Laboratory and Environemantal Assessment Division

Quality Manual

State of Oregon
Department of
Environmental
Quality

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DEQ is a leader in restoring, maintaining and enhancing the quality of Oregon's air, land and water.

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Scott Hoatson, Agency Quality Assurance Officer (and Land Quality)	Date
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1 INTRODUCTION AND SCOPE (V1M2: 1.0 – 3.0):

The overall DEQ agency quality system is described in the Oregon Department of Environmental Quality (DEQ) Quality Management Plan (QMP) (**DEQ03-LAB-0006-QMP**). The plan is approved by the executive management team (EMT) for DEQ. The EMT is comprised of, among others, The director and deputy director of the agency as well as all of the division administrators, and the managers for OCE, OCO, VIP, and Government Relations. The QMP provides DEQ's overall policy for quality and is the blueprint for DEQ planning, implementation, and assessment of the quality system for the environmental work the agency performs as part of our mission. The QMP also describes the agency's commitment to use The NELAC Institute (TNI) 2009 standards to evaluate a laboratory's ability to generate quality data.

This Laboratory Quality Manual (LQM) describes the quality system (policies and procedures) by which, Laboratory and Environmental Assessment Division (LEAD) operations are performed including the organization, objectives, and operating philosophy. All personnel within the LEAD are required to follow the policies and procedures that are contained in this document. The policies and procedures in the LQM shall guide LEAD personnel in collecting, producing, maintaining, and reporting data of known quality and, where applicable, demonstrate regulatory compliance. The quality assurance officers (QAOs) are responsible for the review and revision of the LQM. LEAD personnel must keep abreast of changes in quality policy and procedures, and therefore, must read and implement the changes to the LQM with each revision. This LQM is written to meet NELAC standards and will be reviewed and revised as needed on an annual basis

The objectives of the LEAD quality system, with the commitment of management, are to consistently provide DEQ and the EPA with data of known and documented quality that meets their requirements. Our policy is to use good professional practices, to maintain quality, to provide the highest quality of service, and to comply with the TNI Standard. The LEAD management ensures that personnel are free from any commercial, financial, and other undue pressures, which might adversely affect the quality of work. This policy is implemented and enforced through the commitment of management, at all levels, to the quality assurance (QA) principles and practices outlined in this manual. However, the primary responsibility for quality rests with each individual within the laboratory organization. Every LEAD employee must ensure that the generation and reporting of quality analytical data is a fundamental priority. Every laboratory employee is required to familiarize themselves with the quality documentation and to implement the policies and procedures in their work. All employees are trained annually on ethical principles and procedures surrounding the data that is generated. The laboratory maintains a strict policy of client confidentiality where applicable, however since DEQ is a state agency, almost all records are available to the public.

The objectives of the LQM are to describe this quality system, to document the LEAD quality policies and procedures, and to provide a tool for ensuring personnel are both knowledgeable of and committed to these policies and procedures. The LQM as a whole, is designed to ensure that all events affecting the quality of data are known and properly documented. All laboratory personnel must read this document and sign an attestation memo **(DEQ06-LAB-0016-FORM** or **Appendix B)** that they shall implement the policies and procedures contained in the LQM in their work practices. These policies and procedures ensure:

- laboratory personnel have appropriate training and supervision (**Section 2** *Organizational Roles and Responsibilities*);
- the implementation of proper procedures for sample collection, preservation, storage, record keeping, analysis, and reporting (**Section 4.1** *Standard Operating Procedures*);
- where applicable data is traceable to acceptable reference standards (**Section 20** *Measurement Traceability*);

- the degree of precision, accuracy, and bias of the analyses is known and documented (**Section 18** *Test Methods and Method Validation*, and **Section 22** *Quality of Test Results*)
- that analytical equipment is properly used, calibrated, and maintained (**Section 19** *Equipment and Calibrations*);
- and data is reported in useful and comparable formats (**Section 23** *Reporting of Results*).

