Laboratory Quality Manual

FOR THE OREGON DEPARTMENT OF ENVIRONMENTAL QUALITY

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State of Oregon
Department of
Environmental
Quality

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Acronyms and Abbreviations

AR	Analytical Reagent grade
CCB	Continuing Calibration Blank
CCV	Continuing Calibration Verification
CA	Corrective Action
CAA	Clean Air Act
CERCLA	Comprehensive Environmental Response, Compensation and Liability Act
CWA	Federal Water Pollution Control Act (Clean Water Act)
DAR	Data Analysis Report
DAS	Oregon Division of Administrative Services
DEQ	Oregon Department of Environmental Quality
DOC	Demonstration Of Capability
DQO	Data Quality Objectives
EMT	DEQ's Executive Management Team
EQC	Oregon's Environmental Quality Commission
EPA	Environmental Protection Agency
FIFRA	Federal Insecticide, Fungicide and Rodenticide Act
LASAR	Laboratory Analytical Storage and Retrieval (archive database for DEQ)
LCS	Laboratory Control Sample
LIMS	Laboratory Information Management System (active database)
LMT	Laboratory Management Team
LQAO	Laboratory Quality Assurance Officer
MB	Method Blank
MDL	Method Detection Limit
MRL	Method Reporting Limit
NELAC	National Environmental Laboratory Accreditation Conference
NELAP	National Environmental Laboratory Accreditation Program
NIST	National Institute of Standards and Technology
ORELAP	Oregon Environmental Laboratory Accreditation Program
PCS	Procurement & Contract Specialist
PDF	Portable Document Format
PDR	Property Disposition Request
QA	Quality Assurance
QAO	Quality Assurance Officer
QAPP	Quality Assurance Project Plan
QAT	Quality Assurance Team
QC	Quality Control
QMP	Quality Management Plan
RCRA	Resource Conservation and Recovery Act
SAP	Sampling and Analysis Plan
SDWA	Safe Drinking Water Act
SOP	Standard Operating Procedure

Glossary[1]

Accuracy:	the degree of agreement between an observed value and an accepted reference value. Accuracy includes a combination of random error (precision) and systematic error (bias) components which are due to sampling and analytical operations; a data quality indicator. (QAMS)
Analyst:	the designated individual who performs the "hands-on" analytical methods and associated techniques and who is the one responsible for applying required laboratory practices and other pertinent quality controls to meet the required level of quality. (NELAC)
Audit:	a systematic evaluation to determine the conformance to quantitative *and qualitative* specifications of some operational function or activity. (EPA-QAD)
Batch:	environmental samples that are prepared and/or analyzed together with the same process and personnel, using the same lot(s) of reagents. A preparation batch is composed of one to 20 environmental samples of the same NELAC-defined matrix, meeting the above mentioned criteria and with a maximum time between the start of processing of the first and last sample in the batch to be 24 hours. An analytical batch is composed of prepared environmental samples (extracts, digestates or concentrates) which are analyzed together as a group. An analytical batch can include prepared samples originating from various environmental matrices and can exceed 20 samples. (NELAC Quality Systems Committee)
Chain of Custody Form:	record that documents the possession of the samples from the time of collection to receipt in the laboratory. This record generally includes: the number and types of containers; the mode of collection; collector; time of collection; preservation; and requested analyses. (NELAC)
Clean Air Act:	the enabling legislation in 42 U.S.C. 7401 et seq., Public Law 91-604, 84 Stat. 1676 Pub. L. 95-95, 91 Stat., 685 and Pub. L. 95-190, 91 Stat., 1399, as amended, empowering EPA and its delegates to promulgate air quality standards, monitor and to enforce them. (NELAC/DEQ)
Comprehensive Environmental Response, Compensation and Liability Act (CERCLA/Superfund):	the enabling legislation in 42 U.S.C. 9601-9675 et seq., as amended by the Superfund Amendments and Reauthorization Act of 1986 (SARA), 42 U.S.C. 9601et seq., to eliminate the health and environmental threats posed by hazardous waste sites. (NELAC)

[1] Sources in ( ) are presented at the end of the glossary.

Conformance:	an affirmative indication or judgment that a product or service has met the requirements of the relevant specifications, contract, or regulation; also the state of meeting the requirements. (ANSI/ASQC E4-1994)
Federal Insecticide, Fungicide and Rodenticide Act (FIFRA):	the enabling legislation under 7 U.S.C. 135 et seq., as amended, that empowers the EPA to register insecticides, fungicides, and rodenticides. (NELAC)
Federal Water Pollution Control Act (Clean Water Act, CWA):	the enabling legislation under 33 U.S.C. 1251 *et seq.*, Public Law 92-50086 Stat. 816, that empowers EPA and its delegates to set discharge limitations, write discharge permits, monitor, and bring enforcement action for non-compliance. (NELAC/DEQ)
Field Measurement:	The determination of physical, biological, or radiological properties, or chemical constituents that are measured on-site, close in time and space to the matrices being sampled/measured, following accepted test methods. This testing is performed in the field outside of a fixed-laboratory or outside of an enclosed structure that meets the requirements of a mobile laboratory. (NELAC)
Holding Times (Maximum Allowable Holding Times):	The maximum times that samples may be held prior to analysis and still be considered valid or not compromised. (40 CFR Part 136)
Integrity:	The quality or state of being complete or uncompromised. (DEQ)
Legal Chain of Custody Protocols:	Procedures employed to record the possession of samples from the time of sampling until analysis, performed at the special request of the client. These protocols include the use of a Chain of Custody Form that documents the collection, transport, and receipt of compliance samples by the laboratory. **In addition, these protocols document all handling of the samples within the laboratory.** (NELAC)
Must:	Denotes a requirement that must be met (Random House College Dictionary); to be distinguished from “shall” in that “shall” implies a policy requirement and “must” implies a standard requirement. (NELAC/DEQ)
National Institute of Standards and Technology (NIST):	An agency of the US Department of Commerce’s Technology Administration that is working with EPA, States, NELAC, and other public and commercial entities to establish a system under which private sector companies and interested States can be accredited by NIST to provide NIST-traceable proficiency testing (PT) to those laboratories testing drinking water and wastewater. (NIST)
National Environmental Laboratory Accreditation Conference (NELAC):	A voluntary organization of State and Federal environmental officials and interest groups purposed primarily to establish mutually acceptable standards for accrediting environmental laboratories. A subset of NELAP. (NELAC)

National Environmental Laboratory Accreditation Program (NELAP):	The overall National Environmental Laboratory Accreditation Program of which NELAC is a part. (NELAC)
National Voluntary Laboratory Accreditation Program (NVLAP):	A program administered by NIST that is used by providers of proficiency testing to gain accreditation for all compounds/matrices for which NVLAP accreditation is available, and for which the provider intends to provide NELAP PT samples. (NELAC)
Negative Control:	Measures taken to ensure that a test, its components, or the environment do not cause undesired effects, or produce incorrect test results. (NELAC)
NELAC Standards:	The plan of procedures for consistently evaluating and documenting the ability of laboratories performing environmental measurements to meet nationally defined standards established by the National Environmental Laboratory Accreditation Conference. (NELAC)
NELAP Recognition:	The determination by the NELAP Director that an accrediting authority meets the requirements of the NELAP and is authorized to grant NELAP accreditation to laboratories. (NELAC)
Nonconformance:	Event that does not meet laboratory requirements prescribed by policies or procedures (see conformance). (DEQ)
Programs:	Agency work units with the authority to implement state rules and regulations created through the CAA, CWA, CERCLA, FIFRA, RCRA, or SDWA.
Protocol:	A detailed written procedure for field and/or laboratory operation (e.g., sampling, analysis) which must be strictly followed. (EPA-QAD)
Quality Assurance (QA):	An integrated system of activities involving planning, quality control, quality assessment, reporting and quality improvement to ensure that a product or service meets defined standards of quality with a stated level of confidence. (QAMS)
Quality Assurance [Project] Plan (QAPP):	A formal document describing the detailed quality control procedures by which the quality requirements defined for the data and decisions pertaining to a specific project are to be achieved. (EPA-QAD)
Quality Control (QC):	The overall system of technical activities whose purpose is to measure and control the quality of a product or service so that it meets the needs of users. (QAMS)
Quality Control Sample (QCS):	A sample used to assess the performance of all or a portion of the measurement system. QC samples may be Certified Reference Materials, a blank matrix fortified by spiking, or actual samples fortified by spiking. (NELAC)

Quality Manual:	A document stating the management policies, objectives, principles, organizational structure and authority, responsibilities, accountability, and implementation of an agency, organization, or laboratory, to ensure the quality of its product and the utility of its product to its users. (NELAC)
Quality System:	A structured and documented management system describing the policies, objectives, principles, organizational authority, responsibilities, accountability, and implementation plan of an organization for ensuring quality in its work processes, products (items), and services. The quality system provides the framework for planning, implementing, and assessing work performed by the organization and for carrying out required QA and QC. (ANSI/ASQC E-41994)
Quality System Matrix:	These matrix definitions are an expansion of the field of accreditation matrices and shall be used for purposes of batch and quality control requirements (see Appendix D of NELAC standards Chapter 5). These matrix distinctions shall be used: (NELAC)
Air and Emissions:	whole gas or vapor samples including those contained in flexible or rigid wall containers and the extracted concentrated analytes of interest from a gas or vapor that are collected with a sorbent tube, impinger solution, filter, or other device.
Aqueous:	any aqueous sample excluded from the definition of Drinking Water matrix or Saline/Estuarine source. Includes surface water, groundwater, effluents, and TCLP or other extracts.
Drinking Water:	any aqueous sample that has been designated a potable or potential potable water source.
Saline/Estuarine:	any aqueous sample from an ocean or estuary, or other salt water source such as the Great Salt Lake. Non-aqueous Liquid: any organic liquid with <15% settleable solids.
Biological Tissue:	any sample of a biological origin such as fish tissue, shellfish, or plant material. Such samples shall be grouped according to origin.
Solids:	includes soils, sediments, sludges and other matrices with >15% settleable solids.
Chemical Waste:	a product or by-product of an industrial process that results in a matrix not previously defined.
Resource Conservation and Recovery Act (RCRA):	The enabling legislation under 42 USC 321 *et seq*. (1976), that gives EPA the authority to control hazardous waste from the “cradle-to-grave”, including its generation, transportation, treatment, storage, and disposal. (NELAC)
Safe Drinking Water Act (SDWA):	The enabling legislation, 42 USC 300f *et seq*. (1974), (Public Law 93-523), that requires the EPA to protect the quality of drinking water in the U.S. by setting maximum allowable contaminant levels, monitoring, and enforcing violations. (NELAC)

Sample Tracking:	Procedures employed to record the possession of the samples from the time of sampling until analysis, reporting, and archiving. These procedures include the use of a Chain of Custody Form that documents the collection, transport, and receipt of compliance samples to the laboratory. In addition, access to the laboratory is limited and controlled to protect the integrity of the samples. (NELAC)
Section Manager:	The individual designated as being responsible for a particular area or category of scientific analysis. This responsibility includes direct day-to-day supervision of technical employees, supply and instrument adequacy and upkeep, quality assurance/quality control duties and ascertaining that technical employees have the required balance of education, training and experience to perform the required analyses. (NELAC/DEQ)
Shall:	Denotes a requirement that is mandatory whenever the criterion for conformance with the specification requires that there be no deviation. This does not prohibit the use of alternative approaches or methods for implementing the specification so long as the requirement is fulfilled. (ANSI)
Should:	Denotes a guideline or recommendation whenever noncompliance with the specification is permissible. (ANSI)
Standard Operating Procedures (SOPs):	A written document which details the method of an operation, analysis or action whose techniques and procedures are thoroughly prescribed and which is accepted as the method for performing certain routine or repetitive tasks. (QAMS)
Standard Method:	A test method issued by an organization generally recognized as competent to do so. (NELAC)
Toxic Substances Control Act (TSCA):	The enabling legislation in 15 USC 2601 *et seq.*, (1976), that provides for testing, regulating, and screening all chemicals produced or imported into the United States for possible toxic effects prior to commercial manufacture. (NELAC)
Traceability:	The property that allows a measurement to be related to appropriate standards, generally international or national standards, through an unbroken chain of comparisons. (VIM-6.12/DEQ)
United States Environmental Protection Agency (EPA):	The federal governmental agency with responsibility for protecting public health and safeguarding and improving the natural environment (i.e., the air, water, and land) upon which human life depends. (US-EPA)
Validation:	The confirmation by examination and provision of objective evidence that the particular requirements for a specific intended use are fulfilled. (NELAC)

Verification:	Confirmation by examination and provision of objective evidence that specified requirements have been met. (NELAC/DEQ) NOTE: In connection with the management of measuring equipment, verification provides a means for checking that deviations between values indicated by a measuring instrument and corresponding known values of a measured quantity are consistently smaller than the maximum allowable error defined in a standard, regulation or specification peculiar to the management of the measuring equipment. Verification leads to a decision either to restore in service, to perform adjustment, to repair, to downgrade, or to declare obsolete. In all cases, it is required that a written trace of the verification performed shall be kept on the measuring instrument's individual record.

Sources:

40 CFR Part 136

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US EPA Quality Assurance Division (QAD)

Webster's New World Dictionary of the American Language

1 Quality System

1.1 *Introduction:*

The Oregon Department of Environmental Quality (DEQ) was formally established in 1969 as a product of a 1938 citizen initiative known as the Water Purification and Prevention of Pollution Bill. In 1970 the Environmental Protection Agency (EPA) was created thereby making available federal funds for environmental programs. The DEQ receives a significant portion of its funding through the EPA; consequently many of the DEQ's programs are designed to meet EPA requirements. To qualify for this federal funding the EPA has made specific requirements of the recipients, such as having a Quality System, which must be documented in the organizations Quality Management Plan (QMP). The scope of the QMP is defined by EPA's memorandum 5360 A1.

> *The QMP is a policy statement describing how an EPA organization shall comply with the requirements of EPA Order 5360.1 CHG 2. Quality systems encompass the management and technical activities necessary to plan, implement, and assess the effectiveness of QA and QC operations applied to environmental programs. The QMP provides the blueprint for how an individual EPA Program Office, Region, and National Laboratory or Center EPA Quality Manual for Environmental Programs will plan, implement, and assess its quality system for the environmental work to be performed as part of its mission. QMPs are reviewed and approved by the OEI. Approval is valid for a period of up to five years.*[2]

The DEQ first wrote its Quality Management Plan in 1982 to meet the standards at the time. The DEQ's latest QMP can be found on Q-Net and is intended to meet EPA's current (R-2) standard. The QMP describes the DEQ's policies, objectives, principles, authority responsibility and implementation of the agency's Quality Management system. EPA has signed and approved the DEQ's QMP allowing the EPA programs to grant funds and accept data from the DEQ. EPA requires the QMP be signed by the agency's senior management. DEQ's Executive Management Team (EMT), which includes the director, the deputy director, and division administrators have signed the QMP giving their concurrence. This commitment by top management is essential for the success of the agency's Quality System.

The QMP describes the use of other Quality Systems documents including this manual. The Laboratory Quality Manual (LQM) shall describe the policies and, where appropriate, procedures for all personnel within the Laboratory Division to follow. The LQM is not new to the Laboratory. Its history is similar to that of the QMP and was formerly titled the DEQ Laboratory Quality Assurance Manual. The policies and procedures in the LQM shall guide laboratory personnel in collecting, producing, maintaining, and reporting data of known quality. The LQM is continuously evolving with the discovery of omissions or undocumented policies, changes in the agency's Quality System, and adoption of new quality standards. The Laboratory Quality Assurance Officer is responsible for the review and revision of the LQM. Laboratory Division personnel must keep abreast of changes in quality policy and procedures, and therefore, must read the changes to the LQM with each revision.

The QMP also describes the agency's commitment to use National Environmental Laboratory Accreditation Conference (NELAC) standards to evaluate a laboratory's ability to generate quality data. This LQM is written to meet NELAC standards and will be reviewed and revised as required by the adoption of revised NELAC standards. NELAC standards may be revised at the annual meeting, which may require annual modifications to the Laboratory Quality Manual, however as the standards become more refined fewer and fewer changes shall come about. NELAC standards are adopted two years after they are approved at the annual meeting, thus

[2] **EPA Quality Manual for Environmental Programs 5360 A1,** 5/5/2000 US EPA, pg 2-8.

allowing laboratories and accrediting authorities the opportunity to make the appropriate adjustments in their systems. NELAC 2002 was used for the revision of this DEQ LQM.

1.2 *DEQ's Quality Management Policy*

In 2002 and re-written in 2004, as part of an investment in environmental excellence, the DEQ identified its Strategic Directions and defined the Agency's priority work. Incumbent to these Strategic Directions and DEQ's vision to work cooperatively with all Oregonians for a healthy, sustainable environment are four priorities:

- Deliver Excellence in Performance and Product;
- Protect Oregon's Water;
- Protect Human Health and the Environment from Toxics; and
- Involve Oregonians in Solving Environmental Problems.

