORY QUALITY CONTROL

Intra laboratory quality control is performed as described in the Standard Methods for the Examination of Water and Wastewater, 20th and 21st Editions, Environmental Protection Agency Methods for Organics in Finished Drinking Water, and Test Methods for Evaluating Solid Waste, 3rd Edition. Quality Control data is used to review and determine method precision and accuracy.

11.3 FIELD QC SAMPLES

The following QC check samples are the basic requirements needed to ensure the reliability and integrity of field data. For details, see the Quality Control Field Samples SOP located in Appendix B.

11.3.1 Equipment/Rinsate Blanks

These are field QC check samples used to ensure non-dedicated sampling devices (bailers, filtering equipment, pumps, etc.) have been effectively decontaminated. After field washing and decontamination, sampling equipment should be rinsed with reagent free water. This rinse water is then transferred to a sample bottle, and returned to the laboratory for analysis. A representative number of the equipment / rinsate blanks should be analyzed, depending on the SOW.

11.3.2 Field Reagent Blanks (FRB)/ Field Transfer Blanks (FTB)

These types of QC samples are reagent grade water placed in a sample container at the laboratory and treated exactly as a sample in all respects, including exposure to sampling site conditions, storage, preservation and all analytical procedures. The purpose of the FRB/FTB is to determine if the method analytes or other interferences are present as air-borne constituents in the field environment. These types of QC samples are analyzed for project specific compounds.

11.3.3 Trip Blanks (TB)

Trip Blanks (TB) are analogous to FRBs in all respects except they are not opened or exposed to the field environment at any time. The purpose of the TB is to determine if method analytes or other air-borne interferences are present in the environment to which actual samples were exposed, and perhaps contaminated or cross-contaminated samples by air-borne infusion through the sample septum. Trip Blanks are most typically sent in coolers with samples requesting VOC analysis.

11.3.4 Field Duplicates

Field duplicates are two separate samples collected at the same time, under identical circumstances, and treated exactly the same throughout the field and laboratory procedures. Analyses of field duplicates give a measure of the precision associated with sample collection, preservation and storage, as well as with laboratory procedures. Field duplicates are typically collected for an analysis at a frequency of 10% of the total samples taken for each parameter group.

11.4 LABORATORY QUALITY CONTROL SAMPLES

Laboratory control samples are those samples introduced into the train of environmental samples to function as monitors on the performance of the analytical method. All QC samples are prepared and processed through the complete analytical method. Stock solutions used to spike QC samples are prepared independently of stock standards used for calibration standards.

Method Blanks (negative controls) and laboratory control samples(positive controls) are used to monitor day-to-day performance of routine analytical methods. Internal standards are used to monitor the performance of the instrument. Surrogates, matrix spikes and sample duplicates are used to assess the effects of extraction efficiency, sample matrix and sampling, respectively, on the analytical data. The following descriptions indicate the types of QC samples that are included in an analytical or an extraction batch.

11.4.1 Method Blank (MB)

11.4.1.1 Definition

Method blanks are QC samples that have not been exposed to the analyzed sample stream in order to monitor contamination during sampling, transport, storage or analysis. The method blank is subjected to the usual analytical and measurement process to establish a zero baseline or background value and is sometimes use to correct routine analytical results.

11.4.1.2 Purpose

The method blank is used to assess the preparation batch for possible

contamination during the preparation and processing steps. The method blank is processed along with and under the same conditions as the associated samples to include all steps of the analytical procedure.

11.4.1.3 Frequency

The method blank is prepared and analyzed at a minimum of 1 per preparation batch not to exceed 20 samples. In those instances for which no separate preparation method is used (example: volatiles in water) the batch is defined as the group of environmental samples that are analyzed together with the same method and personnel, using the same lots of reagents, not to exceed the analysis of 20 environmental samples.

11.4.1.4 Composition

Method blanks are prepared from a matrix similar to the batch of associated samples (e.g., reagent grade water for water matrices, or ottowa sand, sodium sulfate or teflon chips for soil matrices) that is free from the analtyes of interest and is processed simultaneously with and under the same conditions as samples through all steps of the analytical procedures, and in which no target analytes or interferences are present at concentrations that impact the analytical results for sample analyses.

11.4.1.5 Evaluation Criteria

The goal for the method blank is to have no detectable contamination, however each method blank is critically evaluated as to the nature of interferences and the effect on the analysis of each sample within the batch. The source of contamination is investigated and measures are taken to minimize or eliminate the problem.

11.4.1.6 Corrective Actions

If a blank exceeds the detectable limit for reporting of target analytes, then the analytical and extraction systems are evaluated and the appropriate corrective actions are taken. This may include such things as:

a) cessation of further sample analysis to determine the source of contamination;

b) reanalyses of all samples processed with that blank; or

c) reporting the results with the appropriate footnotes.

11.4.2 Laboratory Control Sample (LCS)

11.4.2.1 Definition

Laboratory control samples are prepared from a sample matrix, similar to the batch of associated samples (e.g., reagent grade water for water matrices, or ottowa sand, sodium sulfate, or teflon chips for soil matrices), free from the analytes of interest and spiked with verified known amounts of analytes or material containing known or verified amounts of analytes.

Note: The matrix spike may be used in place of the LCS as long as the acceptance criteria are as stringent as for the LCS. Alternatively the LCS may be prepared from a matrix which contains known and verified concentrations of analytes or as a Certified Reference Material (CRM). Spiked analyte concentrations must be within the range of calibration.

11.4.2.2 Purpose

The LCS is used to evaluate the performance of the total analytical system, including all preparation and analysis steps.

11.4.2.3 Frequency

An LCS is analyzed at a minimum of 1 per preparation batch. Exceptions for this frequency are for those methods/analytes for which no spiking solutions are available such as pH. In those instances for which a separate preparation method is not used (Example: volatiles in water) the batch is defined as the group of environmental samples that are analyzed together with the same method and personnel, using the same lots of reagents, not to exceed the analysis of 20 environmental samples.

11.4.2.4 Composition

The components spiked into the LCS are those specified by the mandated test method or other regulatory requirement or as requested by the client. In the absence of specified spiking components LCS samples are spiked as follows:

a) For those components that interfere with an accurate

assessment (i.e., spiking multi-component analytes together such as spiking simultaneously with technical chlordane, toxaphene and PCBs) the spike is chosen that represents the chemistries and elution patterns of the components to be reported.

b) For those test methods that have extremely long lists of analytes, a representative number may be chosen. The selected analytes are chosen in a manner representing all reported analytes. The following criteria are used for determining the minimum number of analytes to be spiked. However, all target analytes are spiked and evaluated over a 2 -year period.

1) For methods that include 1-10 target analytes, all compounds are spiked and evaluated;

2) For methods that include 11-20 target analytes, at least 10 or 80% of the compounds, whichever is greater, are spiked and evaluated;

3) For methods that include more than 20 target analytes, at a minimum 16 compounds are spiked and evaluated.

11.4.2.5 Evaluation Criteria

11.4.2.5.1 The results of the individual batch LCS analytes are calculated in percent recovery and compared to established acceptance criteria. These calculations are documented on method worksheets and/or on LIMS generated summary LCS reports. LCS results are compared to acceptance criteria as follows:

a) If LCS criteria is method specified, than the LCS is compared to the criteria as published in the mandated test method.

b) If LCS criteria is not method specified, than the LCS is compared to laboratory derived criteria, and the method used to establish the limits are documented.

c) Client specified criteria.

A LCS that is determined to be within the criteria effectively establishes that the analytical system is in control and validates system performance for the samples in the associated batch.

Samples analyzed along with a LCS determined to be "out-of-control" are considered suspect and the samples are reprocessed and re-analyzed or the data reported with the appropriate footnote.

11.4.2.5.2 Marginal Exceedance Limits (Optional)

11.4.2.5.2.1 If a large number of analytes are in the LCS, it becomes statistically likely that a few will be outside the control limits. This many not indicate that the system is out of control, therefore, corrective action may not be necessary. Upper and lower marginal exceedance (ME) limits may be established to determine when corrective action is necessary. A ME is defined as being beyond the LCS control limit (3 standard deviations), but within the ME limits. ME limits are between 3 and 4 standard deviations around the mean.

11.4.2.5.2.2 The number of allowable marginal exceedances is based on the number of target analytes evaluated in the LCS. If more analytes exceed the LCS control limits than are allowed, or if any one analyte exceeds the ME limits, the LCS fails and corrective action is necessary. This marginal exceedance approach is relevant for methods with long lists of analytes such as Methods 8260B and 8270C. This evaluation does not apply to target analyte lists with fewer than 11 analytes.

11.4.2.5.2.3 The number of allowable marginal exceedances is as follows:

1) >90 analytes in LCS, 5 analytes allowed in ME;
2) 71-90 analytes in LCS, 4 analytes allowed in ME;
3) 51-70 analytes in LCS, 3 analytes allowed in ME;
4) 31-50 analytes in LCS, 2 analytes allowed in ME;
5) 11-30 analytes in LCS, 1 analytes allowed in ME;
6) <11 analytes in LCS, no analytes allowed in ME.

11.4.2.5.2.4 Marginal exceedances must be random. Analytes which repeatedly exceed the ME are not random and may be an indication of a systemic problem. If this occurs, the source of the problem is located and corrective action is taken.

11.4.2.6 Corrective Actions

Any affected samples associated with an out-of-control LCS is reprocessed for re-analysis or the results are reported with the appropriate footnotes.

11.4.3 Matrix Spike (MS)

11.4.3.1 Definition

Matrix spike samples are prepared by adding a known mass of target analytes to a specified amount of the sample targeted for spiking where an independent estimate of target analyte concentration is available. Matrix spikes are used to determine the effect of the matrix on the method spike recovery efficiency.

A matrix spike duplicate (MSD) is a replicate aliquot of the same sample taken through the entire analytical procedure. The results form this analysis indicates the precision of the results for the specific sample using the selected method.

Client samples are used as the batch MS/MSD and are randomly picked to ensure spiked samples are rotated through the client sample stream.

11.4.3.2 Purpose

Matrix spike samples indicate the effect of the sample matrix on the precision and accuracy of the results generated using the selected method. Therefore, the MS is extracted and analyzed exactly like a sample, and its explicit purpose is to determine whether the sample matrix contributes bias to the analytical results.

Note: The information from this control is sample and matrix specific and is not normally used to determine the validity of the entire analytical batch or to judge laboratory performance.

11.4.3.3 Frequency

Each preparatory batch, not to exceed 20 samples, must contain an associated MS and MSD using the collected project samples. If adequate sample material is not available, then the lack of a matrix spike/matrix spike duplicate is noted. Additionally, MS/MSD frequency may be project or test method specified.

11.4.3.4 Composition

The components spiked into the MS are those specified by the mandated test method or other regulatory requirement or as requested by the client. In the absence of specified spiking components MS samples are spiked as follows:

a) For those components that interfere with an accurate assessment (i.e., spiking multi-component analytes together such as spiking simultaneously with technical chlordane, toxaphene and PCBs) the spike is chosen that represents the chemistries and elution patterns of the components to be reported.

b) For those test methods that have extremely long lists of analytes, a representative number may be chosen. The selected analytes are chosen in a manner representing all reported analytes. The following criteria are used for determining the minimum number of analytes to be spiked. However, all target analytes are spiked and evaluated over a 2-year period.

1) For methods that include 1-10 target analytes, all compounds are spiked and evaluated;

2) For methods that included 11-20 target analytes, at least 10 or 80% of those compounds, whichever is greater, are spiked and evaluated;

3) For methods that include more than 20 target analytes, at a minimum 16 compounds are spiked and evaluated.

11.4.3.5 Evaluation Criteria

The results from matrix spike/matrix spike duplicate are primarily designed to assess the precision and accuracy of analytical results in a given matrix and are expressed as percent recovery (%R), relative percent difference (RPD), or other appropriate statistical technique that allows comparison to established acceptance criteria. These calculations are documented on method worksheets and/or on LIMS generated summary MS reports. MS/MSD results are compared to acceptance criteria as follows:

a) If MS criteria is method specified, than the MS is compared to the criteria as published in the mandated test method.

b) If MS criteria is not method specified, than the MS is compared to laboratory derived criteria, and the method used to establish the limits are documented.

c) Client specified criteria.

The sample is first analyzed in a separate non-spike aliquot, to determine if any analytes are in the sample which may contribute as background. The sample is then spiked and the measured value(s) in the spiked MS/MSD is corrected for background concentrations.

11.4.3.6 Corrective Actions

If MS/MSD results are outside the established criteria, for either accuracy or precision, corrective action is documented or the results are reported with the appropriate footnotes.

Often-times, MS recoveries exhibit matrix interference and are outside the range of acceptability. In these cases the LCS is used to qualify analytical data; therefore, the recovery problem encountered with the spiked sample is judged to be matrix related, not system related, provided both LCS and MS were extracted in the same extraction batch.

11.4.4 Matrix and Sample Duplicate

11.4.4.1 Definition

The analyses or measurements of the variable of interest performed identically on two sample aliquot of the same sample.

11.4.4.2 Purpose

A spiked matrix or sample duplicate is a replicate aliquot of the same sample taken through the entire analytical procedure. The results form this analysis indicates the precision of the results for the specific sample using the selected method. The spiked matrix or sample duplicate provides a usable measure of precision only when target analytes are found in the sample chosen for duplication.

Note: The information from this control is sample and matrix specific and is not normally used to determine the validity of the entire analytical batch or to judge laboratory performance.

11.4.4.3 Frequency

If target analytes are known in a sample duplicate then it may be analyzed in place of an MSD. Duplicate analysis whether an MSD or sample duplicate must be performed at a minimum frequency of once per preparatory batch not to exceed 20 samples.

Note: Since it not typically known if a sample contains target analytes, preparing and analyzing a sample duplicate in place of an MSD is impractical and is discouraged. However, if a client or a project requires a sample duplicate, then both a sample duplicate and MSD are prepared and analyzed.

11.4.4.4 Composition

Laboratory duplicates are two sample aliquots taken from the same sample and analyzed separately with identical procedures. The composition is usually not known. Client samples used as sample duplicates are randomly picked to ensure they are rotated through the client stream.

11.4.4.5 Evaluation Criteria

The results from the spiked matrix or sample duplicate are primarily designed to assess the precision in a given matrix and are expressed as relative percent difference (RPD). These calculations are documented on method worksheets and/or on LIMS generated summary sample duplicate reports. Duplicate results are compared to acceptance criteria as follows:

a) If sample duplicate criteria is method specified, than the duplicate data is compared to the criteria as published in the mandated test method.

b) If sample duplicate criteria is not method specified, than the duplicate data is compared to laboratory derived criteria, and the method used to establish the limits are documented.

c) Client specified criteria.