1.1 SCOPE OF TESTING

This LQM applies to the list of tests that are contained in (**Appendix G**)

1.2 GLOSSARY

Quality control terms are generally defined within the section that describes the activity. Additionally, **Appendix A** Glossary provides definitions for terms used in the LQM. The is also a glossary of quality related terms that can be found on DEQ's intranet (Q-Net) at the following link http://deq05/lab/QA/labQSdefns.asp

1.3 ACRONYMS

A list of acronyms and abbreviations used in this document and their definitions are

A2LA	The American Association for Laboratory Accreditation
AQM	Air Quality Monitoring
AR	Analytical Reagent grade
CCB	Continuing Calibration Blank
CCV	Continuing Calibration Verification
CA	Corrective Action
CAA	Clean Air Act
CDOC	**Continuing Demonstration of Capability**
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
DU	Laboratory Duplicate Sample
EMT	DEQ's Executive Management Team
EQC	Oregon's Environmental Quality Commission
EPA	Environmental Protection Agency
FD	Field Duplicate sample
FIFRA	Federal Insecticide, Fungicide and Rodenticide Act
IDOC	**Initial Demonstration of Capability**
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
LOD	Limit of Detection
LOQ	Limit of Quantitation
MB	Method Blank
MDL	Method Detection Limit (the preferred terminology is to use LOD)
MRL	Method Reporting Limit (the preferred terminology is to use LOQ)
MS	Matrix Spike
MSD	Matrix Spike Duplicate
NELAP	National Environmental Laboratory Accreditation Program
NIST	National Institute of Standards and Technology
OCE	Office of Compliance and Enforcement
OCO	Office of Communications and Outreach
ORELAP	Oregon Environmental Laboratory Accreditation Program
PCS	Procurement & Contract Specialist
PDF	Portable Document Format
PDR	Property Disposition Request
PT	Proficiency Testing
QA	Quality Assurance
QAO	Quality Assurance Officer
QAPP	Quality Assurance Project Plan
QC	Quality Control
QCS	Quality Control Sample
QMP	Quality Management Plan
RSD	Relative Standard Deviation
RCRA	Resource Conservation and Recovery Act
SAP	Sampling and Analysis Plan
SDWA	Safe Drinking Water Act
SOP	Standard Operating Procedure
TNI	The NELAC Institute
VIP	Vehicle Inspection Program
WQM	Water Quality Monitoring

2 ORGANIZATIONAL ROLES AND RESPONSIBILITIES (V1M2: 4.0 -4.2)

Through application of the policies and procedures outlined in this chapter, the laboratory assures that it is impartial and that personnel are free from undue commercial, financial, or other undue pressures that might influence their technical judgment. The laboratory is responsible for carrying out testing activities that meet the requirements of the TNI Standard and that meet the needs of DEQ and the EPA.

2.1 DEQ'S LEAD'S QUALITY POLICY (V1M2: 4.2.2)

The DEQ *Mission* "…is to be a leader in restoring, maintaining and enhancing the quality of Oregon's air, land and water. DEQ works collaboratively with Oregonians for a healthy, sustainable environment." Along with the mission, we at DEQ all have a ***Shared Vision*** with one of the major components: "*We base our work on good science"*. As part of our investment in environmental excellence, the EMT identified its ***Core Processes*** to better define the Agency's priority work. The DEQ LEAD plays an important role in this mission and is the foundation for the core operational processes (*Assessing Environmental Conditions*), in that LEAD provides the other operational processes with the information they need to make sound decisions.

Simply put, in order to deliver good science, the DEQ LEAD has a policy that all of our activities shall result in products and decisions of known and acceptable quality. The LEAD's policy on quality and the quality system design reflects the same strategic direction of the agency as a whole. It is intended to provide a path to 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. Thus, data used by the agency shall be of known accuracy, precision, completeness, representativeness, comparability, 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.