To deliver excellence the DEQ has a policy that Agency activities shall result in products and decisions of known and acceptable quality. Consequently, the Laboratory's management practices are intended to document and ensure that all environmental data generated, stored, reported, or used by DEQ is of known and adequate quality to fulfill the needs of the primary data user. Moreover, data used by the Agency shall be accurate, precise, complete, representative, comparable, and when required, legally defensible. This policy applies to data generated both internally within DEQ through the direct efforts of Agency personnel, and data that is generated external to the Agency, arising from regulated activities, contracts, inter-agency agreements, grants, and/or cooperative agreements.[3]

Management and Staff Responsibilities: As a key action element described in the agency's Strategic Directions, the agency recognizes the importance of motivated DEQ employees to deliver excellence in their work. Laboratory management shall play an active role in supporting laboratory staff and providing a work climate that fosters excellent service and high quality work. Personnel honesty is of utmost importance for producing data of know quality. Laboratory staff shall perform their duties with the intention to meet the policies and procedures in this Quality Manual. Personnel shall not receive incentive rewards where to do so would mean that Quality Assurance (QA) policies and or procedures could not be followed, nor shall staff receive punitive actions for failing to generate data by applying QA policies and procedures. Laboratory management shall not attempt to coerce staff into reporting data of uncertain quality as if it were known to be acceptable. Personnel should contact a Quality Assurance Officer (QAO), should they feel that pressure to generate data is compromising quality. The QAO shall document the event and conduct an internal audit of the allegations. The identity of the person making the observation shall remain anonymous if so desired. It is recommended that such issues be brought before the Laboratory Quality Assurance Team (QAT). Personnel may contact any member of the QAT whom they feel most comfortable with or a Union representative to discuss their concerns.

LQM Objectives: The objectives of the LQM are to document the laboratory's quality policies and procedures, and to provide a tool for ensuring personnel are both knowledgeable of and committed to these policies and procedures. All laboratory personnel must read this document and sign an attestation memo that they shall implement the policies and procedures contained in the LQM in their work practices. These policies and procedures shall ensure:

- Laboratory personnel have appropriate training and supervision (Chapter 2 Laboratory Management Structure);

[3] "QUALITY MANAGEMENT PLAN FOR THE OREGON DEPARTMENT OF ENVIRONMENTAL QUALITY", introduction and chapter 1.1 DEQ's Management Policy.

- the implementation of proper procedures for sample collection, storage, preservation, sample tracking, analysis, and reporting (Chapter 3 Standard Operating Procedures);
- where applicable data is traceable to acceptable reference standards (Chapter 4 Measurement Traceability);
- the degree of precision, accuracy, and bias of the analyses is known and documented (Chapter 5 Assuring the Quality of Analytical Data);
- that analytical equipment is properly used, calibrated, and maintained (Chapter 6 Equipment);
- all items influencing the quality of data are properly documented (Chapter 7 Quality Assurance);
- data is reported in useful and comparable formats (Chapter 8 Reporting Results).

2 Laboratory Management Structure

The laboratory shall have managerial and technical personnel with the authority and resources needed to carry out their duties and to identify the occurrence of departures from the quality system or from the procedures for performing environmental tests, and to initiate actions to prevent or minimize such departures. The laboratory shall have technical management who shall have overall responsibility for technical operations and can allocate the necessary resources to ensure the required quality of laboratory operations. The laboratory shall also appoint a Laboratory Quality Assurance Officer (LQAO) who shall ensure NELAC standards are implemented in the laboratory.

The laboratory's organizational structure and its place in the agency supports this policy by giving the laboratory autonomy with executive leadership.

The DEQ is organized into the Office of the Director and seven Regions/Divisions which report to either the Director or Deputy Director. The Director is required to answer to the members of the Environmental Quality Commission (EQC), who are appointed by the governor and serve as an external oversight board. For a more in depth description of the Director's Office and the agency's divisions other than the Laboratory Division refer to the agency's QMP.

The Laboratory Division is organized into six structural groups under the direction of the Laboratory Administrator. The Administration section of the laboratory is under the direct supervision of the Laboratory Administrator as are the section managers for Air Quality Monitoring, Watershed Assessment, Inorganic Laboratory, Organic & General Chemistry Laboratory, and Technical Services. Each section manager is responsible for the proper management of his/her section and compliance with the LQM. Even though the Air Quality Monitoring and Watershed Assessment sections generate data while not physically in the laboratory, they must also comply with the policies and procedures of the LQM. The Laboratory Organizational Chart (Appendix H) illustrates the present structure of the laboratory. The technical requirements and training documentation for each position are described in section 2.8 below.

Section managers are responsible for the training needs of each person within their sections. Through the use of the laboratory's personnel training database, managers shall monitor required and received training. Section managers shall also submit to the LQAO documentation of personnel receiving training; and, where pertinent, certificates of Demonstration Of Capability (Appendix A). The LQAO shall maintain administrative files containing this documentation and a signature log with initials and date. These files should not be confused with other personnel files, which are maintained by the laboratory executive staff and the Human Resources Division. The laboratory executive staff must maintain files containing personal information on employees. This personal information is not available for QA review, whereas the QA administrative files shall be available for quality audits. The LQAO shall routinely destroy laboratory QA administrative documentation older than five years.

2.1 *Laboratory Quality Assurance Team*

Laboratory QA activities are processed through the laboratory Quality Assurance Team (QAT). The QAT is made up the Quality Assurance Officers (QAOs) and representatives from each section of the laboratory. The QAT may assist the QAOs with internal audits, review of documents, and QA/QC policy. The QAT shall recommend QA decisions to the Laboratory Management Team (LMT) and the Division Administrator, whereupon the Administrator shall adopt or rescind the decision. The LMT shall support and abide by all adopted QA decisions.

The mission of the laboratory QAT is to continually review quality with respect to the functions of the laboratory. The QAT shall meet routinely to discuss and implement action items, and to

develop systems to help prevent quality related problems. Laboratory personnel are encouraged to discuss Quality Management issues with the QAT members. When the QAT recognizes the need to change or adopt new quality polices or practices, the QAT shall prepare an action plan to ensure that the changes are implemented and monitored. The QAT meeting minutes shall include the perceived need of a plan, the procedure for initiating the plan, and application of controls to ensure the preventative measure is effective.

2.2 *Administration*

The administrative section of the laboratory provides support for the laboratory in the areas of Human Resources/Accounting, special projects, and Quality Management.

An executive support specialist offers administrative support to all sections of the laboratory in addition to the administrator (e.g. travel and time accounting) and serves as a liaison to PSU facilities.

Special projects include updating monitoring strategies, assisting with laboratory and monitoring operational analysis, legislative coordination for the Lab Division (including grant applications and reports), and assistance to the Administrator on unanticipated requests from outside stakeholders or colleagues.

The oversight of quality assurance issues is delegated to three QAOs. There is a QAO for each DEQ program: Air Quality, Land Quality, and Water Quality. The QAOs provide technical assistance in the development and implementation of QA project plans; audits monitoring network; performs assessments of self-monitoring activities under air, NPDES, and RCRA permits; participates in Oregon Laboratory Accreditation Program (ORELAP) activities; reports to programs documenting project data quality; and ensures corrective action procedures are followed when data quality criteria are not met. Additionally one Quality Assurance Officer shall be assigned the responsibility of ensuring that NELAC standards are implemented at the DEQ laboratory and is recognized in this document as the Laboratory Quality Assurance Officer (LQAO).

2.3 *Air Quality Monitoring*

The Air Quality Monitoring section operates and maintains the DEQ ambient air monitoring/sampling network, calibrates air monitors/samplers, collects samples, maintains equipment, and reports air monitoring data to EPA. The Air Quality Monitoring (AQM) section may audit ambient air monitoring and meteorological monitoring activities by permitted sources and Prevention of Significant Deterioration applicants.

2.4 *Watershed Assessment*

Watershed Assessment staff collect data and samples for ambient water quality monitoring of both surface and groundwater, for monitoring biological activity, and for groundwater monitoring at solid waste sites. The Watershed Assessment section maintains field equipment and instruments, provides technical assistance for the collection of water, soil, and sediment samples, performs audits of self-monitoring programs, collects split samples, and reports on audit findings and project studies.

2.5 *Inorganic Laboratory*

Inorganic personnel conduct analyses on Air / Emissions, Aqueous, Drinking Water, Saline/Estuarine, Biological Tissue, Solids, and Chemical Waste samples for inorganic constituents such as metals, nutrients, and particulate mass. They also provide technical assistance in the preparation of QA project plans relating to sampling requirements, and laboratory capabilities; and interpret inorganic analytical data.

2.6 *Organic Laboratory*

Organic personnel conduct analyses on Air / Emissions, Aqueous, Drinking Water, Saline/Estuarine, Biological Tissue, Solids, and Chemical Waste samples for organic constituents and physical properties. They also provide technical assistance relating to preparation of QA project plans, sample collection requirements, and laboratory capabilities; and interpret organic analytical data.

2.7 *Technical Services*

The Technical Services section maintains the laboratory computer network, Laboratory Information Management System (LIMS), and the laboratory data stored in these systems. Technical Services also responds to customer service and data interpretation requests. After the Laboratory Administrator approves the final release of data, the Technical Services section ensures the report recipient (programs and other agencies) receives the data in a usable format. Technical Services ensures samples are received in compliance with requirements of the Quality Assurance Project Plan (QAPP) and that sample integrity is maintained through the completion of the analytical work or that data is flagged. Technical Services also maintains an inventory control system for equipment and supplies; processes supply requisitions for the laboratory; provides clerical support; and controls access to the laboratory.

2.8 *Personnel*

The DEQ laboratory hires employees with the necessary training and experience through the recruiting procedures required by the State of Oregon. The minimum qualifications of agency positions are defined by Oregon State Division of Administrative Services (DAS). The DEQ must adhere to strict and consistent processing of all recruitments. A prospective employee must complete a State application form where he/she lists his/her qualifications and certify the information he/she gives as true and complete. All applications and recruitment materials are kept in the Human Resource office in confidential files. These files are available for internal review upon request. In order for an applicant to advance to the interview stage of a recruitment, his/her application is first reviewed by the agency's Human Resources department to insure he/she meets the minimum qualifications for the classification as determined by DAS (refer to Table 1). An applicant is required to meet the established minimum qualifications in order to proceed in the recruiting process. The DEQ laboratory management shall assume that any person who has been selected for the opportunity to interview for a particular position has successfully met or exceeded the minimum qualifications required for that position.

Many of the skilled laboratory positions require special training. Management shall ensure personnel receive the special training required for these positions. This training shall be documented and submitted to the LQAO. During an internal audit the QAO shall review these records to ensure personnel have had the training to perform their duties as required by programs, NELAC standards, and laboratory policy.

Table 1 Minimum Qualification for Laboratory Personnel

Position	Minimum Qualification
Laboratory Division Administrator (P E/Mgr G)	Qualifications will be determined by the appointing authority based on the duties and responsibilities of the position. Four years of management experience in a public or private organization which included responsibility for each of the following: a) development of program rules and policies; b) development of long- and short-range goals and plans; c) program evaluation; and d) budget preparation. Graduate level courses in management may be substituted for one year of the required experience.
Quality Assurance Officer (NRS5, Chem3, NRS3)	Three years of experience in the program area. At least one year of the experience must be at a technical or professional level performing activities in the program such as researching and analyzing data, conducting investigations, applying pertinent laws and regulations, **OR** coordinating and monitoring project activities; **AND** a Bachelor's degree in chemistry or environmental disciplines, **OR** three additional years of related (pertinent) experience. A Master's degree in chemistry or environmental disciplines will substitute for up to one year of the required experience. A Doctorate degree in chemistry or environmental disciplines will substitute for up to two years of the required experience. QAOs have day-to-day responsibility for agency program QA needs and frequently function as a team leader to fulfill program responsibilities, or frequently lead other staff and coordinate actions to accomplish central projects or studies.
Section Manager (P E/Mgr E)	Three years of management experience in a public or private organization which included responsibility for each of the following: a) development of program rules and policies, b) development of long- and short-range goals and plans, c) program evaluation, and d) budget preparation. Graduate level courses in management may be substituted for one year of the required experience. Laboratory Section managers must have the technical expertise for the sections they supervise. Experience in the field of expertise may be used to qualify a candidate.

Position	Minimum Qualification
Senior Monitoring Specialist/Chemist, and Toxics Coordinator (NRS4)	Three years of experience in the program area. At least one year of the experience must be at a technical or professional level performing activities in the program such as researching and analyzing data, conducting investigations, applying pertinent laws and regulations, **OR** coordinating and monitoring project activities; **AND** a Bachelor's degree in chemistry or environmental disciplines, **OR** three additional years of related (pertinent) experience. A Master's degree in chemistry or environmental disciplines will substitute for up to one year of the required experience. A Doctorate degree in chemistry or environmental disciplines will substitute for up to two years of the required experience. Senior Monitoring Specialist and Chemist have day-to-day responsibility as a team leader to fulfill program responsibilities, or frequently lead other staff and coordinate actions to accomplish central projects or studies.
Lead Chemist (Chem3)	Two years of experience independently performing analytical chemistry procedures which included designing, developing, and implementing analytical methods and procedures **AND** a Bachelor's degree in Chemistry Three additional years of pertinent experience may substitute for the Bachelor's. In general the lead chemist serves as a specialist with expertise in a specialty area of chemistry involving the design, development, and application of the state of the art analytical methods and procedures to complex and unusual problems and may serve as a team leader to fulfill program responsibilities.
Chemist (Chem2 & Chem1)	One year of experience independently performing analytical chemistry procedures and a Bachelor's degree in Chemistry. Three additional years of pertinent experience may substitute for the Bachelor's degree.

Position	Minimum Qualification
Monitoring Specialist (NRS3)	Three years of experience in the program area. At least one year of the experience must be at a technical or professional level performing activities in a natural resource program such as researching and analyzing data, conducting investigations, applying pertinent laws and regulations, **OR** coordinating and monitoring project activities; **AND** a Bachelor's degree in environmental related disciplines, **OR** three additional years of related (pertinent) experience. A Master's degree in environmental related disciplines will substitute for up to one year of the required experience. A Doctorate degree in environmental related disciplines will substitute for up to two years of the required experience. Monitoring Specialists frequently function as a team leader to fulfill program responsibilities.
Monitoring Technician (NRS2)	Two years of experience in environmental work, **AND** a Bachelor's degree in environmental related disciplines, or three additional years of related (pertinent) experience; OR one year of experience in environmental related disciplines, and a Master's degree in environmental related disciplines; OR a Doctorate degree in environmental related disciplines.
Project Coordinator (NRS5)	Three years of experience in the program area. At least one year of the experience must be at a technical or professional level performing activities in the program such as researching and analyzing data, conducting investigations, applying pertinent laws and regulations, **OR** coordinating and monitoring project activities; **AND** a Bachelor's degree in chemistry or environmental disciplines, **OR** three additional years of related (pertinent) experience. A Master's degree in chemistry or environmental disciplines will substitute for up to one year of the required experience. A Doctorate degree in chemistry or environmental disciplines will substitute for up to two years of the required experience.
LIMS Coordinator (ISS5)	Three years of professional consultative, technical, or administrative experience which includes designing, constructing, or analyzing information systems. Experience must include activities in Laboratory Information Systems; **AND either** (a) at least 30 quarter (20 semester) credits in computer science; **OR** (b) two more years of experience providing a knowledge of information systems theory and principles;

Position	Minimum Qualification
Database Specialist (ISS4)	Two years of professional information systems experience which includes developing, maintaining, and installing information systems, and analyzing systems. Experience must include activities in database maintenance; **AND either** (a) at least 30 quarter (20 semester) credits in computer science; **OR** (b) two more years of information systems experience;
Sample Tracker (NRS3)	Three years of experience in the program area. At least one year of the experience must be at a technical or professional level performing activities in the program such as researching and analyzing data, conducting investigations, applying pertinent laws and regulations, **OR** coordinating and monitoring project activities; **AND** a Bachelor's degree in chemistry or environmental disciplines, **OR** three additional years of related (pertinent) experience. A Master's degree in chemistry or environmental disciplines will substitute for up to one year of the required experience. A Doctorate degree in chemistry or environmental disciplines will substitute for up to two years of the required experience.
Procurement & Contract Specialist (PCS1)	Three years experience in the procurement of goods and services through purchase orders and contract agreements, or tracking and preparing simple or standard contracts or agreements **OR** A Bachelor's Degree in Business or Public Administration, or a related degree that included course work in general business management, contract or business law, accounting, finance or economics.
Document Control Coordinator (OS2)	Two years of general clerical experience, one year of which included typing, word processing, or other experience generating documents; **OR** an Associate's degree in Office Occupations or Office Technology; **OR** graduation from a private school of business with a Certificate in Office Occupations or Office Technology **AND** one year of general clerical experience. College courses in Office Occupations or Office Technology will substitute for the required experience on a year-for-year basis. Laboratory management may supplement the minimum qualifications to require specific knowledge and skills as specified in the Classification Specification, i.e. knowledge of database structure and maintenance.

Position	Minimum Qualification
Data Clerk (OS2)	Two years of general clerical experience, one year of which included typing, word processing, or other experience generating documents; **OR** an Associate's degree in Office Occupations or Office Technology; **OR** graduation from a private school of business with a Certificate in Office Occupations or Office Technology **AND** one year of general clerical experience. College courses in Office Occupations or Office Technology will substitute for the required experience on a year-for-year basis. Laboratory management may supplement the minimum qualifications to require specific knowledge and skills as specified in the Classification Specification, i.e. knowledge of Chemical naming conventions.

3 Standard Operating Procedures

The Laboratory shall provide personnel with written instructions that shall be used to ensure the correctness and reliability of test results. The laboratory shall write Standard Operating Procedures (SOPs) for repetitive routine tasks, which shall describe the techniques and procedures for an analysis or action.

Laboratory section managers shall ensure SOPs are written and controlled using the laboratory's Document Control procedure (DEQ02-LAB-0004-SOP). The Quality Assurance Team (QAT) will help identify work requiring development of an SOP. The following activities must be described in Laboratory SOPs. This list is not designed to be exhaustive. Additional activities may also need to be documented in SOPs:

- Watershed Assessment and Air Quality Monitoring section managers shall ensure that sample collection and field analysis procedures are written to cover their respective fields.
- The Laboratory Quality Assurance Officer (LQAO) shall revise the Field Sampling Reference Guide used for field operations typically performed by regional staff.
- The organic and inorganic section managers shall ensure SOPs are written for sample analyses and data transfer to appropriate databases.
- Technical Services shall ensure SOPs are written and revised for a series of routine work performed by the Technical Services section. Technical Services will make sample storage, preservation, and tracking SOPs available on Q-net.
- Technical Services shall write SOPs for the control of electronic information within the laboratory; i.e. posting documents on the web, securing servers for defined uses, maintaining tables within LIMS, creating reports from LIMS, and the reporting of results.