11.4.4.6 Corrective Actions

If sample duplicate data results are outside of the established criteria, corrective actions are documented or the data is reported with the appropriate footnotes.

11.4.5 Surrogate Spikes

11.4.5.1 Definition

A substance with properties that mimic the analytes of interest which is unlikely to be found in environmental samples and is added to samples for quality control purposes.

11.4.5.2 Purpose

Surrogates are used most often in organic chromatography test methods and are chosen to reflect the chemistries of the targeted components of the method. Surrogates are added prior to sample preparation/extraction and provide a measure of recovery for every sample matrix.

Note: The information from this control is sample and matrix specific and is not normally used to determine the validity of the entire analytical batch or to judge laboratory performance.

11.4.5.3 Frequency

Surrogates are added to all samples, standards, and blanks for all appropriate test methods, except where the matrix precludes its use.

11.4.5.4 Spike Composition

Surrogate compounds are chosen to represent the various chemistries of the target analytes in the method. They are most often specified by the mandated method and are deliberately chosen for their being unlikely to occur as an environmental contaminant. Often this is accomplished by using deuterated analogs of selected compounds.

11.4.5.5 Evaluation Criteria

The results from surrogate spikes are used to monitor the effect of the matrix on the accuracy of the analysis. Surrogates are also used to assess the recovery of the method and to detect any systematic extraction problems. Results are reported in terms of percent recovery. These calculations are documented on method worksheets and/or on LIMS generated summary reports. Surrogate results are compared to acceptance criteria as follows:

a) If surrogate criteria is method specified, than the surrogate

recovery data is compared to the criteria as published in the mandated test method.

b) If surrogate criteria is not method specified, than the surrogate recovery data is compared to laboratory derived criteria, and the method used to establish the limits are documented.

c) Client specified criteria.

11.4.5.6 Corrective Action

Surrogates outside the acceptance criteria are evaluated to determine if the aberration is indicating an effect on the individual sample results. The appropriate corrective action is generally guided by the data quality objectives or other site specific requirements. Results reported from analyses with surrogate recoveries outside the acceptance criteria are reported with the appropriate data qualifiers.

11.4.6 System Monitoring Compounds

System Monitoring Compounds are added to every blank, sample, matrix spike, matrix spike duplicate and standard for volatile organic analysis, and are used to evaluate the performance of the entire analytical system. These compounds serve essentially the same purpose as the surrogates used in extractable analysis.

11.4.7 Internal Standards

11.4.7.1 Definition

A known amount of standard is added to a known volume of sample to be analyzed as a reference for evaluating and controlling the precision and bias of the applied analytical method.

11.4.7.2 Purpose

Internal standards are used in internal standard calibration methods to correct sample results affected by changing instrument sensitivity, injection and purging losses, by measuring and comparing the relative responses of method analytes that are components of the same solutions.

11.4.7.3 Frequency

Internal standards are added to all samples, standards, and blanks for all appropriate methods, except where the matrix precludes its use.

11.4.7.4 Composition

Internal standards are chosen to represent the various chemistries of the target analytes in the method. They are most often specified by the mandated method and are deliberately chosen for their being unlikely to occur as an environmental contaminant. Often this is accomplished by using deuterated analogs of selected compounds.

11.4.7.5 Evaluation Criteria

The results from internal standards are used to monitor the effect of the matrix on the accuracy of the analysis. Internal standard results are compared to acceptance criteria as published in the mandated test method.

11.4.7.6 Corrective Action

Internal standard results outside the acceptance criteria are evaluated to determine if the aberration is an effect of the individual sample matrix or an instrument problem. Typically samples which have exceeded the method criteria are re-analyzed. If the reanalysis indicates a matrix effect, then results reported from analyses with internal standard recoveries outside the acceptance criteria are footnoted appropriately.

QUALITY ASSURANCE MANUAL
VOLUME I

Section 12

Data Reduction, Review and Storage

12.0 DATA REDUCTION, REVIEW AND STORAGE

12.1 CONTROL OF DATA

The data reduction, review, verification and reporting procedures described in this section are established to help ensure data are correctly reported. The primary focus of this multi-tiered peer review is as follows:

a) Reported data are free from transcription and calculation errors;

b) Quality control measures are reviewed, and evaluated before data is reported;

c) Calibrations, calculations and manual integrations are reviewed for correctness; and

d) Complete documentation trail is maintained.

Laboratory data reduction and review procedures are required to ensure that the overall objectives of analysis and reporting meet method or project specifications.

All laboratory activities and procedures are documented where possible to ensure maximum sample integrity. Data reduction, review, and reporting activities are included in all client data files and/or the Analytical Data Record Keeping System.

The overall Data Quality Objectives (DQOs) for the analytical activities can only be met if the data generated can be proven to be valid.

12.2 DATA REDUCTION

Alpha Analytical maintains written Standard Operating Procedures (SOPs) governing all aspects of the data acquisition and reporting process. This record keeping procedure makes it possible to reanalyze data at a future date and is used in support of the experimental conclusions.

Data reduction is the process of converting measurements collected by analytical data systems into an expression of parameters and information from which conclusions about the sample or site can be made. This process must be performed with acceptable precision and accuracy. All calculations and data entries are reviewed to maintain the accuracy of this process.

Laboratory QC samples (such as matrix spikes, matrix spike duplicates, method blanks, surrogate spikes, and laboratory control samples) are analyzed and data generated to evaluate and assess the accuracy and precision of the data. Accuracy and precision data are used to determine if errors are produced through the analytical procedure.

In addition, the QC field samples (such as trip, equipment/rinse blanks, and field duplicate samples) are analyzed to determine any systematic or random errors introduced by field procedures.

12.2.1 Compound Identification

When appropriate, identification and quantitation is based on internal standards, such as those specified in EPA Methods 524.2, 624, 625 and 200.8. When internal standards methods are not satisfactory, external standard methods are used to quantitate analytical data.

Chromatographic compound identification is routinely accomplished by comparison of its retention time to the retention times of standard reference chromatograms. If the retention time of an unknown compound in a sample corresponds, within the retention time (RT) limits which are established by standard calibrations, then identification is considered positive for the analytical column only.

12.2.2 Analytical Column

For GC analysis of single response analytes, which use RT as the only source of compound identification, an alternative technique is employed to confirm peak identification. When possible, sample confirmation is determined by GC/MS. This may be limited by trying to confirm compounds at very low levels of detection where other GC detectors are more sensitive.

12.2.3 Confirmation Column

The second technique that Alpha uses for positive peak identification is through the use of a confirmation column. Each confirmation column is selected with a different polarity than the analytical column; therefore, the retention time and the retention time order are different for each column. If the unknown compound is within the prescribed retention time windows on the confirmation column and compound quantitation is similar to the analytical column, then compound identification is considered positive.

Multi-response analytes (i.e., TPH and PCBs) do not require additional confirmation because they each have a unique chromatographic signature that positively identifies each of these types of compounds.

12.2.4 Retention Time Windows

Retention time windows are calculated and used in chromatographic methods of analysis for qualitative identification of analytes. They are generally calculated from the analyses of standards over the course of an analytical sequence. The standard deviation of the retention time of multiple injections for each single component or

analyte in question is calculated. Typically plus or minus three times the standard deviation from the mean of the retention time of each standard is generally used to define the retention time window.

In those cases where the standard deviation is zero, the standard deviation of a similar close eluting compound is used to develop a valid retention time window. The procedure and calculation methods are given in SW 846, Method 8000. This is not a hard and fast criteria, and often compounds and instrument behavior are more heavily used in the interpretation of chromatography and the establishment of retention time windows.

12.2.5 Compound Quantitation

Analyte concentrations in the sample are calculated from the response of those analytes used in the calibration procedures.

If an internal standard calibration procedure is used, the concentration (C) in the sample is calculated using the Response Factor (RF) ratio to the appropriate internal standard. RF is calculated for each analyte and surrogate using the following equations:

$$RF = \frac{(A_s)(C_{is})}{(A_{is})(C_s)},$$

where: A_s = Response of the analyte;
A_{is} = Response of the internal standard;
C_{is} = Concentration of the internal standard; and,
C_s = Concentration of the analyte to be measured.

and:

$$C = \frac{(A_s)(I_s)}{(A_{is})(RF)(V_o)},$$

where: I_s = Amount of internal standard added to each extract; and,
V_o = Volume or weight extracted or purged.

If the external standard calibration procedure is used, calculate the peak area response by using the calibration curve or the calibration factor determined from the initial calibration. The concentration (C) in the sample can then be calculated from the following equations:

$$C = \frac{(A)(V_t)}{(V_i)(V_s)},$$

where : A = Calculated peak area response;
V_i = Volume of extract injected;
V_t = Volume of total extract; and,
V_s = volume or weight of sample extracted.

12.3 DATA VERIFICATION (Review)

Technical third-party data validation reviews are not performed by the laboratory. However, Alpha does perform a technical data review/verification to assure the supporting analytical documentation is correct and the analysis was carried out in accordance with the data user's project specifications so that future third-party data validation reviews may be performed. Records are maintained by Alpha in sufficient scientific detail to recreate each extraction and analytical event during a sample's progress throughout the laboratory. Physical or hard-copy data records are assembled and maintained in the client file folder and scanned as a permanent digital pdf file for future electronic storage and data retrieval.

12.3.1 Data Integrity

The following is a list of the more important data records that are checked and maintained by Alpha:

a) Chain-of-Custody forms;
b) Extraction and Analytical Sequence Logbooks;
c) Instrument and Document Control Logbooks;
d) Initial and continuing calibration records;
e) Standard Preparations Logbooks;
f) Internal standard results;
g) Surrogate recovery charts;
h) Method blank analyses;
i) Matrix spike and matrix spike duplicate records and results;
j) Initial method demonstrations; and
k) Raw data including instrument printouts, chromatogram and quant reports.

Alpha maintains and uses written procedures for analytical QA/QC functions when appropriate.

12.3.2 Data Review

Before releasing any analytical data for reporting, it is our policy to review and verify the data has met the method criteria and is scientifically correct and/or has been footnoted properly describing those method criteria which were not met and are noted as deficiencies.

Data reviews include the evaluation of information, as presented by the analyst or staff member, for accurate representation of the samples submitted. Analytical data are generally subjected to a four-person tiered review before it is released with each successive check performed by a different staff person.

12.3.2.1 Analyst Review (Tier-1)

First the analyst will run, quantitate, and review analytes found in a particular sample or sample set. This includes reviewing and performing the following activities:

a) Calibrations, tunes, blanks, and any other instrument quality control criteria were met and were in-control during the sample analysis or analytical event;

b) Calculations of individual analytes and reporting limits were met;

c) Verify holding times or extraction times were met; and,

d) Make notes or footnotes on the quantitation report if abnormalities occurred during the sample analysis or describes any other QA/QC problems associated with the sample that should be documented.

12.3.2.2 QC/Peer Review (Tier-2)

This person may be another analyst, a person in the QC department, the person who performs the final QC upload or an assistant to the person signing the final report.

Samples pass through a two way QC review prior to final sample signature. The first half of this review includes a QC review of the calibration data and the other half of the review is a general QC review of all other QC batch data. Often times this review occurs simultaneously. These reviews are as follows:

a) Calibration Review - Initial calibrations, initial calibration verifications, and daily calibration verifications are reviewed for correctness against the method criteria or other in-house established criteria prior to releasing the analytical data associated with that particular calibration; and,

b) Batch QC Review - Once the data has been worked-up by the

analyst and the data has passed the first phase of the calibration review the data proceeds to the QC review department. The QC review person, then verifies that all dates, sample identification, reporting limits, reported analyte values, concentration units, header information, and footnotes or comments were transcribed accurately. All information on the final report that can be verified against the chain-of-custody is checked for errors and completeness.

12.3.2.3 General Data Review (Tier-3)

This person is typically a second person in the QC department, the person who performs the final QC upload or an assistant to the person signing the final report.

When this step is completed and the client file is a fully assembled, it will include the chain-of-custody records, chromatograms, quantitation report, QC reports, final reports etc. This person may review such things as:

a) Chain-of-Custody records were analytically followed;

b) Calculations and quantitation were performed correctly;

c) Analytical holding times were met;

d) Correct methods were used;

e) Quality control criteria were met;

f) Reporting limits were calculated properly;

g) Correct concentration units were reported; and,

h) Follow up and verify that any abnormalities which may have occurred during the analysis did not affect the final report. If abnormalities did occur, this person verifies the QA documentation and footnotes to determine if they are appropriate.

12.3.2.4 Final Review/Data Signature (Tier-4)

The Laboratory Director, Laboratory Manager, or QA Officer may at any time a problem is encountered, question the appropriate staff members and make determinations concerning the quality of the data.

Finally, the client file is checked and verified by the Laboratory Director or other designee who is signing final reports. This person spot checks analytical events and activities associated with the log-in,

tracking, extraction, sample analysis, and final reporting for technical and scientific soundness.

Many of the same activities reviewed by the Laboratory Director are also spot checked by the QA Officer, or designee as part of the internal quality assurance program. The QAO or the appointed designee reviews approximately 10% of all data for technical completeness and accuracy. Once this has been accomplished, the final reports or summary reports are signed indicating they have been approved for release to the client.

12.3.3 Quality Control Data Reporting

The results for each analyte in the spiked QC samples are calculated using the same calibration curve used for the analysis of samples in the associated analytical batch. Values less than the reporting limit are reported as "Not Detected" (ND).

QC data is typically reported in terms of accuracy and precision, which translates into percent recovery of spiked compounds and relative percent difference of spiked analytes between duplicate analyses. This is the most commonly accepted practice for all analytical data. Alpha reports QC data in several different formats depending on the type of analysis that the client or regulatory agency requests. We frequently modify or change QC summary reports to satisfy the requirements of a particular SOW.

12.3.4 Transcription and Calculation Errors

It is a primary goal of Alpha Analytical to minimize any and all errors associated with data reduction, review, validation and the reporting procedures including data transcription errors.

If transcription errors are discovered during any part of the data review process, those errors challenged by the reviewer are taken to the person where the error was first initiated or propagated and discussed. During this discussion, the alleged transcription error will either prove to be a transcription error and corrected or not.