2.2 LABORATORY QUALITY ASSURANCE TEAM

The core laboratory QA Team is comprised of the 3 quality assurance officers (QAOs) and the laboratory QA chemists. Other laboratory section managers are brought in on specific issues that may affect their areas. The mission of the QA team is to:

- Ensure that the LEAD will produce data of known quality
- Identify QA/QC needs for the laboratory
- Prioritize QA/QC projects
- Review and recommend approval of QA documents
- Develop QA/QC Policy
- Request internal audits and review audit reports.

2.3 MANAGEMENT AND STAFF RESPONSIBILITIES:

As a key action element described in the agency's ***Shared Vision*** and ***Core Process Map*** the agency recognizes the importance of motivated DEQ employees to deliver excellence in their work. DEQ LEAD management carries a commitment to meet the requirements of the TNI standard and continually improve the effectiveness of the management system. 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 integrity is of utmost importance for producing data of known quality.

Laboratory staff shall perform their duties with the intention to meet the policies and procedures in this Quality Manual. 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 QAO in the event they feel 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. Personnel may contact any QAO whom they feel most comfortable with or a union representative to discuss their concerns.

2.4 LEAD ORGANIZATIONAL STRUCTURE

The DEQ LEAD organization is structured to minimize the potential for conflicting or undue interests that might influence the technical judgment of analytical personnel. The LEAD has 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 has technical management who have overall responsibility for technical operations and can allocate the necessary resources to ensure the required quality of laboratory operations. The laboratory has a Laboratory Quality Assurance Officer (LQAO) who ensures TNI standards are implemented in the laboratory.

The LEAD's organizational structure and its place in the agency support 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 LEAD is organized into structural groups under the direction of the LEAD administrator. The Administration Section (including the QAOs) of the laboratory is under the direct supervision of the LEAD administrator as are the section managers for Air Quality Monitoring, Water Quality Monitoring, Inorganic Laboratory, Organic Laboratory, and Technical Services (See LEAD organizational chart Appendix I**).** 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 Water Quality Monitoring sections generate data while not physically in the laboratory; they must also comply with the policies and procedures of the LQM. The technical requirements and training documentation for each position are described in **Section 16** below. Position descriptions for all staff are maintained by the agency Human Resources Department and are available on the agency intranet site (QNet) ***HR Info Center***

2.4.1 Administration

The Administration Section of the laboratory provides support for the laboratory in the areas of human resources/accounting, special projects, and quality management.

Special projects include, assisting with laboratory operational procedures and analysis, legislative coordination for the Laboratory and Environmental Assessment Division (including grant applications and reports), and assistance to the administrator on unanticipated requests from outside stakeholders or colleagues.

An executive assistant offers administrative support to the division administrator and limited support to the other sections of the laboratory and serves as a liaison to DEQ EMT. The executive support specialist also acts as the LEAD records management coordinator.

Quality assurance oversight is delegated to three QAOs. There is a QAO for each DEQ program: Air Quality, Land Quality, and Water Quality. The QAOs report directly to the LEAD Administrator and provide technical assistance in the development and implementation of QA project plans; audits monitoring network; perform assessments of self-monitoring activities under air, NPDES, and RCRA permits; may participate in Oregon Laboratory Accreditation Program (ORELAP) activities; report to programs documenting project data quality; and ensure corrective action procedures are followed when data quality criteria are not met.

Additionally one QAO is assigned the responsibility of ensuring that TNI standards are implemented at the DEQ laboratory and is recognized in this document as the Laboratory Quality Assurance Officer (LQAO). The LQAO maintains the administrative files containing this documentation and a signature log with initials and dates. These files should not be confused with other personnel files, which are

maintained by the laboratory executive assistant and the Human Resources Division. The laboratory executive assistant 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 works with the LEAD records management coordinator to schedule the destruction of laboratory QA administrative documentation older than five years (past last use) or as otherwise specified in the DEQ record retention schedule and with prior authorization from the agency records management officer.