SOPs shall be available to all Laboratory and Agency personnel. Hardcopies may be requested through the LQAO and electronic version will be available whenever possible on Q-net.

3.1 *Sampling*

The DEQ laboratory primarily conducts analytical tests on samples collected by the different regions of the agency and by the Laboratory sections of Watershed Assessment (WA) and Air Quality Monitoring (AQM). WA has prepared a Mode of Operations Manual (MOM) to cover sampling procedures and analytical work performed in the field. AQM staff have prepared SOPs for each of the sampling techniques they use. Sampling procedures shall instruct personnel on how to collect representative samples, record data, and when applicable submit properly preserved samples to the laboratory for further analysis. Data recorded during sampling should include the sampling procedure used, the identification of sampling equipment and personnel, environmental conditions (if relevant), the time sample collection started and stopped, the date, and the sampling location.

The LQAO shall maintain the Field Sampling Reference Guide, which instructs personnel outside the laboratory on how to collect and submit samples to the DEQ laboratory. The Technical Services section shall make these documents available on Q-Net.

The laboratory must have on file copies of Quality Assurance Project Plans (QAPPs) as required by the agency's QMP. The QAPP should describe the methods used to collect samples, ensuring the quality of the data and, if appropriate, the statistics used to develop the sampling procedures. The QAPP shall provide sampling information or the appropriate template for writing a Sampling & Analysis Plan (SAP). SAPs are a subgroup of the larger more encompassing QAPP. There are cases where the QAPP shall describe the elements within the

SAP and thus a SAP may not be necessary for all samples. SAPs are also controlled documents (refer to the agency's QMP for more information). The SAP should primarily describe the schedule for collecting samples, the location of the sample, and the requested test methods. The laboratory should receive the SAP and/or QAPP before sampling begins so the different sections of the laboratory can make the necessary arrangements to perform the work.

If the project coordinator requires a deviation from the QAPP, a SAP may describe the deviation without revising the QAPP. Technical Services shall devise a system for ensuring that all appropriate personnel are advised of SAPs and that they shall have access to SAPs.

3.2 *Sample Handling*

Technical Services shall receive samples submitted to the laboratory. The Technical Services manager shall ensure the Sample Receiving SOP is controlled as with all other SOPs. The procedure shall describe required documentation and information on the transportation, receipt, handling, protection, storage, retention and/or disposal of samples, including all provisions necessary to protect the integrity of the sample.

3.2.1 Sample Identification

Upon sample receipt, the sample tracker shall assign a sampling event number to the group of samples packaged together. Each container is inspected to ensure the container identification number appears on the form used to submit the samples. Typically labels are made of a durable plastic material with pre-printed numbers or are engraved into the container. The container number along with the sampling event number uniquely identifies each sample/sub-sample received. The sample tracker logs the sampling event number and container number into LIMS, which assigns an item number to each sample linking sample, sub-samples and sample location. This combination of numbers is used in LIMS to minimize confusion regarding the identity of samples at any time. The combined sampling event number, item number and/or container number are used for the identification of all subsequent extracts and/or digestates to link the sample with all related laboratory activities.

As the supply of containers diminishes over time new containers are purchased and label numbers are engraved into them. The numbers assigned to the container should not exceed four digits to minimize transcription errors. The laboratory has used several thousand containers over the years and the numbering system has rolled over since its conception. Because it is not clear whether the container was disposed of prior to reusing a number, it is possible for the same container number to appear in the system at the same time. Technical Services shall maintain a controlled log of container numbers to manage the process of discarding old containers and labeling new containers. In the event that more than one sample with the same container number is entered into LIMS at the same time, the LIMS system will catch the problem and prompt the sample tracker to verify the numbers. The sample tracker may append a suffix to the number or re-label the container to create a unique combination of numbers.

3.2.2 Sample Receipt

LIMS shall track who received the samples and the date and time the sample is entered into the database. This record will be the official sample receipt date and time. Because some methods require short holding times it is important for the project coordinator to account for transportation time from the shipper and the time it takes for the tracker to log in the sampling event. The project coordinator shall discuss these details with the QAO during the development of the QAPP.

Upon receipt of samples, the sample tracker shall fill out a "Sample Receipt Checklist" that will identify the number of shipping containers, the internal temperature of those containers, the condition of the samples, the status of preservation checks, and whether or not the samples were shipped and received with a Chain-of-Custody form. Any abnormalities or departures from acceptable conditions as described in the QAPP or SOPs shall be noted on the checklist. This checklist may be used in lieu of the Nonconformance forms in Appendix C, however the procedures in section 7.5.3 below must be followed to ensure the proper information is collected and documented. The sample tracker must consult with the project coordinator and document all correspondence to determine the course of action, i.e. to resample, void, or proceed with sample analysis. The project coordinator's instructions must be determined prior to proceeding with the analyses. The sample tracker's Sample Receiving procedure shall be provided to project coordinators during the development of their QAPP so they may add any specific needs for handling samples. The project coordinator shall either accept the sample trackers procedure or address procedures in the QAPP for avoiding deterioration, contamination, loss or damage to the sample during storage, handling, preparation and testing. Without explicit sample handling procedures described in the QAPP, the laboratory shall assume that there is no deviation from the tracker's Sample Receiving SOP. Should the sample tracker discover special sample handling instructions at the time of receipt, the sample tracker shall communicate the sample handling deviation to laboratory managers using the proper Nonconformance documentation procedures (refer to Appendix C: Nonconformance Report).

The laboratory uses a special Legal Chain Of Custody procedure for handling samples collected for the use of enforcement investigations. This procedure documents the location of the sample and who has removed it from its locked location to perform his/her work. The sample tracker shall record the "Legal" status of the sample in LIMS. The tracker, Technical Services manager, and the assistant to the laboratory director shall be the only personnel to have a key to the locked "Legal" storage areas.

3.2.3 Sample Integrity

The sample tracker shall place all samples in specified storage areas. The sample tracker shall ensure that these areas are maintained and the environmental conditions are monitored, and recorded. The tracker's Sample Receiving SOP shall describe how to check, record, and identify the types of samples requiring the following prescribed preservation protocols:

1) Samples which require thermal preservation shall be stored under refrigeration which is +/-2°C of the specified preservation temperature unless method specific criteria exist. For samples with a specified storage temperature of 4°C, storage at a temperature above the freezing point of water to 6°C shall be acceptable.
    a) Samples that are hand delivered to the laboratory immediately after collection may not meet this criterion. In these cases, the samples shall be considered acceptable if there is evidence that chilling has begun such as arrival on ice.
2) Samples requiring pH preservation shall be checked either at the time of sample receipt or by the laboratory analysts; the point at which preservation is checked will be documented on the Sample Receipt Checklist. All preservation checks will be electronically documented in the LIMS.
3) Samples shall be stored away from all standards, reagents, food and other potentially contaminating sources. Samples shall be stored in such a manner to prevent cross contamination.
    a) Sample fractions, extracts, leachates and other sample preparation products shall be stored as described above or according to specifications in the test method or QAPP.

b) Test method SOPs shall describe the process for the disposal of digestates, leachates and extracts or other sample preparation products.

c) The Technical Services section shall maintain the SOP for the disposal of samples. Samples identified as Hazardous Waste shall be collected for disposal at an appropriate facility, refer to the laboratory's Chemical Waste Management SOP (DEQ04-LAB-0014-SOP).

4) After logging in samples the tracker shall notify appropriate personnel of the receipt of samples with short holding times (for methods with holding times of less than or equal to 48 hours refer to the sample tracker's Sample Receiving SOP). Chemists shall begin these test methods within the cited holding times.

Personnel who find that any of the above sample integrity policies has been compromised shall initiate the corrective action procedure described in this document and begin the completion of the Nonconformance Report. In most cases the data shall be reported as an estimate with an appropriate comment attached to the result.

3.3 *Test Methods and Method Validation*

3.3.1 Analytical SOPs

Appendix G lists the analytical test methods the laboratory performs and cites the EPA reference test method. The laboratory shall have controlled (refer to section 2.8) written Standard Operating Procedures (SOPs) for each of these test methods. Authors of test method SOPs shall write or revise the SOP to conform to the cited procedure, but any revision shall not alter the chemistry involved in the cited method. Copies of the cited reference materials shall be controlled and retained for the same period as the referencing SOP. Laboratory SOPs shall clarify ambiguities and explicitly identify options used in the referenced method. The author shall clearly note any deviation from the referenced test method.

The DEQ laboratory shall attempt to use peer-reviewed and validated methods published by international, national, or regional authorities. The laboratory shall ensure it uses the latest valid edition of a standard unless it is not appropriate or possible to do so. Deviations from this policy shall require discussions between the project coordinator, laboratory section managers, and the QAO. When necessary, the Laboratory-specific SOP shall supplement the reference method with additional details to ensure consistent application. The DEQ shall only use EPA promulgated test methods for programs that require such methods. The QAOs shall ensure QAPPs cite the appropriate analytical methodologies.

Test method SOPs shall instruct the reader on the use and operation of all relevant equipment, and on the handling and preparation of samples (refer to Preparing Standard Operating Procedures: DEQ04-LAB-0001-SOP for creating an acceptable SOP). During the annual internal audit, the auditors shall cite, when necessary, the deficiency of failing to meet criteria described in the Preparing Standard Operating Procedures. The corrective action plan for this deficiency shall be to revise the SOP to meet the current NELAC standards and to set a schedule for completing the revision.

There are occasions where projects require technical deviations from the laboratory's documented procedures. Deviation from cited methods shall occur only if the deviation is documented in the QAPP. The deviation shall be technically justified, authorized, and accepted by appropriate personnel (typically the signatories on the QAPP). The QAO shall ensure that method deviations required by the project are documented in the QAPP and communicated to all appropriate personnel, which includes personnel identified in the QAPP and all appropriate sections of the laboratory. Test results that are reported using the altered procedure shall be

recorded and tracked in LIMS by creating a unique “Standard Parameter” for the new procedure.

During the development of the QAPP, the QAO shall advise the project coordinator if test methods appear to be inappropriate or out of date. If a decision is made to continue with an undocumented procedure, the DEQ laboratory will then develop its own procedure. The QAO shall assign the task of creating the new SOP to the appropriate laboratory section. The section manager shall delegate the responsibility for developing the method and writing the SOP to appropriate personnel.

The analytical sections of the laboratory shall play the most significant role in the development and documentation of new test methods; however, they must maintain open communication with the QAO and management. The development of new methods shall be a planned activity, which will require routine meetings between analytical staff, management, and the QAO. The meeting minutes shall become part of the SOP control document file. The meetings shall cover the process of developing the method and its progress. QAT members shall put method development on their meeting agenda to share the progress of their new methods with other sections of the laboratory. New methods will be based on the laboratory’s best available technologies and preferably on test methods published by reputable organizations or instrument manufacturers. Communication between project coordinators and QAO is essential to ensure laboratory procedures conform to program requirements.

3.3.2 Demonstrations of Capability

Prior to using a procedure, all test methods must be validated through completion of an Initial Demonstration of Capability (IDOC). A summary of the IDOC data shall be included in the "Method Performance" section of the analytical SOP. The IDOC should confirm the laboratory’s ability to meet QC criteria cited in referenced methods. Ongoing QC should ensure that each batch of data produced continues to meet expected quality. Continuing Demonstration Of Capability (CDOC) studies are required annually and shall demonstrate the laboratory’s continued proficiency to perform the analytical method. DOC records (refer to Appendix A) will be kept on file for all personnel who perform analytical test methods. SOPs shall be reviewed annually and DOC procedures shall be verified to comply with the current NELAC standards. Details of the Laboratory's DOC procedures will be written by the LQAO.

3.3.3 Data Validation

Laboratory personnel will ensure that all reported data satisfies – at a minimum – the appropriate analytical Quality Control requirements. The typical DEQ analytical procedure must satisfy specific QC requirements, which are usually cited in the reference method. In the event that data does not meet the requisite QC criteria, results will be reported with data qualifiers. During the development of the QAPP the QAOs will discuss the laboratory's QC procedures and establish if they are appropriate for the project needs. In many instances, the Laboratory's QC requirements and control limits will meet or exceed project needs and the Laboratory's default procedures will be used. However, if the project requires the reporting of data that falls outside the Laboratory's default control limits, the laboratory shall develop and document procedures for reporting such data in the QAPP. By default, data that falling outside the default control limits will be reported as estimates ("est"), and the data flagged with in LIMS with the QA status code of “B”. LIMS status codes (Appendix D) are used to track the quality of analytical results in the Laboratory Analytical Storage and Retrieval (LASAR) database. This coding system allows the secondary and LASAR data users to quickly and easily assess data quality using the Laboratory's default QC criteria. The status code of "B" is used to mark data as failing to meet the Laboratory's default QC standards. Data users should carefully review data and the associated QC information before using it.

The Laboratory will not provide detailed data quality assessment services to the Agency by default. Generally, Project Coordinators sending samples to the Laboratory shall be responsible for the statistical validation of data for their projects. Project Coordinators submitting samples to the lab should first identify their Data Quality Objectives (e.g. verifying a null hypothesis) and establish the appropriate the statistical approach for assessing the data set. Laboratory assistance with data quality assessments, can be arranged between the project coordinator and a QAO or the Technical Services section manager. Data quality concerns should be reported to the QAOs so that DQOs can be refined in future QAPPs. Project Coordinators are encouraged to request QC data with the analytical report, so they can more readily evaluate the effectiveness of the project.

3.3.4 Estimation of Uncertainty of Measurement

With adoption of the 2002 NELAC standards, the DEQ Laboratory is required to establish a system for estimating measurement uncertainty. Analytical SOPs written or revised after adoption of these standards (01 July 2004) shall discuss the sources of measurement uncertainty. If rigorous, metrologically and statistically valid, calculations of uncertainty cannot be described, the SOP shall attempt to identify all the major components of analytical uncertainty and make a reasonable estimation of the expected uncertainty. Under no circumstances should the Laboratory misrepresent the estimation of measurement uncertainty; reasonable attempts must be made to establish the limits of measurement uncertainty for all analytical methods. Reasonable attempts at estimation shall be based on knowledge of the method performance and on the measurement scope, and shall make use of previous experience and validation data.

When estimating measurement uncertainty, all uncertainty components which are important in a given situation shall be taken into account using appropriate methods of analysis.

3.3.5 Data Control

Employees performing new functions shall have their work reviewed by their peers. Section managers may assign this task to other staff members or review the work themselves. Typically the frequency of review diminishes as personnel become more competent. However comfort can lead to a level of complacency and a system of routine checks is needed.

Section mangers shall write a procedure for data control within their sections. Since different analytical methods and programs require different recording procedures, a laboratory-wide generic data control procedure is not practical. All data control procedures shall comply with section 7.2 of this document (Analytical Records). The procedure shall assign responsibility for the review of integrations, data transfer, and manual calculations; the frequency of these reviews; and the implementation process for potential corrective actions.

As described in the Nonconformance Investigations and Corrective Action procedure an analyst shall typically review data for transfer and calculation errors prior to completing the work lists in LIMS. The lead chemists, section managers and QAO shall then examine quality control measures prior to approving the data during the review processes.

Most of the analytical equipment used by the laboratory is controlled by computers. The software on these computers must have sufficient documentation, which is adequate for its intended use. Personnel purchasing new systems shall ensure they come with appropriate documentation. Commercial off-the-shelf software (e. g. word processing, database and statistical programs) in general use within the designed application range may be considered to be sufficiently validated. Personnel shall maintain automated equipment to ensure they function properly. The laboratory facility shall provide the necessary environmental and operating conditions to maintain the integrity of data stored in these computer systems.

The Technical Services section shall develop a SOP for ensuring the data integrity of electronic data. This SOP shall describe the process used to maintain, document, and store data ensuring that it can not be altered, is transmitted correctly, and processing does not produce misinformation. Data shall be stored such that security can be monitored and access controlled.

4 Measurement Traceability

The laboratory shall ensure analytical measurements are traceable to acceptable reference standards.

4.1 *Documentation*

In order to ensure data is of known quality, laboratory staff must be able to document the source and tolerance levels of reference standards, and reagents. To the best of its ability, the laboratory shall use National Institute of Standards and Technology (NIST) traceable standards and contract services that use NIST traceable standards. The laboratory must be able to show a paper trail from the test result of a sample back to the NIST standard for the analyte and all measurement devices used in the analysis. Analysts performing analytical work shall follow the laboratory's documentation procedures ensuring that analytical results are easily linked to calibration data, which is linked to reference material certificates described in Purchasing Services and Supplies section below.

There are occasions when certificates are not available in which case the analyst must validate the suitability of the reference. The analyst should obtain approval from his/her section manager prior to testing the suitability of a product, since the Laboratory should be able to purchase materials with certificates. This suitability testing procedure must be documented and approved by the Quality Assurance Team (QAT) or test method signatories. As a final measure for verifying the suitability of uncertified materials, the analyst must evaluate the result of a Laboratory Control Sample (LCS) using the material in question. Results of the suitability testing shall be documented as other test method results, thus the first occurrence of the product identification number in the analytical records will be that of the suitability test.

- ***Glassware, Chemicals, Reagent Water, Gases, and Reagents***

The laboratory will purchase supplies of the highest quality needed to ensure minimal interference or contamination with a procedure. As appropriate Chemists and Technicians will:

- use "Class A" volumetric glassware for the preparation and dilution of reagents, standards, and samples;
- ensure non-volumetric glassware is of an appropriate quality;
- ensure compressed gases are of known purity and guaranteed by the supplier;
- ensure chemicals are dated on receipt, stored according to chemical properties, and discarded when shelf life is exceeded (for chemicals where shelf life is defined);
- ensure solvents employed in organic analyses are "HPLC" or "Pesticide grade" and stored in ventilated explosion-proof cabinets; and,
- ensure analytical reagents or solvents are never stored with samples awaiting analysis.

