

A&L Great Lakes Laboratories, Inc.

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Laboratory Quality Assurance Manual

(Revision 7)

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Revision History:

Revision 1 – Additions to address components required for NELAC Accreditation.

Revision 2 – Updated facilities map to reflect construction addition/expansion, correction to BOD holding time

Revision 3 –

1. Correction to Table of Contents
2. Terms and Definitions – Deleted reference to Laboratory Control Sample and added Method Control to stay consistent with terminology used in the laboratory.
3. Addition of the “Note” to Systems Audit definition (page 10).
4. Corrected spelling of “day-to-day” on page 11.
5. Added item #10 to section 4.2.2 (page 14).
6. Update to point 5 under section 5.0 and paragraph under section 5.1 (page 18).
7. Update to section 5.4 (page 19).
8. Updates to Table 7.1. (pp. 25-26) reflecting Method Update Rule.
9. Additions to sections 8.1, 8.3 and 8.4 (page 29).
10. Added clarification to section 12.1.1 (page 39).
11. Added section 13.1.4 “Analytical Method Audits” to page 44.
12. Added analytes to section 13.3.5 Manure Analysis Proficiency Testing Program (page 45).
13. Updates to section 18.0 (pages 54-55).
14. Updated Facility Map (page 57).

Revision 4 -

1. Updated Forward (section 1.1)
2. Updated Instrument Detection Limit (section 1.4).
3. Addition of Limit of Detection definition (section 1.4).
4. Additions to Ensuring Appropriate Facilities and Resources (section 6.5).
5. Addition of Section 6.5.1.
6. Updated Procurement/Inventory (section 8.1).
7. Grammatical change to section 9.0.
8. Updated Performance Evaluation Samples (PE) Studies (Section 13.3.1.).
9. Addition of Pennsylvania Department of Environmental Protection under section 13.3.
10. Updated section 16.3.4.
11. Addition of Questionable Lab Results and Client Notification (Section 16.5).

Revision 5 -

1. Updated Title Page
2. Addition to Purpose (section 1.2.)
3. Updated Quality Assurance Documents (section 1.3.)
4. Addition to Reporting Limit definition (section 1.4.)
5. Addition of annual ethics training in section 3.0
6. Addition of Technical Director to QA Organization and Personnel (section 4.0)
7. Changes to Laboratory Organization (section 4.1)
8. Addition of Technical Director position to section 4.2.1.
9. Addition of education requirement to Description of Responsibilities (section 4.2.2.)
10. Addition to duties for Technical Director and Quality Manager (sections 4.2.1.)
11. Changes to Training and Orientation (section 4.3.)
12. Addition to Sample Receipt (section 7.1.)
13. Addition to Chain of Custody (section 7.2.)
14. Subtracted education requirements from the Quality Assurance Unit (section 13.1.1.)
15. Additions to Specific Guidelines for Corrective Action (section 16.2)

16. Changes to Treatment of Sample Data Collected in Instances where Procedural or QC Data or Non-Conformances Exist (section 16.3.3.)
17. Changes to Questionable Lab Results and Client Notification (section 16.5)

Revision 6 –

1. Updated Title Page
2. Changes to The Quality Assurance Manual (section 11.6.1)
3. Addition of AFPC Phosphate Fertilizer Check Sample Program (section 13.3.11)
4. Addition of Agricultural Laboratory Proficiency Program (section 13.3.12)
5. Changes to Methods Under which the Laboratory Performs its Testing (section 18.0)
6. Additions to Major Equipment (section 19.0)
7. Changes to Chromium VI (Table 7.1)

Revision 7 –

1. Updated Title Page
2. Changes to Table of Contents
3. Changes to Foreword (1.1)
4. Changes to Ethical and Legal Responsibility (section 3.0)
5. Changes to QA Organization and Personnel (section 4.0)
6. Changes to Laboratory Organization (section 4.1)
7. Changes to Technical Director (section 4.2.1)
8. Changes to Table 7.1 in Sample Handling and Custody (section 7.0)
9. Changes to Standards (section 12.2)
10. Changes to Indiana State Board of Health – Public Water Supply (section 13.3.3)
11. Changes to Data Archive (section 15.4)
12. Changes to Testing Methods (section 18.0)
13. Additions to Major Equipment (section 19.0)
14. Changes are highlighted

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1.0 INTRODUCTION

1.1. FOREWORD

A&L Great Lakes Laboratories, Inc. is an independently owned and operated agricultural and environmental analytical laboratory located in Fort Wayne, Indiana. The laboratory operates from a 18,000 sq. ft. facility which was built in 1987, and houses approximately 50 professionals and technicians.

A&L Great Lakes Laboratories was established in 1976 by Mr. Don Ankerman and Dr. Richard Large as part of a regional network of nine A&L laboratories in North America. A&L Great Lakes Laboratories became an independent lab from the A&L license group in 2002, but still benefits from cooperative efforts with the other regional labs.

A&L Great Lakes Labs analyzes many types of sample matrices including soils, plants, feedstuffs, fertilizers, waters, wastewaters, manures, composts, sludges, and hazardous wastes. The labs testing capabilities include a wide variety of organic and inorganic parameters.

1.2. PURPOSE

The Quality Assurance Manual provides guidelines to ensure that A&L Great Lakes Laboratories, Inc. employees and management work to generate analytical data that is scientifically valid, legally defensible, accurate, impartial, and of known and documented quality in accordance with standards developed by the National Environmental Laboratory Accreditation Conference (NELAC) and any applicable state or EPA regulations or requirements.

The purpose of the quality assurance program is to achieve a level of data quality that meets customer requirements for completeness, precision, accuracy, comparability, and dependability. The purpose of this written quality assurance manual is to summarize and document A&L Great Lakes Laboratories, Inc. quality assurance practices. The Quality Assurance (QA) Manual provides the foundation for the establishment and maintenance of the QA Program. A&L Great Lakes Laboratories' management is committed to promoting and ensuring quality throughout the organization and is committed to compliance with NELAC and other applicable regulatory accreditation/certification standards.

1.3. QUALITY ASSURANCE DOCUMENTS

1.3.1. Quality Manual

This document describes management policies concerning laboratory quality assurance and related operations of A&L Great Lakes Laboratories, Inc. Environmental Department. It provides an operational framework within which the Quality Assurance Program can operate. This document outlines and discusses each element of the Quality Assurance Program. All employees are required to adhere to the policies set forth in the Quality Assurance Manual. Changes or Revisions to the manual can be made only with the approval of the Quality Assurance Unit and Management.

1.3.2. Standard Operating Procedures

Standard Operating Procedures (SOP's) are intended to provide specific written guidelines as to how processes and procedures are carried out by company personnel. These documents also provide historical information as to how procedures were carried out in the past. Every procedure related to sample collection, storage, preparation, analysis, disposal, validation, data reporting, etc. will be contained in written Standard Operating Procedures (SOP's). Each SOP is written according to guidelines set forth in SOP **ADM-0-001** "Writing and Administration of Standard Operating Procedures". Both upper level management and the Quality Assurance Unit must approve all SOP's placed in the laboratory. Each SOP is uniquely numbered according to category codes. SOP document distribution is controlled by the Quality Assurance Unit. No copies, other than official copies will be used. A unique copy number is assigned to each SOP. The Quality Assurance Unit will also keep a master SOP listing (SOP number, revision number, title and effective date). The master list is updated whenever SOP's are written or revised. All retired revisions of SOP's are maintained in our secure archive. All laboratory SOP's are reviewed annually and documented on the SOP review log attached to the copy in the Quality Manager's office.

1.3.3. Statement of Laboratory Qualifications

This summary document contains information such as "About A&L Great Lakes Laboratories, Inc.", "Quality Assurance", "Organizational Chart", "Staff Information", "Instrumentation", and a layout of the laboratory floor plan.

1.3.4. Protocols

Protocols govern the conduct of the study. The protocol contains study specific instructions directed by the Sponsor. Work done by A&L Great Lakes Laboratories, Inc. under GLP "Good Laboratory Practice" regulations (EPA 40 CFR Part 160) requires a protocol. All protocols are maintained by the Quality Assurance Unit. Additional EPA or regulatory work outside the scope of the GLP's may also require a protocol.

1.4. TERMS AND DEFINITIONS

Accuracy:	The degree of agreement between a measured value and the true or expected value.
Aliquot:	A measured portion of a sample taken for analysis
Analyte:	An analyte is the element, compound, or species that is detected and determined through analysis. Analytical methods require calibration for quantitation of specific analytes.
Batch:	A grouping of samples of similar matrix which are prepared and/or analyzed together with the same method within the same time frame, as designated by the method.
Blank:	A blank is a sample designated to detect and/or monitor the contribution of analyte and non-analyte contamination, instrumental background and sample processing to the measurement system.

Blind Sample:	A sample submitted for analysis whose composition is known to the submitter but unknown to the analyst.
Calibration:	The process of establishing the relationship between instrument response and known, traceable quantities of analytes of interest.
Calibration Check:	Verification of the ratio of instrument response to analyte amount. A calibration check is done by analyzing for analyte standards in an appropriate solvent. Calibration check solutions should be made from a stock solution which is different from the stock used to prepare standards if available.
Continuing Calibration:	The process of analyzing standards periodically to verify the maintenance of calibration of the analytical system.
Control Chart:	A graphical representation of test results with respect to time or sequence of measurement, together with limits within which they are expected to lie when a system is in a state of statistical control.
Data Quality Objective (DQO):	Systematic strategic planning tool based on the scientific method that identifies and defines the type, quality, and quantity of data needed to satisfy a specified use.
Detection Limit:	The minimum concentration of a substance that can be measured and reported with 99% confidence that the analyte concentration is greater than zero, expressed either as method detection limit (MDL) or instrument detection limit (IDL).
Dry Weight:	The weight of a sample based on percent solids. The sample weight after drying in an oven at a specified temperature.
Duplicate Analysis:	A second measurement made on the same sample extract or digestate to assist in the evaluation of analysis precision.
Duplicate Sample:	A second aliquot of the same sample that is treated the same as the original sample in order to determine the precision of the method.
Environmental Sample:	<p>An environmental sample or field sample is a representative sample of any material (aqueous, non-aqueous, or multimedia) collected from any source for which determination of composition or contamination is requested or required. Environmental samples can generally be classified as follows:</p> <ul style="list-style-type: none">Surface Water and Ground WaterDrinking Water - Delivered (treated or untreated) water designated as potable water.Water/Wastewater - Raw source waters for public drinking water supplies, ground waters, municipal influents/effluents, and industrial influents/effluents.Sludge - Municipal and industrial sludges.

Soil - Predominately inorganic matter ranging in classification from sands to clays.

Waste - Aqueous and non-aqueous liquid wastes, chemical solids, and industrial liquid and solid wastes.

Field Blank: A quality control sample that is used to assess the contamination effects on accuracy due to the combined activities of sampling and analysis. Typically, it is composed of analyte free matrix (e.g., de-ionized water).

Field Sample: A portion of material received by the laboratory to be analyzed, that is contained in single or multiple containers and identified by a unique ID.

Holding Time: The elapsed time from the date/time of sample collection by the field personnel until the date/time of its processing/analysis. Holding time requirements are defined by the method or protocol.

Homogeneity: The degree to which a property or substance is evenly distributed throughout a material.

Instrument

Detection Limit (IDL): The smallest signal above background noise that an instrument can reliably detect. The instrument detection limit is determined by replicate analyses of a standard solution prepared on the instrument. The IDL is generally more sensitive than the MDL because its determination does not include sample preparation steps.

Initial Calibration: The process of analyzing standards, prepared at specific concentrations, to define the quantitative response, linearity and dynamic range of the instrument to the analytes of interest.

Internal Standards: Analytes added to every standard, blank, lab control sample, matrix spike, duplicate, and sample at a known concentration, prior to analysis for the purpose of adjusting the response factor used in quantifying target analytes. Internal standards are used as the basis for quantitation of the target compounds, and are generally applicable to organic analyses.

Limit of Detection (LOD): For the purposes of A&L Great Lakes Laboratories, Inc. certification, the LOD is approximately equal to the MDL for those tests which the MDL can be calculated.

LIMS: Laboratory Information Management System.

Lot: A quantity of bulk material of similar composition processed or manufactured at the same time.

Matrix: The sample matrix is the component or substrate, which contains the analyte(s) of interest. Examples include: groundwater, soil, drinking water, sediment and sludge.

Matrix Spike:	A matrix spike is an aliquot of sample that is spiked with a known concentration of target analyte(s) prior to sample preparation. The recovery of target analyte(s) from the matrix spike sample is used to determine the bias of the method in the specific sample matrix.
Method Blank:	An analytical control consisting of a blank matrix containing all reagents, internal standards and surrogate standards, that is carried through the entire analytical procedure. The method blank is used to define the level of laboratory background and contamination, and to demonstrate that this level does not exceed acceptance limits.
Method Control (MControl):	An MControl is a quality control sample that consists of a known matrix spiked with a known amount of targeted analytes. The MControl is carried through the entire sample preparation and analysis procedure, and is used to monitor the overall accuracy of the analytical measurement process. Control limits for MControl recovery, typically expressed as a % recovery, serve as acceptance criteria for determining whether an analytical run is in control.
Method Detection Limit (MDL):	The minimum concentration of a substance that can be measured with 99% confidence that the analyte concentration is greater than zero. MDL's are determined using replicate spike samples prepared by the lab and taken through all preparation and analysis steps of the method. The MDL is calculated using the appropriate Students' t value times the standard deviation of a series of spiked samples.
Performance Evaluation (PE) Samples:	Samples provided by an outside source which are run as either known, single, or double-blind fashion. The results of PE samples provide data to evaluate the proficiency of an analyst or laboratory method.
Precision:	The measurement of agreement of a set of replicate results among themselves without any prior information as to the true result. Precision is assessed by means of duplicate/replicate sample analysis.
Protocol:	A stated plan that clearly defines the objectives, methods and procedures for accomplishing a task.
Quality Assurance:	A system of policies and procedures whose purpose is to ensure, confirm, and document that the product or service rendered fulfills the requirements of A&L Great Lakes Laboratories, Inc. and its client. Quality Assurance includes quality planning, quality assessment (auditing), quality reporting and corrective action.
Quality Control:	A system of checks and corrective measures, integrated with the activities that directly generate the product or service that serves to monitor and adjust the process to maintain conformance to predetermined requirements.