2.4.2 Management

For the purposes of this LQM, "management" is comprised of the Division administrator and the various section managers. Management bears specific responsibility for maintenance of the quality system and ultimately responsible for compliance with the TNI standard. This includes defining roles and responsibilities to personnel (all DEQ position classification descriptions define the minimal level of education, qualifications, experience, and skills necessary for each position), certifying position description requirements are appropriate to perform NELAP accredited analytical test methods, approving documents, providing required training, providing a procedure for confidential reporting of data integrity issues, and periodically reviewing data, procedures, and documentation.

Management ensures that audit findings and corrective actions are completed within required time frames.

Designated alternates are appointed with full signature authority by management prior to or during the absence of the Division administrator or section managers. This is done via email to all staff and the agency accounting section. The Division administrator also notifies the EMT. The QA officers are the designated back-ups for each other. In the event that no QA officers are available, a back-up will be designated via email to the LEAD staff.

The section managers ensure the technical competence of personnel operating equipment, performing tests, evaluating results, or signing reports, and limit the authority to perform laboratory functions to those appropriately trained and/or supervised.

Section managers shall submit to the LQAO documentation of personnel receiving training; and, where pertinent, certificates of demonstration of capability (DOC).

2.4.3 Air Quality Monitoring (AQM)

The Air Quality Monitoring section operates and maintains the state-wide DEQ ambient air monitoring/sampling network, managers the data from continuous monitors, calibrates air monitors/samplers, collects samples, maintains equipment, provides technical assistance, 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* (PSD) applicants.

2.4.4 Water Quality Monitoring (WQM)

The Water Quality Monitoring section serves a dual role with staff performing watershed assessment and water quality monitoring functions. Staff involved with watershed assessment collect data and samples for ambient water quality monitoring of surface water for, monitoring biological integrity and for the calculation of the load and wasteload allocations for specific pollutants in surface water. The watershed assessment staff maintain field equipment and instrumentation, provide technical assistance for the planning and collection of water, biological and sediment samples, perform audits of self-monitoring programs, collect split samples, and report on audit findings and project studies.

Staff involved with water quality monitoring collect data and samples for groundwater, pollution source monitoring (toxics and pesticides), and for groundwater monitoring at solid waste and environmental cleanup sites. The water quality monitoring staff maintain field equipment and instruments, provide

technical assistance for the collection of water, soil, and sediment samples, perform audits of self-monitoring programs, collect split samples, and report on audit findings and project studies.

2.4.5 Inorganic Laboratory

Inorganic personnel conduct analyses on air, aqueous, drinking water, saline/estuarine, biological tissue, solids, air, and chemical waste samples for inorganic constituents such as metals, nutrients, and particulate mass. They may also provide technical assistance in the preparation of QA project plans (relating to sample collection requirements and laboratory capabilities) and inthe interpretation of inorganic analytical data.

2.4.6 Organic Laboratory

Organic personnel conduct analyses on air, aqueous, drinking water, saline/estuarine, biological tissue, solids, and chemical waste samples for organic constituents and physical properties. They may also provide technical assistance in the preparation of QA project plans (relating to sample collection requirements and laboratory capabilities) and in theinterpretation of organic analytical data.

2.4.7 Technical Services

The Technical Services Section maintains the laboratory computer network, ELEMENT® (a Laboratory Information Management System), and the laboratory data stored in these systems. Technical Services also responds to customer service and data interpretation requests. 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 sample acceptance policy 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 service and supply requisitions for the laboratory; provides clerical support; controls access to the laboratory; and ensures facility maintenance is performed and facility related contracting is completed in accordance with agency policy and procedures.

3 QUALITY SYSTEMS

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

As stated in **Section 1**, the LEAD quality system is based on the agency quality management plan (QMP) and is described in this LEAD quality manual (LQM) along with associated quality system documents. Together they describe the policies, objectives, principles, organizational authority, responsibilities, accountability, and implementation plan of the organization for ensuring quality in its work processes, products, and services.

The pedigree of analytical data is dependent upon the laboratory's ability to determine the quality of the analytical result; this cannot be done without the proper documentation of all observable information. A successful QA system will produce data of known quality, not necessarily that data will be of superior quality in terms of accuracy and precision. With the knowledge of the sources of error, the laboratory may engineer systems to improve data quality.