If the alleged transcription error is found to be a true error than the following are required:

12.3.4.1 LIMS Transcription Errors

a) LIMS generated COC are amended with the correct information. The mistake is annotated in the comments section, with the name of the person making the correction, and a new blue colored COC is issued to all affected personnel.

b) The preferred procedure to correct a LIMS generated extraction batch report is to amend the original batch report with the correct information, dated and initialed by the extraction/digestion chemist. After the mistake is annotated, a mistake-free batch report is generated. The new batch report is attached to the old batch report as a way to track the initial error.

c) LIMS generated final reports are reissued with the correct information. The mistake is annotated as a footnote which clearly indicates the mistake, i.e., "This report replaces the report issued 1/10/11 due to a change of client ID"

12.3.4.2 Non-LIMS Transcription Errors

a) Instrument quantitation reports and any other non-LIMS generated data are amended (hand written) with the correct information, dated and initialed by the analyst.

12.4 DATA STORAGE

12.4.1 Client File Data Assembly

Following final data review, client files are organized in a manner to enhance future referencing of the data. Data files are organized by the DCO to facilitate easy data review and reconstruction of laboratory activities. Data files are generally organized in the following order:

a) Computer generated chain of custody;

b) Client chain of custody;

c) Sub-contract lab chain of custody (if present);

d) Work order information (if present);

e) Sample receipt checklist;

f) Sample receipt checklist fax confirmation;

g) Final Alpha analytical reports;

h) Alpha QA/QC data reports (if present);

i) Final sub-contract lab reports (if present);

j) Sub-contract lab QA/QC data reports (if present);

k) Alpha invoice-always make sure than an invoice is present;

l) Sub-contract lab invoices (if present);

m) Raw data;

n) Final report raw data, (the initials of the analyst should be indicated somewhere on all of the sheets and should be paper clipped together);

o) Screen reports, re-runs, etc. this is indicated on the top sheet and should be clipped together;

p) Air bill;

q) Correspondence; and,

r) Confirmation the report was faxed, e-mailed or scanned and a pdf copy sent.

If there were amendments made to the work order, put the amended COC on top of the previous COCs. Put amended final reports on top of other lab reports.

12.4.2 Archival Storage

12.4.2.1 Analytical Data

All analytical instrument data is permanently archived on the LIMS Server Disk as well as on Compact Disks (CD's) as a backup to hard copy data. The archival storage of data allows samples to be reevaluated at any time, providing proof of previous identifications, and the ability to search for other non-target compounds if GC-MS or ICP-MS data is being reevaluated.

12.4.2.2 Client File Data

All client data, which include final reports, calculation sheets, chains-of-custody, records, chromatograms, quantitation reports, correspondence, and other associated data, are maintained by Alpha Analytical, Inc. for no less than five years. This hard-copy data is stored on location at the laboratory for approximately one year. After this period of time, the data is scanned and stored electronically as a PDF file at an off-site location.

12.5 COLLECTIONS AND VALIDATIONS OF COMPUTERIZED DATA

12.5.1 In a computerized environment there are unique problems which must be considered in order to assure data integrity. Particularly when computers, automated equipment, or microprocessors are used for data acquisition, processing, recording, reporting, storage or retrieval or environmental test data.

12.5.2 The computerized data collection and handling systems used by Alpha are designed such that data entries and data files are uniquely identified so that data can be reliably stored and retrieved without loss. Additionally each datum is supported by at least one hard copy output or laboratory notebook entry.

12.5.3 It is the responsibility of the LIMS Administrator to ensure the computerized data handling systems are used by trained personnel such that data corruption is prevented. The LIMS Administrator is responsible for ensuring the following:

a) computer software developed by Alpha is documented in sufficient detail and is suitably validated as being adequate for use;

b) established procedures are implemented for protecting the data to include: integrity and confidentiality of data entry and/or collection, data storage, data transmission and data processing;

c) computers and automated equipment are maintained to ensue proper functioning and are provided with the environmental and operating conditions necessary to maintain the integrity of the test data; and

d) to establish and implement appropriate procedures for the maintenance of data security, including the prevention of unauthorized access to, and the unauthorized amendment of computer records.

Note: Commercial off-the-shelf software (e.g., word processing, database and statistical programs) used within their designated application range are considered sufficiently validated. However, analytical data acquisition software only needs to be validated initially, and again if modifications have been made to the software.

12.5.4 A complete description of Alpha's Software Quality Assurance Plan (SQAP) is found in Appendix F of the QAM. Specific items found in the SQAP are as follows:

a) Computer Software/Hardware Operations;
b) Data Collections and Storage;
c) Data File Uploading;
d) Electronic Diskette Deliverables (EDD's);
e) MSAccess 97 DataBases;
f) Data Archiving;
g) PC Server Integrity and Software Validation;
h) Sample Log-In; and
i) Sample Prep Omega SOP.

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Section 13

Corrective Actions and Control of Nonconforming Environmental Test Work

13.0 CORRECTIVE ACTIONS AND CONTROL OF NONCONFORMING ENVIRONMENTAL TEST WORK

13.0.1 Policy

13.0.1.1 It is Alpha's policy to designate the responsibilities and authorities for the management of nonconforming work and to define the necessary actions when nonconforming work is identified.

13.0.1.2 It is Alpha's policy to make an evaluation of the significance of the nonconforming work.

13.0.1.3 It is Alpha's policy to take immediate corrective actions, and make appropriate decisions regarding the acceptability of the nonconforming work.

13.0.1.4 It is Alpha's policy to notify the client when data quality has been impacted by nonconforming work and affected their data results.

13.0.1.5 It is Alpha's policy to define the responsibility for authorizing the resumption of work.

13.0.2 Summary

13.0.2.1 Analytical methods require strict adherence to initial calibration, calibration verification, defined accuracy and precision limits, as well as a host of other critical QC elements with prescribed criteria. These QC elements are continuously monitored by the analyst, supervisors and QA Officer to ensure they are conforming to the prescribed method criteria.

13.0.2.2 In an imperfect world, occasionally, one or more of these QC elements do not comply with the method and/or project defined criteria and are identified as a nonconforming sample. There are four primary areas where a sample may be noncompliant or nonconforming. They are generally described as:

a) the sample is nonconforming due to a sampling or sample receiving issue;
b) the sample is nonconforming due to a sample matrix effect issue;
c) the sample is nonconforming due to a sample preparation issue; or
d) the sample is nonconforming due to a instrument issue.

In addition, samples may be nonconforming due to a combination of the potential issues or causes as described above.

13.0.3 Types of Corrective Actions

13.0.3.1 The Laboratory Director, Laboratory Manager, analyst, and QA Officer may all be involved in corrective actions. Corrective actions are of two kinds:

13.0.3.1.1 Immediate, used to correct or repair non-conforming equipment and systems. The need for this type of action is most frequently identified by the analyst as a result of calibration checks and QC sample analyses.

13.0.3.1.2 Long-term, to eliminate causes of non conformance. The need for such actions is normally identified by audits. Examples of this type of action include:

a) staff training in technical skills or in implementing the QA Program;

b) rescheduling of laboratory operations to ensure analysis within allowed holding time;

c) identifying vendors who supply reagents of sufficient purity; and

d) revision of the QA Program or replacement of personnel.

13.0.4 Procedural Outline

13.0.4.1 The following procedure is implemented when samples are determined to be noncompliant either by method, by laboratory defined criteria, or by client requirements. The following outline describes this procedure:

13.0.4.1.1 the responsibilities and authorities for the management of nonconforming work are designated and actions (including halting of work and withholding test reports, as necessary) are defined and taken when nonconforming work is identified;

13.0.4.1.2 an evaluation of the significance of the nonconforming work is determined;

13.0.4.1.3 corrective actions are taken, together with any decision about the acceptability of the nonconforming work;

13.0.4.1.4 where the data quality is impacted, the client is notified; and

13.0.4.1.5 the responsibility for authorizing the resumption of work is defined.

13.0.5 Conclusion

13.0.5.1 A comprehensive discussion which would include a compendium of situations, various scenarios to resolve these issues, ways to improve or minimize these issues, the corrective actions associated with these issues, and the respective follow up is beyond the scope of this discussion.

However, the following discussion, identifies the key elements of our policy and defines the procedures to implement these findings in a manner which would correct and minimize nonconforming samples.

13.0.5.2 The following discussion is an extremely important aspect of environmental testing and is critical to improving our quality systems. If the evaluation of nonconforming work indicates the issue could reoccur or a systemic problem is discovered, the corrective actions described below are promptly followed.

13.1 CORRECTIVE ACTIONS

When, as a result of audits or QC sample analysis, analytical systems are shown to be unsatisfactory, a corrective action is implemented. Generally, quality control information is reviewed by several individuals. The responsibility for the initial assessment of the failed quality control measure lies with the analyst who identifies the problem with the sample or procedure and has access to the test results.

13.1.1 Initial QC Assessment and Assignment of Responsibility and Authority

The individual responsible for operating the analytical instrument is responsible for performing the first initial data review (Tier-1 review).

Table 13-1 identifies typical quality control checks that may be required by the various test methods. The typical acceptance range or the source of the acceptance range is also identified in this table. This table does not include sample receiving criteria but focuses on sample matrix, sample preparation and instrument criteria.

If one or more of these key QC elements fail, than the sample is noncompliant. The

root cause of the failure is investigated, and corrective actions are taken to resolve these issues. The following QC samples are typically reviewed by the analyst:

a) Calibrations, which may include initial calibration, initial calibration verification, continuing calibration verification and tuning standards when specified;

b) Blank, which may include, method or reagent blanks, equipment and trip blanks;

c) Spikes, which may include matrix spikes, control spikes, duplicate spikes; and

d) Surrogate and internal standards when required.

13.1.2 Secondary QC Assessment and Assignment of Responsibility and Authority

In addition to the analyst, the following controls are also reviewed by a second person (Tier-2 review) such as a QC assistant. This generally includes the following:

- standards,
- blanks,
- spiked samples (matrix and blank), and
- duplicates.

13.1.3 Corrective actions are ultimately the responsibility of the individual in oversight authority (i.e., supervisor, Laboratory Director, Laboratory Manager, or QA Officer).

13.1.4 Cause Analysis

Finding the source of a QC problem involves identifying probable sources of error and checking each source to determine if the protocols were properly followed. Common sources of error and expected follow-up protocols are outlined in Tables 13-2, 13-3, 13-4, and 13-5.

13.1.5 Selection and Implementation of Corrective Actions

When the source of a QC error has been identified, a corrective action is selected and implemented. The selection of a particular corrective action and its implementation is premised on the action(s) most likely to eliminate the problem and to prevent future recurrence.

Corrective actions are selected and implemented to match the degree that is appropriate to the magnitude and the risk of the problem.

GENERAL ACCEPTANCE CRITERIA FOR QUALITY CONTROL CHECKS TABLE 13-1	
QC CHECKS	**ACCEPTANCE CRITERIA**
Calibrations	
Initial Calibration	Method Acceptance Criteria
Initial Calibration Verification	
Continuing Calibration Verification	
Tuning	
Blanks	
Method Blanks	Method blank - < reporting limit and < 1/10 of the amount measured in any batch sample, unless it is a common laboratory solvent. Other blanks - < Reporting Limit or Client Defined
Reagent Blanks	
Equipment Blanks	
Trip Blanks	
Spikes	
Matrix Spikes	Method/Laboratory/Client Specified Accuracy Limits
Laboratory Control Samples	
Duplicates	
Laboratory Duplicates	Method/Laboratory/Client Specified Precision Limits
Matrix Spike Duplicates	
Field Duplicates	
Others	
Surrogates	Method /Laboratory/Client Specified Accuracy Limits
Internal Standards	Method Acceptance Criteria
Split Samples	Meets Precision Criteria

13.1.6 Documentation of Corrective Actions

13.1.6.1 If a quality control measure fails to meet acceptance criteria (i.e., immediate or short-term corrective actions), the QC measure and the procedures used to correct the problem is typically documented using analytical corrective action worksheets.

If system changes are required resulting from corrective action investigations (i.e., long-term corrective actions), these changes and the implementation of these changes are also documented.

13.1.6.2 The selection and implementation of corrective actions are documented by the appropriate individuals and procedures. For example, documentation does not imply a formal memo but may be documented in the following fashion:

a) Corrective actions that are initiated during an on-going analytical run typically documented on the chromatogram as well as in the instrument analytical log book; and

b) Corrective actions that require input or intervention of more than one individual is typically documented in the related log books and records.

Both of these types of immediate or short-term corrective actions are documented using the instrument corrective action worksheets.

13.1.6.3 If an identified quality control problem affects more than one set of data or multiple projects, then the documentation associated with identifying and resolving the problem is cross-referenced to all associated projects.

13.1.7 Monitoring of Corrective Actions

For either immediate or long-term corrective actions, a closed-loop corrective action system is employed comprising the following steps:

a) Define the problem;

b) Assign responsibility for investigating the problem;

c) Investigate and determine the cause of the problem;

d) Assign and accept responsibility for implementing the corrective action;

e) Establish effectiveness of the corrective action and implement the corrections; and

f) Monitor and verify that the corrective action has eliminated the problem.

Note: This is the same as described as our policy statement found and the procedural outline also described above.

The QA Officer and/or the Laboratory Director examine the occurrence of QC problems and the corrective actions employed to verify the problem has been eliminated. Historical corrective action items are periodically reviewed during the internal data quality audits to monitor conformity and to identify long-term trends or recurring problems.

13.2 Additional Audits

13.2.1 Internal Audits

When the identification of nonconformance or departures cast doubts on method and/or program compliance, or with policies and procedures established by Alpha, additional audits of the effected areas of activity are initiated as soon as possible. A detailed description of our audit policies and procedures are found in section 14.

13.2.2 External Audits

The need to initiate a corrective action may be the result of activities or audits from external sources. Sources include system audits, performance audits, split samples, blind QC samples, and findings from project or data validation review. A description of external audits policies and procedures are also found in section 14.

13.3 Technical Corrective Action

A generalized explanation of probable sources and expected corrective action for each QC measure is included below. A more detailed description is found in individual analytical SOPs in tables identified as Summary of Calibration Procedures and Summary of QC Procedures listing method specific calibration and batch QC criteria. Since many QC problems have unique solutions, the corrective action protocols are not limited to those listed below, and further assessment, based on an individual's experience and knowledge, may be warranted.

13.3.1 Calibrations

The first QC measure focuses on calibrations. Some probable sources of calibration QC problems are outlined in Table 13-2 as well as their expected review procedures.