Reagent Grade:	Analytical reagent (AR) grade, ACS reagent grade, and reagent grade are synonymous terms for reagents, which conform to the current specifications of the Committee on Analytical Reagents of the American Chemical Society.
Replicate Samples:	Samples collected at the same time, from the same place, for the same analysis, as the original sample in order to determine precision between the samples.
Reporting Limit (RL):	The level at which method, permit, regulatory and client specific objectives are met. The reporting limit may never be lower than the statistically determined MDL, but may be higher based on any of the above considerations. Reporting limits are corrected for sample amounts and dilutions, including the dry weight of solids, unless otherwise specified. RL's are usually 2-10 times the MDL. Additionally, the RL is never below the lowest calibration standard.
Sensitivity:	Capability of methodology or instrumentation to discriminate between samples having differing concentrations or containing differing amounts of an analyte.
Split Sample:	A split sample may be used to assess intra- or inter-laboratory precision of the measurement process. Field split samples are obtained by preparing two (or more) individual sample aliquots after thorough homogenization, in the field, of a single sample. A field split sample may be used to determine intra-laboratory precision if the split samples are submitted to a single laboratory. A field split sample may be used to determine inter-laboratory precision if the split samples are submitted to different laboratories. The degree to which split precision data represent a true measure of laboratory precision is limited by the degree to which the sample is homogenized in the field. If the field sample is not effectively homogenized, the resultant data may not be used to assess laboratory precision.
Standard:	A substance or material, the properties of which are known with sufficient accuracy, used to evaluate the same property in a sample.
Standard Blank:	A calibration standard consisting of the same solvent/reagent matrix used to prepare the calibration standards without the analytes. It is used to construct the calibration curve by establishing instrument background.
Standard Curve:	A standard curve is the graphical representation of known analytes standard concentrations versus the instrument response to the analytes.
Surrogates:	A surrogate analyte is used to monitor method performance on a matrix-specific basis. A surrogate is a pure analyte which is added to the sample aliquot in known amount, prior to sample extraction. The surrogate, which is similar to the Method target analytes in composition and behavior, is not ordinarily found in environmental samples. Because surrogates are generally added to each sample in a batch, they can be

- used to monitor recovery on a sample-specific, rather than batch-specific basis.
- Systems Audit:** An on-site inspection or assessment of a laboratory's quality system.
Note: *This differs from an analytical method audit in which adherence to a particular method or procedure is assessed.*
- Traceability:** The ability to trace the source and accuracy of a material (i.e. standard) to a recognized primary reference source such as the National Institute of Standards and Technology (NIST) or USEPA.
- Warning Limits:** The limits (typically +/- 2 standard deviations) shown on a control chart within which most results are expected to lie (within 95% probability) while the system remains in a state of statistical control.

2.0 QUALITY SYSTEM COMPONENTS AND OBJECTIVES

A&L Great Lakes Laboratories, Inc. QA/QC plans and procedures provide a framework that guides our chemists and technicians, as well as administrative personnel, to achieve our Data Quality Objectives, thereby ensuring the precision, accuracy, completeness, and consistency of the analytical data reported to our clients. This framework is referred to as the Quality System. The Quality System encompasses every quality assurance (QA) policy and quality control (QC) procedure and guides all technical and administrative laboratory operations by documenting and specifying standardized procedures to control both the short-term and long-term activities influencing the quality and defensibility of our testing services. The Quality System is not a static entity. It is continually evolving to allow for consistent improvement of all aspects of A&L Great Lakes Laboratories, Inc.

The management of A&L Great Lakes Laboratories, Inc. is fully committed to the continual development and improvement of the Quality System and ensures that all aspects of the day-to-day laboratory operations are conducted following the requirements outlined in the QA Manual.

A&L Great Lakes Laboratories, Inc. management also ensures that the objectives of the Quality System are communicated to and understood by all A&L employees. All aspects of the Quality System have an impact on our ability to provide our clients with technically sound and legally defensible data that is generated by well-trained and qualified analysts and is delivered in an efficient and timely manner.

We strive for consistent standards of quality that conform to each client's overall project quality control requirements. Quality control limits are established and published for most EPA methodology. State agencies and clients will occasionally set limits different from those in the published methods, and we attempt to adhere to the most stringent limits as a method of meeting all agency and/or client requirements.

3.0 ETHICAL AND LEGAL RESPONSIBILITY

All employees of A&L Great Lakes Laboratories, Inc. have an ethical and legal responsibility to produce data that is accurate and legally defensible. All aspects of the ethics and data integrity program are addressed in **Appendix A**. All employees are educated with regards to A&L Great Lakes Laboratories, Inc. Ethics Policy and are required to sign a current Ethics Acknowledgement Statement (**Appendix A**) upon employment. The original form is kept in the employee's training file and a copy is given to the employee.

A&L Great Lakes Laboratories, Inc. employees receive initial ethics training on their first day of employment, or as soon as feasibly possible thereafter. Employees also receive annual ethics re-training. A&L Great Lakes Laboratories, Inc. provides a way for any individual facing an ethical problem or other concern to address it anonymously, if they wish to do so, by using an "Open Line" form supplied by the office.

Unethical activities are defined as intentional falsification of records. Records may be personal credentials, resumes or educational transcripts, instrument logbooks, maintenance logbooks, raw data and data reports. Scientific misconduct is defined as intentionally not adhering to the prescribed method or Standard Operating Procedure (SOP). Common falsifications include, but are not limited to:

- **Falsifying data** – The process of making up/creating data without performing the procedure.
- **Improper peak integration** – Intentionally integrating data chromatograms so quality control samples meet acceptance criteria. This is also known as peak shaving or peak enhancing.
- **Improper clock setting** – Readjusting the computer clock so that it appears samples were analyzed within hold times. This is also known as time traveling.
- **Improper representation of Quality Control samples** – Misrepresenting analytical spikes as matrix (digested) spikes, analyzing a blank or LCS without processing it using the correct prep procedure, and treating a QC sample differently from a client sample.
- **Improper calibration** – Manipulating the calibration or tune so that it meets QC criteria. Examples are deleting/discarding calibration points along a curve or forging tuning data so that it appears to have met calibration criteria.
- **File substitution** – Substituting invalid calibration data with valid data from a different time so that the analysis appears to be successful.
- **Hiding or concealing a problem** – Concealing a known analytical or sample problem or ethical problem.

A&L Great Lakes Laboratories, Inc. will not tolerate these or any other unethical or improper activities or behavior. Violation of this policy may lead to repercussions ranging from a severe reprimand to immediate termination, and possible criminal prosecution if warranted by the situation. Allegations of misconduct shall be dealt with in a fair, unbiased and timely manner and all parties involved shall be advised of the procedures available to them. Persons against whom allegations of misconduct have been made shall be advised of the allegations, and shall be afforded the opportunity to respond. A&L Great Lakes Laboratories, Inc. has access to many other resources that may be utilized at any time to help clarify a situation determined to be a "gray area." Employees are strongly encouraged to seek further guidance whenever doubt is raised.

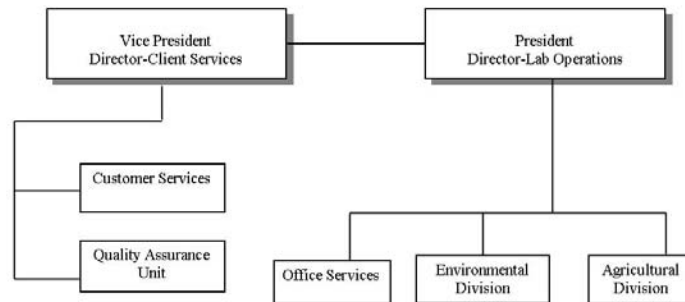
4.0 QA ORGANIZATION AND PERSONNEL

The organization of A&L Great Lakes Laboratories, Inc consists of five operating departments or divisions (Figure 1). Personnel from each department or division answer to a supervisor, manager or directly to the President.

The Agricultural and Environmental Divisions are responsible for all sample processing and laboratory analyses. Data processing, reporting and billing, are the responsibility of the Office Services section. Customer services include Marketing and Technical Services.

The Quality Manager is a staff level position working under and answering directly to the President and/or Vice-President.

A & L GREAT LAKES LABORATORIES, INC. Organization Chart



4.1. LABORATORY ORGANIZATION

All employees engaged in making decisions affecting the quality of laboratory output undergo training programs designed to be commensurate with their positions, duties, and responsibilities. It is the responsibility of each analyst to perform their assigned task according to the applicable SOP's, Protocols, Work Plans, etc. This responsibility includes performing quality control analyses as specified in the SOP, Protocol or Work Plan. Any out-of-control results are reported immediately to the analyst's supervisor.

The Technical Director and Department Managers / Supervisors ensure that analysts and technicians are trained in all applicable QA methods and procedures. The Technical Director and Department Managers / Supervisors review QC data at frequent intervals designed to ensure that QC analyses are being performed at the required frequency, data are properly documented and established corrective action procedures for out-of-control situations are followed and the results documented.

The Director of Laboratory Operations has overall responsibility for all activities in the operation of the laboratory. The Director determines the effectiveness of the QA program, recommends changes in the QA program to the QA Manager, approves changes to the Quality Assurance Manual and SOPs and serves as the focal point for the reporting and disposition of all non-conformance. The Director also ensures that there is a qualified Technical Director and Quality Manager, that personnel and other resources are adequate and that personnel have been informed of their responsibilities.

The Quality Assurance Unit is responsible for conducting systems audits and inspections for compliance with this manual, SOP's, protocols, and any other regulatory requirements.

4.2. DESCRIPTION OF RESPONSIBILITIES

Job descriptions are located in each employee's respective training file.

4.2.1. Technical Director

The A&L Great Lakes Laboratories, Inc. Technical Director is responsible for NELAC Accredited laboratory operations. Responsibilities include but are not limited to:

1. Monitoring standards of performance in quality control and quality assurance
2. Monitoring the validity of the analyses performed and data generated in the laboratory to assure reliable data
3. Certifying and documenting that only personnel with appropriate educational and/or technical background perform all tests for which the laboratory is accredited

The Technical Director shall have at least a bachelor's degree in the chemical, environmental, biological, physical sciences or engineering, with at least 24 college semester credit hours in chemistry and 16 college semester credit hours in general microbiology and biology. The Technical Director shall also have at least 2 years of experience in the environmental analysis of representative inorganic, organic and microbiological analytes for which the laboratory seeks or maintains accreditation. A masters or doctoral degree in one of the above disciplines may be substituted for one year of experience.

The Quality Manager will serve as Deputy to the Technical Director for chemical analysis in the event that he/she is absent for a period of time exceeding 15 consecutive calendar days. If this absence exceeds 65 consecutive calendar days, the primary accrediting authority shall be notified in writing.

4.2.2. Quality Manager

The Quality Manager and his or her designees shall:

1. Develop and carry out quality control programs, including statistical procedures and techniques, which will enable the laboratory to meet desired quality standards
2. Monitor quality control activities of the laboratory to determine conformance with authorized policies and procedures as well as good laboratory practices

3. Maintain all records relating to Quality Assurance needs of the company
4. Maintain and operate that portion of the Laboratory Information Management System that is concerned with quality control monitoring
5. Perform monthly inspections to ensure that quality control in all departments meets requirements set forth by management
6. Perform study progress and final report inspections for all GLP (40 CFR 160) work
7. Assist company personnel in developing SOP's in a consistent format
8. Serve as a liaison for all audits and inspections by A&L customers or regulatory agencies
9. Perform bi-annual facility inspections and report findings to management
10. Perform periodic analytical method and procedural inspections and report findings to management.
11. Notify laboratory management of deficiencies in the quality system and monitor corrective action.
12. Have functions independent of laboratory operations in which they have quality assurance oversight to ensure non-bias and objectivity.
13. Have a general knowledge of the analytical test methods for which data review is performed and be able to evaluate that data objectively and perform assessments without outside influence.
14. Have documented training and/or experience in QA/QC procedures and be knowledgeable in the quality system as defined under NELAC.

The Quality Manager has a bachelors degree in the chemical, environmental, biological, physical sciences or engineering, with 24 college semester credit hours in chemistry and 2 years of experience in the environmental analysis of representative inorganic and organic analytes for which the laboratory seeks or maintains accreditation. A masters or doctoral degree in one of the above disciplines may be substituted for one year of experience.

If the Quality Manager is absent for a period of time exceeding 15 consecutive calendar days the Quality Assurance Coordinator will serve as the Deputy Quality Manager.

4.2.3. Department Managers / Supervisors

Department Managers / Supervisors oversee the day-to-day production and quality activities of the laboratory. Specific duties include:

1. Provide Supervision of laboratory operations
2. Ensure proper scheduling and execution of testing programs
3. Assess data quality and take corrective action if needed
4. Coordinate management of projects
5. Lead the training of analysts in laboratory operations and analytical procedures
6. Organize and schedule sample analysis with consideration to sample holding times
7. Evaluate instrument performance and supervise instrument calibration and preventative maintenance programs
8. Report non-compliance situations to the Quality Manager and Director of Laboratory Operations
9. Ensure samples are received properly and that Chain of Custody documentation is complete

Department managers will have at least an associate's degree in chemical, environmental, biological, physical sciences or engineering and at least 1 year of experience in the environmental analysis of representative inorganic and organic analytes for which they are managing. A masters or doctoral degree in one of the above disciplines may be substituted for one year of experience.

4.2.4. Analysts

Analysts, who include chemists and technicians, are responsible for tasks identified in the scope of work. Specific duties include:

1. Assist in planning for each phase of their tasks and in defining objectives and activities
2. Train alternate analysts in specified laboratory QC and analytical procedures
3. Verify that laboratory QC procedures are being followed
4. Review QC data (includes examination of raw data such as chromatograms as well as inspection of reduced data, calibration curves and laboratory bench sheets/notebooks)

4.3. TRAINING AND ORIENTATION

Upon hire, each new employee undergoes training in several areas of the laboratory. A&L Great Lakes Laboratories, Inc. has a continual safety program in place that begins when a person enters into the employment of the company. The employees are trained and educated in safety procedures (e.g. proper handling of chemicals, operation of safety equipment, general safety policies, Chemical Hygiene Plan and emergency procedures) with the help of video tape programs, slides, wall charts, and on-hand experiences in dealing with the prevention of potential hazards. Safety training is conducted by the Safety Officer and is consistent with the requirements of OSHA's Hazard Communication Program (29 CFR 1920.1200). Safety policies are detailed in the A&L Great Lakes Laboratories, Inc. Chemical Hygiene Plan. Safety awareness meetings for all laboratory personnel are held to focus on one or more aspects of safety that are of special interest. Safety training is documented in each employee's respective personnel file. "On-going" safety training is documented in each employee's respective training record.

The general quality assurance training introduces the new employee to A&L Great Lakes Laboratories, Inc. quality assurance procedures. Each applicable facility SOP must be "Read & Understood" and documented in the training record. The quality assurance training focuses on the A&L Great Lakes Laboratories, Inc. Quality Assurance Manual. This training is based on the most current version of the QA Manual and is documented on the employee's training log which is kept in the employee's training file after it is signed and dated by the trainee and the Quality Assurance Unit. Each employee is made aware of new revisions and documents in their training record that they have "read and understood" any changes or updates to the Quality Assurance Manual. Employees are encouraged to use the QA Manual as a reference at any time.

A&L Great Lakes Laboratories, Inc. has taken steps to prevent fraud, falsification of data or other illegal actions (See section 3.0 and Appendix A). Employee training includes review of the A&L Great Lakes Laboratories, Inc. ethics policy statement. Each employee must sign and date a document that denotes understanding and agreement with the A&L Great Lakes Laboratories, Inc. ethics policy statement including knowledge of the penalties involved with improper, unethical or illegal actions. Additionally, during quality assurance audits and daily data review by management

an explicit effort will be made to discover any illegal or unethical actions. Any such behavior will be immediately communicated to the laboratory director.