Many factors determine the correctness and reliability of the environmental data reported by the laboratory. These factors include contributions from:

- human factors (TNI V1M2 5.2);
- accommodation and environmental conditions (TNI V1M2 5.3);
- environmental test methods and method validation (TNI V1M2 5.4);
- equipment and instrument calibrations (TNI V1M2 5.5);
- measurement traceability (TNI V1M2 5.6);
- collection of samples (TNI V1M2 5.7);

- the handling of samples (TNI V1M2 5.8).
- quality assurance of the testing (TNI V1M2 5.9)
- reporting of the results (TNI V1M2 5.10)

Because these factors contribute to the pedigree of the analytical data quality, the laboratory shall attempt to apply quality control measures for each of these factors with the intent to identify and reduce systematic errors. Each of these areas is discussed in this Quality Manual.

3.1 QUALITY SYSTEM DOCUMENTATION

The LEAD's Quality System is communicated through a variety of documents:

- DEQ agency Quality Management Plan (QMP)
- LEAD Quality Manual (LQM) - this document
- DEQ agency policies and procedures
- Quality related forms - Forms associated with a specific administrative or technical procedure (e.g., data review checklists, sample log-in checklists, etc).
- LEAD SOPs – administrative and technical
- DEQ agency or DEQ LEAD QA/QC policy memo's

3.2 QUALITY ASSURANCE OFFICERS

The QAOs have the authority and responsibility for ensuring that the quality system is implemented and followed. The QAOs along with the laboratory section managers comprise the QA Team (see **Section 2.2)**

Quality Assurance Officers:

- are the focal point for the quality system and have oversight of quality control data.
- evaluate data objectively and performs assessments without managerial influence.
- arrange for, or conduct, internal audits annually; and, notify LEAD management of deficiencies (or opportunities for continuous improvement) and monitor corrective actions. (note: field audits for air quality monitoring sites occur quarterly or semi-annually).
- coordinate the scheduling of PT studies.
- are responsible to keep the *Quality Manual* current.
- assist in the preparation of QAPPs and SAPs
- coordinate the annual quality management review

3.3 QUALITY MANUAL

- Management ensures the laboratory's policies and objectives for quality are documented by reference or by inclusion in the LEAD *Quality Manual* (LQM), and that the LQM is communicated to, understood by, and implemented by all personnel concerned. The LQM is controlled following the procedures outlined in **Section 4** below.
- Where the *Quality Manual* documents laboratory requirements, a separate SOP or policy is not required.All employees sign a form, kept with their training records that states that they have read and understood the *Quality Manual*, including the quality policy.The *Quality Manual* is maintained current and up-to-date by the LQAO.

4 CONTROL OF QUALITY DOCUMENTS (V1M2; 4.3)

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. At a minimum the laboratory will have documented procedures prescribed by TNI, which includes analytical test method SOPs and administrative 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 document control process ensures QA objectives are met and that the laboratory's Quality Assurance Team (QAT) has input. The most current electronic version of the documents are tracked, maintained and controlled by the QAOs through an access database located on a restricted server. The current documents are made accessible to all DEQ staff through **Q-Net.**

The author, technical reviewer(s), and QAO shall sign controlled documents of manuals, policies, and procedures. The cover page of controlled quality documents shall contain the document title, control number, the version number, the effective date. Page numbers will be on each page. Controlled documents are identified by the signatures and colored title page and stored in a file cabinet in the QA area. 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 is following 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 are responsible for ensuring that all personnel are kept current with specific controlled documents that are relevant to their DEQ activities. Section managers and employees shall update form **DEQ06-LAB-0016-FORM** in **Appendix B** (maintained in training file), as needed, to document the employee's attestations that they will follow the policy and procedures described in controlled documents.

Note: The disposition of analytical records and data reports is discussed in sections Section 14 Control of Records.