SOURCES AND EXPECTED REVIEW PROCEDURES FOR CALIBRATIONS TABLE 13-2

SOURCES	EXPECTED REVIEW PROCEDURES
Improperly prepared or outdated standards	Review preparation logs for calculations or dilution errors and use of expired standards
Improperly prepared or outdated check standards	Verify check standard
Poor instrument response	Determine if preventative maintenance is required
Incorrect calculations	Review and verify all calculations
Contamination problems	See Table 13-3

The following is a description of expected corrective actions for calibrations:

a) Recalculate calibration curve;

b) Prepare fresh standards;

c) Re-calibrate instrument;

d) Perform preventative maintenance;

e) Perform mass calibration and retune the instrument;

f) Reanalyze all samples bracketing those from the previous acceptable QC check through the next acceptable QC check; and

g) Take measures to eliminate sources of contamination.

13.3.2 Blanks

The second QC measure focuses on blanks. While the goal is to have no detectable contaminants, each method blank is critically evaluated as to the nature of compound interferences and the effect on the analysis of each sample within the batch. The source of the blank contamination is investigated and measures are taken to minimize or eliminate the problem and the affected samples in the batch are reprocessed or the data is appropriately qualified if:

a) The concentration of a target analyte in the blank is at or above the reporting limit, AND is greater than 1/10th of the amount measured in the associated sample batch.

Note: If sample concentrations are significantly higher than the blank concentrations, or contaminants found in the blank are not detected in the sample, then report the sample data with no data qualifiers and flag the analytes in the blank appropriately.

b) The blank contamination otherwise affects the sample results as per the test method requirements or the individual project objectives.

c) When a blank is determined to be contaminated, the cause investigated, measures taken to eliminate the problem, then the samples associated with the contaminated blank are evaluated as to the best corrective action and may be reported with data qualifiers.

Measures are taken to determine the source of the problem and eliminate any future problems during the corrective action process. The following table describes some

probable sources of blank contamination and the procedures that should be reviewed during this corrective action review.

SOURCES AND EXPECTED REVIEW PROCEDURES FOR BLANKS TABLE 13-3

SOURCES	EXPECTED REVIEW PROCEDURES
Contaminated reagents	Verify reagent sources
Environmental cross contamination (sample collection, and sample and analysis conditions)	Review sample handling and storage protocols
Improper or incomplete laboratory or field decontamination procedures	Review cleaning protocols
Contaminated sample containers	Verify source and storage conditions
Contaminated source water	Verify water source

13.3.3 QC Spikes (Surrogates, Spikes and Internal Standards)

One of the central themes of our QA program is to document procedures for determining the effect of the sample matrix on method performance. Therefore, the third QC measure focuses on the analyses of matrix specific QC samples and are designed as data quality indicators (DQIs) for a specific sample using the designated test method.

The information from these controls are sample and matrix specific and are not normally used to determine the validity of the entire batch or to judge laboratory performance. Examples of matrix specific QC include:

a) matrix spike,
b) matrix spike duplicate, and
c) surrogate spikes.

While not universal, some auditors believe this list should also include the following with the list of matrix specific QC samples:

a) sample duplicates, and
b) internal standard spikes

The following table describes some probable sources of QC spike errors and the common corrective action procedures that should be reviewed during this review.

SOURCES AND EXPECTED REVIEW PROCEDURES FOR SPIKES, SURROGATE SPIKES, AND INTERNAL STANDARDS TABLE 13-4

SOURCES	EXPECTED REVIEW PROCEDURES
Error in calculation	Review and recheck all calculations
Error in preparing or using spike solution	Review all preparation logs and analytical logs for proper dilutions, solvents, etc.
Outdated standards	Review expiration dates and standard preparation logs
Contamination problems	See blanks above
Poor instrument response	Determine if preventative maintenance is required

The following is a description of expected corrective actions for nonconforming spikes, surrogate spikes, and internal standards:

a) Take measures to eliminate contamination problems, reprocess or reextract samples, and reanalyze as necessary;

b) Perform required maintenance and revise PM schedules;

c) Review preparations, calculations, and record keeping to determine if additional training or more stringent protocols are required; and,

d) If the sample matrix produces consistently unacceptable recoveries, and none of the sources discussed above are responsible for the problem, then the sample should be re-extracted and re-analyzed. If reanalysis produces the same results, then the associated samples should be reported with a footnote to qualify the results.

13.3.4 Duplicates

The fourth QC measure focuses on duplicate issues. The results of laboratory duplicate analyses, i.e., Laboratory Control Sample and Laboratory Control Sample Duplicate, are used to evaluate analytical or measurement precision, and the results of field or sample duplicate analyses are used to help evaluate precision of sampling, preservation and storage. Some probable sources of duplicate errors are outlined in Table 13-5 as well as their expected review procedures.

SOURCES AND EXPECTED REVIEW PROCEDURES FOR DUPLICATES TABLE 13-5

SOURCES	EXPECTED REVIEW PROCEDURES
Non-representative sample	Review sample collection procedures
Error in calculations	Recheck calculations
Contamination problems	See blanks above
Error in preparing or using spike solutions	Review all preparation logs and analytical logs for proper dilutions, solvents, etc.
Outdated standards	Review expiration dates and standard preparation logs
Poor instrument response	Determine if preventative maintenance is required

The following is a description of expected corrective actions for nonconforming duplicate analyses:

a) Report data with a footnote and explanation;

b) Revise sample collection or sample processing procedures to assure a representative sample;

c) Take measures to eliminate contamination problems; and

d) Reprocess and re-analyze the sample set.

13.4 CLIENT NOTIFICATION OF NONCONFORMITY

13.4.1 Nonconformance Associated with Sample Receiving

There are many potential levels of sample nonconformance which may be the result of the submitted sample or a laboratory error. In general, if a sample is received and noted that it contains a nonconforming item, then the client is notified of the samples nonconformance.

It is the responsibility of Alpha to identify and notify the client of the sample nonconformance. Subsequently once the client has been notified it is the responsibility of the client to determine the final status of that sample.

13.4.2 Nonconformance Associated with Analytical Data

13.4.2.1 It is Alpha's policy to report, to the extent possible, sample data only if all quality control measures are acceptable.

13.4.2.2 Nonconformance associated with analytical data production is not always black and white, but is an issue of the significance of the nonconformity.

13.4.2.3 Critical QC Elements

Analytical data associated with the following items are not released as final data until the nonconforming item is technically reviewed and if possible, all associated data is reanalyzed with the analytical QC in control or the data appropriately footnoted. These critical items are:

- BFB or DFTPP;
- Initial calibration;
- Calibration verification;
- Internal standards;
- Method blank; and
- LCS recovery.

13.4.2.4 Reporting Nonconforming Data Results Associated with Critical QC Elements

It is our first priority to identify the problem, correct the problem, and if possible, reanalyze all associated samples prior to releasing data.

a) If data are analyzed during a nonconforming situation, and it is impossible to reanalyze those affected samples; then it is the decision of the client, if possible, to decide the fate of that data.

b) If the data is to be released, then at a minimum all affected data are footnoted with a description of the failed QC parameters.

c) If the problem cannot be corrected in a timely manner they are reanalyzed on a second instrument. It is one of the primary objectives of our laboratory contingency plan to have redundant back-up instruments available for just this type of case.

13.4.2.5 Less Critical QC Elements

There are additional analytical QC items which are not as critical. These items are generally attributed to the sample matrix which may cause analytical QC parameters to fail. These less critical QC elements are described as follows:

- The ending calibration fails for one or more compounds;
- One or more surrogates are not recovered within the QC limits of acceptability; and
- Matrix Spike recovery values are not within the QC limits of acceptability.

13.4.2.6 Reporting Nonconforming Data Results Associated with Less Critical QC Elements

a) If these less critical QC criteria fails, then a decision is made regarding the scientific defensability, technical soundness, and end users data quality objectives, whether these samples will be reanalyzed or not.

b) Most of the time these problems are matrix related and reanalysis will confirm the nonconformity is matrix related by the repeated QC failure.

c) In either case this data is footnoted with a description of the failed QC parameters and the data is released.

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Section 14

System and Technical Audits

14.0 SYSTEMS AND TECHNICAL AUDITS

14.1 DEFINITIONS

14.1.1 Audit - a systematic evaluation to determine the operational quality of a particular system or function.

14.1.2 System audits - verify compliance with our laboratories quality system (e.g., QA Manual, Vol I and II) based on the NELAC Quality System. Examples of these types of audits would include audits such as sample acceptance policies, and sample tracking procedures.

14.1.3 Technical audits - verify compliance with method-specific requirements, as well as operations related to the test method (e.g., sample preparation).

Note: NELAP makes a distinction between the two types of internal audits, however, in practice most internal audits are verifying the operational quality of the entire laboratory (i.e., both system and technical audit elements are intertwined).

14.2 INTERNAL AUDITS

14.2.1 Internal audits are an independent check used to verify that laboratory policies and procedures continue to comply with the requirements of the quality system as defined by the NELAP standards and detailed in this QA Manual.

14.2.2 Internal audits are conducted to encourage staff members to adopt good quality assurance practices at all levels of the organization. Staff members are also encouraged to use these audits as an educational opportunity.

14.2.3 The internal audit program addresses all elements of the quality systems, including the environmental testing activities and is conducted on an annual basis.

14.2.4 It is the responsibility of the QA Officer to plan, organize and schedule internal audits. Audits not conducted by the QA Officer or carried out by trained and qualified personnel who are, wherever resources permit, independent of the activity to be audited.

Technical directors of the laboratory including the Laboratory Director, Laboratory Manager and QA Officer are qualified to carry out all internal audits. The technical staff to include analysts, extraction personnel and ancillary officers are qualified to carry out internal audits of the laboratory commensurate to their skill level.

14.2.5 These audits take into account reports from laboratory personnel, the outcome of recent internal audits, assessments of external auditing agencies, the results of inter-

laboratory comparisons or proficiency tests, changes in the volume and type of work undertaken, feedback from clients, corrective actions, and other relevant factors.

14.2.6 Audit Findings and Corrective Actions

14.2.6.1 When audit findings cast doubt on the effectiveness of the operations or validity of the test results, corrective actions are taken and clients are notified if investigations indicate results were affected. All internal audit review finding and any corrective actions that may arise from them are documented accordingly.

14.2.6.2 The time-frame for notifying a client is based on a number of potential considerations such as: a) the magnitude of the problem; b) the potential impact of the problem data; and c) the use of the data.

14.2.6.3 If corrective actions were taken, follow-up audits are conducted to verify and record the implementation and effectiveness of the corrective action taken.

14.2.7 Preventative Action

14.2.7.1 Preventive action is a pro-active process used to identify opportunities for improvement rather than a reaction to the identification of problems or complaints.

Section 13 is a description of corrective actions and is the reactionary process after a problem has been identified. This section, section 14 describes the audit and review process which, by its very nature, is a preventive action procedure.

14.2.7.2 Once potential sources of nonconformance, either technical or concerning the quality system, is identified, preventive action is taken. If preventive action is required, a plan of action is developed, implemented and monitored to reduce the likelihood of the occurrence of the nonconformance and to take advantage of the opportunities for improvement. The procedure as described in section 13, Corrective Action, is the same basic procedure as used for preventive actions.

14.3 INTERNAL SYSTEMS AUDITS

14.3.1 Quality Assurance Manual Audit

The EPA requires that each laboratory must have a written and approved Quality Assurance Manual as well as individual analytical Standard Operating Procedures. The EPA and NELAP specifies the QA Manual must present in specific terms, the

policies, organizations, objectives, functional activities, and specific QA/QC activities designed to achieve the data quality goals of specific methods or projects.

14.3.1.1 One of the primary duties and responsibilities of the QA Officer is to prepare, review, approve, revise and distribute the Quality Assurance Manual, Standard Operating Procedures and other technical documents and to ensure they are kept current. The QA manual, including Volume I and Volume II, is reviewed annually for accuracy and updated as appropriate. Reviews are documented and made available for assessment.

14.3.1.2 All staff members are required to attend annual training sessions covering selected material outlined in the chapters, or sections, contained in Volume I of the QA Manual. This required training is described in the training SOP. Internal system audits are typically conducted simultaneously with this training to encourage feedback and to address any shortcomings with the QA Manual and related laboratory procedures.

14.3.1.3 The documentation that the internal systems audits have been conducted regarding Volume I of the QA Manual, is outlined in Table 14-1 and is available for external audits and reviews.

14.3.2 Staff Audits

Training and auditing of the systems and procedures described in Volume II of the QA Manual are generally conducted directly with the staff members assigned to those procedures.

14.3.2.1 These audits are conducted with individual staff members covering the various aspects of their work or work related activities that have a bearing on the overall quality of the data produced. Internal staff audits consist of observations and verification to the adherence of approved practices and procedures. Deviations are noted and discussed with the affected staff members.

14.3.2.2 The documentation that the internal system audits have been conducted regarding Volume II of the QA Manual, is outlined in Table 14-2 and is available for external audits and reviews.

14.4 INTERNAL TECHNICAL AUDITS

14.4.1 Internal systems audits tend to focus on laboratory procedures rather than data quality. Therefore, these types of systems audits do not completely address and identify potential data quality issues and problems.

Data quality (technical) audits are conducted for the purpose of determining whether data of acceptable quality is being generated. There are three general types of internal technical audits:

- SOP document audits,
- Methods audits, and
- Data and records audits.

14.4.2 SOP documents are reviewed for accuracy and adequacy annually or whenever procedural method changes have occurred and updated as appropriate. Typically, method audits are conducted simultaneously with the SOP document audits. The documentation that the internal technical audits were conducted and their audit schedules are outlined in Table 14-3 and 14-4. The documentation of specific audit topics is outlined in Tables 14-5 and 14-6. These types of technical reviews are documented and made available for assessment.

14.4.3 Records and Data Audits

An internal records audit is conducted to verify the QA record systems is functioning properly and is being adequately filed and maintained for protection and accessability. Records audit are also conducted to ensure data packages as generated by Alpha are adequate and fulfill the QA deliverables package as requested by the client or project. The QA Officer typically reviews records to verify the following general items:

- QA contents,
- QA format, and
- Completeness of data package in relation to the appropriate deliverable requirement.

14.5 PERFORMANCE EVALUATION REVIEWS

14.5.1 Alpha Analytical participates in a proficiency-testing program to help ensure our laboratory has adequate quality control procedures in place for monitoring the validity of environmental test methods and procedures.

14.5.2 It is a policy of Alpha and a NELAC requirement that laboratories participate in two single-blind, single-concentrate Proficiency Testing (PT) studies, per year for each field of testing to maintain accreditation.

14.5.3 Performance Evaluation (PE) samples are purchased and prepared as follows:

14.5.3.1 Performance evaluation samples are obtained from a laboratory accredited as a provider of Proficiency Testing (PT) samples, under the auspices of the National Institute of Standards and Technology

(NIST), the USEPA and the National Voluntary Accreditation Program (NVLAP);

14.5.3.2 Aqueous samples are typically prepared in analyte-free water or prepared as whole volume samples by the PT provider, and soil samples are typically sent in a pre-spiked soil matrix.