Employees beginning a new job function or learning a new method must undergo specific training for the methods to be used. This training begins with instruction and demonstration of techniques and continues with hands-on experience in the laboratory with close observation by a qualified analyst.

After initial training, each analyst performing a test completes an initial demonstration of capability. This test involves analyzing a sample spike or standard (for analytes which do not lend themselves to spiking) four times and determining an average recovery and a relative standard deviation. These results are compared with available method performance criteria. The completed proficiency forms document that the analyst has successfully demonstrated a level of proficiency with the method. Each analyst will perform an annual ongoing demonstration of capability for each method performed. Only analysts who have completed training may independently conduct analytical methods.

Additional quality assurance training is provided to those employees who work with GLP (40 CFR 160) samples or other regulatory type samples.

The following types of training will be made available to personnel when necessary to carry out assigned job functions:

- On the job training will be provided for specific tasks. No person shall be permitted to do reportable work without appropriate on-the-job training.
- In-house classroom study
- Outside seminars / training courses
- University Classes
- Specialized training by instrument manufacturers
- Participation in proficiency programs

Current Curricula Vitae and records of training will be kept for all personnel actively involved with generating lab data, supervising laboratory personnel or performing quality assurance functions.

4.4. LABORATORY SAFETY

At A & L Great Lakes Laboratories, the safety of its employees has top priority. It has a current safety program organized, maintained and updated by the safety officer. The purpose of this program is to train and educate employees about the known and potential hazards throughout the laboratory. Safety guidelines have been written and safety equipment has been installed and made available to employees to prevent injury or property damage and loss.

It is the responsibility of all managers, supervisors, and employees to follow the safety guidelines and to properly carry out the safety program.

4.5. SECURITY

A&L Great Lakes Laboratories, Inc. maintains security in several ways. First, access to the laboratory building is controlled via an alarm system. During operational hours, visitors must sign in the Visitor's Logbook and read the "Guidelines for Visitors" SOP **ADM-0-006**. They must also wear a Visitor's Badge and either be authorized (maintenance and delivery personnel) or be accompanied by an A&L Great Lakes Laboratories, Inc. employee. Upon leaving the laboratory, the visitor must then sign out on the Visitor's Logbook. The Data archive is locked and access strictly controlled by an authorization list.

4.6. CONFIDENTIALITY

All information and documents pertaining to client specific analyses are held in the highest confidence. Data is only released to the owner of the account under which data was generated. If anyone other than the client is to receive data, permission must be granted from the client via a signed and dated authorization form. Additionally, A&L Great Lakes Laboratories, Inc. has entered into confidentiality (or secrecy) agreements with several clients whose work is considered proprietary in nature. In the event of a regulatory audit, SOP **ADM-0-006** covers how A&L Great Lakes Laboratories, Inc. handles request for client data by regulatory agencies.

5.0 QUALITY ASSURANCE OBJECTIVES

The objective of the Laboratory Quality Assurance Program is to ensure that only data of known quality is reported by the laboratory. All data released to clients of A&L Great Lakes Laboratories must be scientifically valid, defensible and of known precision and accuracy.

Specific objectives:

1. To develop and put into service rugged methods capable of meeting the user's needs for precision, accuracy, sensitivity, and specificity.
2. To ensure that all staff members receive training in basic quality technology, in sufficient depth to enable them to carry out the provisions of this document.
3. To establish the level of quality of the laboratory's routine performance as a baseline against which to measure the effectiveness of quality improvement efforts.
4. To make any changes in routine methodology found necessary to make it compatible with performance needs.
5. To monitor the routine operational performance of the laboratory through participation in PE studies and testing programs from several different agencies and to provide for corrective actions as necessary.
6. To improve and validate laboratory methodologies by participation in method validation studies.

5.1. CONTROL LEVELS

Control data generated by the A&L Great Lakes Laboratories, Inc. is evaluated at the 99% confidence level (mean +/- 3 standard deviations) for the control limits and at the 95% confidence level (mean +/- 2 standard deviations) for warning limits. Controls, check samples, surrogate spike recoveries, matrix spike recoveries, standard reference materials and PE samples are used, where appropriate, to monitor analytical accuracy.

5.2. PRECISION AND ACCURACY

5.2.1. Precision

Precision measures the reproducibility of repetitive measurements. It is strictly defined as the degree of mutual agreement among independent measurements as results of repeated application of the same process under similar conditions. Analytical precision is a measurement of the variability associated with duplicate (two) or a replicate (more than two) analysis of the same sample in the laboratory and is determined by analysis of laboratory duplicate/replicate samples. Total precision is a measurement of the variability associated with the entire sampling and analysis process.

5.2.2. Accuracy

Accuracy is a statistical measurement of correctness and includes components of random error associated with a measurement. The accuracy of a measurement can be determined by how close the measured value is from the known concentration or true value of the spike or standard (usually expressed in units of standard deviation).

5.3. REPRESENTATIVENESS

Representativeness is a qualitative element that is related to the ability to collect a sample that reflects the characteristics of that part of the environment that is to be assessed. Sampling representativeness is largely dependent on the sampling technique used.

5.4. COMPARABILITY

Comparability is how closely comparable data match data generated by different parties. This can be established by independent confirmation or from approved performance evaluation (PE) or proficiency testing (PT) studies (See Section 13.0).

5.5. TRACEABILITY

Traceability is the extent to which results can be substantiated by documentation. Analytical results can be traced through documentation back to measurement standards and then to the original certificates of purity from the manufacturer. Chain of Custody documentation, sample receipt logs, refrigerator/freezer logs, equipment logs and bench sheets provide traceability to the history of the sample from sampling through analysis.

6.0 SAMPLING PROCEDURES

6.1. INTRODUCTION

Obtaining representative samples and maintaining their integrity are critical parts of accurate analyses. Results are only as good as the sampling and preservation methods used in collecting samples. In sampling, the objective is to remove a small portion of an environment that is representative of the entire body. Once the sample is taken, the constituents must stay in the same condition as when collected. The length of time that these constituents will remain stable is related to their character and the preservation method used.

6.2. SAMPLING SUPPORT

A&L Great Lakes Laboratories, Inc. provides clients with shipping containers, Chain-of-Custody documents, coolers, ice packs, bottles, labels, chemical preservatives, etc. in support of sampling procedures.

6.3. PRESERVATION

Although A&L Great Lakes Laboratories, Inc. provides reagent grade chemical preservatives for water samples, those responsible for collecting the samples must properly use the materials to ensure that proper preservation requirements are met, including the maintenance of proper temperature. *See Table 7.1 and 7.2.*

6.4. SAMPLE CONTAINERS

A&L Great Lakes Laboratories, Inc. provides both clean and sterile sample containers. A variety of sizes and types of containers are available. Supplies (bottles) can also be ordered from our web site at <http://www.algreatlakes.com/>. *See Table 7.1 and 7.2 for additional information.*

6.5. ENSURING APPROPRIATE FACILITIES AND RESOURCES

The Laboratory Director and the Department Manager review every prospective project prior to receipt of samples. Each project is evaluated from the perspective of overall workload, appropriateness of test methods, client requirements, deliverables requirements, turnaround-time requirements, information resource requirements and laboratory personnel expertise. ADM-0-009 outlines A & L Great Lakes procedures for the review of requests, tenders, contracts and any associated sub-contracted laboratory work. Any differences between the request or tender and the contract shall be resolved before any work commences. After work has commenced, if it is determined – for any reason – that the project cannot be completed or if the laboratory accreditation status changes, the prospective client is notified within a reasonable time frame.

Reviews cover any work that is subcontracted by the laboratory and the client is informed of any deviation from the contract. The laboratory shall be responsible to the client for subcontracted work, except in the case where the client or a regulatory authority specifies which subcontractor is to be used. Contracts that are amended after work has commenced undergo a second contract review and those amendments are communicated to all affected personnel. Records of project reviews, discussions with clients and any significant changes are maintained.

6.5.1.

Records of reviews, including significant non-NELAP accredited work, whether in-house or sub-contracted are clearly identified by a subcontractor name or accreditation number in final reports. Clients are informed of subcontracting arrangements in writing and the laboratory performing the subcontracted work is indicated in the final report. A register of all subcontractors that the laboratory uses for environmental tests is maintained along with a record of the evidence of compliance with NELAC Quality Systems Revision 16 (5.4.5.1.). REC-9-001 outlines the maintenance and transfer of records in the event that the laboratory transfers ownership or goes out of business.

7.0 SAMPLE HANDLING AND CUSTODY

7.1. SAMPLE RECEIPT

Samples are received and stored in a secured laboratory according to SOP **GEN-2-001** “*Sample Receipt, Handling, and Storage*”. Once received at the sample receiving area, the samples are sorted according to department. Then, the contents are verified, receipt condition noted, and receipt signature/date added to the chain of custody. If there is any discrepancy between the samples received and the chain of custody, or, if there is inadequate sample volume or there is evidence of damage, contamination or inadequate preservation, the areas of concern will be documented and, if applicable, the client will be notified.

7.2. CHAIN OF CUSTODY

A completed chain of custody form must accompany the samples. A&L Great Lakes Laboratories, Inc. can supply clients with a chain of custody form (See **Appendix B**) or the client may use their own chain of custody. It is important that the following items are present and complete in order to accurately track the custody of the sample:

- A&L Account Number
- Company and Contact Information
- Project Related Information
- Unique Sample Identification for All Samples including Split Samples
- Sample Container Type
- Date and Time Sampled
- Sample Type
- Preservative
- Requested Analyses
- Sampled By Signature and Date
- Relinquished By Signature and Date
- Received By Signature and Date
- Receipt Condition

Additional items can be noted on the chain of custody such as special instructions, waybill number, shipping condition, temperature of samples etc. In the event that a sample arrives without a chain of custody, the client will be notified. If the chain of custody can be obtained, the client will be requested to send it to A&L. There may be some circumstances in which A&L will complete the chain of custody by request of the client.

7.4. SAMPLE VERIFICATION

Immediately upon opening a sample shipment, the contents are verified against the chain of custody or sample submittal form. The following represent items checked:

- Condition of the samples (e.g. intact, no broken seals or containers etc.)
- Present and Complete Chain of Custody
- Labeling (container vs. chain of custody)

Any discrepancies found are documented and the client is immediately notified.

7.5. SAMPLE LOGGING

After samples have been properly received, all required information is entered into the A&L Great Lakes Laboratories, Inc. Laboratory Information Management System (LIMS). A written record of sample receipt is also kept, including the date of receipt, sample condition, sample identification, and assigned laboratory number. The LIMS is a tool that tracks the sample as it progresses through all requested analyses through final reporting. Each analysis requested is logged into LIMS and tracked by an analysis code system. Each sample is assigned a unique laboratory ID number and sample containers are labeled with the corresponding laboratory ID number. Once a particular analysis is complete, the result, date and technician are recorded for that sample.

Regulatory samples are logged according to SOP **GEN-4-001** “*Logging Samples Under GLP Protocol*”.

7.6. SAMPLE STORAGE

7.6.1. General Procedures

As a general rule, all samples are processed immediately upon receipt to prevent any sample degradation. A unique laboratory ID number is assigned and attached as a durable label to the sample container for reference.

7.6.2. Refrigerated Storage / Maintenance

Refrigerators are maintained according to SOP **RAM-1-001** “*Maintenance of Refrigerators and Freezers*”. Each unit has a min/max and a submersible thermometer (calibrated annually against NIST traceable thermometer according to SOP **GEN-1-001** “*Thermometer Calibration*”) that is read and recorded on a maintenance log each morning upon opening. The temperature is maintained at 4° Celsius (+/-2° C). Regulatory samples housed in the refrigerators will be logged onto the unit's Sample Residence Log. The log documents sample activity. Security is maintained as a result of limited access, total building security systems and a very strict visitor policy.

In the event that the temperature falls outside tolerance limits, the following corrective action will be taken:

1. Temperature re-checked after waiting 1 hour to verify that temperature is out of tolerance.
2. Any adjustment made to the refrigerator temperature control is recorded on the log.
3. If temperature still cannot be maintained, then the samples will be moved to a properly functioning refrigeration unit.

7.6.3. Freezer Storage / Maintenance

Freezers are handled exactly as specified in section 7.5.2. with the exception that they are maintained at a temperature of -20° Celsius (+/-5° C) and that they require periodic defrosting which is documented on the log.

7.7. SAMPLE / DATA ACCESS

Sample and data access is controlled by A&L Great Lakes Laboratories, Inc. A system of policies and SOP's are in place restricting access to only authorized personnel and protecting client specific data. Only employees and authorized personnel are allowed into the laboratory facilities. Visitors are accompanied by an employee and must wear a "Visitor" badge.

Analytical Data is entered into the LIMS system that has password security built in. Only authorized personnel can access the LIMS system.

7.8. SAMPLE DISPOSAL

Samples not consumed by analysis are disposed according to SOP **GEN-6-001** "*Disposal of Sample Material after Analysis*". The laboratory follows these specific guidelines:

1. Highest priority will be given to waste minimization whenever possible.
2. Agricultural samples from quarantined areas will be autoclaved before discarding.
3. Every effort will be made to minimize the size of incoming samples that may present disposal problems.
4. Routine samples and wastes that are not considered to be hazardous will be disposed of through standard refuse hauling services.
5. Samples that are determined to be hazardous or environmentally unsound for disposal to landfills shall be returned to clients after completion of requested analyses and after reasonable holding time.
6. Reagents, chemicals or samples that cannot be discarded to non-hazardous landfills or returned to clients will be containerized labeled and disposed of through licensed contractors. The preferred method of disposal through these contractors shall be incineration.

7.8.1. Hazardous Samples and Samples of Unknown Origin.

Samples that are potentially heavily contaminated are tagged as "Hazardous", are stored separately and sent back to client after analysis or lab packed for proper disposal. Samples from unknown origin such as foreign soil or a sample from a quarantined area within the United States are autoclaved prior to disposal.