4.1 STANDARD OPERATING PROCEDURES

Standard operating procedures (SOPs) are used to ensure consistency of application of common procedures. They are written procedures that describe in detail how to accurately reproduce laboratory processes, and are of two types: 1) Technical SOPs, which have specifically required details, and 2) Administrative SOPs which document the more general organizational procedures.

The Laboratory maintains documented instructions that accurately reflect administrative and technical laboratory operations, including project planning, sample collection, sample handling, calibration, equipment usage, data review, data processing, data assessment procedures, etc. Where equipment manuals or published methods accurately reflect laboratory procedures in detail, a separate SOP is not required. Circumstances may arise where analyses may be performed without a completed SOP under the supervison of the Technical Manager provided all steps of the process are documented including references to available literature that the procedure is based upon. Deviations from a test method are documented in the SOPs, including both a description of the change made and a technical justification.

The SOPs are prepared, reviewed, revised, approved and controlled in accordance to the DEQ procedure: "*Preparing Standard Operating Procedures"* (**DEQ04-LAB-0001-SOP**). The control procedure for SOPs is the same process as discussed in **Section 4** above.

The following activities must be described in LEAD SOPs. This list is not exhaustive and additional activities may also need to be documented in SOPs:

- Water Quality Monitoring and Air Quality Monitoring Section managers will ensure that sample collection, transportation to the laboratory, and field analysis procedures are written to cover their respective areas.

- The Organic and Inorganic Section managers shall ensure SOPs are written for all procedures under the scope of testing including sample preparation, calibration analyses, and data handling procedures.
- Technical Services shall ensure SOPs are written and revised for a series of routine work performed by the Technical Services Section (including sample receiving, sample storage, preservation and tracking).
- Technical Services –is responsible for the control of electronic information within the laboratory; i.e. posting documents on the web, securing servers for defined uses, maintaining tables within ELEMENT® and data repository, creating reports from ELEMENT®, and the reporting of results.

An independent QAO shall review and sign all controlled documents, for which he/she is not the author to help ensure that the procedure is properly and completelydocumented. SOPs are available to all Laboratory and Agency personnel through **Q-Net**. Staff is encouraged to view the electronic version on screen as there are hyperlinks to associated documents and are easily searchable. However, hardcopies may be printed as uncontrolled documents from the electronic version that is available, whenever possible, on Q-Net.

4.2 TECHNICAL SOPS

Test method SOPs 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). The laboratory shall ensure written procedures are available, which describe how to properly use, calibrate, and maintain analytical equipment. During the annual internal audit, the auditors shall cite, when necessary, the deficiency of failing to meet criteria described in *Preparing Standard Operating Procedures*. The corrective action plan for this deficiency shall be to revise the SOP to meet the current TNI standards and to set a schedule for completing the revision. The QAO or section manager shall assign the task of creating the new SOP to the appropriate laboratory personnel.

Appendix G lists the analytical test methods the laboratory performs and cites the EPA reference test method. The laboratory shall have controlled written 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 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.

5 REVIEW OF REQUESTS, TENDERS AND CONTRACTS (V1M2: 4.4)

The review of all new work assures that oversight is provided so that requirements are clearly defined, the laboratory has adequate resources and capability, and the test method is applicable to DEQ or EPA needs. This process assures that all work will be given adequate attention without shortcuts that may compromise data quality.

Prior to the onset of Air Quality or Water Quality Monitoring projects, a project proposal infopath form (on the **LEAD Share Point site**) is initiated and presented to section managers, QA officer, and the sample coordinator during the weekly status meetings and basics of the project are discussed and capabilities are assessed. With few exceptions, all work falls under a *Quality Assurance Project Plan* (QAPP) and/or a *Sampling and Analysis Plan* (SAP). 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 QAPP and SAP development, the

appropriate QAO reviews the document to determine if the laboratory has the necessary accreditations, resources (including schedule), equipment, deliverables, and personnel to meet the work request.

5.1 QUALITY ASSURANCE PROJECT PLANS (QAPPS)

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 and advise the project coordinator if test methods appear to be inappropriate or out of date. If a decision is made to continue with a non-routine procedure, the DEQ laboratory will then develop its own procedure or subcontract the test.