The analyst shall document reagent preparation. Documentation shall include the source of the reagent, the mass or volume used, and dilution information. The test method SOP should contain specific instructions for reagent preparation and documentation. Reagents shall be prepared from "Analytical Reagent" grade (AR) or higher purity chemicals as required by the method, and shall be stored as recommended in the method, or by the chemical manufacturer.

4.2 *Purchasing Services and Supplies*

For the purpose of this LQM only the purchasing procedures for products that have an effect on data quality are discussed here. For example the purchases of paper and phone services are

not directly related to data quality. Whereas the quality of chemical reagents, concentration of standards, instrumentation, and instrument service contracts do have an impact on the quality of data reported. In order to ensure the integrity of data, laboratory staff must follow these procedures for purchasing services and supplies.

It is the responsibility of the Procurement & Contract Specialist (PCS) to purchase, receive, label appropriate supplies, and to dispose of unused obsolete equipment and supplies by following the State Surplus procedures and completing the Property Disposition Request (PDR) forms. The Technical Services section manager shall ensure the PCS is properly trained and instructed to apply the following Request for Purchase SOP to all chemicals, measuring devices, and service contracts.

4.2.1 Request for Purchase

The PCS, analyst, and the section manager are responsible for the traceability of supplies. The PCS shall maintain the Requisition database, which is used to track the purchase and receipt of all orders.

The chemist or technician, who needs the supplies shall investigate purchase options and request the purchase of appropriate supplies and/or services. Even though some laboratory sections may designate an individual to complete the Requisition forms, the chemist/technician requesting the supplies shall note the quality needed. Signatory authorities who sign test method SOPs shall ensure the SOP specifies the quality of chemicals and instrument to be used. Test method SOPs shall identify chemicals that need not come with certificates or are unlikely to have such certification. Test method SOPs shall describe how these chemicals will be evaluated to assess their suitability.

The purchaser of supplies or services in each section shall use the Requisition database, where the purchaser may retrieve historical data to help determine viable vendors. The list of vendors in the database is maintained by the PCS. Chemists and technicians using the products shall offer feedback to the Technical Services section as to the quality of the product received from the vendor. Technical Services shall document the feedback and link the comments to the vendors within the Requisition database. This information shall be reviewed for the annual QA report to management and for future consideration of a given vendor. Some programs may require the use of specific products available through qualified vendors. The Requisition database shall maintain an auditable link between product and vendor ensuring the use of appropriate vendors for prescriptive program needs.

It is the responsibility of management to either concur with staff requests for purchase or disapprove them. When the section manager signs the Requisition form he/she is agreeing with the chemist's assessment of the appropriate product quality.

4.2.2 Purchase

All purchases have a formal purchase approval process. Expenses over an amount set by the Oregon State Division of Administrative Services (DAS) must be approved by DAS. Less expensive orders may be approved by the DEQ's accounting office or laboratory personnel. The triggers, which identify who is to grant final approval of a purchase, change with time and the PCS shall stay informed of the current policy.

Many purchases are performed using credit cards. Such purchases must be approved by the credit card holder and the section manager. Since these purchases may occur without prior consent, personnel should take caution and management shall inform personnel of the current credit card policy. Personnel may be held responsible for inappropriate purchases.

To ensure products directly related to data quality are tracked; personnel must copy the Requisition by printing it from the database or by filing out the paper form. Products that shall be tracked are often of sufficient value to trigger the requirement to complete a Requisition; however, laboratory staff may use petty cash for inexpensive supplies. Personnel must be mindful of the requirement that certain products must be tracked and the PCS must have a copy of the Requisition to ensure data for such products are entered into the system.

4.2.3 Receipt

Upon receipt of any product the laboratory receptionist shall notify the PCS of its arrival. The PCS shall identify the product and locate a copy of the Requisition form and notify the staff member who requested the purchase. The purchaser shall inspect the product for consistency with the order and possible shipping damage. The purchaser shall also verify the quality of any chemical received and verify that the appropriate certificate of analysis was sent. If the purchaser finds no problems, he/she shall sign the receipt and return it to the PCS. The recipient of contracted services shall request copies of certificates from the contractor to maintain the laboratory's traceability requirements. If there is a problem with the order the PCS shall take appropriate steps to reverse the order or the analyst may attempt to verify the suitability of the product (refer to section 4 Measurement Traceability).

4.2.4 Labeling

If the product is acceptable the PCS shall label the product with a unique identification number. The identification number will be logged into the Requisition database and transcribed to the Requisition form, receipt, and accompanying quality certificates. The PCS shall file the receipt and Requisition form in the appropriate binder. The PCS shall forward quality certificates to the LQAO, who shall file the certificate in the Quality Document file. The requisition binders and Quality Document files shall be retained for at least five years.

4.2.5 Storage

The laboratory has very little storage space; personnel are encouraged to make accommodation for purchases prior to receiving supplies. Chemical reagents and standards must be handled such that their composition shall not be jeopardized. Some chemicals must be preserved and should come with special instructions; chemists shall inform the PCS of special handling procedures when completing their Requisition form. When the section manager signs the Requisition form, he/she should also look for special handling instructions of which the PCS should be aware.

Often Material Safety Data Sheet (MSDS) are packaged with chemicals. The purchaser should forward MSDSs to the laboratory receptionist who will file them in the MSDS notebooks stored in the centralized Safety area.

4.2.6 Use and Consumption

Personnel who find consumable materials or equipment that affect the quality of analytical data without the appropriate laboratory identification label shall notify the PCS and obtain a new label. Personnel shall transcribe the identification number of the chemicals and instrument devices used during the analysis to secondary containers, logs, computer systems used for tracking data output, and bench work sheets. LIMS may also be used to document the use of these materials. Such documentation shall be adequate to verify the calibration of all measuring devices used during the analysis that are critical in the computation of the result.

Equipment and supplies which are not the desired level of quality shall be taken out of service. Instruments, which have served their purpose and are no longer of any use to the agency,

should be disposed of through the State Surplus procedures. Personnel should contact the PCS for the current procedure, which shall include completing the PDR form and moving the equipment to the holding area so that it can not be inappropriately put back into service.

Chemical supplies have a shelf life and shall not be used beyond the recommended holding time unless they are tested and proven to still be suitable for use.

5 Assuring the Quality of Analytical Data

The laboratory shall define Quality Control (QC) measures and provide a process of data review to ensure the degree of precision, accuracy, and bias of the analyses is known and documented.

In addition to maintaining traceability to NIST or like standards to ensure the production of quality data, the laboratory shall participate in Performance Evaluation studies, Inter-laboratory split comparisons, and EPA triennial audits. The LQAO shall use the information collected from these sources with the routine QC data to develop plans to prevent the deterioration of data quality.

The multiple levels of review as described below in section 7.5.3.5 (Technical Corrective Action) plays a significant role in reporting quality data. This review process shall ensure that all QC measures of precision, accuracy, and bias are reviewed by qualified personnel. Test method SOPs shall describe the QC measure to be taken by the analyst. Laboratory section managers shall assign the responsibility of monitoring QC data to the appropriate chemists or technicians, who shall evaluate calibration, Laboratory Control Samples (LCS), Matrix Spikes (MS), blanks, surrogates, and duplicate measurements. Where applicable the chemist shall use statistical techniques to summarize the QC data. During the internal audit of the test method SOP, the auditor shall review the summarized QC data looking for trends. The chemist shall not rely on the internal audit to discover trends in his/her QC data; he/she shall continually review his/her QC data and use the Nonconformance Report form (Appendix C), should he/she discover anomalies or exceed QC limits.

The lead or senior chemist/technician shall validate data per sampling event by reviewing sample history, comparing intra-sample results, and investigating data anomalies. The section manger shall review the comments of the chemists and lead chemist and the decision process when necessary.

Personnel performing an internal audit shall review test method SOPs ensuring all necessary QC measures listed in Appendix F are accounted for in the procedure. The auditor shall consider the essential QC standards outlined in the current NELAC standard, mandated methods or regulations (whichever is more stringent) to determine if any deficiencies exist. If the test method SOP and cited documents do not described how to conduct the QC measures described in Appendix F the auditor shall write a deficiency. Through the corrective action process, the QAO and Laboratory Section Manager shall determine if the cited QC should be included in the SOP.

Specific procedures for conducting each of the QC measures in Appendix F shall be developed by the laboratory's Quality Assurance Team (QAT). These procedures shall be entered into the Document Control database, which shall be linked to the training database.

6 Equipment

The laboratory shall ensure analytical equipment is properly used, calibrated, and maintained

The laboratory is equipped with state-of-the-art analytical instrumentation for analysis of environmental samples. Appendix E lists the major analytical instrumentation used in the DEQ Laboratory (this list does not include field instrumentation). Instruments are maintained in proper operating condition through service contracts on major equipment, and by following maintenance and calibration schedules. No equipment shall be used without first verifying its accuracy. Records of this initial verification and the ongoing maintenance performed on equipment shall be recorded in controlled maintenance/calibration logs (refer to Analytical Records section 7.2 below). Some equipment such as volumetric glassware may only require the purchase receipt for documentation of its accuracy. Should the laboratory use equipment that is not owned by the laboratory, the laboratory shall maintain and calibrate said instrument as prescribed in this section of the LQM. Records and maintenance logs shall be retained by the laboratory, even though the equipment may not be retained.

Calibration procedures must be included in the test method SOPs. Calibration requirements are divided into three parts:

1) requirements for analytical support equipment,
2) requirements for standardizing reagents used for calculating concentration, and
3) requirements for instrument calibration, which is further divided into
    a) initial instrument calibration and
    b) continuing instrument calibration verification

6.1 *Support Equipment*

Support equipment may not be the actual test instruments, but are necessary to support laboratory operations. These include but are not limited to: balances, ovens, refrigerators, freezers, incubators, water baths, temperature measuring devices (including thermometers and thermistors), thermal/pressure sample preparation devices and volumetric dispensing devices (such as Eppendorf®, or automatic dilutor/dispensing devices) if quantitative results are dependent on their accuracy, as in standards preparation and dispensing or dilution into a specified volume.

Often this equipment is shared within the laboratory, however, not usually between sections. The section manager shall ensure all support equipment used in his/her section shall be maintained and in proper working order. Records of all repairs, maintenance activities including service calls, and calibration data shall be recorded in appropriate logs. These logs shall be controlled as described in section 7.2 (Analytical Records).

- Support equipment shall be calibrated or verified at least annually, using NIST traceable references when available, over the entire range of use. The results of such calibration shall be within the specifications required of the application of this equipment or:
    i. the equipment shall be removed from service until repaired; or
    ii. the laboratory shall maintain records of established correction factors to correct all measurements.
- Raw data records shall be retained to document equipment performance.
- Prior to use on each working day, balances, ovens, refrigerators, freezers, and water baths shall be checked in the expected use range, with NIST traceable references where

available. The acceptability for use or continued use shall depend on the needs of the intended analysis or application.

- Mechanical volumetric dispensing devices including burettes (except Class A glassware) shall be checked for accuracy on at least a quarterly use basis. If these devices are not used during the quarter it is not necessary to check their accuracy. Glass micro-liter syringes are to be considered in the same manner as Class A glassware, but must come with a certificate attesting to established accuracy or the accuracy must be initially demonstrated and documented by the laboratory.
- For chemical tests the temperature, cycle time, and pressure of each run of autoclaves must be documented by the use of appropriate chemical indicators or temperature recorders and pressure gauges.

6.2 *Instruments used for the Determinative Step*

This section of the LQM lists and describes the use of equipment required for to perform the analytical methods listed in Appendix G. It should be noted that typically all procedures the lab performs will be developed from EPA reference test methods, which already describe the calibration procedures summarized in this document. This document dictates the criteria for selecting the correct options when further developing methods for laboratory use and writing the SOPs. Each analytical test method SOP shall describe the essential elements for when and how to perform the Initial Calibration, and the Continuing Calibration Verification as well as the type of calibration to be used.

Unless the reference method specifies otherwise, a multipoint calibration curve shall be created for the Initial Calibration. Single point calibrations may not be used except with approval from the LQAO, the section manager, and when one of the following three conditions is satisfied:

- the method does not allow the use of multiple point calibrations;
- instrument manufacturers have validated calibrations techniques for the technology and the promulgated test method cites the technique; or
- the method employs standardization with a single standard i.e. titration techniques.

When using multipoint calibrations, the analytical results must be calculated from the initial instrument calibration using one of these three approaches:

- linear regression (the preferred option);
- second order regressions may be used if during the development of the analytical test method it is demonstrated that a linear response cannot be routinely achieved and a second order regression yields more appropriate results. Appropriate use of second order regressions must be documented in the SOP. Third order regressions and greater are not allowed; or
- a point to point calibration (i.e., a linear regression fit between the two nearest points) may be used if the first and second order curves prove to be inappropriate for the test method.

In each case raw data from the instrument and the knowledge of the standard concentrations are used in the statistical method of least squares to create a calibration curve. The analyst shall record and determine QC control limits for the correlation coefficients. Test method SOPs shall provide the control limits for the coefficients and the number of calibration standards to use. In the event that circumstances do not allow for the proper calibration, the analyst must follow the Nonconformance Investigations and Corrective Action procedure (section 7.5.3) and initiate the completion of the appropriate Nonconformance form. The test method SOP shall

also describe how raw data records are to be retained. Data records shall contain sufficient information to permit the reconstruction of the initial instrument calibration, including:

- calibration date;
- test method;
- instrument;
- analysis date;
- analyte name (or analyst's initials or signature);
- concentration and response of each standard;
- unique equation or coefficient used to convert instrument responses to analyte concentration; and
- calibration assessment factor (i.e., r^2 or other criteria).

Sample results shall be quantitated from the initial instrument calibration and may not be calculated from any continuing instrument calibration verification unless otherwise specified in the QAPP.

With the exception of those test methods that allow for a single point calibration at least three calibration standards shall be used in the initial calibration not including the calibration blank or a zero standard. The concentrations of the standards shall be distributed throughout the calibration range. The lowest calibration standard shall be equal to or less than program required reporting levels when appropriate and the analytical method reporting limit. If specific programs are more prescriptive in establishing the initial calibration, those requirements shall be followed. During the development of the laboratory method, the lowest calibration standard (MRL) shall be determined by using a dilution of the stock standard that is one to five times greater than the Method Detection Limit (refer to Appendix B: 40 CFR Part 136, App. B). If blank samples tend to yield detectable contamination, the lowest calibration standard shall be set to five to ten times greater than the MDL. Refer to the Laboratory's procedures for establishing the Method Detection Limit and Method Reporting Limit for additional details.

For analytical methods that have been approved to use a single point calibration, the following requirements must be satisfied:

1) the linear dynamic range (LDR) of the instrument must be established (and verified annually) by analyzing a series of standards, beginning at the MRL and extending to the highest concentration that will be reported without sample dilution;
2) a new calibration (with a calibration blank and single point calibration standard) must be created for every analytical batch;
3) the calibration curve must be verified immediately following the initial calibration with two second source quality control samples. One QCS must be analyzed at the MRL and the second QCS must be analyzed at a second concentration that will not exceed 90% of the single point standard concentration;
4) Sample concentrations within an analytical batch that exceed the single point calibration concentration must be handled with one of the following procedures:
    a) analysis of a reference material at or above the sample value that meets established acceptance criteria for validating the linearity;
    b) sample dilution such that the result falls below the single point calibration concentration; or

c) use of an appropriate data qualifier and comment explaining the qualifier flag.

For analytical methods that do not require an initial calibration with every analytical batch, a new initial calibration shall be prepared whenever there is sufficient change in instrument setup or reagents used to cause a quantitative change in instrument response, or when QC failures initiate the Corrective Action procedure from which it is determined the instrument should be recalibrated. Immediately following the initial calibration a sample called an Initial Calibration Verification (ICV) shall be run to verify the quality of the calibration. The ICV will be prepared from a second source standard when possible and meet the same quality as the primary calibration standard. The SOP shall cite the control limits set on the ICV.

If it is necessary to report data generated from calibrations that do not meet control limits or the ICV fails to fall within the acceptable range and it is not possible to rerun samples, the analytical results shall be reported as estimates with comments explaining the problem.

6.3 *Accommodation and Environmental Conditions*

6.3.1 Laboratory Facility

The DEQ laboratory is equipped with sufficient power resources to operate instruments and equipment safely for the work noted above. Space is at a premium so personnel must employ good “house keeping” procedures to accommodate other work performed in common areas. When appropriate the test method SOP shall describe “house keeping” procedures. Personnel must communicate with the Quality Assurance Team (QAT) and management should they recognize limits of the PSU facility that hamper the progress of producing quality data. As with all other observations and findings, management shall ensure that this communication is documented. The Nonconformance form shall be completed and an internal audit initiated if appropriate.

The laboratory is structurally organized to isolate similar work to be accomplished in general work areas. Personnel working in these areas are naturally familiar with similar cleaning procedures, sample handling procedures, and instrument requirements. This division helps to ensure that sample integrity and instrument response won’t be compromised. Special care is taken to ensure that cross contamination won’t occur. In particular the Volatile Organic Compounds (VOCs) analysis is easily contaminated by work performed on non-volatile organics. Clean VOC work shall be performed in L-71 A and organic extractions that use solvents, which have VOC contaminants, shall be performed in L-63. Special care is also taken for the analysis of metals by ICP/MS, which is capable of measuring very low levels of metal contamination. The normal laboratory environment will create contamination problems for tests performed by ICP/MS, thus a special clean hood is used for ICP/MS tests.