TABLE 7.1

List of Containers, Preservatives and Holding Times for Inorganic and Organic Analyses of Aqueous Samples:

NAME	CONTAINER ¹	PRESERVATION ²	MAXIMUM HOLDING TIME ³
Bacterial Tests:			
Coliform, total, fecal, & <i>E. coli</i>	PA,G	Cool, ≤10 °C, 0.0008% Na ₂ S ₂ O ₃ ⁸	6 hours
Inorganic Tests:			
Acidity	P,G,FP	Cool, ≤6 °C	14 days
Alkalinity	P,G,FP	Cool, ≤6 °C	14 days
Ammonia	P,G,FP	Cool, ≤6 °C, H ₂ SO ₄ to pH<2	28 days
Biochemical oxygen demand	P,G,FP	Cool, ≤6 °C	48 hours
Bromide	P,G,FP	None required	28 days
Biochemical oxygen demand, carbonaceous	P,G,FP	Cool, ≤6 °C	48 hours
Chemical oxygen demand	P,G,FP	Cool, ≤6 °C, H ₂ SO ₄ to pH<2	28 days
Chloride	P,G,FP	None required	28 days
Chlorine, Total Residual	P,G	None required	Analyze within 15 minutes
Color	P,G,FP	Cool, ≤6 °C	48 hours
Cyanide, total or available (or CATC)	P,G,FP	Cool, ≤6 °C, NaOH to pH>12 0.6g ascorbic acid ⁴	14 days
Fluoride	P	None required	28 days
Hardness	P,G,FP	HNO ₃ to pH<2, H ₂ SO ₄ to pH<2	6 months
Hydrogen ion (pH)	P,G,FP	None required	Analyze within 15 minutes
Kjeldahl and Organic nitrogen	P,G,FP	Cool, ≤6 °C, H ₂ SO ₄ to pH<2	28 days
Metals:			
Chromium VI	P,G,FP	Cool, ≤6 °C pH=9.3-9.7 ⁷	28 days for preserved samples or 24 hours for unpreserved samples
Mercury (SW846)	P,G,FP	HNO ₃ to pH<2	28 days in glass
Mercury (CLP, 200 series)	P,G,FP	HNO ₃ to pH<2	28 days
Metals, except Chromium VI and Mercury	P,G,FP	HNO ₃ to pH<2, or at least 24 hrs. prior to analysis.	6 months
Nitrate	P,G,FP	Cool, ≤6 °C	48 hours
Nitrate-nitrite	P,G,FP	Cool, ≤6 °C, H ₂ SO ₄ to pH<2	28 days
Nitrite	P,G,FP	Cool, ≤6 °C	48 hours
Oil and Grease	G only	Cool, ≤6 °C, H ₂ SO ₄ to pH<2	28 days
Organic Carbon, Total (TOC)	P,G,FP	Cool, ≤6 °C, H ₂ SO ₄ to pH<2	28 days
Orthophosphate	P,G,FP	Filter immediately, Cool, ≤6 °C	48 hours
Phenols	G only	Cool, ≤6 °C, H ₂ SO ₄ to pH<2	28 days
Phosphorus (elemental)	G only	Cool, ≤6 °C	48 hours
Phosphorus, Total	P,G,FP	Cool, ≤6 °C, H ₂ SO ₄ to pH<2	28 days
Residue, Total	P,G,FP	Cool, ≤6 °C	7 days
Residue, Filterable	P,G,FP	Cool, ≤6 °C	7 days
Residue, Nonfilterable (TSS)	P,G,FP	Cool, ≤6 °C	7 days
Residue, Settleable	P,G,FP	Cool, ≤6 °C	48 hours
Residue, Volatile	P,G,FP	Cool, ≤6 °C	7 days
Silica	P or quartz	Cool, ≤6 °C	28 days

A&L GREAT LAKES LABORATORIES, INC.

(Cont.) List of Containers, Preservatives and Holding Times for Inorganic and Organic Analyses of Aqueous Samples:

NAME	CONTAINER ¹	PRESERVATION ²	MAXIMUM HOLDING TIME ³
Inorganics Continued:			
Specific conductance	P,G,FP	Cool, ≤6 °C	28 days
Sulfate	P,G,FP	Cool, ≤6 °C	28 days
Sulfide	P,G,FP	Cool, ≤6 °C, add zinc, acetate & sodium hydroxide to pH>9	7 days
Sulfite	P,G,FP	None required	Analyze within 15 minutes
Surfactants	P,G,FP	Cool, ≤6 °C	48 hours
Turbidity	P,G,FP	Cool, ≤6 °C	48 hours
Organic Tests:			
Purgeable Halocarbons	G, FP-lined septum	Cool, ≤6 °C, 0.008% Na ₂ S ₂ O ₃ , HCl ^{5,6} to pH 2	14 days
Purgeable Aromatic Hydrocarbons	G, FP-lined septum	Cool, ≤6 °C, HCl ^{5,6} to pH 2	14 days
Acrolein and Acrylonitrile	G, FP-lined septum	Cool, ≤6 °C	2 days
Phenols	G, FP-lined cap	Cool, ≤6 °C	7 days until extraction, 40 days after extraction
Benzidines	G, FP-lined cap	Cool, ≤6 °C	7 days until extraction ⁹
Phthalate esters	G, FP-lined cap	Cool, ≤6 °C	7 days until extraction, 40 days after extraction
Nitrosamines	G, FP-lined cap	Cool, ≤6 °C, store in dark	7 days until extraction, 40 days after extraction
PCBs	G, FP-lined cap	Cool, 4° C	1 year until extraction, 1 year after extraction
Nitroaromatics and cyclic ketones	G, FP-lined cap	Cool, ≤6 °C, store in dark	7 days until extraction, 40 days after extraction
Polynuclear aromatic hydrocarbons	G, FP-lined cap	Cool, ≤6 °C, store in dark	7 days until extraction, 40 days after extraction
Haloethers	G, FP-lined cap	Cool, ≤6 °C	7 days until extraction, 40 days after extraction
Chlorinated Hydrocarbons	G, FP-lined cap	Cool, ≤6 °C, HCl or H ₂ SO ₄	7 days until extraction, 40 days after extraction
Dioxins and Furans	G, FP-lined cap	Cool, ≤6 °C, 0.008% Na ₂ S ₂ O ₃ ⁴ , pH<9	1 year until extraction, 1 year after extraction
Total Organic Halides (TOX)	G, FP-lined cap	Cool, ≤6 °C, HCl or H ₂ SO ₄ to pH <2	28 days
Pesticides	G, FP-lined cap	Cool, ≤6 °C, pH 5-9	7 days until extraction, 40 days after extraction

Table Footnotes:

1. Polyethylene (P), Glass (G), or Fluoropolymer (polytetrafluoroethylene (PTFE; Teflon®) (FP); "PA" is any plastic that is made of a sterilizable material (polypropylene or other autoclavable plastic).
2. Sample preservation should be performed immediately upon sample collection.
3. Holding times are based from time of sample collection.
4. Should only be used in the presence of residual chlorine.
5. Free chlorine must be removed prior to addition of HCl by the appropriate addition of Na₂S₂O₃
6. Sample receiving no pH adjustment must be analyzed within seven days of sampling.
7. To achieve the 28-day holding time, use the ammonium sulfate buffer solution specified in EPA Method 218.6. The allowance in this footnote supersedes preservation and holding time requirements in the approved hexavalent

chromium methods, unless this supersession would compromise the measurement, in which case requirements in the method must be followed.

8. Add a reducing agent only if an oxidant (a.g., chlorine) is present. Reducing agents shown to be effective are sodium thiosulfate ($\text{Na}_2\text{S}_2\text{O}_3$), ascorbic acid, sodium arsenite (NaAsO_2), or sodium borohydride (NaBH_4). However, some of these agents have been shown to produce a positive or negative cyanide bias, depending on the other substances in the sample and the analytical method used. Therefore, do not add an excess of reducing agent. Whatever agent is used, it should be tested to assure that cyanide results are not affected adversely.
9. Extracts may be stored up to 30 days at $<0^\circ\text{C}$.

TABLE 7.2

Required Containers, Preservation Techniques and Holding Times for Non-Aqueous, Soil or Solid Matrices (as specified in SW-846)

Name	Container	Preservation	Maximum Holding Time ¹
Semi-volatile Organics / Organochlorine Pesticides / PCBs and Herbicides			
Concentrated waste samples	8 oz. wide mouth glass w/Teflon liner	None	14 days until extraction, 40 days after extraction
Liquid samples, no residual Chlorine present	1 gal. or 2 ½ gal. amber glass w/Teflon liner	Cool, 4° C	Samples must be extracted within 7 days & extracts analyzed within 40 days
Residual Chloride, present	1 gal. or 2 ½ gal. amber glass w/Teflon liner	Add 3mL 10% sodium thiosulfate	Samples must be extracted within 7 days & extracts analyzed within 40 days
Soil/sediments and sludges	8 oz. wide mouth glass w/Teflon liner	Cool, 4° C	14 days until extraction, extracts analyzed within 40 days
Volatile Organics			
Concentrated waste samples	8 oz. wide mouth glass w/Teflon liner	None	14 days
Liquid samples, no residual Chlorine present	2x40 mL vials w/Teflon lined septum caps	Cool, 4° C ²	14 days
Residual Chlorine, present	2x40 mL vials w/Teflon lined septum caps	Collect sample in a 4oz. Soil VOA container which has been pre-preserved w/4 drops of 10% sodium thiosulfate. Gently mix sample & transfer to a 40 mL VOA Vial ² . Cool to 4° C	14 days
Acrolein & Acrylonitrile	2x40 mL vials w/Teflon lined septum caps	Adjust to pH 4-5, Cool to 4° C	14 days
Soil/sediments and sludges	4 oz. (120mL), wide mouth glass w/Teflon liner or wide mouth glass container sealed w/a septum	Cool to 4° C	14 days

Table Footnotes:

1. Holding times are based from time of sample collection.
2. Adjust pH<2 w/H₂SO₄, HCl or solid NaHSO₄

8.0 PURCHASING SERVICES AND SUPPLIES

8.1 PROCUREMENT / INVENTORY

A&L Great Lakes Laboratories, Inc. purchases analytical supplies from a variety of reputable vendors. All vendors are evaluated based on service, quality, response time, and price. Records of evaluations of vendors that supply critical consumables, supplies and services affecting the quality of environmental testing and a list of those approved is maintained. Purchasing documents for items affecting the quality of laboratory output shall contain data describing the services and supplies ordered. Each department manager has the authority to determine the vendor best suited to meet their needs and shall review and approve the technical content of all said purchasing documents prior to release. Original containers received from the vendor must be labeled with an expiration date. Chemicals, standards, and other laboratory consumables are stored in the various chemical and flammables storage cabinets. Lot numbers of chemicals or reagents found to be contaminated are disposed of immediately, and a replacement is ordered. Material Safety Data Sheets (MSDS) are kept on the networked computer system for all chemical purchases. Certificates of Analysis (where applicable) are kept on file. Certificates of Analysis for standards provide a traceable record of the standard to a NIST standard. Chemical Certificates of Analysis (for non-standards) provide information on its constituents and concentrations in the chemical. A&L SOP **GEN-3-001** “*Labeling of Reagents and Reagent Solutions*” details the procedure for labeling chemicals received by the laboratory.

8.2 GLASSWARE

A&L Great Lakes Laboratories, Inc. uses only laboratory grade glassware and supplies purchased from nationally known supply houses. Prior to use, all glassware is cleaned using Contrad NF®, Chemsolve® or other appropriate laboratory detergent and rinsed thoroughly with deionized water according to SOP **GEN-1-003** “*Maintenance of Laboratory Glassware*”. Additional glassware preparation (removal of adsorptive site) may be necessary depending on the method. Glassware is stored in a clean environment.

8.3 DEIONIZED WATER

SOP **GEN-1-004** “*Deionized Water System Maintenance*” details the procedure for maintaining the deionized water ion exchange system.

8.4 HIGH PURITY WATER

SOP **ENV-1-006** “*Operation and Maintenance of the Barnstead B-Pure Water Purification System*” and SOP **ENV-1-046** “*Operation and Maintenance of the Barnstead EASY Pure II LF*” detail the procedures for maintaining the ultra-pure water filtration systems.

9.0 INSTRUMENTATION, EQUIPMENT & PREVENTATIVE MAINTENANCE

There are two objectives of A&L Great Lakes Laboratories, Inc. preventive maintenance program. First, to establish a system of instrument care that maintains equipment at desired levels of calibration and sensitivity. The second objective is to minimize loss of productivity due to repairs. The program includes a system for documenting all routine and non-routine instrument maintenance and repairs.

See **Section 19.0** for a listing of instrumentation and major pieces of lab equipment. Each piece of laboratory equipment or analytical instrumentation is calibrated prior to each use to ensure proper functioning. All instrument calibrations are performed by an experienced analyst. Instruments are calibrated using certified standards traceable to recognized national standards or using reference standards whose values have been statistically validated. Many of the instruments in the laboratory are covered by a service contract that involves preventative maintenance checks by a service technician. All maintenance is documented and can be used as a source of information in solving instrument problems.

Additionally, the laboratory stocks many parts considered consumable for most instruments in order to minimize downtime. A&L Great Lakes Laboratories, Inc. utilizes equipment redundancy when possible. In the event one instrument goes down, another instrument is available to meet hold times and client due dates. Support equipment is monitored regularly to confirm proper functioning. The temperature of all drying ovens, refrigerators, freezers, and incubators must be checked each working day (not on weekends or holidays). The accuracy of each thermometer associated with a piece of support equipment is checked annually using calibrated thermometers.

9.1 MAINTENANCE RESPONSIBILITIES

The laboratory Operations Manager and department Manager/Supervisors are responsible for providing technical leadership to all staff, including serving as a technical resource to help solve equipment and method problems, evaluating and recommending investments in new technologies, improving efficiency, and coordinating instrument repair and maintenance. The analysts have a primary responsibility to perform routine maintenance.

9.2 MAINTENANCE SCHEDULES

To minimize downtime and interruption of analytical work, preventive maintenance is routinely performed on analytical instrumentation. Standard Operating Procedures (SOP's) are written for each instrument that covers basic operation, maintenance procedures, and maintenance schedules.

9.3 MAINTENANCE DOCUMENTATION

9.3.1 Routine Maintenance

All routine and non-routine instrument maintenance is documented in maintenance and calibration logbooks assigned to each instrument. Maintenance logs will document the date and extent of all maintenance and repairs. Maintenance will be performed by qualified laboratory personnel or a certified manufacturer's representative.

9.3.2 Non-Routine Maintenance and Repair

When non-routine maintenance or repair is performed on an instrument, SOP **GEN-1-002** "*Non-Routine Maintenance / Repair*" is followed. The following items are recorded:

- Equipment / Instrument Name
- Serial Number
- Nature of the defect or problem
- How the defect or problem was discovered
- When the defect or problem was discovered
- Remedial action taken in response to the defect or problem
- Person responsible signature / date

Under no circumstances will defective or malfunctioning instrumentation be used in the generation of analytical data. A defective instrument will be immediately removed from production until successful repair and/or calibration can be positively verified.

9.4. SPARE PARTS

When economically feasible, A&L Great Lakes Laboratories, Inc. maintains an adequate inventory of spare parts to minimize downtime in the event of instrument or equipment failure. Parts that most frequently wear out or that have limited useful lifetimes are most frequently inventoried. Department Managers/Supervisors are responsible for maintaining adequate inventories.

10.0 CALIBRATION PROCEDURES AND FREQUENCY

10.1. STANDARDS AND TRACEABILITY

Analytical standards are prepared from quality grade reagent compounds, are purchased as neat chemicals or diluted standard solutions from reputable vendors who can provide a certificate of analysis. They are used to prepare calibration and spiking standards. The preparation information is recorded on either the bench sheet or into a "Standard Preparation Log".

Each standard is assigned a unique identification number using a numbering system detailed in SOP **ENV-3-001**. Each prepared standard is traceable back to the original certificate of purity where available. Expiration dates are assigned as follows: If the manufacturer provides a date of expiration, that date will be recognized as the actual expiration date. For intermediate mixes, spiking solutions, working stocks, or daily calibration solutions, the expiration date will be no more than six months from the date of preparation. Under no circumstance should expired standards be used.