14.5.3.3 These are blind PE samples; therefore, the analyst is not aware of the analyte concentration values in the PE audit sample. PE samples are inserted into the routine stream of laboratory sample analysis.

14.5.4 Performance Evaluation Findings and Corrective Actions

If Alpha's PE study results, determined by score of pass/fail criteria is deemed fully acceptable, corrective actions are not required. However, if Alpha's performance is determined to be unacceptable on any individual fractions, then corrective actions are taken to locate the problem, identify the problem, implement corrective actions and to document these corrective actions. Once the problem has been identified and the corrective action implemented, a remedial PE sample is analyzed for that fraction.

14.6 GENERAL REVIEWS

14.6.1 Internal audits and reviews are conducted not only on PE samples, QA documents and analytical procedures, but also audits are conducted on many other quality control procedures. These procedures are continuously reviewed to ensure quality data is being provided to clients. The following quality control procedures are regularly reviewed:

- Use of certified reference material or second source for QC sample analysis,
- Replicate testing such as quarterly QC samples, and/or annual DOC studies,
- Re-testing of retained samples, and
- Calculation of results for different parameters of a sample (i.e. comparing gravimetrically determined TDS results with a cross check calculation using conductivity data).

14.7 ANNUAL MANAGEMENT REVIEW

14.7.1 The analytical quality systems and all other ancillary quality systems are reviewed annually by management to ensure their continuing suitability and effectiveness. If systems are found to be ineffective; than this review will discuss and introduce necessary changes or improvements to the quality systems and/or laboratory operations.

14.7.2 This review takes into account:

a) the suitability of policies and procedures;

b) reports from management and supervisory personnel;

c) the outcome of recent internal audits;

d) corrective and preventive actions;

e) assessments by external bodies;

f) the results of inter-laboratory comparisons or proficiency tests;

g) changes in the volume and type of work undertaken;

h) client feedback;

i) customer complaints; and

j) other relevant factors such as quality control activities, resources and staffing.

14.7.3 The documentation that management reviews have been conducted are outlined in Table 14-7.

14.8 EXTERNAL AUDITS

14.8.1 External Systems Audit

External audits are conducted by individual clients or the various regulatory agencies (i.e., state certifying agencies, EPA regional agencies, etc.). This is an on-site inspection and review of our quality control system. Their visit to the laboratory is to review and discuss any shortcomings and discrepancies in an actual sample walk through. Audits performed by an external Quality Assurance Officer normally will address all applicable elements of this QA Plan or contract requirements as it pertains to their QAPP or SOW. It is Alpha's policy to comply fully with audits conducted by certifying agencies, regulatory agencies and clients.

14.8.2 External Performance Audits

External performance audits are Performance Evaluation (PE) samples submitted and analyzed as unknown sample concentrates as double-blind samples. These PE samples are typically obtained from proficiency test (PT) providers and submitted as samples within the clients normal sample stream.

Internal System Audit
Document Review
QA Manual Volume I
Table 14-1

Section	Audit Schedule (suggested)	Year		Year Additional Review (if required)	
		Review		Review	
		Date	Revision No.	Date	Revision No.
Section 1	January (week 1)				
Section 2	January (week 1)				
Section 3	January (week 1)				
Section 4	January (week 3)				
Section 5	January (week 3)				
Section 6	February (week 1)				
Section 7	February (week 1)				
Section 8	February (week 3)				
Section 9	February (week 3)				
Section 10	March (week 1)				
Section 11	March (week 1)				
Section 12	March (week 3)				
Section 13	March (week 3)				
Section 14	April (week 1)				
Section 15	April (week 1)				
Section 16	April (week 1)				
Section 17	April (week 1)				
Section 18	April (week1)				

Comments:

Internal System Audit
Document Review
QA Manual Volume II
Table 14-2

Section	Audit Schedule (suggested)	Year		Year Additional Review (if required)	
		Review		Review	
		Date	Revision No.	Date	Revision No.
Appendix A	November (week 1)				
Appendix B	November (week 2)				
Appendix C	April (week 3)				
Appendix D	November (week 4)				
Appendix E	Jan./April (week 3)				
Appendix F	December (week 2)				
Appendix G	January (week 1)				
Appendix H	December (week 4)				

Comments:

Internal Technical Audit
Document Review
QA Manual Volume III
Table 14-3

SOP	Method	Comments	Audit Schedule (suggested)	Year		Year Additional Review (if required)	
				Review		Review	
				Date	Revision No.	Date	Revision No.
E.20	524.2		May (week 1)				
E.30	Pesticides		April (week 2)				
E.31	PCBs		April (week 4)				
E.33	VOCs		May (week 4)				
E.34	SVOCs		June (week 1)				
E.35	PNA (SIMs)		June (week 2)				
E.36	Alcohols (SIMs)		June (week 3)				
E.37	TPH-DRO		June (week 4)				
E.38	TPH-GRO		July (week 1)				
E.50	TCLP		July (week 2)				
E.51	SPLP		July (week 3)				
E.52	STLC		July (week 4)				
E.55	ASE (3545)		July (week 4)				
E.56	Liq-Liq (3510)		July (week 4)				

Comments:

Internal Technical Audit
Document Review
QA Manual Volume III
Table 14-4

SOP	Method	Comments	Audit Schedule (suggested)	Year		Year Additional Review (if required)	
				Review		Review	
				Date	Revision No.	Date	Revision No.
E.60	Metals		August (week 1)				
E.64	Organic Acids		August (week 3)				
E.65	Anions		August (week 4)				
E.66	Perchlorate		August (week 4)				
E.67	TOC		August (week 3)				
E.70	3015		August (week 2)				
E.71	3051		August (week 2)				
E.72	200.2		August (week 2)				
E.75	Conductivity		September (week 1)				
E.76	pH		September (week 1)				
E.77	Ammonia/TKN		September (week 2)				
E.78	Turbidity		September (week 2)				
E.80	TDS/TSS/TS		September (week 3)				
E.81	% Moisture		September (week 3)				
E.82	1664A		September (week 4)				
E.85	Alkalinity/Acidity		September (week 4)				
E.90	Chromium VI		October (week 1)				
E.92	Sulfide		October (week 1)				
E.93	Chlorine		October (week 2)				
E.94	COD		October (week 2)				
E.95	Total-P		October (week 3)				
E.96	Fe		October (week 3)				

Comments:

Internal Data Quality Audit
Checklist
Table 14-5

Item	Date Completed	Comments	Initial
1) Method review			
2) CAR external auditiors review			
3) SOP review			
4) IDC review			
5) MDL review			
6) Standards review			
7) Data Package review			
8) PE review			
9) Data Quality Audit review			
10) Analyst training			
11) Report review			
12) DQO review			
13) QC Summary Report review			
14) Bench records review			
15)			
16)			

Comments:

Internal Data Quality Audit
IDC/MDL Review
Table E.14-6A

Date:_____ Method:_____
Analyst: _____ Instrument:_____
Peer: _____ Matrix:_____

DESCRIPTION	Y/N/NA	Corrective Action Due Date	Corrective Action Completion Date	Comments
1) Initial Demonstration of Capability				
a) Was an IDC performed for this method by this analyst?				
b) Was each target analyte included at the required concentrations?				
c) Was each target analyte evaluated for spike value, found value, average percent recovery, standard deviation and percent relative standard deviation?				
d) Does the average percent recovery and standard deviation of each analyte meet the method specified acceptance range?				
e) Is the IDC documented on the standard form with the correct information and required signatures?				
2) Method Detection Limits				
a) Was an MDL study performed for this method by this analyst?				
b) Was each target analyte and data point included?				
c) Was the MDL study generated using the preparatory and cleanup procedures routinely used on samples?				
d) Were any data points deleted from the MDL study?				
e) Was each target analyte evaluated for spike value, found value, average percent recovery, standard deviation and calculated MDL?				
f) Is calculated MDL higher than the spike concentration, and if so was the study repeated at a higher concentration?				
g) Is the calculated MDL greater than 1/10 of the spike concentration, and if not was the study repeated at a lower concentration.				
h) Was a MDL verification check standard analyzed at approximately 2 times the MDL? And was the check standard detected or have a signal to noise ratio greater than 3?				
i) Is the calculated MDL higher than the LOQ, and if so was the study repeated at a lower level or the LOQ elevated above 3 times the MDL?				
j) Was a LOQ verification check standard analyzed at 1-2 times the LOQ and if so were the recoveries within the LCS windows of acceptability?				

Internal Data Quality Audit
Calibration Review
Table E.14-6B

Date:_____ Method:_____
Analyst: _____ Instrument:_____
Peer: _____ Matrix:_____

DESCRIPTION	Y/N/NA	Corrective Action Due Date	Corrective Action Completion Date	Comments
3) Initial Calibration Data				
a) Was an initial calibration performed for each target analyte?				
b) Were all target analytes included in the calibration standard?				
c) Were the concentration values used for each analyte in the calibration table or curve appropriate for the method and do the concentration values in the method table match the actual standard concentrations?				
d) Are analytes reported by GC/MS assigned the right characteristic ions or isotopes by ICP/MS?				
e) Are GC or IC retention time windows correctly calculated?				
f) Is a second GC confirmation column or alternate detector used for analyte confirmation?				
g) Are multi response analytes being evaluated for pattern match?				
h) Was peak integration performed properly with no indication of improper data manipulation?				
i) Were all manual integrations properly documented with a before and after chromatogram and appropriate explanation?				
j) Does the low level standard have a signal to noise response greater than 3?				
k) Are integration routines used on calibration points acceptable?				
l) Do calibration points either high or low need to be deleted from the IC?				
m) Does IC meet method criteria for the chosen mathematical model?				
n) Are the same instrument conditions used for IC the same used for production analysis?				

Notes:

Internal Data Quality Audit
Calibration Review
Table E.14-6B
(Continued)

Date:_____ Method:_____
Analyst: _____ Instrument:_____
Peer: _____ Matrix:_____

DESCRIPTION	Y/N/NA	Corrective Action Due Date	Corrective Action Completion Date	Comments
4) Instrument Performance Check (IPC)				
a) Was the appropriate IPC analyzed? 1) BFB or DFTPP tune standard for GC/MS 2) Metals tune standard for ICP/MS 3) GC or IC method specific IPC	 1) 2) 3)			
b) Was the GC/MS or ICP/MS tune standard analyzed and checked each 12 hours of sample analysis or method required frequency?				
c) For GC/MS are ion abundance and relative ion abundance within the method acceptance criteria?				
d) For ICP are mass calibration, resolution and RSD within method criteria?				
e) For IC or GC methods, does the ICP meet method criteria?				
5) Initial Calibration Verification (ICV)				
a) Was an ICV performed using a standard independent from the IC?				
b) Was a midpoint concentration value used for the ICV?				
c) Are the % recovery values acceptable? (Typically evaluated against the CV criteria?				
6) Calibration Verification (CV)				
a) Are the concentration values appropriate for the CV?				
b) Are high and low concentration values being used for GC methods?				
c) Do assigned concentration values match the actual concentration values provided with the calibration standard?				
e) Are % difference or % recovery values for each target analyte or CCC within method criteria?				

Notes:

Internal Data Quality Audit
Data Package Review
Table E.14-6C

Date:_____ Method:_____
Analyst: _____ Instrument:_____
Peer: _____ Matrix:_____

DESCRIPTION	Y/N/NA	Corrective Action Due Date	Corrective Action Completion Date	Comments
7) Method Blank (MB) [Negative Control]				
a) Are analytes present above the LOQ in the blank? If so did this affect any of the associated samples?				
8) Laboratory Control Spike (LCS) [Positive Control]				
a) Are the appropriate analytes and spike levels included in the LCS?				
b) Was the LCS prepared independently from the IC or from a second source?				
c) Do the assigned analyte concentrations match the values from the source?				
d) Do the % recovery range of each analyte compare with method stated or laboratory derived acceptance ranges?				
9) Matrix Spike (MS) [Sample Specific Control]				
a) Is the same spike mix and spike levels used in the LCS also used in the MS?				
b) Do MS/MSD analytes meet acceptable RPD criteria?				
10) Sample Data				
a) Are surrogates and internal standards recoveries within the method criteia?				
b) If not was sample re-analyzed or was sample data appropriately footnoted?				
c) Were second column confirmation, GC retention time windows, elution order, and pattern recognition criteria correctly used for reported analytes by GC?				
d) Were concentrations for found analytes calculated and reported correctly?				
11) Standards Preparation				
a) Are all standards traceable to the Certificate of Analysis?				
b) Were all standards completely documented with lot numbers, expiration dates, correct concentration units, solvents, initials etc.?				
c) Was the DOC/MDL/ICV/LCS/MS prepared and clearly documented from a standard source independent from the IC?				

Management Review
Table 14-7

Date:_____

Personnel Present:_____/_____/_____/_____

Review Items	Specific Items of Interest	Comments
a) Suitability of policies and procedures		
b) Reports from managerial and supervisory personnel		
c) Outcome of internal audits		
d) Corrective and preventive actions		
e) Outcome of external audits		
f) Results of PE data results		
g) Changes is volume and types of work		
h) Client feedback/complaints		
i) Other		
Recommended Changes		

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Section 15

Quality Assurance Reports

15.0 QUALITY ASSURANCE REPORTS

15.0.1 Quality assurance reports are designed to keep staff members informed of the performance of QA/QC activities. Quality assurance reporting documents the quality control and quality assurance activities in the laboratory and provides communications and an accountability link among analysts, management and clients. Analytical report formats, which include selected quality control data and evaluations, are referred to as Quality Control Reports or QA deliverables. These provide a direct link between the analyst and the data user concerning the quality of the data.

15.0.2 In some instances, projects need to pay attention to particular quality assurance controls and assessment. In such situations Alpha will provide technical guidance and, if requested, periodic quality assurance reports to keep the project on target toward achieving those quality goals. These reports, both verbal and written, may include subjects that address the validity and documentation of data generation activities.

15.0.3 QA reports typically list significant problems and discuss the solutions and corrective actions implemented concerning QA/QC activities. QA reports may include such things as internal and external audits, QA/QC summary data sheets associated with a batch of analytical samples, PE sample results, etc.

15.1 QUALITY CONTROL REPORTING

15.1.1 Data reported for quality control, including MDL studies will have at least three significant figures unless specified otherwise in the method (i.e., any analytical result being used for quality control calculations should carry three significant figures).