In addition to the structural requirements for analytical testing and calibration procedures, the laboratory shall maintain work areas with sufficient access and entryways to the laboratory:

a) sample receipt area(s);
b) sample storage area(s);
c) chemical and waste storage area(s); and,
d) office space for data handling and data storage

Access to the laboratory is controlled by requiring all visitors report to the receptionist and sign in. The receptionist shall page the appropriate personnel, who shall inform the guest of our safety policy, evacuation routes, and location of work areas. The laboratory is divided into an upper and lower floor. Each floor has a main hallway that divides most desk spaces from the calibration and analytical test methods areas. Special safety rules, which also help preserve data quality, are required in the calibration and analytical areas. Personnel are required to read the laboratory’s Chemical Hygiene Plan (CHP: DEQ04-LAB-0025-SFTY), which covers the

safety procedures necessary for the work performed at the laboratory. Documentation that personnel have read the CHP shall be kept in the LQAO's administrative file.

6.3.2 Environmental Controls

Where applicable test method SOPs shall specify requirements for controlling the environment of the laboratory. The laboratory shall monitor, control and record environmental conditions as required by the relevant specifications, methods and procedures or where the environment influences the quality of the result e.g., the weighing of Air Quality particulate samples requires a temperature and humidity controlled environment and virtually all samples are stored in a refrigerator. The temperature and humidity of the Air Quality room is monitored and documented, this process is documented in the Air particulate weighing procedure. Although it may not be documented in a particular procedure, the temperature of the refrigerator is maintained and documented because sample storage temperature is known to influence the quality of numerous test methods.

7 Quality Assurance

The laboratory shall provide a system to ensure all events influencing data quality are documented.

7.1 *Control of Quality Documents*

The laboratory must maintain control of all documents that form the laboratory's Quality System to ensure personnel use and have access to the most recent versions of quality documents and SOPs. The laboratory's document control procedure (DEQ02-LAB-0004-SOP) can be found on Q-Net and in the agency's QMP. This procedure describes the process the laboratory shall use to develop, write, approve, and store controlled documents. The approval process shall ensure QA objectives are met and that the laboratory's Quality Assurance Team (QAT) has input.

The author, technical editor, and QAO shall sign controlled documents of manuals, policies, and procedures. The page header of controlled quality documents shall contain the document title, control number, the version number, the effective date, and page numbers. Controlled documents are identified by the signatures and colored title page. The QAO shall ensure the controlled copy is stored in a locked file cabinet. Photocopies of the signed document and re-printed electronic documents are not controlled. It is the responsibility of the document holder to ensure he/she has the most current document. The QAO shall ensure electronic copies of controlled documents are available on Q-Net and notify the appropriate personnel when revisions are posted.

Section managers shall maintain database documentation identifying personnel who are required to keep current with specific controlled documents. Section managers shall submit to the LQAO records of attestations that employees will follow the policy and procedures described in controlled documents.

7.2 *Analytical Records*

As a public agency the laboratory must also follow state policies for maintaining records.

> *It is the policy of the State of Oregon to assure the preservation of records essential to meet the needs of the state, its political subdivisions, and its citizens, and to assure the prompt destruction of records without continuing value.*

All instrument logs, sample preparation logs, standard/reagent logs, bench work sheets, and data output records from electronic instruments used to generate analytical data must be retained for at least five years. To ensure the laboratory meets this requirement all chemists and field technicians must read and sign memos of attestation for document and record control procedures.

The document control coordinator shall assign control numbers to logs and notebooks prior to use. The document control coordinator shall create control numbers in a database and enter pertinent data describing the content, version, author, and date of the document or records log. Once the logs and/or notebooks are full, the chemists and technicians must return the records to the document control coordinator for archiving. Analytical records shall be archived for a period of five years since the last entry in the log. Refer to the agency's Archiving procedures, for storage and retrieval of archived records.

7.3 *Data Reports and Verification Records*

Technical Services (TS) is responsible for maintaining the integrity of analytical data generated by the laboratory division. The Technical Services section manages analytical data through the use of LIMS. As data progresses through the system from entry to review to reporting, LIMS

adjusts the status code of the result, which restricts what can be done to the data. Chemists or technicians enter data into LIMS and may make corrections to data up until they submit their work for review. Senior chemists review data and may send the data back to the chemist for rework. LIMS users can not alter data during the review process. Once the results are reported and approved for all tests performed on a set of samples, Technical Services shall print an analytical report. The DEQ laboratory shall release the analytical report after the QA Officer and Laboratory Administrator review and sign the document. The release process is completed when Technical Services sends a copy of the report (either electronically or paper) to the primary recipient. Up until that point the report may be edited without an erratum. However, after the report is sent to the primary recipient it must not be altered. Subsequent changes in an analytical report must be made through an erratum. Should an error in the report be detected following the release of the report an erratum must be prepared (refer to DEQ03-LAB-0002-SOP for this procedure).

As with other controlled documents the colored title page and the signatures on the analytical report identifies the report as the official controlled copy. Reprinted electronic reports and photocopies of the analytical report are not controlled. Technical Services shall ensure that PDF copies of the controlled report are available through electronic means. Data users may receive PDF copies of the report through e-mail or retrieve them out of Q-Net. Although the PDF is not the official copy, Technical Services shall ensure the electronic copy of a report is equally maintained. Technical Services shall store electronic copies of the original reports and subsequent errata in a secure server location. The process of maintaining an official controlled document on paper and a secure electronic copy offers a backup system for system failures in the controlled document procedure.

During data review, lead chemists/technicians, senior chemists/technicians, managers, QAO, and the Division Administrator may question the validity of data and initiate an audit. Technical Services is responsible for tracking the audit request, findings, and corrective action. The auditor shall identify what prompted the question in a memo addressed to personnel involved in the corrective action and the QAO. The title of the memo shall contain the sampling event number.

Documentation of this audit must be retained per NELAC 2002 5.4.12.2.4 d, and 5.4.12.2.5.f. Although data may be altered during specific steps of the review process all changes are tracked through the LIMS audit trail.

7.4 *Client Correspondences*

As noted earlier the Director has placed commitment to **Delivering Excellence in Performance and Product** in the agency's Strategic Directions. Responding to laboratory client needs is a top priority for achieving this goal. The laboratory's primary clients are the agency's programs, who in turn have clients of their own. Ultimately it is the public who is our client.

The laboratory shall cooperate with agency personnel in an attempt to clarify work requests and to monitor the laboratory's performance in relation to the work performed. Agency personnel should contact the laboratory's Technical Services manager for information on projects and for retrieving data. The public has access to data stored in the Laboratory Analytical Storage and Retrieval (LASAR) database. All requests from the public for laboratory data should be directed to the Technical Services manager, who shall follow agency policy and procedures for assisting the public in retrieving such data. Not all data shall be available immediately to the public, such as data collected for criminal enforcement or determined confidential for national security. The Technical Services section shall write an analytical report SOP, which shall ensure client confidentiality in these cases. This SOP shall follow the laboratory's document control policy.

7.4.1 Analytical Work Submitted to the Laboratory

Quality Assurance Project Plans

The process of developing QAPPs as prescribed in the agency's QMP enables the laboratory to review work prior to receiving samples, and to support the laboratory's ability to produce a client-defined product. During the development of the QAPP project coordinators shall involve the QAO, who shall ensure the QAPP addresses laboratory requirements as well as the client's interests. The QAPP must meet the QMP policy and structure requirements, which is modeled after EPA's R5 QAPP procedure. The R5 procedure is available on EPA's web page. Personnel developing a QAPP are encouraged to review these procedures as well as the agency's QMP.

The QAOs shall approve all agency QAPPs and ensure that QAPPs include:

- test method requirements:
  1) The QAO shall ensure test methods to be performed are well documented. Agency personnel who are writing QAPPs should review the current test method SOPs , which are posted on Q-Net.
  2) Special projects may require deviations from test method SOPs or new SOPs. The QAPP shall describe test methods in full detail if there is no SOP available. The QAO shall discuss test method proposals with the appropriate section manager and personnel ensuring, the laboratory has the resources and capabilities to make such adjustments. The laboratory shall validate the new procedure as described in section 3.3.1 of this document.
  3) If the DEQ laboratory opts not to develop a special test method, it may subcontract the work to other laboratories with the appropriate capabilities. Subcontracting agreements shall be covered in the QAPP.
- reporting levels required for the project,
- contaminant action levels to be used for decision making,
- the projects accreditation requirements and the laboratory's current status,
  1) The QAO shall track the accreditation status of subcontracted laboratories. If appropriate the QAO shall qualify data and ensure the DEQ analytical report includes the appropriate comments.
  2) The QAPP shall specify if and how analytical data should be flagged when there are changes in accreditation status.
- QC measures to be taken,
- procedures to control nonconforming work, i.e. deviations from the QAPP,
  1) Refer to section C1 of "EPA Requirements for Quality Assurance Project Plans EPA QA/R-5".
  2) The Nonconformance procedure in the QAPP shall describe the process for finding deficiencies in meeting QAPP requirement.
     a) The Nonconformance procedure shall identify management responsibility for taking action, such as halting work until a corrective action plan is determined, and identifying the authority responsible for resuming work.
     b) The QAO and project coordinators shall review QAPPs during the project and they shall have the capacity to make amendments to the QAPP.
     c) If deficiencies are found in DEQ laboratory work, the Nonconformance Investigations and Corrective Action procedure (section 7.5.3) shall be used to document and find solutions for the Nonconformance work.
- procedures for amending the QAPP
  1) The QAO and project coordinator shall review and approve amendments.

7.4.2 Complaints

It is not in the scope of the LQM to address all viable complaints that come to the laboratory. The LQM shall focus on issues that relate to data quality. The laboratory shall respond to inquiries of data anomalies and complaints of report format or content, response time, and laboratory policy using the Nonconformance Investigations and Corrective Action procedures described in section 7.5.3. Complaints that are related to personnel conduct, and agency policy are handled by laboratory management and the DEQ Human Resources Division.

Agency personnel tend to contact the sample tracker, whereas the receptionist usually receives calls from the public. The receptionist and the sample tracker shall attempt to direct the caller to the appropriate employee; however complaints are often difficult to decipher and will be referred to the Technical Services manager or the laboratory Administrator as the default. All laboratory staff members shall ensure the complaints they receive are discussed with the appropriate section manager. If an employee feels uncomfortable with bringing a complaint to his/her supervisor he/she may contact a QAO, the Technical Services section manager, or the Laboratory Division Administrator.

Laboratory personnel should not attempt to resolve complaints without informing management. Section managers shall assess whether the root causes of the complaint puts the integrity of laboratory work into question. If the section manager determines there is a QA problem, the manager shall ensure the Internal Audit procedures below are initiated immediately.

A complaint does not necessarily mean nonconforming work has occurred; however, complaints must be investigated to determine whether or not an error has occurred. The laboratory must also control records of complaints received. Thus, whenever a complaint is received, the section manager shall file a Nonconformance report with the Technical Services section. The Technical Services section manager shall assign a Nonconformance investigation to unbiased personnel who will determine the validity of the complaint and assess whether the event that generated the complaint violated laboratory policy or failed to follow procedures. The QAO team shall participate in the investigation when necessary to guarantee an unbiased evaluation. Technical Services shall track complaints received by recording pertinent information about the complaint such as: description, date, staff assigned to investigate, action taken, and date resolved.

7.5 *Quality Assurance Measures*

The LQAO shall maintain procedures for identification, collection, indexing, access, filing, storage, maintenance and disposal of quality and technical records. Quality records shall include reports from internal audits and management reviews as well as records of corrective and preventative actions. Records may be kept on either paper or electronic media.

7.5.1 Internal Audit

The LQAO shall establish an internal audit schedule, and at least annually, conduct internal audits of the laboratory's activities to verify operations continue to comply with the LQM. The internal audit program shall address all elements of the LQM, including test method activities. It is the responsibility of the LQAO to plan and organize audits as required by the schedule and requested by management. The QAO team shall audit the technical operations of the laboratory, since the QAOs are independent of this laboratory function. Management shall assign other staff members to audit QA activities, which may include members of the QAT. Personnel shall not audit their own activities except when insufficient resources are available and the QAO team is satisfied the individuals involved can carry out an effective audit.

When audit findings cast doubt on the laboratory's operational effectiveness or on the correctness or validity of the laboratory's work, the laboratory shall take timely corrective action. The LQAO shall retain documentation that the project coordinator was notified in writing of findings showing the laboratory results may have been affected.

The audit team shall use the Oregon Laboratory Accreditation Program (ORELAP) database to record assessment findings, from which an Audit report will be printed and submitted to the section managers. The section manager shall ensure that a corrective action plan for his/her section will be written within 30 days of receiving the report. The LQAO and section manager shall negotiate reasonable time frames for completion of corrective action procedures. The LQAO shall record the scheduled completion date of the corrective action procedure in the database. The LQAO will then monitor and routinely report on the status of the corrective action. Subsequent internal audits shall verify and record the implementation and effectiveness of the corrective action taken.

The LQAO shall review NELAC required administrative files. These files should contain documentation of training requirements, training received, Demonstration of Capability, and personnel conduct with respect to compliance to the laboratory data integrity policy. Discovery of potential personnel issues shall be handled in a confidential manner until such time as a follow up evaluation, full investigation including union representation if requested, or other appropriate actions have been completed and the issues clarified. All investigations that result in finding of inappropriate activity shall be documented and shall include corrective actions taken and all appropriate notifications to clients. Any disciplinary actions taken shall be documented and stored in the Human Resources Division's personnel files which are not accessible to the general public. All documentation of these investigations and actions taken shall be maintained for at least five years.

7.5.2 Annual QA Report

The LQAO shall schedule and prepare an annual Laboratory Quality Assurance report for the Laboratory Division Administrator to review. The report shall cover the status of the laboratory's quality system and the laboratory's analytical activities to ensure the continuing suitability and effectiveness of the system, and to introduce necessary changes or improvements. The report will be completed by 31 January and shall take account of QA activities and concerns that occurred during the previous calendar year, including:

1. the suitability of policies and procedures;
2. reports from managerial and supervisory personnel;
3. the outcome of recent internal audits;
4. corrective and preventative actions;
5. assessments by external bodies;
6. the results of interlaboratory comparisons or proficiency tests;
7. changes in the volume and type of work;
8. client feedback;
9. complaints;
10. other relevant factors, such as quality control activities, resources and staff training.

The Division Administrator shall comment on the Annual QA Report and if necessary set a corrective action schedule and assign responsibility. The LQAO shall maintain a file of the Annual QA Reports, the Division Administrator's responses and applicable corrective action plans. The LQAO shall also ensure all section managers receive a copy of this file. The corrective action assigned by the Division Administrator shall be monitored as described in the Corrective Action procedure below (section 7.5.3).

7.5.3 Nonconformance Investigations and Corrective Action Reports

Whenever personnel suspect or identify deviations from the policies or procedures within the LQM, monitoring activities, analytical test method SOPs, or failed QA/QC, they must initiate a Nonconformance investigation. The procedure for documenting Nonconformance events and the subsequent corrective action shall encompass all measures taken to rectify QA/QC problems, which include but are not limited to:

- documentation or evidence of the presumed problem;
- identification of the SOPs not conforming to the LQM;
- a description of how the SOP was not being followed as written; and
- impediments to following the SOP.

Nonconformance Investigations shall be documented using the Nonconformance report form (or equivalent) in Appendix C to record the description of the incident, personnel involved in the investigation, deadlines, Corrective Action (CA) taken, and the dates for completing each event. Section managers may develop their own Nonconformance form for reporting failed QC, which shall be approved by the QAT prior to use. The Technical Services section shall develop a system to capture this information and track the status of these investigations. Technical Services shall also maintain these records for at least five years.

This corrective action procedure is referenced throughout this document and shall be used for all incidences where non-routine work occurs and alternative documentation strategies have not been identified. Although external audits may require a specific format for the corrective action plan, the laboratory shall track the actions taken using the Nonconformance report form or until an alternate electronic Nonconformance information system is developed and implemented.

7.5.3.1 Cause Analysis

Cause Analysis is the initial step in any corrective action that is initiated in response to a Nonconformance Investigation. The Cause Analysis shall start with an investigation to determine the root cause(s) of the problem. Personnel who initiated the Nonconformance Investigation may offer opinions on the possible the root cause. Even if the root cause can not be determined, the investigation and the Nonconformance report must be completed.

Personnel shall begin the process by filling in the applicable fields of the Nonconformance form and forwarding it on to their supervisor or lead worker, who will then evaluate the problem and assign the investigation to appropriate staff. The investigation shall begin with employees whom appear to be most directly related to the Nonconformance.

If the manager determines the Nonconformance is not related to his/her section, he/she shall forward the report onto the appropriate manager. The section manager shall immediately bring deviations from the LQM or the QAPP to the attention of the QAO team, who shall conduct these Nonconformance investigations. The QAO shall ensure the project coordinator is involved with the corrective action plan. Generally, Nonconformance Investigations will be handled by the appropriate laboratory sections:

- Technical Services section shall investigate and perform corrective actions that relate to LIMS functions, sample receipt, sample integrity, and complaints;
- the analytical sections of the laboratory shall conduct the investigation and complete the necessary corrective actions for deviations from analytical test methods and failed QC measures within their sections; and,
- the monitory sections shall investigate nonconformance events related to sample collection, continuous monitoring, and other field operations;

Cause analysis should be conducted through data review and QA audits. The data review process may reveal a Nonconformance which is of a technical nature and a suite of corrective action procedures to follow (refer to Technical Corrective Action section 7.5.3.5).

Section managers may delegate the responsibility of reviewing data and assigning corrective action tasks to whom ever they choose in their staff. Since it is the managers' prerogative to assign investigations and corrective action tasks as they see fit, each section shall write procedures for Data Control, which will describe who is responsible for implementing corrective action procedures for data quality problems.