10.2. CALIBRATION PROCEDURES

Calibration standards for each parameter are chosen to bracket the expected concentration range of those parameters in the sample and to operate within the linear response range of the instrument. Samples that fall outside the calibration range are diluted until bracketed by the calibration standards. Calibration standards are prepared typically at a minimum of three concentration levels (app. 2-5x (or at the RL), 5-10x and up to 20x the MDL).

Instrumental responses to calibration standards for each parameter are subjected to an appropriate statistical test of fitness (least squares linear regression, quadratic equation, or relative standard deviation of response factors) or as required by the method. The calibration must reflect an acceptable correlation of data points or linearity to be acceptable. In cases where the calibration data are outside these criteria, the analyst must rerun the calibration standards and/or prepare new standards, changing instrumental conditions as necessary.

10.2.1 Analytical Balances

Every 12 months a professional outside vendor services each balance. The balances are cleaned and the calibration is checked throughout the entire analytical range. This service is documented on the maintenance and calibration record for each respective balance. Daily, before use, each balance is checked using weights traceable to NIST (calibrated annually by an outside certifying agency). All information regarding balance maintenance and calibration is recorded on the maintenance and calibration logbook and/or is maintained on file in the QA office.

10.2.2 Thermometers

Laboratory working thermometers are calibrated annually against certified or reference thermometers according to SOP **GEN-1-001**. Reference thermometers are traceable to NIST. Working thermometers used in refrigerators, freezers, incubators and water baths are also calibrated annually at the working temperature.

Every five years all reference thermometers are professionally calibrated.

Each thermometer is uniquely identified and, if necessary, tagged with the appropriate correction factor. Calibration temperatures and acceptance criteria are based upon the working range of the thermometer and the accuracy required in its use. Laboratory thermometer identification and calibration information are recorded in logbooks.

10.2.3 pH Meters

pH meters are calibrated before use each day using fresh buffer solutions (usually 4.0, 7.0 and/or 10.0). The efficiency of the meter is calculated during the calibration and must be between 92-102% for the calibration to be considered successful.

10.2.4 Spectrophotometers

During use, spectrophotometer performance is verified at established frequencies with calibration verification standards or control solutions.

10.3. GC/MS CALIBRATION PROCEDURES

The minimum operations necessary to satisfy analytical requirements associated with the determination of organic compounds in water and soil/sediment samples are listed below. The following operations are performed routinely in the laboratory:

- Documentation of GC/MS mass calibration and abundance pattern
- Documentation of GC/MS response factor stability
- Internal standard response and retention time

Prior to initiating data collection, it is necessary to establish that a given GC/MS meets the standard mass spectral abundance criteria. This is accomplished through the analysis of decafluorotriphenylphosphine (DFTPP) for the base/neutral and acid (BNA) compounds or p-bromofluorobenzene (BFB) for volatile compounds. Each GC/MS system used for analysis of volatile or a semivolatile organic compound is tuned to meet method or program specific ion abundance criteria before analysis of standards, blanks, or samples can proceed.

Prior to the analysis of samples and after tuning criteria have been met, the GC/MS system is initially calibrated using multiple concentrations to determine the linearity of response. The number of concentration points used for calibration depends on the specific method criteria and client project requirements. USEPA criteria specify both the concentration levels for initial calibration and the specific internal standards to be used on a compound-by-compound basis for quantitation.

A calibration check standard containing all compounds of interest as well as all required surrogates is performed each day of analysis. The RF data from the standard is compared against the average RF from the initial calibration for a specific instrument. The response must be within +/- 20% (or within tolerances stated in the referenced method) or corrective action must be taken.

10.4 NON GC/MS CHROMATOGRAPHY CALIBRATION PROCEDURES (i.e., GC and HPLC)

Initially, a three or five point calibration curve, consisting of all compounds of interest is established to define the usable range of the instrument. Calibration is established as best-fit line, quadratic equation, or average response factors (RF). Response factors are calculated per the analytical method used.

10.5 CALIBRATION OF INDUCTIVELY COUPLED ARGON PLASMA SPECTROPHOTOMETER (ICP) AND ATOMIC ABSORPTION SPECTROPHOTOMETER (AAS)

The ICP and AAS are standardized for the analyte of interest by the analysis of a set of calibration standards prepared by diluting a stock solution of known concentration. Working standards are prepared by dilution of the stock standard. After working standards have been prepared, they are analyzed on the ICP or AAS and the instrument response is calibrated to provide a direct readout of concentration. Independent controls are run to verify the calibration curve within the working range of the instrument. Any run that is out of tolerance will be re-run after the calibration problem is corrected.

11.0 ANALYTICAL PROCEDURES

A&L Great Lakes Laboratories, Inc. is capable of analyzing a wide range of environmental and agricultural samples from all media including surface and groundwater, soil, sediment, tissue, waste, plant tissue, feed and fertilizer. Methodologies are employed with guidance from federal agencies such as EPA and ASTM and, in certain instances, state regulatory agencies such as the Indiana State Department of Health. Methodologies are also used from widely accepted sources such as Standard Methods and SW846. A partial listing of references follows:

1. Handbook for Quality Control in Water and Wastewater Laboratories, U.S. EPA 600/4-79-019, March, 1979
2. Federal Register, 40 CFR Part 136, October 26, 1984.
3. Method Update Rule, Federal Register, Vol. 72, No. 47. March 12, 2007.
4. Method Update Rule, Federal Register, Vol. 72, No. 57. March 26, 2007.
5. Test Methods of Evaluating Solid Waste, Physical/Chemical Methods, SW-846 CDROM version 2.0, December 1997.
6. Standard Methods for the Examination of Water and Wastewater, APHA, AWWA, WPCF, current edition.
7. Methods for Chemical Analysis of Water and Wastes, EPA-600/4-79-020, 1983.
8. Pesticide Analytical Manual, FDA 3rd Edition, 1994.

11.1 ANALYTICAL METHODS

The choice of analytical methodology used depends on how the data will be used, the "type" of sample and regulatory requirements. A&L Great Lakes Laboratories, Inc. may modify existing methods based on the following considerations:

- To address unusual matrices not covered in available methods
- To meet project specific objectives
- To incorporate modifications or improvements in technology
- To comply with changing regulations and requirements

A&L Great Lakes Laboratories, Inc. will make every effort to disclose to its clients any instances in which modified methods are used in the analysis of samples.

11.2 PROCEDURE DOCUMENTATION AND DOCUMENT CONTROL

Standard Operating Procedures (SOPs) are intended to provide specific written guidelines as to how processes and procedures are carried out by company personnel. These documents also provide historical information to how procedures were carried out in the past. All processes involved with the production and reporting of analytical data must be documented through the use of SOPs.

1. A document (ADM-0-001 "Writing and Administration of Standard Operating Procedures") must be maintained as a guideline for the development of all SOPs.
2. SOP's must be maintained in a current fashion.
3. A historical file of SOPs must be kept in a secured place.

4. Out of date SOPs will be collected by the Quality Assurance Unit and destroyed (except for the historical file copy).
5. All substantial processes or procedures carried out by company personnel must be governed by current, written SOPs.

11.2.1 SOP Organization

Standard Operating Procedures (SOPs) are organized according to various departmental categories. Each SOP contains the revision number and date at the top of the SOP. It also has a unique number at the bottom of the page (e.g. 1 of 3, 2 of 4, etc.).

11.2.2 Review, Revision and Distribution

Each Standard Operating Procedure (SOP) undergoes an annual review. SOP review is documented on a document review log attached to the copy of the SOP that resides in the Quality Manager's office. Retired SOPs are maintained in the A&L Great Lakes Laboratories, Inc. secure archive with the date of retirement. The master SOP list additionally lists all current SOPs as well as retired SOP's.

11.2.3 Document Control

Standard Operating Procedures are controlled documents in which the following rules apply:

- Retirement and maintenance of the previous version
- Replacement of all controlled copies and destruction of retired version copies
- Notification of all affected personnel that a revision has been issued

11.3 METHOD VALIDATION

There are occasions when A&L Great Lakes Laboratories, Inc. performs method development for specific projects of interest. In these cases, method validity must be established by meeting certain criteria for precision and accuracy specified in data quality objectives set by the end user of the data.

11.4 METHOD DETECTION LIMITS

A&L Great Lakes Laboratories, Inc. determines method detection limits according to SOP **GEN-5-001** "*Determination of the Method Detection Limit*".

Estimate the MDL (Method Detection Limit) by multiplying 10 x the IDL (Instrument Detection Limit).

The IDL can be determined by using one of the following:

1. The concentration value that corresponds to an instrument's known signal/noise ratio.
2. The concentration value that corresponds to three times the standard deviation of replicate instrumental measurements for the analyte in reagent water.
3. The concentration value that corresponds to known instrumental limitations.

Prepare a laboratory standard (analyte in reagent water) at a concentration that is at least equal to or in the same concentration range as the estimated MDL. (1-5 times estimated MDL is recommended.)

Take seven aliquots of the standard to be used to calculate the MDL and process each through the entire analytical method.

Compute the Method Detection Limit (MDL) as follows:

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

Where t = Students' t value appropriate for a standard deviation estimate with n-1 degrees of freedom.

Where s = standard deviation

For reference information see:

1. *EPA 600/4-82-057 Methods for Organic Chemical Analysis of Municipal & Industrial Wastewater, Appendix A.*
2. *40 CFR 136, Appendix B.*

11.5 RETENTION OF RAW DATA AND SUPPORTING RECORDS

All Environmental raw data supporting an analytical result will be retained. Under no circumstances will raw data be destroyed prior to 5 years from the date of analysis (See SOP for record retention **REC-0-001**). Supporting documentation should allow for the reconstruction of an analytical result. The following records will be retained:

1. MDL Studies
2. Initial Demonstrations of Performance
3. Calibrations and standardizations
4. On-going calibration checks/verifications
5. Method Control, Method Blank and Matrix Spike/MSD data
6. Standard preparation data
7. Instrument Maintenance Logs
8. PE results
9. Temperature Logs: Refrigerator, Freezer, Incubator, Oven etc.
10. Analytical balance checks, calibrations and maintenance
11. Analytical reports
12. Waste management, Safety and Regulatory affairs

11.6 DOCUMENT CONTROL

A&L Great Lakes Laboratories, Inc. has a system in place to control revisions and distribution of controlled documents. The following summarize control procedures:

11.6.1 The Quality Assurance Manual

This document is maintained by the Quality Assurance Unit. It is revised as needed and approved by the signature of the Laboratory Director. Revisions are numbered and dated. A number of copies sufficient for each department are distributed after each revision is complete. A historical summary is included to easily track changes and updates made to the QA Manual. Earlier revisions are returned to the quality officer and destroyed. A copy of each earlier revision is maintained in archive.

11.6.2 Standard Operating Procedures (See section 11.2)

11.6.3 Calibration and Maintenance Logs

The date performed, analyst's initials, and any pertinent identifying material or observations are noted. Each log book is assigned a sequential number and pages are also numbered sequentially. These logs, when full, are transferred to A&L Great Lakes Laboratories, Inc. secure archives.

11.6.4 Organizational Charts

Organizational charts are updated as employees join, leave, or are promoted within the company. Each organizational chart has the effective date printed on it enabling the recreation of the organizational structure at any given period in time. Organizational charts that have been revised are stored in A&L Great Lakes Laboratories, Inc. secure archives.

11.7 COMPLIANCE

Compliance is the proper execution of recognized, documented procedures, which are either approved or required. Adherence to these procedures is required in order to provide data products acceptable to a regulatory body. Compliance is separate from, but not inconsistent with, technical scientific quality. A&L Great Lakes Laboratories, Inc. understands that the expectations of our clients commonly include the assumption that data and reports will satisfy a regulatory purpose and will be found acceptable and compliant with regulatory requirements for the performance of tests and generation of data.

It is our policy to disclose in a forthright manner any detected noncompliance that may affect the usability of data produced by A&L Great Lakes Laboratories, Inc.

12.0 ELEMENTS OF QUALITY CONTROL

A quality control (QC) program is a systematic process that controls the validity of analytical results by measuring the accuracy and precision of each method and matrix, developing performance based control limits and, using these limits to detect errors or out-of-control events. QC procedures are implemented in order to ensure that sample data meet the quality objectives of the laboratory and the client. A&L Great Lakes Laboratories, Inc. quality control program involves monitoring measurement processes to determine laboratory performance. Where applicable, quality control samples are analyzed with every batch of samples for every analytical method. Performance control samples demonstrate the precision/accuracy of the analytical run and expose out-of-control events. Matrix-specific control samples document the effect of the matrix on performance and also qualify data as in or out-of-control.

Often, regulatory agencies such as the EPA specify the acceptance criteria for all laboratory performance on a method-to-method basis. If QC acceptance criteria are not specified by regulatory agencies or in the published method, A&L Great Lakes Laboratories, Inc. uses internal quality control to determine data acceptability. Internal QC can consist of instrument calibration, use of NIST traceable standards, analysis of laboratory QC samples and duplicates (when applicable).

12.1 LABORATORY QUALITY CONTROL SAMPLES AND MEASUREMENTS

The results of quality control samples created in the laboratory represent estimates of accuracy and precision for the preparation and analysis steps of sample handling. This section describes the types of quality control samples used to assess the quality control procedure.

12.1.1 Method Blank

Method blanks are analyzed to evaluate the effect of background interferences and/or laboratory contamination on sample results. A method blank or laboratory reagent blank is a volume of deionized water for water samples, or reagent samples (matrix matched using acid washed sand or Teflon™ chips) that are carried through the entire analytical procedure. Blank analysis allows any method interferences caused by contaminants in solvent, reagents, glassware or sample processing hardware to be examined and verified. The volume of the blank must be approximately equal to the sample volume processed. The maximum permissible level of analyte in the method blank is method specific.

If the maximum permissible level of analyte in the blank is exceeded, the source of contamination will be investigated and measures taken to correct, minimize or eliminate the problem. Corrective action shall be performed to eliminate the source of contamination prior to proceeding with analysis. After the source of contamination has been eliminated, all samples in the analytical batch shall be re-prepped, then reanalyzed. No analytical data shall be corrected for the presence of analytes in blanks. When an analyte is detected in the method blank and in the associated samples and corrective actions are not performed or are ineffective, the reported data is appropriately qualified.

12.1.2 Laboratory Control Spikes/Control Spike Duplicates

The laboratory control spike consists of an aliquot of deionized water for liquid matrices and purified solid matrix for soils. If no such soil is available, laboratory pure water is substituted. The aliquot is injected with a known concentration of all the analytes of interest. It is treated exactly like a sample, and its purpose is to determine whether the methodology is in control, and

whether the laboratory is capable of making accurate and precise measurements. Successful analysis of the laboratory control spike indicates correct spiking technique. If the laboratory control spike fails, but associated data is reported since all other laboratory QC passes, then the reported result will be appropriately qualified.