15.1.2 Reported quality control data needs to be expressed in the proper units, i.e. the same units as the samples.

15.1.3 A typical final analytical report does not include quality control information. That information is most typically reported with its associated batch or summary QC data report.

15.2 INTERNAL QA REPORTS

15.2.1 Internal QA reports are distributed as necessary to keep staff members informed of the performance of QA/QC activities. This information is provided in the form of verbal communication, formal memorandums, or reports to ensure sound QA/QC practices.

15.3 DATA DELIVERABLES

15.3.1 In some specific situations, samples from a project may need to be technically

evaluated by a third party review. Methods of analysis, SOW, QAM and other documents are used to assist the evaluator in the technical review of analytical data generated by our QA program.

15.3.2 Data deliverables is the process of gathering and collating analytical data in a manner which helps organize the data in a logical sequence. Table 15-2 through 15-5, gives guidance on collating and organizing a data deliverables package. Data deliverables may be project specific; therefore, it is important to organize the data deliverables specific to the SOW.

QA Summary Reports
DQO Reference Table 15-1

Method	Surrogate Window of Acceptability	LCS Window of Acceptability	MS Window of Acceptability	Matrix
200.8	Not Required	Method Specified	Method Specified	Water
524.2	Method Specific	Method Specified	Not Required	Water
608	Laboratory Derived	Method Specified	Method Specified	Water
624	Laboratory Derived	Method Specified	Method Specified	Water
625	Laboratory Derived	Method Specified	Method Specified	Water
6020	Not Required	Laboratory Derived	Laboratory Derived	Water/Soil
8015B-DRO	Laboratory Derived	Laboratory Derived	Laboratory Derived	Water/Soil
8015B-GRO	Laboratory Derived	Laboratory Derived	Laboratory Derived	Water/Soil
8081A	Laboratory Derived	Laboratory Derived	Laboratory Derived	Water/Soil
8082	Laboratory Derived	Laboratory Derived	Laboratory Derived	Water/Soil
8260B	Laboratory Derived	Laboratory Derived	Laboratory Derived	Water/Soil
8270C	Laboratory Derived	Laboratory Derived	Laboratory Derived	Water/Soil

Example
GC/MS Level IV Deliverables
Table 15-2

Item #	Deliverables	Page
1	Case Narrative	Page_____ through _____
2	Table of Contents	Page_____ through _____
3	Chain of Custody	Page_____ through _____
4	Sample results with analysis and extraction/preparation dates	Page_____ through _____
5	Raw data which includes chromatograms and quantitation reports	Page_____ through _____
6	Summary of MS/MSD/Duplicate recoveries and control limits (linking native samples)	Page_____ through _____
7	Raw data associated with the MS/MSD/Duplicate which includes chromatograms and quantitation reports (linking native samples)	Page_____ through _____
8	Summary of LCS recoveries and control limits	Page_____ through _____
9	Raw data associated with the LCS which includes chromatograms and quantitation reports	Page_____ through _____
10	Summary of method blank results	Page_____ through _____
11	Raw method blank data which includes chromatograms and quantitation reports	Page_____ through _____
12	Summary of internal standard areas/RT's, and summary of surrogate recoveries	Page_____ through _____
13	Summary of initial calibration data (RF, and %RSD)	Page_____ through _____
14	Raw data associated with the initial calibration which includes chromatograms, quantitation reports, and the calibration plots, indicating correlation coefficients if required	Page_____ through _____
15	Summary of continuing calibration data (% Difference reports from calculated concentrations, and from RRF)	Page_____ through _____
16	Raw data associated with the continuing calibration which includes chromatograms and quantitation reports	Page_____ through _____
17	Summary of instrument tuning (listing associated samples and injection times) for all applicable analytical shifts, including those in which initial calibration levels, QC samples, and client samples were analyzed	Page_____ through _____
18	Instrument sequence/injection logs	Page_____ through _____
19	Extraction/preparation logs and sample dilution logs	Page_____ through _____

Example
ICP/MS Level IV Deliverables
Table 15-3

Item #	Deliverables	Page
1	Case Narrative	Page_____ through _____
2	Table of Contents	Page_____ through _____
3	Chain of Custody	Page_____ through _____
4	Sample results with analysis and digestion/preparation dates	Page_____ through _____
5	Raw data which includes quant reports	Page_____ through _____
6	Summary of MS/MSD/Dup recoveries and control limits (linking native samples)	Page_____ through _____
7	Raw data associated with MS/MSD/Dup which includes quant reports (linking native samples)	Page_____ through _____
8	Summary of LCS/LCSD recoveries and control limits	Page_____ through _____
9	Raw data associated with LCS/LCSD which includes quant reports	Page_____ through _____
10	Summary of method blank results	Page_____ through _____
11	Raw method blank data which includes quantitation reports	Page_____ through _____
12	Summary of IC data (CPS, and Linear Regression Equations)	Page_____ through _____
13	Raw data associated with the ICwhich includes quant reports and the cal plots, indicating correlation coefficients if required	Page_____ through _____
14	Raw data associated with ICV which includes quant reports.	Page_____ through _____
15	Raw data associated with the ICSA/ICSB which includes quantitation reports.	Page_____ through _____
16	Raw data associated with ICB/CCB which includes quant reports.	Page_____ through _____
17	Summary of continuing calibration data	Page_____ through _____
18	Raw data associated with the CV which includes quant reports	Page_____ through _____
19	Summary of instrument tuning	Page_____ through _____
20	Instrument sequence/injection logs	Page_____ through _____
21	Digestion/preparation logs	Page_____ through _____

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Section 16

Laboratory Reports and Reporting Procedures

16.0 LABORATORY REPORTS AND REPORTING PROCEDURES

16.1 DATA REPORTING

16.1.1 Policy

It is Alpha's policy to report environmental test results accurately, clearly, unambiguously and objectively, and in accordance with any specific instruction in the test method. Analytical test data is reported on a final analytical test report which summarizes all the information requested by the client and that which is necessary for the interpretation of the test results and all information required by the method.

16.1.2 Test Reports

The generation of analytical data requires the use of computerized data systems for acquisition, storage, retrieval, and reporting of data. After the data has been acquired, reduced, and reviewed, it is then transcribed onto a final analytical report. For most analytical final reports, the report will include the following information:

a) Title: "Analytical Report,"

b) Alpha's Name and Address,

c) A unique serial number (project identification) at the bottom of the report, and page number to ensure that the page is recognized as a part of the test report,

d) Title of project or job number, to include:

   i. Client address,
   ii. Client phone number,
   iii. Point of contact,

e) Analytical method,

f) Sample identification, to include:

   i. Alpha Analytical identification number,
   ii. Client identification number,

g) Dates, to include:

   i. Date and time sample collected,
   ii. Date and time sample received,
   iii. Date sample analyzed,

Note: If the method specified time of sample extraction or analysis is less than 48 hours from sample collection, than both the date and time analyzed are documented on the final report.

h) Sample matrix,

i) Test results, to include:

i. Reporting limits,
ii. Concentration units,
iii. List of compounds analyzed,
iv. Observations or comments such as failed quality control,

j) Signature or approval block, and date of issue,

k) A statement at the bottom of the analytical report that the test results meet all of the requirements of NELAC unless footnoted otherwise, and

l) Alpha holds the appropriate certifications for the reported data.

16.1.3 Test Report Description

16.1.3.1 Footnotes or Data Qualifiers

Data is reported when the sample analysis occurred during periods that the calibration and systems were in-control. If a quality control measure is found to be out-of-control, and the data are to be reported, then the failed QC measure is reported with the appropriate data qualifiers.

16.1.3.2 Concentration Units

All numerical results are reported in terms of concentrations (i.e., ug/Kg for soils or ug/L for waters) in the environmental sample.

16.1.3.3 Reporting Limits

Reporting limits are required for all methods to evaluate method performance. All values less than the reporting limit are reported as Not Detectable, "ND". Reporting limits are dependent on the matrix of the sample that is being tested. Interferences frequently require sample dilution, which may change the analytical reporting limit.

16.1.4 Test Report Format

16.1.4.1 Final reports and reporting formats are designed to facilitate the analytical data review process. These reports are formatted for specific regulatory programs and designed to display information accordingly.

16.1.4.2 Final reports may contain multiple analyses from a set of samples with the same type of analysis on a single report.

16.1.4.3 Final reports may contain analytical information from several methods of analysis from the same sample.

16.1.4.4 Reports can be customized to virtually any client request or QAPP specific request. Not only can reports be customized, data can also be reported on a number of different spreadsheets or databases (i.e., Excel) and transmitted electronically.

16.1.5 Amendments to Test Reports

Any amendments to an analytical report, after it has been issued, are clearly stated that the report is an amended report. The amended report clearly states the following:

a) that the amended report replaces the original report with the date of issue of the original report in order to avoid any possible confusion; and

b) the amended report clearly indicates the reason for amending the original report and is usually expressed as a footnote.

16.1.6 Analytical Reports Signature Block

The following personnel have the authority to sign final analytical data reports and to approve or disapprove other final analytical data reports that do not require a signature block such as Electronic Data Deliverables (EDD) or other data packages.

1) Laboratory Director,
2) Quality Assurance Officer, and
3) Laboratory Manager

16.1.7 Verbal Analytical Data Release

The following additional personnel have the authority to verbally release final analytical data to clients that has previously been signed:

1) Director of Client Services,

2) Project Coordinators,
3) Supervisors, and
4) LIMS Administrator

16.2 SIGNIFICANT FIGURES

16.2.1 Definition

Significant figures are the count of the number of digits in a number which properly represents a measured quantity when the number is expressed in scientific notations.

16.2.2 Purpose

When properly rounded, the result of a measurement is expressed so that the last digit remaining shows where the uncertainty in the measurement begins. The nature of the measurement process, accounting for all cumulative errors from consecutive operations, determines the number of significant figures.

16.2.3 Methods

There are two methods used to signify uncertainty. The most definitive technique is to follow the measured value with the ± sign and the amount of uncertainty (e.g., 10 ± 2). The less informative and more widely used method is to indicate the degree of uncertainty by the number of significant figures in the reported value. The last figure shown is the one in which there is uncertainty.

16.2.4. Procedure

The proper use of the concept of significant figures requires adherence to some conventions.

16.2.4.1 Rules of Significant Figures

Zeros may be significant digits, for example 5<u>0</u>, 2.<u>0</u>, or 5<u>0</u>5. However, there are two different functions for which zeros are not considered significant digits.

a) Zeros may flag a decimal, for example <u>0</u>.52.

b) Zeros may also define magnitude, for example 50<u>0</u>, 5<u>0</u>, or 0.<u>0</u>5.

When reporting data which follows the conventions for significant figures, zeros will not be added to make each number have the same number of digits past the decimal point (i.e., we report 520 not 520.0)

16.2.4.2 Rounding Rules

In reporting results, rounding to the correct number of significant figures occurs only after all calculations and data manipulations are completed. Premature rounding can significantly affect the final result. When the calculation or instrument gives more figures than needed, it is necessary to round off. The following rules shall be used:

Rule 1: If the next digit beyond the rounding point is less than 5, leave the previous digit unchanged (e.g., 21.4 becomes 21).

Rule 2: If the next digit beyond the rounding point is greater than 5, increase the previous digit by one (e.g., 21.6 becomes 22).

Rule 3: If the next digit beyond the rounding point is equal to 5, with no digits other than zero following the 5, round the previous digit to the nearest even number (e.g., 21.5 and 22.5 both become 22).

Rule 4: If the next digit is a 5 followed by other digits, then treat the case as in rule 2 - for greater than 5 (e.g., 21.51 becomes 22).

Rule 5: If there are not enough numbers to get to the required number of significant figures, for example 2.3 when working with three significant figures, do not add extra zeros.

Rule 6: When performing calculations, carry at least one extra significant figure through the process and round only the final result. Rounding data before a calculation introduces a cumulative error. Carrying at least one extra digit minimizes this error.

16.3 CONTRACT LABORATORY PROGRAM (CLP) DATA QUALIFIERS

16.3.1 To the extent possible, samples are reported only if quality control measurements are acceptable. If a quality control measure is found to be out of control, and the data are to be reported, the failed QC measure is reported with the appropriate footnote or data qualifier.

16.3.2 Many times CLP data qualifiers do not accurately describe or represent the associated

sample problem. In these cases custom footnotes or footnotes requested by a state agency which more accurately describe the sample situation is appended to the bottom of the associated sample reports.

16.3.3 The CLP describes a set of data qualifiers that are often requested by the end user. The following definitions provide brief explanations of the CLP qualifiers assigned to results in the data review process. Additional data qualifiers may be requested by data validators when evaluating data usability.

CLP Data Qualifiers	
Flag	Description
U	The analyte was analyzed for, but was not detected above the reported sample quantitation limit.
J	The analyte was positively identified; the associated numerical value is the approximate concentration of the analyte in the sample.
N	The analysis indicates the presence of an analyte for which there is presumptive evidence to make a "tentative identification".
NJ	The analysis indicates the presence of an analyte that has been "tentatively identified" and the associated numerical value represents its approximate concentration.
UJ	The analyte was not detected above the reported sample quantitation limit. However, the reported quantitation limit is approximate and may or may not represent the actual limit of quantitation necessary to accurately and precisely measure the analyte.
R	The sample results are rejected due to serious deficiencies in the ability to analyze the sample and meet quality control criteria. The presence or absence of the analyte cannot be verified.
B	Blank contamination, analyte was detected above the method reporting limit in an associated blank.

16.4 DEPARTMENT OF DEFENSE (DoD) DATA QUALIFIERS

16.4.1 The DoD describes a set of data qualifiers that are similar to the CLP qualifiers but are worded slightly differently. The following definitions provide brief explanations of the DoD qualifiers assigned to results in the data review process.

DoD Data Qualifiers	
Flag	Description
U	Analyte was not detected and is reported as less than the LOD or as defined by the client. The LOD as been adjusted for any dilution or concentration of the sample.
J	The reported result is an estimated value.
N	Nontarget analyte. The analyte is a tentatively identified compound (using mass spectroscopy).
B	Blank contamination. The recorded result is associated with a contaminated blank.
Q	One or mare quality control criteria failed.