7.5.3.2 Corrective Action Selection and Implementation

One the root of the problem has been identified through Cause Analysis, potential solutions shall be proposed an implemented through a Corrective Action Selection and Implementation process. Corrective actions shall be appropriate to the magnitude and risk of the problem. The section manager shall collaborate with the LQAO to identify potential corrective action procedures and to select and implement action(s) most likely to eliminate the problem and to prevent recurrence. The section manager shall record these corrective action options on the Nonconformance report.

Once it has been identified, the appropriate corrective action must be implemented. The first step in implementation will often be the revision or amendment of the relevant Laboratory policy or procedure.

The QAT shall document and ensure implementation of any required changes in the LQM or laboratory policy resulting from corrective action investigations. Section managers shall approve changes in analytical data and in SOPs with QAO concurrence.

7.5.3.3 Monitoring of Corrective Actions

The LQAO or his/her designee shall monitor the effectiveness of corrective actions taken. The Nonconformance report system shall provide sufficient information to assess the effectiveness of corrective action procedures. The Nonconformance report must associated with the appropriate documents through the control numbers described in section 2.8 above. The success and performance of the implemented corrective action plans will be a routine part of the annual Internal Audit (section 7.5.1).

7.5.3.4 Additional Audits

Where the identification of Nonconformances or departures from QAPP, SOP, or LQM casts doubt on the laboratory's compliance with its own policies and procedures, the laboratory shall ensure the appropriate activities are audited in accordance with the Internal Audit procedures as soon as possible.

7.5.3.5 Technical Corrective Action and Data Review Process

Corrective action taken in the course of day to day validation of data may require less of a formal cause analysis than the corrective action taken for the response to an internal audit.

Test method SOPs shall provide procedures for correcting problems which may occur during the analytical process. Moreover, to help avoid inadvertent departures from quality policies and procedures analytical data shall be reviewed at several levels.

1) Chemists/Technicians producing analytical data shall review their work ensuring sample and batch QC measures meet acceptable limits. Chemists shall follow their SOPs to correct any failed QC and repeat the analysis provided there is sufficient sample. If the analysis can not be repeated within the recommended sample holding time, the chemist

must attach a comment to the result in LIMS and report the value as an estimate or void the result. Typically chemists shall void results only due to analytical failures and not to sample or batch QC limit failures. However, the failure to meet multiple QC limits may lead to the action of voiding the result.

a) Sample QC measures may include but are not limited to:
   i) Matrix Spike
   ii) Matrix Spike Duplicate
   iii) Duplicate analysis
b) Batch QC measures may include but are not limited to:
   i) Method Blank
   ii) Laboratory Control Sample
   iii) Continuing Calibration Verification
   iv) Calibration coefficients

As noted previously, if a chemist believes he/she has identified a questionable procedure or feels his/her SOP does not meet the policy or procedures detailed in the LQM, he/she is obligated to bring his/her concerns forward. He/she may contact a QAO, a QAT member, his/her section supervisor, or the Laboratory Administrator.

2) Senior chemist/Lead Chemist/Lead Monitoring Specialists

   a) The lead or senior chemist/technician shall validate data by reviewing sample history, comparing intra-sample contaminants, and investigating data anomalies. The lead chemists shall confer with chemists to evaluate any question he/she may have about the data and may send the results back to the chemist for rework. As above this process is controlled through the DAR approval process. The chemist shall verify the transcription of data from the original source to LIMS, and if necessary and possible, retest the sample.

3) The section manager shall review the decision process, which the chemist/technician used to qualify the data and either approve the outcome or send the results back for rework. The procedure for reworking results through LIMS is described in the DAR SOP. This procedure will ensure NELAC 2002 5.4.12 Control of Records and 5.4.13 Internal Audits are followed.

4) The QAO shall review analytical data packets for completeness and sign analytical reports certifying the controlled document is in compliance with the laboratory's quality policy. The QAO shall re-work analytical reports that do not comply with these policies. If a quality control measure fails, all samples associated with the failed quality control measure shall be reported with the appropriate data qualifier(s).

   In addition to verifying precision and accuracy of analytical data, QC procedures described in test method SOPs shall document the process for obtaining QC data. An independent QAO shall review and sign all controlled documents, for which he/she is not the author to help ensure that QC collection is documented. During review of the controlled documents, problems may arise that warrant the creation of a Nonconformance report. The QAO should solicit an investigation from personnel not related to the issues.

7.5.4 Preventative Measures

Preventative measures are a pro-active process to identify opportunities for improvement rather than a reaction to problems or complaints. The agency's QMP addresses how the Quality Systems shall continue to grow and improve (chapter 10 of the QMP); the laboratory shall

comply with this goal. It is the QAT's responsibility to assess and implement the laboratory's preventative measures (refer to section 2.1 Laboratory Quality Assurance Team).

8 Reporting Results

The laboratory shall report data in useful and comparable formats.

The Technical Services section of the laboratory has the responsibility of creating analytical reports. Technical Services shall ensure that all projects entered into LIMS and assigned a sampling event number shall be reported in a standard format, creating an official report record. The report shall accurately, clearly, unambiguously, and objectively depict the results for requested measurements. The intent of the report design is to minimize the possibility of misunderstanding or misuse of data.

Technical Services shall ensure the integrity of the official report by applying the document control procedures as described in section 7.3 (Data Reports and Verification Records). Even though project coordinators may only use data stored in an electronic database, Technical Services shall create an official paper report for document control.

Analytical reports shall be generated from LIMS and converted to a PDF, which shall be printed on a color printer. Each page of the report shall have the title of the report, the sampling event name and number prominently displayed at the top of the report. The report footer shall contain the name of the PDF file, date and time the report was printed, and the page number with the total number of pages in the PDF. The analytical report shall contain the following sections:

- Title page w/
  1) Primary Report Recipient,
  2) Report Date,
  3) Laboratory Demographics, and
  4) Approval Signatures.
- Narrative page w/
  1) Sampling Event narrative when present,
  2) List of Report Recipients,
  3) List of Sample Collectors, and
  4) List of Analytical Laboratories involved in the analyses.
- Sampling Event Summary page w/
  1) Project ID linking the Sampling Event to the QAPP,
  2) List of Sampling Sites w/
     a) Item number,
     b) Site ID,
     c) Sample Description,
     d) Matrix,
     e) Sample Date,
     f) Sample Time, and
     g) Endnote references
- Analytical Results w/
  1) Sample identification w/
     a) Item Number,
     b) Site ID,
     c) Sample Description,
     d) Sample Date, and
     e) Sample Time
  2) Analytical Parameter w/
     a) Method reference,
     b) Lower Reporting Limit,
     c) Result,

d) Unit,
e) Date and Time of sample preparation for test methods with holding time less than 72 hours, and
f) Endnote references

- Endnotes
- Electronic scanned sample collection form

The Technical Services section shall attach the following original forms and reports to the official analytical report, although they may not be included in the electronic data packet.

- The original sample collection form
- QC reports
- Subcontracted analytical reports

Project coordinators may make special requests to scan documents which could then be attached to the electronic document.

Technical Services shall transcribe pertinent data to LIMS from the sample collection form and subcontracted analytical reports, which shall appear in the official report as well.

The analyst entering data into LIMS shall report results associated with failed QC as estimates and enter a comment explaining his/her decision to qualify data as an estimate. Through the Data Analysis Report (DAR) approval process management shall scrutinize the data and identify which comments will be reported. LIMS will generate endnotes from the approved comments and insert them in the appropriate section of the report.

The Technical Services section shall develop a system for tracking and reporting references to sampling methods. As noted previously Watershed Assessment and Air Quality Monitoring have documented procedures for collecting samples. References to these procedures shall be included in analytical reports where appropriate.

The QAO shall complete the sampling event narrative portion of the analytical report. This section shall be used to clarify QC measures and to offer opinions or interpretations of the data by the QAO.

Project coordinators shall identify in the QAPP or SAP the report recipients and how they will receive the analytical report. Typically agency personnel will receive an e-mail notice with a link to the PDF. Reports may be emailed as a PDF, or photocopied and mailed. LIMS allows the sample tracker to modify the list of report recipients at the time of sample entry. Staff may add or delete personnel to and from the list of people receiving the analytical report. The project coordinator shall be notified of changes to the recipient list. The laboratory shall only send analytical reports to personnel listed as recipients. Laboratory personnel shall instruct the public to request copies of an analytical report from the project coordinator, thus ensuring the project coordinator is made aware of the potential use of their data.

APPENDICES

Appendix A: Demonstration of Capability Certification Statement

Date: _____ Page ____ of ____

Laboratory Name: Oregon Department of Environmental Quality
Laboratory Address: 1712 SW 11th Avenue, Portland OR 97201

Analyst(s) Name(s): _____

Matrix: _____
(Examples: laboratory pure water, soil, air, solid, biological tissue)

Method: _____
Method number, SOP#, Rev#, and Analyte, or Class of Analytes or Measured Parameters
(Examples: barium by 200.7, trace metals by 6010, benzene by 8021, etc.)

We, the undersigned, CERTIFY that:

1. The analysts identified above, using the cited test method(s), which is in use at this facility for the analyses of samples under the National Environmental Laboratory Accreditation Program, have met the Demonstration of Capability.

2. The test method(s) was performed by the analyst(s) identified on this certification.

3. A copy of the test method(s) and the laboratory-specific SOPs are available for all personnel on-site.

4. The data associated with the demonstration capability are true, accurate, complete and self-explanatory (1).

5. All raw data (including a copy of this certification form) necessary to reconstruct and validate these analyses have been retained at the facility, and that the associated information is well organized and available for review by authorized assessors.

Technical Director's Name and Title	Signature	Date
Quality Assurance Officer's Name	Signature	Date

Appendix B: 40 CFR Part 136, App. B

Environmental Protection Agency

[49 FR 43261, Oct. 26, 1984; 50 FR 692, 695, Jan. 4, 1985, as amended at 51 FR 23702, June 30, 1986]

DEFINITION AND PROCEDURE FOR THE DETERMINATION OF THE METHOD DETECTION LIMIT - REVISION 1.11

Definition

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

Scope and Application

This procedure is designed for applicability to a wide variety of sample types ranging from reagent (blank) water containing analyte to wastewater containing analyte. The MDL for an analytical procedure may vary as a function of sample type. The procedure requires a complete, specific, and well defined analytical method. It is essential that all sample processing steps of the analytical method be included in the determination of the method detection limit.

The MDL obtained by this procedure is used to judge the significance of a single measurement of a future sample.

The MDL procedure was designed for applicability to a broad variety of physical and chemical methods. To accomplish this, the procedure was made device- or instrument- independent.

Procedure

1) Make an estimate of the detection limit using one of the following:
   a) The concentration value that corresponds to an instrument signal/noise in the range of 2.5 to 5.
   b) The concentration equivalent of three times the standard deviation of replicate instrumental measurements of the analyte in reagent water.
   c) That region of the standard curve where there is a significant change in sensitivity, i.e., a break in the slope of the standard curve.
   d) Instrumental limitations.

It is recognized that the experience of the analyst is important to this process. However, the analyst must include the above considerations in the initial estimate of the detection limit.

2) Prepare reagent (blank) water that is as free of analyte as possible. Reagent or interference free water is defined as a water sample in which analyte and interferant concentrations are not detected at the method detection limit of each analyte of interest. Interferences are defined as systematic errors in the measured analytical signal of an established procedure caused by the presence of interfering species (interferant). The interferant concentration is pre-supposed to be normally distributed in representative samples of a given matrix.

3)
   a) If the MDL is to be determined in reagent (blank) water, prepare a laboratory standard (analyte in reagent water) at a concentration which is at least equal to or in the same concentration range as the estimated method detection limit. (Recommend between 1 and 5 times the estimated method detection limit.) Proceed to Step 4.
   b) If the MDL is to be determined in another sample matrix, analyze the sample. If the measured level of the analyte is in the recommended range of one to five times the estimated detection limit, proceed to Step 4.

If the measured level of analyte is less than the estimated detection limit, add a known amount of analyte to bring the level of analyte between one and five times the estimated detection limit.

If the measured level of analyte is greater than five times the estimated detection limit, there are two options.

i) Obtain another sample with a lower level of analyte in the same matrix if possible.

ii) The sample may be used as is for determining the method detection limit if the analyte level does not exceed 10 times the MDL of the analyte in reagent water. The variance of the analytical method changes as the analyte concentration increases from the MDL, hence the MDL determined under these circumstances may not truly reflect method variance at lower analyte concentrations.

4)

a) Take a minimum of seven aliquots of the sample to be used to calculate the method detection limit and process each through the entire analytical method. Make all computations according to the defined method with final results in the method reporting units. If a blank measurement is required to calculate the measured level of analyte, obtain a separate blank measurement for each sample aliquot analyzed. The average blank measurement is subtracted from the respective sample measurements.

b) It may be economically and technically desirable to evaluate the estimated method detection limit before proceeding with 4a. This will: (1) Prevent repeating this entire procedure when the costs of analyses are high and (2) insure that the procedure is being conducted at the correct concentration. It is quite possible that an inflated MDL will be calculated from data obtained at many times the real MDL even though the level of analyte is less than five times the calculated method detection limit. To insure that the estimate of the method detection limit is a good estimate, it is necessary to determine that a lower concentration of analyte will not result in a significantly lower method detection limit. Take two aliquots of the sample to be used to calculate the method detection limit and process each through the entire method, including blank measurements as described above in 4a. Evaluate these data:

i) If these measurements indicate the sample is in desirable range for determination of the MDL, take five additional aliquots and proceed. Use all seven measurements for calculation of the MDL.

ii) If these measurements indicate the sample is not in correct range, re-estimate the MDL, obtain new sample as in 3 and repeat either 4a or 4b.

5) Calculate the variance (S^2) standard deviation (S) of the replicate measurements as follows:

[ILLUSTRATION GOES HERE]

ER31AU93.074

$$S^2 = \frac{\sum_{i=1}^{n} X_i^2 - \left(\sum_{i=1}^{n} X_i \right)^2 / n}{n-1}$$

$$S = (S^2)^{1/2}$$

where: X_i for i = 1 to n, are the analytical results in the final method reporting units obtained from the n sample aliquots and Σ refers to the sum of the X values from i = 1 to n.

6)

a) Compute the MDL as follows:

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

where:

MDL = the method detection limit

$t_{(n-1,\ 1-\alpha = 0.99)}$ = the students' t value appropriate for a 99% confidence level and a standard deviation estimate with n-1 degrees of freedom. See 1.

S = standard deviation of the replicate analyses.

b) The 95% confidence interval estimates for the MDL derived in 6a are computed according to the following equations derived from percentiles of the chi square over degrees of freedom distribution (χ^2/df).

LCL = 0.64 MDL

UCL = 2.20 MDL

where:

LCL and UCL are the lower and upper 95% confidence limits respectively based on seven aliquots.

7) Optional iterative procedure to verify the reasonableness of the estimate of the MDL and subsequent MDL determinations.

a) If this is the initial attempt to compute MDL based on the estimate of MDL formulated in Step 1, take the MDL as calculated in Step 6, spike the matrix at this calculated MDL and proceed through the procedure starting with Step 4.

b) If this is the second or later iteration of the MDL calculation, use S^2 from the current MDL calculation and S^2 from the previous MDL calculation to compute the F-ratio. The F-ratio is calculated by substituting the larger S^2 into the numerator S^2A and the other into the denominator S^2B. The computed F-ratio is then compared with the F-ratio found in the table which is 3.05 as follows: if $S^2A/S^2B < 3.05$, then compute the pooled standard deviation by the following equation:

[ILLUSTRATION GOES HERE]

ER31AU93.075

$$S_{pooled} = \left[\frac{6\,S_A^2 + 6\,S_B^2}{12}\right]^{1/2}$$ 1

if $S^2_A/S^2_B > 3.05$, re-spike at the most recent calculated MDL and process the samples through the procedure starting with Step 4. If the most recent calculated MDL does not permit qualitative identification when samples are spiked at that level, report the MDL as a concentration between the current and previous MDL which permits qualitative identification.

c) Use the Spooled as calculated in 7b to compute the final MDL according to the following equation:

$MDL = 2.681\ (S_{pooled})$

where 2.681 is equal to $t_{(12,\ 1-\alpha = .99)}$

d) The 95% confidence limits for MDL derived in 7c are computed according to the following equations derived from percentiles of the chi squared over degrees of freedom distribution.

LCL = 0.72 MDL

UCL = 1.65 MDL

where LCL and UCL are the lower and upper 95% confidence limits respectively based on 14 aliquots.

TABLES OF STUDENTS' t VALUES AT THE 99 PERCENT CONFIDENCE LEVEL

Students' t: Table I.

Number of replicates	Degrees of freedom (n-1)	$t_{(n-1,\ 1-\alpha\ =\ 0.99)}$
7	6	3.143
8	7	2.998
9	8	2.896
10	9	2.821
11	10	2.764
16	15	2.602
21	20	2.528
26	25	2.485
31	30	2.457
61	60	2.390
∞	∞	2.326

Reporting

The analytical method used must be specifically identified by number or title and the MDL for each analyte expressed in the appropriate method reporting units. If the analytical method permits options which affect the method detection limit, these conditions must be specified with the MDL value. The sample matrix used to determine the MDL must also be identified with MDL value. Report the mean analyte level with the MDL and indicate if the MDL procedure was iterated. If a laboratory standard or a sample that contained a known amount analyte was used for this determination, also report the mean recovery.

If the level of analyte in the sample was below the determined MDL or exceeds 10 times the MDL of the analyte in reagent water, do not report a value for the MDL.