12.1.3 Matrix Spikes / Matrix Spike Duplicates

Matrix spikes are similar to control spikes except that the analyte spikes are added to a separate aliquot of the same client sample within an analytical batch. This enables the lab to assess matrix effects. Matrix spike samples are analyzed at a frequency specified by the method, and the results (expressed as % recovery) are checked against control limits. If the recovery is outside of these limits, and all other instrument QC passes, then all samples associated with the matrix spike are appropriately qualified. For the purposes of this document, matrix spike is abbreviated as “MS” and matrix spike duplicate is abbreviated as “MSD.”

The matrix-specific precision associated with the analytical methods is determined and verified through the use of sample duplicates (see section **12.1.6**) and/or spike duplicates (MSD). These are performed at a frequency specified by the method. The method also dictates which type of duplicate is used (sample duplicate or spike duplicate). The results are compared by calculating the relative percent difference (RPD) and percent recovery. The matrix effect on precision and accuracy can then be determined.

12.1.4 Surrogates

Recovery surrogates are compounds chosen to simulate the analytes of interest in organic analyses, but that are not normally found in environmental samples. Recovery surrogates must be added to each sample, including QA/QC samples, prior to extraction. They provide an estimate of bias based on recovery of chemical compounds similar to target analytes, but not occurring in nature. This aids in detecting any effects from the sample matrix. Surrogates are added to environmental samples in accordance with method requirements. Whenever a surrogate recovery is outside the acceptance limit, corrective action must be performed. After the system problems have been resolved and system control has been reestablished, the sample should be re-prepped and retested. If corrective actions are not performed or are ineffective, the appropriate validation flag is applied to the sample results.

12.1.5 Internal Standards

Internal standards are measured amounts of certain compounds having surrogate characteristics but are added to each sample within a batch just prior to analysis. These are primarily used for quantitation in GC/MS and ICP-MS calibrations. It corrects for bias or change in instrument performance from sample to sample, incorporating matrix effects associated with the analytical process only. The internal standard is a compound not likely to be found in the environment and is used in a calibration method to correct sample results affected by column injection losses, purging losses or viscosity effects. The internal standard is added to environmental samples, controls and blanks in accordance with the method requirements.

When the internal standard results are outside of the acceptance limits, corrective actions shall be performed. Once system control has been reestablished, all samples analyzed while the system was malfunctioning shall be reanalyzed. If corrective actions are not performed or are ineffective then the reported data is qualified.

12.1.6 Sample Duplicate

A sample duplicate is prepared by homogenizing and splitting the sample into two equal portions before the method sample preparation process. It measures sample precision associated with preparation as well as matrix-specific precision associated with the analytical method. Also see section 12.1.3.

12.1.7 Accuracy Measurements

Accuracy reflects the degree to which the measured value approximates the actual or "true" value for a given parameter and reflects the influence of systematic biases in the measurement. It is expressed as % Recovery. For laboratory control spikes, surrogates and method blanks, the calculation is as follows:

% Recovery = (the determined concentration / the known spiked concentration) x 100

For matrix spikes, the % Recovery is calculated as:

$$\% \text{ Recovery} = (\text{SSR} - \text{SR}) / \text{SA} \times 100$$

where: SSR = the spiked sample determined result

SR = original sample determined result

SA = the spiked concentration (known)

12.1.8 Precision Measurements

Precision measures the randomness associated with an analytical measurement and reflects the inherent variability in that measurement system.

The comparison of the values determined for a sample and its duplicate is expressed as relative percent difference (RPD). This calculation is as follows:

$$\text{RPD} = \frac{|S - D|}{[(S + D) / 2]} \times 100$$

Where: S = original sample result and D = duplicate result

12.2 STANDARDS

The term standard applies to any analyte solution of known concentration that is traceable to a certified reference material. This includes calibration standards and spiking solutions.

A&L Great Lakes Laboratories, Inc. prepares intermediate and working standard solutions from stock standards purchased from reputable chemical vendors. Stock standards and certified reference

materials are of an analyzed grade and are traceable to NIST standards. Intermediate standards, instrument calibration and check standards are prepared using clean Class A volumetric glassware. Analysts will use either Class A pipettes, or, if the method allows, fixed volume pipettes, or adjustable volume pipettes to measure and dispense reagent aliquots.

Upon receipt, all purchased reference standards for the Environmental Organic Department are logged into the A&L standard library. Items included in the logging of purchased reference standards are the A&L Great Lakes Laboratories, Inc. unique identification number, the name of the compound or solution, manufacture, lot number(s), certificate of purity, place stored and expiration date.

Subsequent preparations of stock, intermediate, and working solutions are also documented in a standard prep log that is ultimately traceable back to the certificate of purity.

12.3 CONTROL CHARTS

Control charts are quality tools that graphically display quality control parameters over time. A&L Great Lakes Laboratories, Inc. uses control charts to maintain control over many of its processes. Control limits are calculated from laboratory generated data at +/- 3 standard deviations from the mean (99% confidence limits). Warning limits are calculated at +/- 2 standard deviations from the mean (95% confidence limits). A&L Great Lakes Laboratories, Inc. generates QC charts using the ControlChart! Pro[®] Plus with Datalink software.

Evaluating data points on a control chart enables the laboratory to detect many types of suspicious and out-of-control situations. Such events can be determined by monitoring outliers, runs and trends. Outliers are points that fall outside the control limits and require immediate corrective action (checking integrity of solutions, instrument conditions, operator error etc.). Runs are a series of points that line up on one side of the mean. A run of 7 points indicates a potential abnormality in the process and should be investigated (e.g. system leak, contamination, wrong dilution). A trend is a series of points that are marked by a continuous rise or fall. A trend with a length of 5 points is a suspicious event and requires investigation. A trend can indicate a change in instrument sensitivity due to conditions, standard degradation, or a dirty injection port. Such are items that can lead to out-of-control situations. The ControlChart! Pro[®] software allows the user to define QC rules to apply to the dataset thereby, allowing a custom evaluation of the data points.

13.0 QUALITY ASSURANCE AUDITS AND PERFORMANCE EVALUATIONS

The use of an effective audit program is necessary to monitor and review the Quality System and to assure that it is continually being improved on all levels. A&L Great Lakes Laboratories, Inc. has instituted a system of audits and performance evaluations in order to evaluate the effectiveness and completeness of various quality control and quality assurance systems in the laboratory. The following describes some of the types of audits and evaluations used by A&L Great Lakes Laboratories, Inc.

13.1 INTERNAL AUDITS

13.1.1 Quality Assurance Unit

The Quality Assurance Unit is responsible for designing and/or performing QA performance and facility audits. Quality Assurance must be an independent function from the laboratory operations in order to ensure non-bias and objectivity. The Quality Assurance Unit must be familiar enough with the objectives, principles, and procedures of laboratory operations to perform a thorough and effective evaluation.

The Quality Assurance Unit reports directly to upper level management and is independent of laboratory operations. The Quality Assurance Unit does not become involved in the generation of any raw data. The Quality Assurance Unit's role is detailed in SOP **QAU-0-001** "*Quality Assurance Unit*".

13.1.2 Scope and Frequency of Internal Facility Audits

Internal systems facility audits are conducted bi-annually (2 times per year) per SOP **QAU-0-002** "*Conducting Facility Inspections & Method Audits*". The following are items contained in the inspection report:

1. Organizational Chart
2. Employee Training Records
3. Adherence to SOPs (annual SOP review documented on master copy)
4. Equipment Maintenance & Calibration Documentation
5. General Facility Cleanliness and Ensuring Doorways and Safety Equipment are unobstructed.
6. Proper Labeling of Reagents, Solutions and Solvents.
7. Standard Preparation Logs and Certificates of Analysis
8. Sample Handling, Receipt and Storage of Samples

Internal audits may be scheduled more frequently depending on the following criteria:

- Number and type of corrective actions filed for a method or activity
- Client complaints
- Continued failure to achieve acceptable results for a Proficiency Testing (PT) sample
- Findings from an external audit
- Request from management

13.1.3 Facility Audit Reports and Corrective Action Plans

The completed facility audit forms list the department audited, date(s) on which the audit was conducted and any findings or observations made during the course of the audit.

Additionally, any recommended courses of action are noted. The Quality Assurance Unit routes the audit report to the department manager as well as upper level management for signatures and date. The Quality Assurance Unit also verifies the responses to all findings and any corrective action taken. All audit reports are maintained in the QA office.

13.1.4 Analytical Method Audits

Periodically, the Quality Assurance Unit will conduct audits of the analytical method (or procedure) itself. Adherence to the SOP, training records, raw data corrections and applicable QC are all items that are observed and documented according to SOP **QAU-0-002** “*Conducting Facility Inspections & Method Audits*”. The following items are contained in the audit report:

1. Employee Training Records
2. Equipment maintenance
3. Adherence to SOPs
4. Proper Documentation Data Corrections and Deviations
5. Preparation of Reagents, Standards, and Solutions
6. Proper Labeling of Reagents, Standards, and Solutions
7. Appropriate QC within Method Specifications

The completed method audit report forms list the analytical method/SOP, date(s) the audit was conducted, technician, auditor, and any findings or comments. Corrective action measures are implemented and the action taken or response is documented on the audit form. The form is signed and dated by the quality assurance unit, department manager, and upper level management. All method audit forms are maintained in the QA office.

13.2 EXTERNAL AUDITS

A&L Great Lakes Laboratories, Inc. is frequently audited by external parties such as regulatory agencies, study sponsors, insurance carriers and various commercial clients. External auditors use their own audit forms to review the areas of the laboratory they are interested in. The A&L Great Lakes Laboratories, Inc. Quality Assurance Unit hosts the auditor(s) and facilitates the audit process. Generally, the external auditor prepares an audit report and often holds an exit interview to discuss any findings and map out a corrective action plan (if required). A&L Great Lakes Laboratories, Inc. Quality Manager ensures that all findings are addressed and that all corrective actions are implemented. All external audit reports are filed in the QA office.

13.3 PERFORMANCE EVALUATION / PROFICIENCY TESTING PROGRAM

13.3.1 Performance Evaluation Samples (PE) Studies

Proficiency Evaluation and Proficiency Test samples shall be handled according to SOP GEN-2-004. A&L Great Lakes Laboratories, Inc. participates in EPA WS (Water Supply - Drinking Water), WP (Water Pollution -Waste Water) and DMR-QA proficiency testing sample programs.

The DMR-QA studies are conducted to measure the proficiency of laboratories performing analyses under the National Pollutant Discharge Elimination System (NPDES). The laboratory analyzes the DMR-QA samples and reports results to each permittee for all analytes it analyzes for the permittee. Performance evaluation reports for each permittee are returned after the study is completed. The reports indicate whether or not the results submitted by the permittees are within the acceptance limits. A&L Great Lakes Laboratories, Inc. investigates any of its not-acceptable results, documents the findings of the investigation and implements any appropriate corrective action.

13.3.2 Pennsylvania Department of Environmental Protection (PA DEP) and NELAP (National Environmental Laboratory Accreditation Program)

A&L Great Lakes Laboratories, Inc. maintains certification as a NELAC and PA DEP certified laboratory for the analysis of bio-solids, composts, sludge and soils. In order to keep up certification, A&L Great Lakes Laboratories, Inc. analyzes performance evaluation (PE) samples and undergoes a facility inspection by the Pennsylvania Department of Environmental Protection as its primary accrediting authority for adherence to the standards set forth by NELAP.

13.3.3 Indiana State Board of Health - Public Water Supply

A&L Great Lakes Laboratories, Inc. maintains certification as a State of Indiana certified microbiological laboratory for the analysis of drinking water. In order to keep up certification, A&L Great Lakes Laboratories, Inc. analyzes performance evaluation (PE) samples and undergoes a facility inspection by the State Department of Health.

13.3.4 North American Proficiency Testing Program (NAPT)

A&L Great Lakes Laboratories, Inc. participates in the NAPT program for agricultural samples such as soils, plants and waters. The program is based on the quarterly submission to participating laboratories of six soil and/or three plant materials for chemical analysis. Quarterly, each laboratory is provided with an evaluation of their individual performance on each of the methods employed. Annually, the program provides a report to each participant of the performance of the individual laboratory and that of the agricultural laboratory industry.

13.3.5 The Compost Analysis Proficiency (CAP) Program for Laboratories

The CAP testing program is an integral part of the US Composting Council Research Education Foundation. The goal of the program is to provide the Laboratory Analysis Industry with an inter-laboratory QC program and develop reference materials.

The CAP program operational guidelines are based on those outlined under ISO 9000, ISO/IEC Guide 43 and Draft ISO/IEC Guide 24, which describe the requirements for proficiency testing schemes.

The CAP program is based upon the tri-annual submission of three compost materials for chemical analysis using reference methods of analysis. Tier II materials are shipped refrigerated overnight mail to maintain integrity.

13.3.6 Manure Analysis Proficiency (MAP) Testing Program

Certificate issued annually for Total Nitrogen (combustion & TKN), Ammonium Nitrogen, Nitrate Nitrogen, Electrical Conductivity, Phosphorus, Potassium, Calcium, Copper, Magnesium, Sulfur, Zinc, Sodium, pH and Total Solids analyses for manure. Certification requires the analysis of eight manure check samples per year. Laboratories with 80% of the analysis values falling within the control limits receive certification for analysis of manure for the parameters reported.

13.3.7 Wisconsin DATCP Soil Testing Laboratory Certification Program

Soil sample exchange program by the University of Wisconsin-Madison Soil & Forage Analysis Laboratory. The certification is required by the Wisconsin Department of Agriculture, Trade and Consumer Protection (WDATCP) for laboratories to conduct analyses for conservation programs in the state of Wisconsin. Laboratories must maintain a minimum of 66% of their semi-annual check sample tests within a standard deviation of the mean to keep certification.

13.3.8 State Specific Accreditation Programs

13.3.8.1 Missouri Soil Testing Accreditation Program (MSTA)

Quarterly soil sample exchange program administered by the University of Missouri using NAPT samples. Successful results allow a laboratory to be certified in the state of Missouri. Results for MSTA are posted in the MU Soil & Plant Testing Laboratory web site at: <http://www.soiltest.psu.missouri.edu/soil/mstacertified.htm>.

13.3.8.2 Minnesota – Quarterly samples through NAPT

13.3.8.3 Indiana – Quarterly samples through NAPT

13.3.8.4 Iowa – Quarterly samples through NAPT

13.3.8.5 Illinois – 3x/year sample exchange through ALP

13.3.9 National Forage Testing Association

The National Forage Testing Association (NFTA) was founded in 1984 as a joint effort of the American Forage and Grassland Council, the National Hay Association, and forage testing laboratories in a concentrated effort to improve the accuracy of forage testing and build grower confidence in testing animal feeds.

NFTA is governed by a 12-member Board of Directors. Six directors represent laboratories, three represent the National Hay Association, and three represent the American Forage and Grassland Council.

Each year the NFTA Board updates the certification program. New methods of grading laboratory performance have been introduced since NFTA was formed. Today, laboratories are evaluated six times a year. Performance grades are provided to laboratories to allow them to better evaluate their testing procedures and methods.