The project coordinators, the QAO, and/or section managers will also 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 document procedures for reporting such data in the QAPP.

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**. There are several templates currently available on **Q-Net** for QAPP/SAP writing. Other templates will be available as they are created, or a QAPP may be developed as a stand alone document without a template:

- Quality Assurance Project Plan Template: **DEQ04-LAB-0029-TMPL**
- Volunteer Monitoring Sampling and Analysis Plan (SAP): **DEQ05-LAB-0072-TMPL**
- Mixing Zone Sampling and Analysis Template: **DEQ06-LAB-0049-TMPL**
- Sampling Analysis Plan Template: **DEQ11-LAB-0026-TMPL**

5.2 QAPP REVIEW:

5.2.1 Test method requirements:

a) The relevant analytical section manager shall ensure the 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 as well as the current available ELEMENT® anlaysis codes, and/or consult with the appropriate tehnical manager to ensure the capability of the laboratory to perform the requested test method.

b) Special projects may require deviations from test method SOPs or the development of new procedures. The QAPP shall describe or refer to the test method to be performed if there is no SOP available. The appropriate section manager shall ensure the laboratory has the resources and capabilities to make such adjustments and discuss test method proposals with the QAO. The laboratory shall validate the new procedure as described in **Section 18.1** of this document.

c) 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 decisions shall be covered in the QAPP.

d) Reporting levels required for the project,

e) Contaminant action levels to be used for decision making,

5.2.2 Projects accreditation requirements and the laboratory's current status

a) The QAO or designee shall track the accreditation status of subcontracted laboratories where available. If appropriate, the QAO or 3rd party data coordinator shall qualify data and ensure the DEQ analytical report includes the appropriate comments.

5.2.3 QC measures to be taken

a) Types and frequency of Field Quality Control
b) Types and frequency of Laboratory Quality Control
c) Control limits

5.2.4 Procedures to control nonconforming work, i.e. deviations from the QAPP

a) Refer to section C1 of "*EPA Requirements for Quality Assurance Project Plans* EPA QA/R-5".
b) The nonconformance procedure in the QAPP shall describe the process for finding deficiencies in meeting QAPP requirements.
c) The nonconformance procedure shall identify management responsibility for taking action, such as halting work until a corrective action plan is determined, and identifying the conditions that would enable the resumption of work.
d) If deficiencies are found in DEQ laboratory work, the procedures (**Section 10** and **Section 12**) shall be used to document and find solutions for the nonconforming work.

5.2.5 Ongoing Review

a) The QAO and project coordinators should review QAPPs during the project and they have the capacity to make amendments to the QAPP.
b) The QAO and project coordinator shall review and approve amendments.

If the review indicates a potential conflict, deficiency, lack of accreditation, or inability of the LEAD to the complete the work satisfactorily, the document is returned to the document author or project manager for revision.

All differences between the initial request and the final QAPP/SAP are resolved and recorded before any work begins. It is necessary that the QAPP/SAP be acceptable to all parties.

The final (QAPP/SAP) is approved with a signature by at least the affected section managers, and at least one QA officer, and the project manager. There are often several other approvals to the documents depending on who is involved. Finalized amendments are controlled by the QAO (following the Documents Control SOP **DEQ02-LAB-0004-SOP**) and made accessible to all staff through **Q-Net**.

When there are amendments or revisions needed to the original QAPP/SAP by the project manager, the review process is repeated. Finalized amendments are controlled by the QAO and accessible to all staff through **Q-Net**.