Appendix C: Nonconformance Report

ID number: _____

Nonconformance recorded by: _____ Date: _____

Description: _____

_____

_____

(Attach separate sheets if needed)

If applicable complaint's name & phone number: _____

Data impacted: _____

Sampling Event(s) Item(s) Container(s) Test(s)/Contaminant

Root cause: _____

_____

_____

Possible Corrective Action (CA): _____

_____

_____

Assigned to: _____ Target Date: _____

CA taken: _____

_____

Management review: _____ Date: _____

Quality Assurance Officer's audit: _____ Date: _____

Subsequent or follow up CA refer to: _____

Non-conformance report ID number

Technical Corrective Action

SECTION: _____

WA, AQM, Inorganic, or Organic/General Chem.

To		From		Date	
Case Number		Case Name			

The review process has revealed the possible problems noted below. Please check bottle numbers, calculations, dilution factors, and data entry for errors. Rerun samples if necessary to resolve the problem(s). If needed, reanalyze another aliquot from another sample container to check for contamination, container or site mix-ups, etc. Complete this report and return it when finished. **This rework is a priority task.**

		New Result		Data Correction	
Item Number		Date		Bottle Number	Test Result
Site Name					
Bottle Number					
Alternate Bottle					
Standard Parameter		Initial Result Verified?		Yes	No
Initial Test Result		Reason Code for Change			
Expected Test Result		Comments:			
Problem Code					

		New Result		Data Correction	
Item Number		Date		Bottle Number	Test Result
Site Name					
Bottle Number					
Alternate Bottle Number					
Standard Parameter		Initial Result Verified?		Yes	No
Initial Test Result		Reason Code for Change			
Expected Test Result		Comments:			
Problem Code					

		New Result		Data Correction	
Item Number		Date		Bottle Number	Test Result
Site Name					
Bottle Number					
Alternate Bottle Number					
Standard Parameter		Initial Result Verified?		Yes	No
Initial Test Result		Reason Code for Change			
Expected Test Result		Comments:			
Problem Code					

Problem Codes
A – Field Duplicate Difference
B – Disagreement with history
C – Disagreement with other tests
D – Irregular Field Blank
E – Disagreement with Ion Balance
F – Other (describe)

Change Codes
a – Data transfer
b – Data transfer error
c – Dilution or correction factor error
d – Bottle Number mix-up
e – Other (describe)

FIELD/LAB QUALITY CONTROL CHECK

Event Number				**Event Name**					
Item Number				**Date Sampled**					
Field Data				**Laboratory Data**					
Test	Meter ID	Result	Units	Bottle Number	Test Date	Result	Units	Confirms Field Data	
pH			SU				SU	Yes	No
Alkalinity			mg/L				mg/L	Yes	No
Turbidity			NTU				NTU	Yes	No
Conductivity			µmhos/cm				µmhos/cm	Yes	No
Comments:									

Item Number				**Date Sampled**					
Field Data				**Laboratory Data**					
Test	Meter ID	Result	Units	Bottle Number	Test Date	Result	Units	Confirms Field Data	
pH			SU				SU	Yes	No
Alkalinity			mg/L				mg/L	Yes	No
Turbidity			NTU				NTU	Yes	No
Conductivity			µmhos/cm				µmhos/cm	Yes	No
Comments:									

Item Number				**Date Sampled**					
Field Data				**Laboratory Data**					
Test	Meter ID	Result	Units	Bottle Number	Test Date	Result	Units	Confirms Field Data	
pH			SU				SU	Yes	No
Alkalinity			mg/L				mg/L	Yes	No
Turbidity			NTU				NTU	Yes	No
Conductivity			µmhos/cm				µmhos/cm	Yes	No
Comments:									

Item Number				**Date Sampled**					
Field Data				**Laboratory Data**					
Test	Meter ID	Result	Units	Bottle Number	Test Date	Result	Units	Confirms Field Data	
pH			SU				SU	Yes	No
Alkalinity			mg/L				mg/L	Yes	No
Turbidity			NTU				NTU	Yes	No
Conductivity			µmhos/cm				µmhos/cm	Yes	No
Comments:									

Corrective Action: Analyze a confirmation sample if the Laboratory Alkalinity, Conductivity, or Turbidity disagrees with the Field results by more than 20% RPD or if the pH value disagrees with the Field results by more than +/-0.5 units. When one or both samples are < 5 times the MRL compare the results using +/- 1 MRL for the average. If one or both of the Turbidity samples are < 10 NTU, then compare the results using +/- 1 MRL for the average + 1 NTU. Another sample container may be required to check for the possibility of site/sample confusion of container handling or cleaning problems. **If the discrepancy is verified, have the analysis assigned and run all the samples for the entire sampling event.**

The Relative Percent Difference (RPD) is calculated using the following equation:

$$RPD = \frac{X_s - X_d}{[X_s + X_d]/2} \times 100\%$$

Where: X_s = field result and
X_d = lab result
The units for X_s must equal those of X_d

Example 1: Given two duplicate results of 18 and 23:

$$\text{RPD} = \frac{23 - 18}{[18 + 23]/2} \times 100\% = \frac{5}{20.5} \times 100\% = 24\%.$$

Therefore, the Quality Control Check would fail. The Laboratory RPD results are over the expectable RPD of 20%.

Example 2: Given two duplicate results of 5 and 7:

The Quality Control Check calculation = (5 + 7)/2 = 6. Therefore, the Acceptance window is 6 ± 1 MRL. Therefore, the Quality Control Check would pass. The Laboratory results are within the calculated range of 5 to 7.

Example 3 (for Turbidity): Given two duplicate results of 5 and 9, the Quality Control Check calculation = (5 + 9)/2 = 7. The acceptance window is 7 ± 2. Therefore, the Quality Control Check would pass. The Laboratory results are within the calculated range 5 to 9.

Appendix D: LIMS status codes

LIMS table: XLU STATUS	
STATUS	**STATUS DESCRIPTION**
A+	Data of known Quality. Presented by DEQ meeting current QC limits as established by the Laboratory's Quality Systems Manual.
A	Data of known Quality. Submitted by entities outside of DEQ meeting current QC limits for external data as established by the DEQ Laboratory.
B	Data of suspect Quality. Data may not meet established QC but is within marginal acceptance criteria or data value may be accurate, however controls used to measure Data Quality Objective elements failed i.e. batch failed to meet blank QC limit.
C	Data of unacceptable Quality. Values are discarded (Void) typically due to analytical failure.
D	No sample collected or no reportable results, typically due to sampling failure.
E	Data of unknown quality. No QA information is available, data could be valid however there is no evidence to prove either way (Educational Only, Very Questionable/Poor QA/QC).
F	Exceptional Event. "A" Quality data but not representative of sampling conditions as required by the project plan.

Appendix E: Major Analytical Equipment

Organic Analytical Section

- ***GC/MS/DS:***
  - Allocated to semi-volatile analyses a HP 5989 Mass Spectrometer Engine with HP 6890 series II GC and HP data system.
  - Allocated to volatile analyses a HP 5972 Mass Spectrometer Detector with HP 6890 GC and HP data system. Capillary/packed column GC with HP Purge Trap Concentrator and a Varian Archon vial autosampler.
  - Allocated for Air toxics HP 5971A Mass Spectrometer Detector with 5890 series II GC and HP data system. Capillary/packed column.
- ***GCs:***
  - HP 6890 series II Plus, FID & NPD, HP 3365 Chemstation data system.
  - Varian 6890 series II Plus; Capillary/packed column, PID and FID dedicated for SUMMA canister analysis.
  - HP 5890 series II; TCD & OI PID/FID; HP 3365 Chemstation data system. With OI 4560 Purge & Trap and 4551 vial autosampler.
  - HP 5890 series II; two ECDs; HP 3365 Chemstation data system.
  - HP 6890 series II; two ECDs.
- ***HPLC:***
  - Agilent Series 1100 equipped with HP Diode Array UV/Vis and Programmable Fluorescence Detectors with an HP Chemstation.
- ***Chromatography Data Stations:***
  - HP 1405 Timeserver and LAN hardware
  - Agilent 1100 LC/MSD Chemstation A.09.03
  - Agilent Environmental Chemstation G1701DA
- ***Total Organic Carbon (TOC) Analyzer:***
  - Tekmar/Dohrman Apollo 9000
- ***COD Reactors:***
  - 3 x HACH COD Reactors.
- ***Total Organic Halogen (TOX) Analyzer:***
  - Mitsubishi TOX-10Σ, Aimed AutoAOX autosampler.
- ***Spectrophotometers:***
  - HACH DR/3000
- ***Fluorometer:***
  - Turner Designs TD-700
- ***Analytical Balances:***
  - Mettler PM4600 Delta Range semi-micro
  - 2 x Mettler AT261 semi-micro

Inorganic Analytical Section

- ***Atomic Absorption Spectrophotometer (AAS):***
  - Varian AA-400. GTA-96 Graphite tube atomizer.
  - Varian AA-400. Zeeman Graphite tube atomizer.
  - CETAC M6000 Mercury Analyzer.

- ***Microwave Digestor***
  - CEM MDS-2100
- ***Autoanalyzer:***
  - Lachat Flow Injection Ion Analyzer: Nitrate + Nitrite, Nitrite, Chloride, TK-N, and Sulfate modules.
  - Alpchem: Fluoride & Ammonia.
- ***Ion Chromatographs:***
  - Dionex Model 120, autosampler and Peaknet software
  - Dionex 600 upgraded with CDM detector, gradient pump, SRS suppresser, AS40 autosampler, and Peaknet software.
- ***X-Ray Fluorescence Spectrometer:***
  - Fisons KEVEX EDX771
- ***Amperometric Titrator:***
  - Wallace & Tiernan Titrator
- ***Spectrophotometers:***
  - Bausch & Lomb: Spectronic 21
  - Perkin Elmer Model Lambda 20 Spectrophotometer
  - Hach DR/700
- ***Inductively Coupled Plasma (ICP):***
  - Simultaneous Perkin Elmer Optima 3000 DV
- ***Inductively Coupled Plasma Mass Spectrometry (ICP/MS):***
  - Thermo Elemental Model VG PQ Excel
- ***Analytical Balances:***
  - Sartorius A200S
  - Mettler H15
  - H6 w/filter weighing chamber
  - Mettler H15, H18
  - Sartorius 1712MP8
  - Ohaus Balance, E4000D
- ***Microbalances:***
  - Cahn C30 - Range 1 μg to 3gm
  - ATI Cahn C-44
- ***Optical Microscope:***
  - Zeiss Standard 18, Polarizing Trinocular with 35-mm camera
  - Spencer Binocular stereo scope
  - Olympus CH2 Phase Contrast microscope

Appendix F: QC definitions

QC Class	QC	LIMS	Frequency	Description/Corrective Action
Analytical	Instrument Blank	IB	1/analytcial batch	Instrument Blank: a clean sample (e.g., distilled water) processed through the instrumental steps of the measurement process; used to determine instrument contamination. (EPA-QAD) QC sample is measured prior to batch analysis. If QC fails to meet assigned control limits and the problem can not be corrected all reported results within the batch shall be flagged with the appropriate data qualifier.
Analytical	Internal Standard	IS	100% per SOP	Internal Standard: pure analyte(s) added to a sample, extract, or standard solution in known amounts and used to measure the relative responses of other method analytes that are components of the same sample or solution. The internal standard must be an analyte that is not a sample component. (EPA Method 200.8) The relative response of this QC element is used to adjust reported results of other analytes. Statistical analysis of the relative response may prove useful for evaluating maintenance schedules.
Analytical	Reagent Blank	RB	1/ preparation batch	Reagent Blank: (method reagent blank): a sample consisting of reagent(s), without the target analyte or sample matrix, introduced into the analytical procedure at the appropriate point and carried through all subsequent steps to determine the contribution of the reagents and of the involved analytical steps. (QAMS) QC sample is measured prior to batch analysis. If QC fails to meet assigned control limits and the problem can not be corrected all reported results within the batch shall be flagged with the appropriate data qualifier.
Calibration	Continuing Calibration Blank	CCB	per SOP	Continuing Calibration Blank: Reanalysis of the calibration blank or equivalent matrix repeated through the analytical batch to establish instrumental bias, drift, and/or carry-over. (DEQ) QC sample is measured during the batch analysis. If QC fails to meet assigned control limits and the problem can not be corrected all reported results bracketed between the RB and CCB or between CCBs shall be flagged with the appropriate data qualifier.
Calibration	Continuing Calibration Verification	CCV	beginning and end of analytical batch	Continuing Calibration Verification: Reanalysis of the initial calibration standards during the course of a calibration batch used to demonstrate continued instrument performance. At a minimum, a CCV must be repeated at the beginning and end of each analytical batch. The concentration of the CCV shall be varied within the established calibration range. However, if an internal standard is used, only one CCV must be analyzed per analytical batch. (DEQ) If QC fails to meet assigned control limits and the problem can not be corrected, all reported results bracketed between the ICV & CCV or between CCV's shall be flagged with the appropriate data qualifier.

QC Class	QC	LIMS	Frequency	Description/Corrective Action
Calibration	Quality Control Sample (QCS) or Initial Calibration Verification	ICV	Immediately following initial calibration	Quality Control Sample: an uncontaminated sample matrix spiked with known amounts of analytes from a source independent from the calibration standards. It is generally used to establish intra-laboratory or analyst specific precision and bias or to assess the performance of all or a portion of the measurement system. (EPA-QAD) When used as the Initial Calibration Verification (ICV), the QCS is used to verify the initial instrument calibration and should be the first sample analyzed in the analytical sequence. (NELAC/DEQ) If the QC fails to meet control limits and the problem can not be corrected all reported results within the analytical batch shall be flagged with the appropriate data qualifier.
Field	Automated Precision	AP	1/ quarter	Changed from Field Control Standard: Used in air sampling. The operator generates a known concentration of the target analyte and analyzes it through the equipment. Similar to a CCV sample, but used to assess field operations. (DEQ) If QC fails to meet assigned control limits and the problem can not be corrected, all reported results bracketed between the ICV & CCV or between CCV's shall be flagged with the appropriate data qualifier.
Field	Equipment Blank	EB	per sampling expedition or per QAPP	Equipment Blank: a sample of analyte-free media which has been used to rinse common sampling equipment to check effectiveness of decontamination procedures. (NELAC) If blank fails QC limits estimate target limits for the sampling event.
Field	Field Audit	FA	per QAPP	Field audit: verification of field measured parameters through the use of a secondary method. (DEQ)
Field	Field Duplicate	FD	10% samples collected during a sampling expedition	Field Duplicate: discrete samples taken from the same field location and processed and analyzed independently by the laboratory. The original sample is identified by space and time. The field duplicate is collected at the same location and within a reasonable lapse of time. (DEQ)
Field	Laboratory Retained Blank	LRB	per SOP	Changed from Lab Stored Blank. Laboratory Retained Blank: a sample of analyte-free matrix that remains in the laboratory and is used as comparison with the blanks carried to the field. (DEQ)
Field	Manual Precision	MP	10% sample sites	Changed from Co-located Sampler. Used in air sampling. A secondary sample collected from a location. Similar to a Field Duplicate. Multiple sampling devices run simultaneously within close proximity. (DEQ)

QC Class	QC	LIMS	Frequency	Description/Corrective Action
Field	Sample	S	100%	Sample identified by space and time. Space is defined by decimal latitude carried to five decimal places and by longitude carried to five decimal places and elevation used in a vertically integrated sample. Time is defined by the time zone using a 24 hour clock to the nearest minute. Start and stop time must be recorded for composite samples.
Field	Transfer Blank	TSFB	per QAPP	Transfer Blank: a sample of analyte-free matrix which has been carried to the field and transferred to a sample bottle in the field. (DEQ)
Field	Transport Blank	TNPB	per QAPP	Transport Blank: a sample of analyte-free media which has been carried to the field and returned to the laboratory. (DEQ)
Operations	Automated Accuracy	AA		Used in air sampling. Generally a gas from a secondary source analyzed on-site by a secondary auditor. Very similar to a 2nd source QC in many respects. (DEQ)
Operations	Blind Sample	BLND		Blind Sample: a sub-sample for analysis with a composition known to the submitter. The analyst/laboratory may know the identity of the sample but not its composition. It is used to test the analyst's or laboratory's proficiency in the execution of the measurement process. (NELAC)
Operations	Certified Reference Material	CRM		Certified Reference Material (CRM): a reference material one or more of whose property values are certified by a technically valid procedure, accompanied by or traceable to a certificate or other documentation which is issued by a certifying body. (ISO Guide 30 - 2.2)
Operations	Inter-Lab Split Sample	SPLT		Changed from Split: Samples split with an external laboratory. Laboratory audit or Split sample: verification of field and/or laboratory performance through the collection and analysis of field duplicate samples by an alternate laboratory. (DEQ)
Operations	Manual Accuracy	MA		Used in air sampling. A secondary auditor collects audit samples on equipment using equipment with known properties, essentially the collection of an audit sample. (DEQ)
Operations	Proficiency Test Sample	PT		Proficiency Test Sample (PT): a sample, the composition of which is unknown to the analyst and is provided to test whether the analyst/laboratory can produce analytical results within specified acceptance criteria. (QAMS)
Operations	Reference Material	RM		Reference Material: a material or substance one or more properties of which are sufficiently well established to be used for the calibration of an apparatus, the assessment of a measurement method, or for assigning values to materials. (ISO Guide 30-2.1)