13.3.10 Magruder Check Program for Fertilizer

Each subscribing laboratory receives one or two samples each month. Each month's samples contain the primary plant nutrients N, P and K, and about four samples each year contain secondary and minor nutrients, as well as contaminants of interest. Samples are analyzed by methods chosen by the individual participating laboratories. Their results, with designation of method used, are reported directly to the statistician. A comprehensive statistical report is

prepared noting inter-laboratory bias, precision and accuracy for each method and analyte, and ranking of coded individual laboratories. These reports are sent to the subscribers to allow them to evaluate their performance. If out-of-control situations are discovered, a potential problem exists that is then investigated and corrected. The Magruder Check Sample results can be found on the internet site of Magruder (<http://www.magruderchecksample.org>).

13.3.11 AFPC Phosphate Fertilizer Check Sample Program

AFPC Phosphate Chemicals Check Sample Program - Prepares, distributes, evaluates monthly samples of DAP, MAP, and Feed Grade Phosphates for participating labs. These programs and reference materials are used to evaluate overall lab performance, individual technician training, and method performance. Program includes State Regulatory labs, referee laboratories, and phosphate mining and chemical manufacturing labs. (<http://www.afpc.net>).

13.3.12 Agricultural Laboratory Proficiency Program (ALP)

The ALP program distributes and evaluates samples of soil, plant tissue and water three times per year. Each round of soil samples contains five prepared soils that are chosen specifically to represent soils found in different regions of North America. Soil methods covered by ALP include all those designated in US regional publications and Canada. Each round of plant tissue contains three processed, homogenous botanical samples. Both the soil analysis and plant tissue analysis evaluate precision and bias. The agricultural contribution of water is a key component in precision agriculture. Three (3) water samples are contained in each round. (<https://www.collaborativetesting.com>).

14.0 DATA QUALITY ASSESSMENT

Data quality assessment requires the review of quality control samples for precision, accuracy, representativeness, completeness, and comparability. Precision and accuracy data are used to determine the acceptability of analytical results.

Representativeness, completeness, and comparability are used to ascertain the level at which the client's data quality objectives (DQO's) have been satisfied. To the extent possible, client samples are reported only if all QC measures are acceptable. If a quality control measure is outside of acceptance criteria, and the data must be reported, then all samples associated with the failed QC must be reported with the appropriate data qualifier(s).

14.1 PRECISION

Precision is the degree to which the measurement is reproducible. Precision is assessed by duplicate measurements of a laboratory control sample or an environmental sample. The precision of laboratory analytical data can be expressed in several forms including: (1) standard deviation, (2) range or R-charts, (3) relative standard deviation, also known as the coefficient of variation, and (4) relative percent difference (RPD). Control charts can be constructed using all the aforementioned forms. If acceptance criteria are not specified in either the method or in regulations, then it can be determined using control charts and historical data.

$$CV = \frac{S}{\bar{X}} \times 100$$

Where: CV = coefficient of variation
X = mean for the dataset

$$S = \sqrt{\frac{\sum (X_i - \bar{X})^2}{n - 1}}$$

Where: S = standard deviation
 \bar{X} = mean
 X_i = individual observation
n = number of samples

$$RPD = \frac{|X_1 - X_2|}{\left(\frac{X_1 + X_2}{2}\right)} \times 100$$

Where: X_1 = measured conc. of the first sample aliquot
 X_2 = measured conc. of the second sample aliquot

14.2 ACCURACY

Accuracy measures the degree of difference between the observed and true values. The actual test result is compared to the theoretical result of 100% recovery and the percent recovery calculated. The accuracy of sample data is assessed using the laboratory control spike, matrix spike or surrogate standards.

- Percent Recovery for a laboratory standard:

$$\%R = \frac{X_1}{X_2} \times 100$$

Where: X_1 = measured concentration of standard
 X_2 = true value of standard

- Percent Recovery for matrix spike:

$$\%R = \frac{S_1 - S_2}{S_3} \times 100$$

Where: S_1 = measured concentration of the spiked sample
 S_2 = measured concentration of the sample
 S_3 = true value of the spike concentration

14.3 REPRESENTATIVENESS

Representativeness is a qualitative element related to the ability to collect a sample that reflects the characteristics of that part of the environment being assessed. Sample representativeness is dependent on the sampling techniques used.

14.4 COMPLETENESS

Completeness is a measure of the amount of valid data obtained from a measurement system compared to the amount that was expected to be obtained under normal conditions.

14.5 COMPARABILITY

Comparability expresses the confidence with which one data set can be compared to another data set measuring the same property. For example, the use of EPA approved methods and procedures ensure comparability with other data from previous or following studies using the same methods.

15.0 DATA REDUCTION, VERIFICATION AND REPORTING

Data reduction, verification and reporting are the processes that result in the delivery of quantitative analytical data to the data user. These processes include calculation of raw data into final concentration units, reviewing results for accuracy and assembling the technical report contents for delivery to the data user.

All analytical data generated by A&L Great Lakes Laboratories, Inc. undergo a well-defined, documented review process before being reported to the client. The following describes procedures used by A&L Great Lakes Laboratories, Inc. for translating raw analytical data into accurate, finished sample reports and describes data storage policies.

15.1 DATA REDUCTION

When an analyst manually generates data, it is recorded in either a bound notebook or on a specific bench sheet. Alternatively, the data may be automatically collected and entered via the computer system. Laboratory notebooks and bench sheets are kept in accordance with the Standard Operating Procedure on documentation (SOP **GEN-4-003** "*Documentation of Data*"). The primary analyst is responsible for the initial reduction and review of the data. This can include entering analytical data into the Laboratory Information Management System (LIMS), confirming compliance with required methodology, checking calculations, checking quality control data against known criteria, and documenting any discrepancies or deviations. Also, all relevant data is gathered such as chromatograms, spectral data, computer printouts, chain of custody, bench sheets, etc.

15.2 DATA VERIFICATION

Data verification is the process of examining data and accepting or rejecting it based on pre-defined criteria. This review step is designed to ensure that the reported data are free from calculation and transcription errors; that quality control parameters are evaluated; and that any discrepancies are clearly documented. Data will be considered valid if it is generated under acceptable operating conditions as specified in the analytical SOP used during the determination of the data. Acceptable operating conditions include not only instrument and analyst performance but also the maintenance of quality control sample results within predetermined control limits.

15.3 DATA REPORTING

After verifying a report's completeness and accuracy, the manager initials the report authorization. One additional layer of authorization is within LIMS. All environmental reports must have an approval from management before they are allowed to be reported or sent out via e-mail. This approval field is saved along with the results. Minimally, environmental reports will provide the following information:

- a) Report number
- b) Client identification number
- c) Sample identification or description
- d) Laboratory number of each sample
- e) Analyte identification
- f) Analytical result in appropriate units
- g) Method identification
- h) Analyst identification
- i) Sampling date
- j) Receipt date

- k) Analysis date
- l) Report date

All final reports will be inspected, verified and initialed by a qualified person other than the analyst(s) before the report is released to the client.

Final reports are prepared according to the level of reporting required by the client. A&L Great Lakes Laboratories, Inc. develops many different types of reports specifically tailored to the needs of their clients.

Results questioned by the client will be re-assayed upon request. No charge will be made unless the re-assay confirms the original results.

15.4 DATA ARCHIVE

A&L Great Lakes Laboratories, Inc. maintains a secure, fireproof archive. The following specific guidelines govern record retention:

1. Records will be kept in a secured place, accessible only to authorized personnel.
2. All Environmental records relating to reports, laboratory data, instrumentation, standards, etc. will be kept for a minimum of 5 years.
3. Records relating to GLP or regulatory studies shall be kept according to SOP **REC-0-001** "*Management of Record Archives for GLP Studies*" or until released to the project sponsor.
4. A record archivist shall be appointed to organize and supervise the filing of documents.

16.0 CORRECTIVE ACTION

16.1 PURPOSE AND SCOPE

To provide guidelines for timely response when errors, deficiencies, or out-of-control situations develop.

16.2 SPECIFIC GUIDELINES

1. All analytical SOP's must contain or refer to specific instructions associated with corrective actions to be taken to prevent the production of poor quality data.
2. When data from the analysis of spikes, duplicates, or control samples exceeds established control limits or shows a pattern indicating there is a problem, the process will be stopped immediately. Immediate action will be taken to determine the cause of the problem.
3. In the event that data of questionable quality is produced and the analyst is able to detect and correct the cause of the problem, the analyses will be rerun to correct the data.
4. If the analyst is unable to determine the cause of the problem, the department supervisor/manager will be notified immediately.
5. Performance of the method will be validated after correction of the problem and before sample analysis is resumed.
6. Non-conformances or corrective actions can be initiated from client, project management, sales, analysts, supervisors, lab managers or QA personnel. Whenever there are non-conforming conditions resulting from data integrity or data quality concerns, the lab supervisor / manager or the Quality department must review all associated data, document findings and the Quality department will perform a follow-up audit. The lab supervisor / manager or QA department will respond to the client within 3 working days.
7. The lab director or similar upper level management shall monitor corrective actions taken to ensure that results are effective and shall determine when to resume work.

16.3 DISCREPANCIES AND/OR DEPARTURES FROM DOCUMENTED POLICIES AND PROCEDURES

16.3.1 Individuals Responsible for Assessing Procedural Compliance and QC Data:

Procedures, method acceptance criteria and QC (method blanks, method controls, spike recoveries and duplicate results) review are the responsibility of the analyst performing the test. Additionally, when procedures are deviated from or batch QC is questionable, the department manager will be notified. It is the responsibility of the analyst to communicate any noncompliance issues to the department manager.

16.3.2 Individuals Responsible for Initiating Corrective Actions:

Department managers are responsible for ensuring that the analysts are properly assessing procedural compliance and QC data through daily oversight. Department managers are responsible for initiating and/or recommending corrective actions. In an effort to continuously improve processes, managers will make every effort to eliminate root causes of any recurring problem.

16.3.3 Treatment of Sample Data Collected in Instances Where Procedural or QC Data Non-Conformances Exist

Whenever procedural or QC data non-compliances occur, the testing process must stop immediately and the analyst must inform the department supervisor/manager for immediate investigation into the cause of the problem. When testing discrepancies are detected, or when departures from documented policies and procedures occur, the following steps may be taken to resolve the situation:

1. If there is sufficient sample to reanalyze, the problem will be corrected and the samples retested. The original results will be rejected and not reported. Providing that the QC data is in conformance and the procedures followed, the reanalysis values will be reported.
2. If the sample is exhausted or if the holding times have expired, the Laboratory Director is informed of the problem. The dynamics of the problem are investigated and the problem's effect on the result is assessed. If the sample data is qualified (e.g. a quality control measure is found to be out of control, and the data has to be reported), then the client is informed within 3 working days, and the results are reported with qualifications documented in the sample comments. On certain occasions the client will resubmit samples for analysis.
3. When procedural or QC data non-compliances occur, samples will not be analyzed or reported until the cause of the problem has been investigated, corrective action documented and a follow-up audit performed.

16.3.4 Review of Corrective Actions by the Laboratory Director or Quality Manager

The Laboratory Director or Quality Manager will review records of corrective action to ensure that the corrective action taken was appropriate, timely and effective. The department manager is required to communicate any data-qualifying issues immediately to the Laboratory Director and/or the Quality Manager.

16.3.5 Management's Procedures for Permitting Exceptional Departures from Documented Policies and Procedures

Whenever departures from documented policies and procedures or from QC specifications result in a compromised analytical result, the data is qualified in the comments section of the report. There are times when such departures do not result in a compromised result. Under these circumstances the laboratory director may permit such departures providing there is a reasonable advantage to do so. Samples occasionally fail to conform to typical matrix expectations to such an extent that they are unable to be analyzed by the method without modification. If the modification is significant, it will be documented in the comments section of the analytical report.

16.4 HANDLING COMPLAINTS

Handling client complaints is a joint effort between the Quality Assurance Unit, Department Managers, and Client Service representatives. If a client has a concern or complaint, either a Department Manager or Client Service Representative takes the call and initiates the complaint procedure. If the A&L Great Lakes Laboratories, Inc. employee who took the call cannot easily resolve the problem or complaint, the complaint is routed from the initiator to other appropriate

parties, including the Quality Manager and/or Laboratory Director if necessary. An investigation into the nature of the complaint or question ensues. Communication with the customer and any resolution is documented and filed with the report. All client complaints are submitted to upper management.

16.5 QUESTIONABLE LAB RESULTS AND CLIENT NOTIFICATION

When audit findings cast doubt on the effectiveness of the operations or on the correctness or validity of the laboratory's environmental test results used to generate any report, test certificate or amendment to a report or certificate such as the identification of defective measuring or test equipment, the laboratory shall take timely corrective action, and shall notify clients in writing within 3 working days if investigations show that the laboratory results may have been affected.

17.0 QUALITY ASSURANCE REPORTS TO MANAGEMENT

As per many regulatory requirements, A&L Great Lakes Laboratories, Inc. requires periodic written quality assurance reports to management. All in-phase or "progress" analytical audit findings are summarized on an audit form and routed to management for acknowledgement signatures and date. Additionally, all facility inspection audit reports are routed to management for signatures / date. These reports not only include all findings, but all corrective action taken to address the findings.

Additionally, as per GLP requirements, project requirements, or client request, quality assurance reports may be sent to the client, sponsor representative, study director and/or study director management for review. In the event that copies of quality assurance reports are sent out, a "receipt and acknowledgement" form accompanies the report(s) and provides documentation that the report(s) was received, by whom and on what date.

18.0 TESTING METHODS

Metals

Soil, Sediment, Groundwater, Wastes (RCRA)

SW846 Methods

Digestions

Method 3050B: Acid Digestion of Sediments, Sludges, and Soils

Method 3051: Microwave Assisted Acid Digestion of Sediments, Sludges, Soils, and Oils.