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Section 17

List of Acronyms and Abbreviations

17.0 LIST OF ACRONYMS AND ABBREVIATIONS

A

AAI	Alpha Analytical, Inc.
ACS	American Chemical Society
ALS	Automatic liquid sampler
A2LA	American Association for Laboratory Accreditation
AIHA	American Industrial Hygiene Association
ANSI	American National Standards Institute
APHA	American Public Health Association
ASCII	American Standard Code Information Interchange
ASE	Accelerated solvent extraction
ASQ	American Society for Quality
ASQC	American Society for Quality Control
ASTM	American Society for Testing and Materials
AWWA	American Water Works Association

B

BFB	4-bromofluorobenzene
BLK	Blank
BN	Base/neutral
BNA	Base/neutral acid
BOD	Biological oxygen demand
BTEX	Benzene, toluene, ethyl benzene, xylene

C

C	Concentration
CAS	Chemical Abstract Service
CCC	Calibration check compounds
CCV	Continuing Calibration Verification
CV	Calibration verification
CERCLA	Comprehensive Environmental Response, Compensation, and Liability Act
CF	Calibration factor
CFR	Code of Federal Regulation
CLP	Contract Laboratory Program
COC	Chain-of-Custody
COD	Chemical oxygen demand

D

DAD	Diode-array detector
DCBP	Decachlorobiphenyl
DCO	Document control officer
DDD	Dichlorodiphenyldichloroethane

DDE	Dichlorodiphenyldichloroethene
DDT	Dichlorodiphenyltrichloroethane
DFTPP	decafluorotriphenylphosphine
DHS	Department of Health Services
DI	De-ionized
DL	Detection Limit
DOC	Demonstration of Capability
DoD	Department of Defense
DQO	Data quality objective
DRO	Diesel range organics

E

EC	Electrolytic conductivity
ECD	Electron capture detector
EDB	Ethylene dibromide
EDD	Electronic diskette deliverables
EDL	Estimated detection limit
EICP	Extracted ion current profile
EMSL	Environmental Monitoring Support Laboratory
EPA	Environmental Protection Agency
EQL	Estimated quantitation limits
ERM	Environmental Resource Management

F

FID	Flame ionization detector
FRB	Field reagent blank
FSP	Field sampling plan
FTB	Field transfer blank

G

G	Glass
GC	Gas chromatography
GC/MS	Gas chromatography/mass spectroscopy
GLP	Good laboratory practice
GRO	Gasoline range organics
GS	Gas spike

H

HCl	Hydrochloric acid
HDPE	High density polyethylene
HNO_3	Nitric acid
HP	Hewlett Packard
HPLC	High-performance liquid chromatography
H_2SO_4	Sulfuric acid

I

IB	Instrument blank
IC	Inorganic carbon
ICL	Inner control limit
ICP	Inductively coupled plasma
ICP/MS	Inductively coupled plasma-mass spectrometer
ICS	Interference check standard
ICV	Initial calibration verification
ID	Identification
IDC	Initial demonstration of capabilities
ILAC	International Laboratory Accreditation Cooperation
IOC	Inorganic compounds
IPC	Instrument performance check
IR	Infrared
IS	Internal standard
ISO	International Organization for Standardization

K

K-D	Kuderna-Danish

L

LC	Liquid chromatography
LCL	Lower control limit
LCS	Laboratory control sample
LCS-CL	LCS Control Limit
LFB	Laboratory fortified blank
LFM	Laboratory fortified matrix
LIMS	Laboratory information management systems
LLD	Lower Limit of Detection
LOD	Limit of Detection
LOQ	Limit of Quantitation
LPC	Laboratory performance check sample
LRB	Laboratory reagent blank
LSC	Liquid sample concentrator
LUFT	Leaking underground fuel tanks

M

MB	Method blank
MBAS	Methylene blue active substances
MDL	Method detection limit
ME	Marginal Exceedance
MeOH	Methanol
$MeCl_2$	Methylene chloride

MRL	Method reporting limit
MS	Matrix spike
MSD	Matrix spike duplicate
MSD	Mass selective detector
MTBE	methyl-tert-butyl ether

N

N	Normal
N_2	Nitrogen
NA	Not applicable
NaCl	Sodium chloride
NaOH	Sodium hydroxide
$Na_2S_2O_3$	Sodium thiosulfate
Na_2SO_3	Sodium thiosulfite
Na_2SO_4	Sodium sulfate
NBS	National Bureau of Standards
ND	Not detected
NEDTS	Navy Environmental Data Transfer Standard
NEIC	National Enforcement Investigations Center
NELAC	National Environmental Laboratory Accreditation Conference
NELAP	National Environmental Laboratory Accreditation Program
NDEP	Nevada Department of Environmental Protection
NH_4Cl	Ammonium chloride
NIH	National Institute of Health
NIOSHA	National Institute of Occupational Safety and Health Administration
NIST	National Institute of Standards and Technology
NMI	National Metrology Institute
NPD	Nitrogen phosphorus detector
NPDES	National Pollution Discharge Elimination System
NPOC	Non-purgeable organic carbon
NVLAP	Nevada Laboratory Accreditation Program

O

QAMS	Quality Assurance Management Section
OJT	On-the-job training
ORO	Oil range organics
OSHA	Occupational Safety and Health Administration
OSWER	Office of Solid Waste Environmental Regulations

P

P	Polyethylene
PAH	Polynuclear aromatic hydrocarbon
PARCC	Precision, accuracy, representativeness, comparability, and

		completeness
	PCB	Polychlorinated biphenyl
	PE	Performance evaluation
	PFE	Pressurized fluid extraction
	PHP	Potassium hydrogen phthalate
	PI	Principle investigator
	PID	Photo ionization detector
	POC	Point of contact; purgeable organic carbon
	ppb	Parts per billion
	ppm	Parts per million
	PQL	Practical quantitation limit
	PSI	Pounds per square inch
	PT	Proficiency testing
	PTOB	Proficiency testing oversight body
	PTPA	Proficiency testing provider accreditor
Q		
	QA	Quality assurance
	QAMS	Quality assurance management system
	QAP	Quality assurance plan
	QAPP	Quality assurance project plan
	QAO	Quality assurance officer
	QC	Quality control
	QCS	Quality control sample
	QSM	Quality Systems Manual
R		
	R	Recovery
	RAAS	Robotic arm automatic sampler
	RCRA	Resource Conservation and Recovery Act
	RF	Response factor
	RIC	Reconstructed ion chromatograph
	RL	Reporting limit
	RPD	Relative percent difference
	RRF	Relative response factor
	RRT	Relative retention time
	RSD	Relative standard deviation
	RT	Retention time
S		
	S	Soil
	SAP	Sampling and Analysis Plan
	SARA	Superfund Amendments and Reauthorization Act
	SB	Storage blank

SCO	Sample custody officer
SD	Standard deviation
SDWA	Safe Drinking Water Act
SI	International System of Units
SLC	Software life cycle
SOP	Standard operating procedure
SOW	Statement of work
SPCC	System performance check compound
SPE	Solid phase extraction
SPLP	Synthetic precipitation leaching procedure
SQAP	Software quality assurance plan
SRS	Self regenerating suppressor
STD	Standard Deviation
STP	Sample tracking plan
SVOC	Semivolatile organic compound

T

TAT	Turn-around time
TB	Trip blank
TCL	Target compound list
TCLP	Toxicity characteristic leaching procedure
TCMX	Tetrachloro-meta-xylene
TDS	Total dissolved solids
THMs	Trihalomethanes
TIC	Tentatively identified compound
TOC	Total organic compounds
TPH	Total petroleum hydrocarbon
TS	Total solids
TSS	Total suspended solids

U

UCL	Upper control limit
UFP-QAPP	Uniform Federal Policy for Quality Assurance Project Plans
USEPA	United States Environmental Protection Agency
UV	Ultraviolet

V

V	Volume
VIM	International Vocabulary of Basic and General Terms in Metrology
VOA	Volatile organic analysis
VOC	Volatile organic compound
VOST	Volatile organic sampling train

W

W	Water
WET	Waste extraction test
WP	Water Pollution Study
WPCF	Water Pollution Control Federation
WS	Water Supply Study

Z

ZHE	Zero headspace extraction

17.1 SYMBOLS

°C	degrees Celsius
g	gram
Kg	kilogram
L	liter
µg	microgram
µl	microliter
mg	milligram
ml	milliliter
mm	millimeter
mg/Kg	milligrams per kilogram
mg/L	milligrams per liter
ng	nanogram
nm	nanometer
oz	ounce
µg/Kg	micrograms per kilogram
µg/L	micrograms per liter

QUALITY ASSURANCE MANUAL
VOLUME I

Section 18

Glossary of Terms

18.0 GLOSSARY OF TERMS

The following definitions are used in the text of the NELAP Quality System and Alpha's Quality Assurance Manual.

Acceptance Criteria - Specified limits placed on characteristics of an item, process, or service defined in required documents. (ASQC)

Accreditation - The process by which an agency or organization evaluates and recognizes a laboratory as meeting certain predetermined qualifications or standards, thereby accrediting the laboratory. In the context of the National Environmental Laboratory Accreditation Program (NELAP), this process is a voluntary one. (NELAC)

Accrediting Authority - The Territorial, State, or Federal agency having responsibility and accountability for environmental laboratory accreditation and which grants accreditation. (NELAC)

Accreditation Body - Authoritative body that performs accreditation. (ANSI/ASQ-2004)

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

Aliquot - A discrete, measured, representative portion of a sample taken for analysis. (DoD)

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

Analyte - The specific chemicals or components for which a sample is analyzed; it may be a group of chemicals that belong to the same chemical family, and which are analyzed together. (EPA Risk Assessment Guide for Superfund; OSHA Glossary)

Analytical Method - A set of written instructions completely defining the procedure to be adopted by the analyst in order to obtain an analytical result.

Assessment - The evaluation process used to measure or establish the performance, effectiveness, and conformance of an organization and/or its systems to define criteria (to the standards and requirements of NELAC). (NELAC)

Audit - A systematic check to determine the quality of operation of some function or activity. (EPA-QAD).

Autozero - Zeroing the instrument (typically a spectrophotometer for inorganic analysis) at the proper wavelength. It is equivalent to running a standard blank with the absorbance set at zero.

Bar Graph Spectrum - A plot of the mass-to-charge ratio (m/e) versus relative intensity of the ion current.

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

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

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

4-Bromofluorobenzene (BFB) - The compound chosen to establish mass spectral instrument performance for volatile analyses.

Calibration - A set of operations that establish, under specified conditions, the relationship between values of quantities indicated by a measuring instrument or measuring system, or values represented by a material measure or a reference material, and the corresponding values realized by standards. (VIM: 6.11)

1) In calibration of support equipment, the values realized by standards are established through the use of Reference Standards that are traceable to the International System of Units (SI).

2) In calibration according to test methods, the values realized by standards are typically established through the use of Reference Materials that are either purchased by the laboratory with a certificate of analysis or purity, ir prepared using support equipment that has been calibrated or verified to meet specifications.

Calibration Curve - The graphical relationship between the known values, such as concentrations, of a series of calibration standards and their instrument response. (NELAC)

Calibration Method - A defined technical procedure for performing a calibration. (NELAC)

Calibration Range - The range of values (concentrations) between the lowest and highest concentration standards of a multi-level calibration curve. For metals analysis with a single-point calibration, the low-level calibration check standard and the high standard establish the linear calibration range, which lies within the linear dynamic range. (DoD)

Calibration Standard - A substance or reference material used to calibrate an instrument. (QAMS)

Certification - Approval by a certifying agency to use an analytical method for analysis of specific analytes following submission of a performance data package.

Certified Reference Material (CRM) - A reference material one or more of whose property values are certified by a technically valid procedure, accompanied by or traceable to a certificate or other documentation which is issued by a certifying body. (ISO Guide 30-2.2)

Chain of Custody - A record that documents the possession of the samples from the time of collection to receipt in the laboratory. This record generally includes: the number and types of containers; the mode of collection; collector; preservation; and requested analyses. (NELAC)

Comparability - Confidence with which one data set can be compared to another.

Confirmation - Verification of the identity of a component through the use of an approach with a different scientific principle from the original method. (NELAC)

Conformance - An affirmative indication or judgment that a product or service has met the requirements or the relevant specifications, contract, or regulation; also the state of meeting the requirements. (ANSI/ASQC E4-1994)

Congener - A member of a class of related chemical compounds (e.g., PCBs, PCDDs) (DoD)

Control Samples - Samples introduced into the train of environmental samples as monitors of the performance of the analytical method.

Corrective Action - The action taken to eliminate the causes of an existing nonconformity, defect or other undesirable situation in order to prevent recurrence. (ISO 8402)

Data Audit - A qualitative and quantitative evaluation of the documentation and procedures associated with environmental measurements to verify that the resulting data are of acceptable

quality (i.e., that they meet specified acceptance criteria). (NELAC)

Data Quality - Totality of features and characteristics of a data set that bears on its ability to satisfy a given purpose.

Data Reduction - The process of transforming raw data by arithmetic or statistical calculations, standard curves, concentration factors, etc., and collation into a more useable form. (EPA-QAD)

Data Validation - A systematic process for reviewing a body of data against a set of criteria to provide assurance that the data are adequate for their intended use. Data validation consists of data editing, screening, checking, auditing, verification, certification, and review.

Decafluorotriphenylphosphine (DFTPP) - A compound chosen to establish mass spectral tuning performance for semi-volatile analysis.

Demonstration of Capability - A procedure to establish the ability of the analyst to generate acceptable accuracy. (NELAC)

Detection Limit - The lowest concentration or amount of the target analyte that can be identified, measured, and reported with confidence that the analyte concentration is not a false positive value. (NELAC)

Digestion - A process in which a sample is treated (usually in conjunction with heat) to convert the sample to a more easily measured form. (DoD)

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

Dry Weight - The weight of a sample based on percent solids. The weight after drying in an oven.

Duplicate - The analyses or measurements of the variable of interest performed identically on two subsamples of the same sample. The results of duplicate analyses are used to evaluate analytical or measurement precision but not the precision of sampling, preservation or storage internal to the laboratory. (EPA-QAD)

Eluent - A solvent used to carry the components of a mixture through a stationary phase.

Elute - To extract; specifically, to remove (adsorbed material) from an adsorbent by means of a solvent.

Elution - A process in which solutes are washed through a stationary phase by the movement of a mobile phase.

Environmental Data - Any measurements or information that describes environmental processes, locations, or conditions; ecological or health effects and consequences; or the performance of environmental technology. (ANSI/ASQ E4-2004)

Environmental Monitoring - The process of measuring or collecting environmental data. (UFP-QAPP)

Equipment Blank - Usually an organic or aqueous solution that is free of analytes and is transported to the sampling site, opened in the field, and poured over or through the sample collection device, collected in a sample container, and returned to the laboratory. This serves as a check on sampling device cleanliness.

Extractable - A compound that can be partitioned into an organic solvent from the sample matrix and is amenable to gas chromatography. Extractable compounds include BNA, pesticide and PCB compounds.