QC Class	QC	LIMS	Frequency	Description/Corrective Action
Operations	Reference Standard	RS	per QAPP	Reference Standard: a standard, generally of the highest metrological quality available at a given location, from which measurements made at that location are derived. (VIM-6.08)
Preparation	Dilution	DR	as required	Dilution: additional measurement made from a diluted sample aliquot. Used with the undiluted sample or other dilutions to establish analytical precision, evaluate matrix interferences, and/or bring the analyte concentration to within the instrument's calibration range. (DEQ)
Preparation	Laboratory Confirmation	LCON	per SOP	Confirmation: verification of the identity of a component through the use of an approach with a different scientific principle from the original method. These may include, but are not limited to: Second column confirmation Alternate wavelength Derivatization Mass spectral interpretation Alternative detectors or Additional cleanup procedures. (NELAC)"
Preparation	Laboratory Control Sample	LCS	1/ analytical batch	Laboratory Control Sample (also known as laboratory fortified blank, spiked blank, or QC check sample): a sample matrix, free from the analytes of interest, spiked with verified known amounts of analytes or a material containing known and verified amounts of analytes. It is generally used to establish intra-laboratory or analyst specific precision and bias or to assess the performance of all or a portion of the measurement system. (NELAC) The LCS requirements for analytical QC may be satisfied through the use of a Quality Control Sample (QCS).
Preparation	Laboratory Control Sample Duplicate	LCSD		Laboratory Control Sample Duplicate: a sample matrix, free from the analytes of interest, spiked with verified known amounts of analytes or a material containing known and verified amounts of analytes. It is used with the LCS to establish intra-laboratory or analyst specific precision and bias when more traditional methods are unavailable. (DEQ)
Preparation	Laboratory Duplicate	LD	10% / preparation batch	Changed from Analytical Replicate. Laboratory Duplicate: aliquots of a sample taken from the same container under laboratory conditions and processed and analyzed independently. (NELAC)
Preparation	Matrix Spike	MS		Matrix Spike (spiked sample or fortified sample): a sample prepared by adding a known mass of target analyte to a specified amount of matrix sample for which an independent estimate of Target analyte concentration is available. Matrix spikes are used, for example, to determine the effect of the matrix on a method's recovery efficiency. (QAMS)

QC Class	QC	LIMS	Frequency	Description/Corrective Action
Preparation	Matrix Spike Duplicate	MSD		Matrix Spike Duplicate (spiked sample or fortified sample duplicate): a second replicate matrix spike prepared in the laboratory and analyzed to obtain a measure of the precision of the recovery for each analyte. (QAMS)
Preparation	Method Blank	MB	1/ preparation batch	Method Blank: a sample of a matrix similar to the batch of associated samples (when available) that is free from the analytes of interest and is processed simultaneously with and under the same conditions as samples through all steps of the analytical procedures, and in which no target analytes or interferences are present at concentrations that impact the analytical results for sample analyses. (NELAC)
Preparation	Standard Reference Material	SRM	per QAPP	Standardized Reference Material (SRM): a certified reference material produced by the U.S. National Institute of Standards and Technology or other equivalent organization and characterized for absolute content, independent of analytical method. (EPA-QAD)
Preparation	Surrogate	SS	100% per SOP	Surrogate: a substance with properties that mimic the analyte of interest. It is unlikely to be found in environment samples and is added to them for quality control purposes. (QAMS)

Appendix G: Analytical Methods

ANALYTE	Standard Methods[i, ii, iii]	EPA[, iv]	EPA[v, vi, vii]	SW-846[viii]	TECHNIQUE
METALS					
Aluminum	***3120 B***	**200.7**	*200.7*	6010	ICP
Antimony	***3113 B***	**204.2**	*200.9*	7041	AA FURNACE
Antimony	**3120 B**	**200.7**		6010	ICP
Arsenic	***3113 B***	**206.2**	*200.9*	7060	AA FURNACE
Arsenic	***3114 B,4.d***	**206.3**		7061	AA HYDRIDE
Arsenic	***3120 B***	**200.7**	*200.7*	6010	ICP
Barium	***3120 B***	**200.7**	*200.7*	6010	ICP
Beryllium	***3113 B***	**210.2**	*200.9*	7091	AA FURNACE
Beryllium	***3120 B***	**200.7**	*200.7*	6010	ICP
Boron	**3120 B**	**200.7**		6010	ICP
Cadmium	***3113 B***	**213.2**	*200.9*	7131	AA FURNACE
Cadmium	**3120 B**	**200.7**	*200.7*	6010	ICP
Calcium	***3120 B***	**200.7**	*200.7*	6010	ICP
Chromium	***3113 B***	**218.2**	*200.9*	7191	AA FURNACE
Chromium	***3120 B***	**200.7**	*200.7*	6010	ICP
Chromium^{+6}		218.5			COPRECIPITATION
Cobalt	**3113 B**	**219.2**		7201	AA FURNACE
Cobalt	**3120 B**	**200.7**		6010	ICP
Copper	***3120 B***	**200.7**	*200.7*	6010	ICP
Iron	***3120 B***	**200.7**	*200.7*	6010	ICP
Lanthanum		200.7		6010	ICP

ANALYTE	Standard Methods[i, ii, iii]	EPA[i, iv]	EPA[v, vi, vii]	SW-846[viii]	TECHNIQUE
Lead	***3113 B***	**239.2**	*200.9*	7421	AA FURNACE
Lead	**3120 B**	**200.7**		6010	ICP
Lithium	3120 B	200.7		6010	ICP
Magnesium	**3120 B**	**200.7**	*200.7*	6010	ICP
Manganese	***3120 B***	**200.7**	*200.7*	6010	ICP
Mercury	***3112 B***	**245.1**	*245.1*	6010	Manual Cold Vapor
Molybdenum	**3120 B**	**200.7**		6010	ICP
Nickel	***3120 B***	**200.7**	*200.7*	6010	ICP
Potassium	**3120 B**	**200.7**		6010	ICP
Selenium	***3113 B***	**270.2**	*200.9*	7740	AA FURNACE
Selenium	*3114 B*	**270.3**		7741	AA HYDRIDE
Selenium	**3120 B**	**200.7**		6010	ICP
Silica	***3120 B***	**200.7**	*200.7*	6010	ICP
Silver	***3113 B***	**272.2**	*200.9*		AA FURNACE
Silver	***3120 B***	**200.7**	*200.7*		ICP
Sodium	**3120 B**	**200.7**	*200.7*	6010	ICP
Strontium	3120 B	200.7		6010	ICP
Thallium		**279.2**	*200.9*	7841	AA FURNACE
Thallium	**3120 B**	**200.7**		6010	ICP
Tin		**200.7**		6010	ICP
Titanium				6010	ICP
Tungsten				6010	ICP
Vanadium	**3120 B**	**200.7**		6010	ICP

ANALYTE	Standard Methods[i, ii, iii]	EPA[i, iv]	EPA[v, vi, vii]	SW-846[viii]	TECHNIQUE
Zinc	***3120 B***	**200.7**	*200.7*	6010	ICP
Zirconium				6010	ICP
NON METALS					
BOD_5	**5210 B**				Winkler-Azide Modification
$CBOD_5$	**5210 B**				Winkler-Azide Mod./Inhib
Dissolved Oxygen	**4500-O C**	**360.2**			Winkler-Azide Modification
Dissolved Oxygen	**4500-O G**	**360.1**			Membrane Electrode
Bromide	4110 B	300			Ion Chromatography
Chloride	**4500-Cl^- C**	**325.1**			Auto Ferricyanide
Chloride	***4110 B***		*300.0*		Ion Chromatography
Chlorine	**4500-Cl D**	**330.1**			Amperometric Titration
Chlorine	**4500-Cl G**	**330.5**			DPD Colorimetric
Fluoride	***4500-F E***	**340.3**			Auto Complexone
Ammonia-Nitrogen	**4500-NH_3 H**	**350.1**			Auto Phenate
Total Kjeldahl Nitrogen		**351.2**			S-Auto Block Digestion
Nitrate-Nitrogen	***4500-NO_3 F***	**353.2**			Auto Cadmium Reduction
Nitrite-Nitrogen	***4500-NO_3 F***	**353.2**			Auto colorimetric
Nitrite-Nitrogen	***4110 B***		*300.0*		Ion Chromatography
Ortho Phosphate-P	***4500-P E***	**365.2**			Colorimetric Ascorbic Acid
Ortho Phosphate-P	***4110 B***		*300.0*		Ion Chromatography
Total Phosphate-P	**4500-P E,5**	**365.2**			Colorimetric Ascorbic Acid
Sulfate	*4500-SO_4^2 F*	375.2	*375.2*		Auto Methylthymol Blue

ANALYTE	Standard Methods[i, ii, iii]	EPA[, iv]	EPA[v, vi, vii]	SW-846[viii]	TECHNIQUE
Sulfate	4110 B	300.0	*300.0*		Ion Chromatography
PHYSICAL PARAMETERS					
Acidity	**2310 B**	**305.1**			Titration
Alkalinity	***2320 B***	**310.1**			Titration
Color	***2120 C***	**110.3**			Colorimetric Pt/Co
Conductivity	***2510 B***	**120.1**			Wheatstone Bridge
Flash Point				1010	Pensky-Martens closed-cup
pH	***4500-H$^+$***	**150.1**	*150.1*		Electrometric
Filterable (TDS)	***2540 C***	**160.1**			Gravimetric, 180^{o}C
Nonfilterable (TSS)	**2540 D**	**160.2**			Gravimetric, 103-105^{o}C
Total (TS)	**2540 B**	**160.3**			Gravimetric, 103-105^{o}C
Volatile		**160.4**			Gravimetric, Ignition @ 550^{o}C
Temperature	**2550 B**	**170.1**			
Turbidity	**2130 B**	**180.1**			Nephelometric
ORGANICS					
Adipates		**625**	*525.2*	8270	GC/MS
BTEX		**602**		5020A	GC/PID
Chlorinated Pest/PCB		**608**	*508*	8081	Solvent Extr., GC/ECD
PCB as DCBP			*508A*		Solvent Extr., GC/ECD
Chlorinated Herbicides			*515.1*	8150A	Solvent Extr., GC/MS
EDB, DBCP			*504.1*		GC/ECD

ANALYTE	Standard Methods[i, ii, iii]	EPA[i, iv]	EPA[v, vi, vii]	SW-846[viii]	TECHNIQUE
Herbicides			*515.1*	8150A	Solv, Ext., derivation, GC/MS
Hydrocarbon ID (HCID)				8015 modified	GC/FID
Pentachlorophenol		**625**	*525.2*	8270B	GC/MS
PAHs		**610**	*525.2*	8270, 8310	Solv, Extr., GC/MS
Phthalates		**625**	*525.2*	8270	Extr., GC/MS
PPOE,BNA		**625**	*525.2*	8270	Extr., GC/MS
Trihalomethanes		**624**	*524.2*	8260	P&T, GC/MS
VOCs/VOAs		**624**	*524.2*	8260	P&T, GC/MS
N-P Pesticides				8141A	GC/NPD
Miscellaneous					
Chemical Oxygen Demand	**5220 D**	**410.4**			Colorimetric (Chromate)
Chlorophyll *a*/Pheophytin *a*	**10200-H**	**445**			Fluorometric
Cyanide, Total/Amenable	***4500-CN E***	**352.2**		9010	Colorimetric
MBAS (Surfactants)	**5540 C**	**425.1**			Methylene Blue Active Substance
NW-TPH: Gas					
NW-TPH: Diesel					
NW-TPH: Oil					
Gas					
Diesel					
Oil					
Oil and Grease		1664[ix]			Gravimetric (Hexane)
Particulate Fall Out (PFO)					Gravimetric
Percent Fat[x]					Hexane Extraction/Gravimetric

ANALYTE	Standard Methods[i, ii, iii]	EPA[, iv]	EPA[v, vi, vii]	SW-846[viii]	TECHNIQUE
Phenolics		**420.1**		9066	Colorimetric (Pyrine)
Sediment Sizing[xi]					Gravimetric
Total Organic Halides (TOX or AOX)				1650	
Total Organic Carbon	**5310 B**	**415.1**			Persulfate Oxidation/NDIR Detection
Total Organic Carbon (Soil)[xii]					Walkley/Black Titration

[i] Approved Waste Water Methods promulgated in 40 CFR Part 136 appear in **"Bold"**.

[ii] Approved Drinking Water Methods promulgated in 40 CFR Part 141 appear in *"Italic"*.

[iii] **Standard Methods for the Examination of Water and Wastewater, 18th Edition, APHA, AWWA, WPCF, 1994.**

[iv] **Methods for Chemical Analysis of Water and Wastes, EPA-600/4-79-020, Revised 3/83.**

[v] 300.0: **Methods for the Determination of Inorganic Substances in Environmental Samples, EPA-600/R-93-100, August 1993.**

[vi] 200.7 & 200.9: **Methods for the Determination of Metals in Environmental Samples - Supplement I, EPA-600/R-94-111, May 1994.**

[vii] Five hundred series: **Methods for the Determination of Organic Compounds in Drinking Water, EPA-600/4-88-039, December 1988. Methods for the Determination of Organic Compounds in Drinking Water - Supplement I, EPA-600/4-90-020, July 1990. Methods for the Determination of Organic Compounds in Drinking Water - Supplement II, EPA-600/R-92-129, August 1992.**

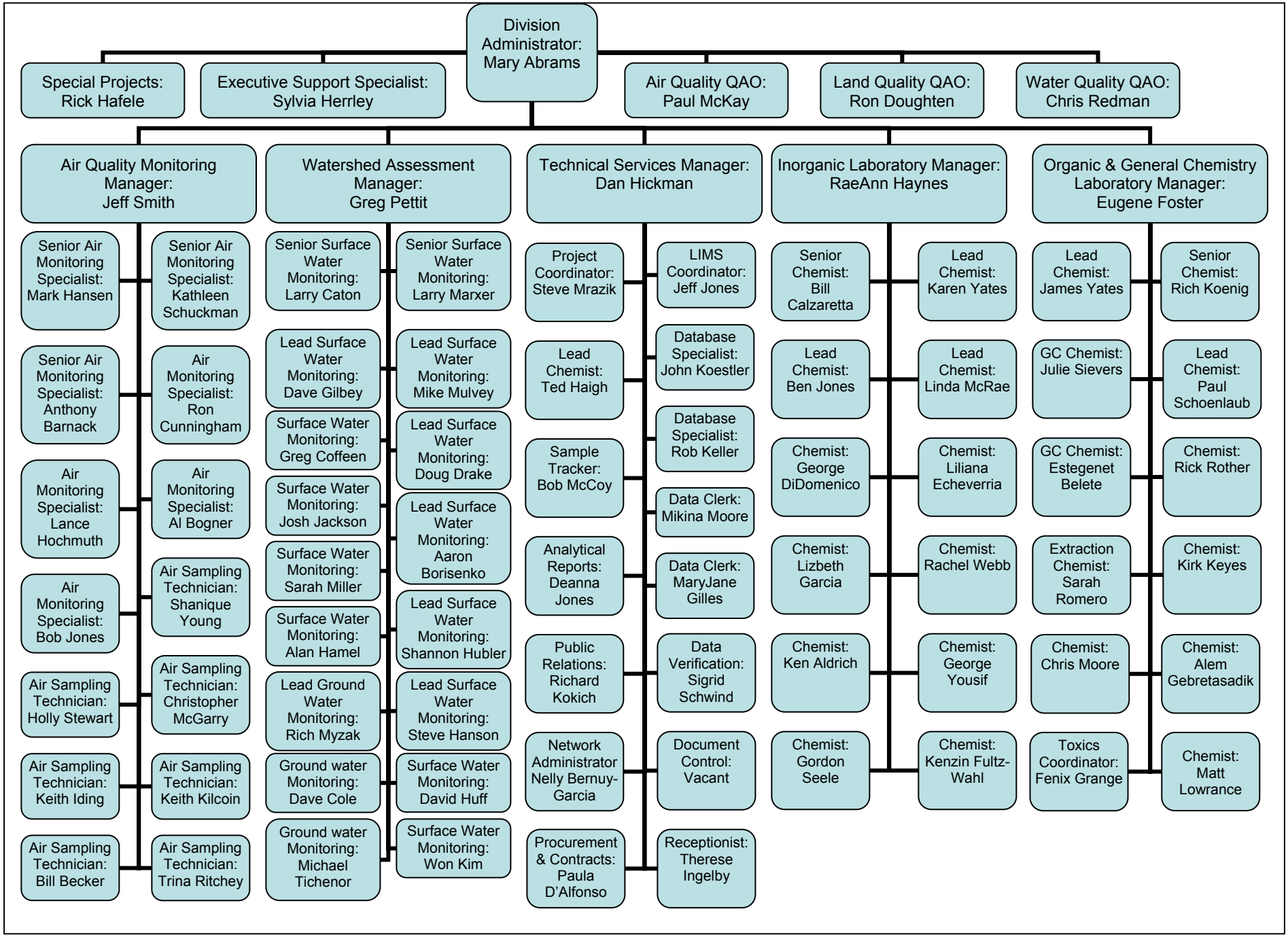
[viii] **Test Methods for Evaluating Solid Waste: SW-846, Third Edition, USEPA, November 1986.**

[ix] Chuck Clarke, Regional Administrator for EPA, Region 10 has sent a letter of approval for an alternate test procedure. EPA will recognize results from Method 1664, Hexane Extractables for all Oil and Grease analyses performed on samples that originate in Region 10.

[x] DEQ Method Revised 2/3/98.

[xi] DEQ Method developed 3/12/1987

[xii] DEQ Method Developed 8/29/1989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Lead Chemist: Linda McRae
Chemist: George DiDomenico
Chemist: Liliana Echeverria
Chemist: Lizbeth Garcia
Chemist: Rachel Webb
Chemist: Ken Aldrich
Chemist: George Yousif
Chemist: Gordon Seele
Chemist: Kenzin Fultz-Wahl
Lead Chemist: James Yates
Senior Chemist: Rich Koenig
GC Chemist: Julie Sievers
Lead Chemist: Paul Schoenlaub
GC Chemist: Estegenet Belete
Chemist: Rick Rother
Extraction Chemist: Sarah Romero
Chemist: Kirk Keyes
Chemist: Chris Moore
Chemist: Alem Gebretasadik
Toxics Coordinator: Fenix Grange
Chemist: Matt Lowrance