Method 3015: Water and Wastewater Digestion

Determinations

Method 6020: Inductively Coupled Plasma – Mass Spectrometry

Method 6010B: Inductively Coupled Plasma – Atomic Emission Spectrometry

Method 7471A: Mercury in Solid or Semisolid Waste (Manual Cold-Vapor Technique)

Characteristics

Method 1020B: Small Scale Closed Cup Method for Determining Ignitability

Sec 8.3: Reactivity

Sec 7.3.3: Reactive Cyanide

Sec 7.3.4: Reactive Sulfide

Method 1311: Toxicity Characteristic Leaching Procedure

Wastewater

EPA Methods

Method 200.7: Metals by ICP

Method 245.1: Mercury by CVAA

Method 200.8: Metals by ICP-MS

Inorganics

Soil, Sediment, Groundwater, Wastes

SW846 Methods

Method 9010B: Total and Amenable Cyanide: Distillation

Method 9030A: Acid-Soluble and Acid-Insoluble Sulfides: Distillation

Method 9038: Sulfate (Turbidimetric)

Method 9040C: pH Electrometric Measurement

Method 9045D: Soil and Waste pH

Method 9065: Phenolics (Spectrophotometric, Manual 4-AAP with Distillation)

Method 9071B: n-hexane Extractable Material (HEM) for Sludge, Sediment, and Solid Samples

Method 9214: Potentiometric Determination of Fluoride in Aqueous Samples with Ion-Selective Electrode

Organic Instrumentation

Method 8270C: Semi-Volatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS)

Method 8260B: Volatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS)

Method 8081A: Organochlorine Pesticides by Gas Chromatography (PCB's)

Method 8021B: Aromatic and Halogenated Volatiles by Gas Chromatography Using Photoionization and/or Electrolytic Conductivity Detectors

Method 8015B: Non-halogenated Organics Using GC/FID (Total Petroleum Hydrocarbons)

Wastewater, Drinking Water and Sludge

Acidity: EPA Method 305.1

Alkalinity: SM(20th)–2320B

Arsenic (ICP/MS): EPA Method 200.8

Biochemical Oxygen Demand (5 Days): SM(20th)–5210B

Conductance (Specific conductance, umhos at 25 C): EPA Method 120.1

Chemical Oxygen Demand (Hach): O.10

Chloride: SM(20th)-4500-Cl⁻ G
Chlorine, Total Residual: SM(20th)-4500-Cl G
Cyanide, Total: EPA Method 335.4 Rev 1.0 (drinking water: SM4500CN-E)
Cyanide, Amenable to Chlorination: EPA Method 335.1/335.4 Rev.1
Fluoride: SM (20th)-4500F-C
Hardness, Total (mg/L as CaCO₃) (Titrimetric, EDTA): SM(20th)-2340
Mercury (ICP/MS): EPA Method 200.8
Nitrogen-Ammonia: SM(20th)-4500-NH₃ B/C (Water: EPA 350.1 Rev.2.0)
Nitrogen-Kjeldahl: SM(20th)-4500-Norg B
Nitrogen, Nitrate-Nitrite (Colorimetric, Automated, Cadmium Reduction): EPA Method 353.2, rev 2.0
Oil and Grease: EPA Method 1664
pH: SM(20th)-4500-H⁺ B
Phosphorus: EPA Method 365.1, rev 2.0
Phenolics: EPA Method 420.4 Rev.1
Selenium (ICP/MS): EPA Method 200.8
Sulfate (Turbidimetric): EPA Method 375.4
Sulfide: Hach S.5
Sulfite (Titrimetric): SM (20th)-4500 SO₃²⁻B
Residue, Filterable Solids (TDS): SM(20th)-2540C
Residue, Non-filterable Solids (TSS) (Gravimetric, Dried at 103-105 C): SM(20th)-2540D
Residue, Total Solids (TS) (Gravimetric, Dried at 103-105 C): SM(20th)-2540G
Residue, Volatile Solids (TVS) (Gravimetric, Ignition at 550 C): EPA Method 160.4

Metals and Trace Elements in Water (All EPA Method 200.7 or 200.8)

Aluminum
Antimony
Arsenic
Barium
Beryllium
Boron
Cadmium
Calcium
Chromium
Cobalt
Copper
Iron
Lead
Lithium
Magnesium
Manganese
Mercury
Molybdenum
Nickel
Phosphorus
Potassium
Selenium
Silicon
Silver
Sodium
Strontium
Sulfur
Thallium
Titanium
Vanadium
Zinc
Zirconium

19.0 MAJOR EQUIPMENT

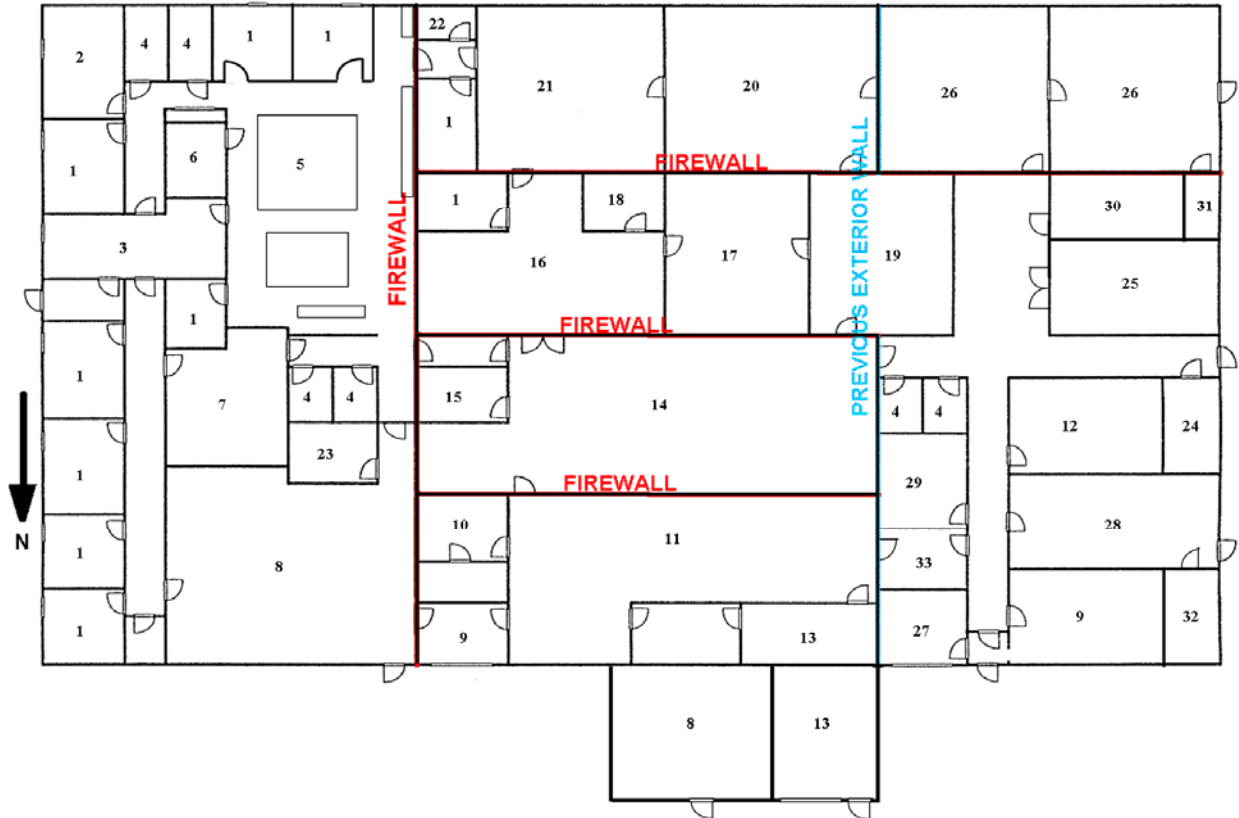
Quantity	Item
ORGANICS	
1	Perkin Elmer Clarus 500 with EC and FI Detectors
1	Perkin Elmer Clarus 500 with FP and FI Detectors
1	Varian 3900 GC/MS with 2100T Ion Trap
1	Perkin Elmer Autosystem with NP and EC Detectors
1	Hewlett Packard 5890 with NP Detector
1	Hewlett Packard 5890 with 5971 Mass Selective Detector
2	Perkin Elmer Series 200 LC with LC 200 UV-VIS Detector
1	Perkin Elmer Autosystem XL with NP and EC Detectors
METALS	
1	Varian SpectrAA Atomic Absorption Spectrometer
1	Perkin Elmer Elan DRC plus ICP/MS
1	Varian Vista-MPX CCD Simultaneous ICP-OES
2	Anton Paar Multiwave 3000 Microwave Sample Prep System
1	Varian 820 ICP/MS
INORGANICS	
3	Leco FP-428 Nitrogen Analyzer
1	GBC Cintra 10 UV/VIS Spectrometer
2	FIALab 2500 Flow Injection System
1	Thermo Orion model 550A pH/Conductivity meter
1	VWR SympHony SB80PC pH and Conductivity Meter
1	HACH HQ40d Dissolved Oxygen Meter
1	YSI 5000 Dissolved Oxygen meter
1	Fisher Scientific Accumet model 50 pH meter
1 each	Lachat QuickChem 8000 and 8500
1	Hach COD reactor
1	Hach DR/3000 Spectrophotometer
1	Sheldon Manufacturing VWR 14-5783 Incubator
1	VWR 3015 Incubator
1	Fisher Scientific 3530 Isotemp Incubator
2	Seward Stomacher 400 Circulator
1	DIONEX Ion Chromatography System ICS-2100 & AS-DV ICS Series
OTHER	
1	TCLP extractor and ZHE extractors
1	Varian Cary 50 UV-Vis Spectrophotometer
2	Automated pH Analyzers
1	Market Forge Sterilmatic Autoclave
1	Baxter Scientific Product DN-63 Oven and Curtin Matheson EQUATHERM Oven
9	Mettler Analytical Balance (various models)
4	AND Analytical Balances (various models)
4	OHAUS Analytical Balances (various models)
1	Phenol distillation unit
2	Precision Coliform Incubator baths
1	Horizon SPE-DEX 3000XL
1	Lachat Micro Dist Cyanide distillation unit
1	Jouan C412 Centrifuge
1	Quebec Darkfield Colony Counter

(Cont.) List of Major Equipment:

A&L GREAT LAKES LABORATORIES, INC.

Quantity	Item
OTHER	
1	VWR Scientific Products water bath
1 each	Branson 3210 & 5510 ultrasonic waterbath
1	Retsch fertilizer grinder and riffler/splitter
1	50 node PC based LAN with custom designed LIMS system
3	Labconco Rapidstill II
1	Barnstead B-Pure Pressure Cartridge System
1	Barnstead EASYpure II Water System
6	THERMO iCAP 6000 Series ICP Spectrometer

20.0 FACILITIES



- | | | | |
|--------------------|-----------------------|----------------------------|----------------------------|
| 1. Office | 9. Receiving | 17. Sample Digestion | 25. ICP Room |
| 2. Conference | 10. Plant Preparation | 18. Weighing | 26. Environmental Lab |
| 3. Reception | 11. Soil Preparation | 19. Fertilizer Analysis | 27. Shop |
| 4. Rest Room | 12. Drying Room | 20. Organic Prep. | 28. Soil Library |
| 5. Data Processing | 13. Grinding | 21. Chromatography | 29. Env. Sample Receipt |
| 6. Computer/Server | 14. Soil Extraction | 22. Gas Storage | 30. ICP/MS Room |
| 7. Employee Lounge | 15. Chemical Storage | 23. Utility Room | 31. Chiller/pump/switch |
| 8. Storage | 16. Instrumentation | 24. Doc. Storage / Archive | 32. Fert Grinding/Riffling |
| | | | 33. Germination Hall |

A&L Great Lakes Laboratories, Inc. uses the following services/systems in conducting tests:

1. Electricity
2. Water (includes de-ionized water system and ultra-pure water system)
3. Sewer
4. Various compressed gases and liquid gas delivered by local vendor
5. HVAC
6. Telephone
7. T-1 High speed internet connection

APPENDIX A Ethics Policy

A&L Great Lakes Laboratories, Inc. holds honesty and integrity in generating data in highest regard. The following corporate policy describes the standards of ethics and behavior that A&L Great Lakes Laboratories, Inc. expects of all staff working in its employ.

Honesty:

It is expected that all employees who contribute to generating analytical data at A&L Great Lakes Laboratories, Inc. maintain the highest possible standards of integrity. Fraud, falsification of data, and other forms of dishonesty shall not be condoned and are cause for disciplinary action.

Dishonesty is defined as misrepresentation with intent to deceive with regard to the accuracy of statements, reports or findings. It may include such things as distorting or concocting laboratory results, reports or projects; copying, plagiarizing, or otherwise representing the work of another as one's own; aiding another employee's dishonesty (or being aware of another employee's dishonesty and not reporting such to management); and similar offenses.

Plagiarism, piracy and fraud:

A&L Great Lakes Laboratories, Inc. expects all staff working in its employ to be aware that plagiarism, piracy, fraud and/or the fabrication of results, or committing any of these actions is regarded as a serious disciplinary offence.

A&L Great Lakes Laboratories, Inc. adopts the following definitions:

- Fraud involves deliberate deception, including the invention of data, and the omission of analysis details and documentation important for the re-creation of the data.
- Plagiarism involves stealing and passing off of the ideas or words of another as if they were one's own new and original ideas or words
- Piracy involves the unauthorized use of another's work, invention or conception.

Supervision:

Individuals in authority set the culture of any organization. At A&L Great Lakes Laboratories, Inc. it is the responsibility of management to create an environment of trust and mutual co-operation.

A&L Great Lakes Laboratories, Inc. recognizes the need for new employees to be properly supervised and mentored during their training time. Careful supervision of new employees is in the best interests of A&L Great Lakes Laboratories, Inc., the trainee, and the supervisor. Supervisors should have an active mentoring role in the training process, monitor employees data collection, and review interpretation of all data.

Collection, processing and interpretation of results:

A&L Great Lakes Laboratories, Inc. requires employees to keep clear and accurate records of the analytical procedures followed and of the results obtained. This is necessary to demonstrate good scientific practice and is also an important safeguard should questions be asked about the conduct of analyses or results obtained.

Disciplinary Action:

Misconduct in analysis is an offense which, depending on its severity, is subject to a range of disciplinary measures up to and including dismissal. Allegations of misconduct shall be dealt with in a fair, unbiased and timely manner and all parties involved shall be advised of the procedures available to them. Persons against whom allegations of misconduct have been made shall be advised of the allegations, and shall be afforded the opportunity to respond.

A & L GREAT LAKES LABORATORIES, INC.

3505 Conestoga Dr. • Fort Wayne, Indiana 46808-4414 • Phone (260)483-4759 • FAX (260)483-5274



QUALITY ASSURANCE MANUAL TRAINING AND ETHICS

ACKNOWLEDGEMENT STATEMENT

I have read and understood the A&L Great Lakes Laboratories, Inc. Quality Assurance Manual and ethics policy. I also understand the severity of the consequences of unethical actions such as dishonesty, piracy, fraud and/or plagiarism. In order to maintain A&L Great Lakes Laboratories, Inc. reputation for providing excellence in data quality, I pledge to do my best to consciously perform my job function in an ethical and honest manner.

Signature

Date

APPENDIX B

CHAIN OF CUSTODY DOCUMENTATION

A & L GREAT LAKES LABORATORIES, INC.
 3505 Conestoga Drive • Fort Wayne, Indiana • 46808-4413
 Phone (260) 483-4759 • Fax (260) 483-5274



ACCT#	
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SUBMITTED BY								PROJECT REFERENCE:									
Company																	
Contact Name																	
Address																	
City, State, Zip																	
Phone#																	
Sample Identification	Container Number	Container Type		Date Sampled	Time Sampled	Type Sample		Sample Preservation								Requested Analysis	
		Glass	Plastic			Grab	Composite	Cool 4° C	Frozen	H ₂ SO ₄ PH<2	HNO ₃ PH<2	HCl PH<2	NaOH PH>12	.008% Na ₂ S ₂ O ₃	Ascorbic Acid		
Sampled by						(signature)	Special Instructions										
						(printed name)	Shipper/Waybill #										
Relinquished by						(signature)	Date:		Shipping Condition								
						(printed name)	Time:										
Received by						(signature)	Date:		Receipt Condition								
						(printed name)	Time:										