False Negative - An item incorrectly identified as present in the sample, resulting in a high reporting value for the analyte of concern.

Field Blank - A sample to which no analytes of interest have been added. It is transported to the sampling site and back to ensure that no contamination is introduced during shipment. This sample may be opened near the sampling location to determine if air-borne contaminants are contributing to the sample contaminations.

Field Duplicate - Two samples, collected at the sample site, that are treated exactly the same throughout field and laboratory procedures. Analysis of field duplicates provides a measure of the precision associated with sample collection, preservation and storage, as well as with laboratory procedures.

Finding - An assessment conclusion, referenced to a regulatory standard and supported by objective evidence that identifies a deviation from that regulatory requirement.

Heavy Metals - Metallic elements with high atomic weights, such as mercury, chromium, cadmium, arsenic, and lead. They can damage living things at low concentrations and tend to accumulate in the food chain.

Holding Time - The maximum times that samples may be held prior to analysis and still be considered valid or not compromised. The time elapsed from the time of sampling to the time of extraction or analysis as appropriate.

Homologue - One in a series of organic compounds in which each successive member has one or more chemical group in its molecule than the next preceding member. For instance CH_3OH (methanol), C_2H_5OH (ethanol), C_3H_7OH (propanol), C_4H_9OH (butanol), etc., form a homologous series.

Hydrocarbons - Any of a series of chemical compounds that consist entirely of carbon and hydrogen.

Initial Calibration - The analysis of analytical standards for a series of different specified concentrations; used to define the linearity and dynamic range of the response of the instrument to the target compounds.

Inspection - An activity such as measuring, examining, testing or gauging one or more characteristics of an entity and comparing the results with specified requirements in order to establish whether conformance is achieved for each characteristic. (ANSI/ASOC E4-1994)

Instrument Blank - A clean sample processed through the instrumental steps of the measurement process; and used to determine instrument contamination. (EPA-QAD)

Instrument Performance Check - A solution of method analytes, used to evaluate the performance of the instrument system with respect to a defined set of method criteria.

Interferents - Substances which affect the analysis for the analyte of interest.

Internal Standards - A known amount of standard added to a test portion of a samples as a reference for evaluating and controlling the precision and bias fo the applied analytical method. (NELAC)

Internal System of Units - The coherent system of units adopted and recommended by the General Conference on Weights and Measures (CCGPM) (VIM 1.12)

Isomer - One of two or more compounds, radicals, or ions that contain the same number of atoms of the same elements but differ in structural arrangement and properties.

Laboratory Control Sample (LCS) - A sample matrix, free from the analytes of interest, spiked with verified known amounts of analytes or material containing known and verified amounts of analytes. It is generally used to establish intra-laboratory or analyte-specific precision and bias or to assess the performance of all or a portion of the measurement system. (NELAC)

Laboratory Duplicate - Aliquots of a sample taken from the same container under laboratory conditions and processed and analyzed independently. (NELAC)

Laboratory Fortified Blank (LFB) - An aliquot of laboratory reagent water to which known

quantities of the method analytes are added in the laboratory. The LFB is analyzed exactly like a sample, and its purpose is to determine whether the method is in control and whether the laboratory is capable of making accurate and precise measurements.

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

Limit of Detection (LOD) - An estimate of the minimum amount of a substance that an analytical process can reliably detect. An LOD is analyte and matrix specific and may be laboratory dependent.

Limit of Quantitation (LOQ) - The minimum levels, concentrations, or quantities of a target analyte that can be reported with a specified degree of confidence.

Linear Dynamic Range - (Inorganic Analysis) The concentration range over which the ICP or IC analytical curve remains linear.

Matrix - The predominant material of which the sample to be analyzed is composed. For most purposes, a sample matrix is either water or soil/sediment.

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

Matrix Spike Duplicate - A second replicate matrix spike prepared in the laboratory and analyzed to obtain a measure of precision of the recovery for each analyte. (QAMS)

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

Method Detection Limit (MDL) - One way to establish a limit of detection, defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the analyte concentration is greater than zero. The MDL is statistically determined from analysis of samples in a given matrix containing the analyte at a predefined low concentration level.

National Environmental Laboratory Accreditation Conference (NELAC) - A voluntary organization of State and Federal environmental officials and interest groups purposed

primarily to establish mutually acceptable standards for accrediting environmental laboratories. A subset of NELAP. (NELAC)

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

Negative Control - Measures taken to ensure that a test, its components, or the environment do not cause undesired effects, or produce incorrect test results. (NELAC)

Nonconformance - An indication or judgement that a product or service has not met the requirements of the relevant specifications, contract or regulation; also the state of failing to meet the requirements.

Outlier - An extreme observation that is shown to have a low probability of belonging to a data population.

Percent Difference (%D) - An arithmetic calculation to compare two values. The percent difference indicates both the direction and the magnitude of the comparisons, i.e., the percent difference may be either negative, positive, or zero.

Percent Moisture - An approximation of the amount of water in a soil/sediment sample obtained by drying an aliquot of the sample at 105°C. The percent moisture determined in this manner also includes contributions from all compounds that may volatilize at 105°C, in addition to water.

Percent Solids - The proportion of the solid in a soil sample determined by drying an aliquot of the sample.

Performance Audit - The routine comparison of independently obtained qualitative and quantitative measurement system data with routinely obtained data in order to evaluate the proficiency of an analyst or laboratory. (NELAC)

Performance Evaluation (PE) Sample - A sample of known composition provided by a third party (unknown composition by the laboratory) used to evaluate laboratory performance.

Positive Control - Measures taken to ensure that a test and/or its components are working properly and producing correct or expected results from positive test subjects. (NELAC)

Precision - The degree to which a set of observations or measurements of the same property, obtained under similar conditions, conform to themselves; a data quality indicator. Precision is usually expressed as standard deviation, variance or range, in either absolute or relative terms. (NELAC)

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

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

Proficiency Testing Oversight Body/Proficiency Testing Provider Accreditor (PTOB/PTPA) - An organization with technical expertise, administrative capability and financial resources sufficient to implement and operate a national program of PT provider evaluation and oversight that meets the responsibilities and requirements established by NELAC standards. (NELAC)

Proficiency Testing Program - The aggregate of providing rigorously controlled and standardized environmental samples to a laboratory for analysis, reporting of results, statistical evaluation of the results and the collective demographics and results summary of all participating laboratories. (NELAC)

Proficiency Testing Study Provider - Any person, private party, or government entity that meets stringent criteria to produce and distribute NELAC PT samples, evaluate study results against published performance criteria and report the results to the laboratories, primary accrediting authorities, PTOB/PTPA and NELAP. (NELAC)

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

Protocol - A detailed written procedure for field and/or laboratory operation (e.g., sampling, analysis) which must be strictly followed. (EPA-QAD)

Purge and Trap (device) - An analytical technique (device) used to isolate volatile organics by stripping the compounds from water or soil by a stream of inert gas, trapping the compounds on a porous polymer trap, and thermally desorbing the trapped compounds onto the gas chromatographic column.

Quality Assurance (QA) - An integrated system of activities involving planning, quality control, quality assessment, reporting and quality improvement to ensure that a product or service meets defined standards of quality with a stated level of confidence. (QAMS)

Quality Assurance Project Plan (QAPP) - A formal document describing the detailed quality control procedures by which the quality requirements defined for the data and decisions pertaining to a specific project are to be achieved. (EPA-QAD)

Quality Control (QC) - The overall system of technical activities whose purpose is to measure and control the quality of a product or service so that it meets the needs of users. (QAMS)

Quality Control Sample - A sample used to assess the performance of all or a portion of the measurement system. QC samples may be Certified Reference Materials, a quality system matrix fortified by spiking, or actual samples fortified by spiking.

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

Quality System - A structured and documented management system describing the policies, objectives, principles, organizational authority, responsibilities, accountability, and implementation plan of an organization for ensuring quality in its work processes, products, and services. The quality system provides the framework for planning, implementing, and assessing work performed by the organization and for carrying our required QA and QC. (ANSI/ASQC E-4 1994)

Quantitation Range - The range of values in a calibration curve between the LOQ and the highest successfully analyzed initial calibration standard. The quantitation range lies within the calibration range. (DoD)

Random Error - The deviation in any step in a procedure that can be explained by standard statistical techniques.

Raw Data - Any original factual information from a measurement activity or study recorded in a laboratory notebook, worksheets, records, memoranda, notes, or exact copies thereof that are necessary for the reconstruction and evaluation of the report of the activity or study. (EPA-QAD)

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

Reagent Water - Water in which an interferant is not observed at or above the minimum reporting limit for the parameters of interest.

Reconstructed Ion Chromatogram (RIC) - A mass spectral graphical representation of the separation achieved by a gas chromatograph; a plot of total ion current versus retention time.

Recovery - A determination of the accuracy of the analytical procedure made by comparing

measured values for a fortified sample against the known spike values.

Reference Material - A material or substance one or more properties of which are sufficiently well established to be used for the calibration of an apparatus, the assessment of a measurement method, or for assigning values to materials. (ISO Guide 30-2.1)

Reference Standard - A standard, generally of the highest quality available at a given location, from which measurements made at that location are derived. (VIM 6.08)

Relative Percent Difference (RPD) - An analytical technique used to compare two values. The relative percent difference is based on the mean of the two values, and is reported as a relative value.

Replicate Analyses - The measurements of the variable of interest performed identically on two or more sub-samples of the same sample within a short time interval. (NELAC)

Resolution - The separation between peaks on a chromatogram, calculated by dividing the height of the valley between the peaks by the average peak height of the two peaks being resolved, multiplied by 100.

Retention Time Window - Usually defined as three times the standard deviation of the absolute or relative RT of an analyte, positioned around a defined absolute retention time.

Sample - A portion of material to be analyzed that is contained in single or multiple containers and identified by a unique sample number. (DoD)

Sample Number - (AAI Sample Number) - A unique identification number designated by Alpha for each sample. The sample number appears on the final report which documents information on that sample.

Second Source Calibration Verification (ICV) - A standard obtained or prepared from a source independent of the source of standards used for the initial calibration. Its concentration should be at or near the middle of the calibration range. It is done after the initial calibration. (DoD)

Selectivity - The capability of a test method or instrument to respond to a target substance or constituent in the presence of non-target substances. (EPA-QAD)

Semi-volatile Compounds - Compounds amenable to analysis by extraction of the sample with an organic solvent. Used synonymously with Base/Neutral/Acid (BNA) compounds.

Sensitivity - The capability of a method or instrument to discriminate between measurement responses representing different concentrations of a variable of interest. (NELAC)

Serial Dilution - The dilution of a sample when corrected by the dilution factor, the diluted sample must agree with the original undiluted sample within specified limits.

Signal to Noise Ratio - The signal carries information about the analyte, while the noise is made up of extraneous information that is unwanted because it degrades the accuracy and precision of an analysis and also places a lower limit on the amount of analyte that can be detected. In most measurements, the average strength of the noise is constant and independent of the magnitude of the signal. Thus, the effect of noise on the relative error of the measurement becomes greater and greater as the quantity being measured (producing signal) decreases in magnitude.

Significant Figures - The number of digits used to express a result in scientific notation. All digits are expected to be known definitely, except the last digit, which may be in doubt.

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

Standard (Document) - The document describing the elements of laboratory accreditation that has been developed and established within the consensus principles of NELAC and meets the approval requirements of NELAC procedures and policies. (ASQC)

Standard (Chemical) - Standard samples are comprised of a known amount of a standard reference material in the matrix undergoing analysis. A standard reference material is a certified reference material produced by the US National Institute of Standards and Technology (NIST) and characterized for absolute content, independent of analytical test method. (DoD)

Standard Deviation - The positive square root of the expected value of the square of the difference between a random variable and its mean.

Standard Method - A test method issued by an organization generally recognized as competent to do so.

Standard Operating Procedure (SOP) - A written document which details the method of an operation, analysis or action whose techniques and procedures are thoroughly prescribed and which is accepted as the method for performing certain routine or repetitive tasks. (QAMS)

Standardized Reference Material (SRM) - A certified reference material produced by the U.S. National Institute of Standards and Technology or other equivalent organization and characterized for absolute content, independent of analytical method. (EPA-QAD)

Supervisor - The individual(s) designated as being responsible for a particular area or category of scientific analysis. This responsibility includes direct day-to-day supervision of

technical employees, supply and instrument adequacy and upkeep, quality assurance/quality control duties and ascertaining that technical employees have the required balance of education, training and experience to perform the required analyses. (NELAC)

Surrogate - A substance with properties that mimic the analyte of interest. It is unlikely to be found in environmental samples and is added to them for quality control purposes. (QAMS)

System Monitoring Compounds - Compounds added to every blank, sample, matrix spike, matrix spike duplicate, and standard for volatile analysis and used to evaluate the performance of the entire purge and trap, gas chromatograph/ mass spectrometer system. These compounds are brominated or deuterated compounds not expected to be detected in environmental media.

Target Analyte - Specific analytes reported for every sample analyzed by a given method.

Target Concentration - Known spiked concentration.

Technical Director - Individual(s) who has overall responsibility for the technical operation of the environmental testing laboratory. (NELAC)

Tentatively Identified Compounds (TIC) - Compounds detected in samples that are not target compounds, internal standards, system monitoring compounds, or surrogates. These peaks are subjected to mass spectral library searches for tentative identification.

Test - A technical operation that consists of the determination of one or more characteristics or performance of a given product, material, equipment, organism, physical phenomenon, process or service according to a specified procedure. The result of a test is normally recorded in a document sometimes called a test report or a test certificate. (ISO/IEC Guide 2-12.1, amended)

Test Method - An adoption of a scientific technique for performing a specific measurement as documented in a laboratory SOP or as published by a recognized authority.

Traceability - The property of a result of a measurement whereby it can be related to appropriate standards, generally international or national standards, through an unbroken chain of comparisons. (VIM - 6.12)

Tune - An injected standard required by the method as a check on instrument performance for mass spectrometry.

Twelve-hour Time Period - The twelve (12) hour time period for GC/MS system tuning and standard calibrations which begins at the moment of injection of the DFTPP or BFB. The time period ends after 12 hours has elapsed according to the system clock.

Validation - The confirmation by examination and provision of objective evidence that the particular requirements for a specific intended use are fulfilled.

Verification - Confirmation by examination and provision of evidence that specified requirements have been met. (NELAC)

Volatile Compounds - Compounds amenable to analysis by the purge and trap technique. Used synonymously with purgeable compounds.

Work Cell - A well-defined group of analysts that together perform the method of analysis. The members of the group and their specific functions within the work cell must be fully documented. (NELAC)