6 SUBCONTRACTING OF TESTS (V1M2: 4.5)

A subcontract laboratory is defined as a laboratory external to the LEAD laboratory that performs analyses for the LEAD. Whenever possible, laboratories performing subcontracted analyses must be accredited under NELAP or another nationally recognized accreditation program approved by the agency QAO. Note that there will be instances where an accredited laboratory may be unavailable or cost prohibitive. These situations are handled on a case by case basis in consultation with a QAO. Subcontracting of analyses are determined at the time of QAPP development and, as stated in **Section**

5.2.2 above, the QAO, through the ORELAP program, will monitor the accreditation status of subcontract laboratories as needed. The DEQ Purchasing department develops contracts with subcontract laboratories, where accreditation is a prerequisite, and the accreditation status is verified at that time as well. In the rare situation if there is catastrophic equipment failure or a situation arises where analyses must be subcontracted by the laboratory, the project manager of the affected samples must be notified via email as well as Technical Services (sample coordinator, sample custodian, and/or third party data coordinator). Verbal notification, is suggested as well.

In all cases where work is performed by another laboratory, the laboratory performing the subcontracted work is identified in the final report.

7 PURCHASING SERVICES AND SUPPLIES (V1M2: 4.6)

The LEAD ensures that purchased supplies and services that affect the quality of environmental tests are of the required or specified quality by using approved suppliers and products. 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.

One of the responsibilities of the Technical Services manager is to coordinate the purchase and receipt of supplies, capital equipment, and the disposal of unused obsolete equipment and supplies by following the State surplus procedures and completing the *Property Disposition Request* (PDR) forms.

7.1 REQUEST FOR PURCHASE

LEAD staff and their section managers are responsible for the traceability of supplies. The Technical Services Section maintains the ***Requisition Database***, which is used to track the purchase and receipt of all orders. Some purchase are made using an agency issued credit card (aka SPOTS card) when purchases need to be made more quickly than the standard requition process and the amounts are below the thresholds required for use of these cards. Both SPOTS and standard purchases are documented in the requition database. Complete detail and documentation of all agency procurement policies and procedures is beyond the Scope of this document. The interested reader is referred to the Management Services division (MSD) Q-Net site for additional details.

The LEAD staff person 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 staff person requesting the supplies shall note the quality needed. Signatory authorities who sign test method SOPs ensure the SOP specifies the quality of chemicals and instrument to be used. Certificates of analysis (COA) are maintained in the lab sections for standards, and critical reagents and specific calibrated support equipment (e.g. micro syringes, thermometers, etc). Suitability is assessed based on visual inspection and demonstrated during the initial use of the chemical as well as review of the certificate of analysis.

The purchaser of supplies or services in each section 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 Technical Services Section. LEAD staff using the products notify the product suppliers immediately if the quality of the product is found to be unsatisfactory for their needs. Department of Administrative Services (DAS) maintains contracted price agreements with selected approved suppliers. Complaints about these suppliers are documented and submitted to DAS. In rare instances, for suppliers not under contract, the LEAD Section manager will take action as necessary up to and including eliminating the vendor as a DEQ supplier. Some programs (e.g. Air) may require the use of specific

products available through qualified vendors. The requisition database can be used to maintain the 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. To ensure specifications are met, approval from the laboratory staff must be obtained before any substitutions of requested products are made.

7.2 PURCHASE

All purchases have a formal agency ***purchase approval process procedur***e. In cases where purchases may occur without prior consent, personnel should take caution and management shall inform personnel of the current credit card policy.

To ensure products directly related to data quality are tracked; personnel must copy the requisition by printing it from the database or by filling 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 (under $25.00). Personnel must be mindful of the requirement that certain products must be tracked and a copy of the requisition is maintained to ensure information for the products and suppliers is retrievable.

7.3 RECEIPT

Upon receipt, 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 a Technical Services Section OS2.

The recipient of contracted services shall request copies of the service form (describes services that were performed) from the contractor to maintain the laboratory's traceability requirements.

If there is a problem with the order or services rendered, the DEQ Procurement section (HQ) shall take appropriate steps to reverse the order or the LEAD staff may attempt to verify the suitability of the product (refer to **Section 20** *Measurement Traceability*).

7.4 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 (e.g. chemicals that come packaged in cans should not be opened in receiving); the LEAD staff that ordered the product is familiar with the special requirements that may be required. When the section manager signs the Requisition form, he/she should also look for special handling instructions. Packages that are received without a packing list on the outside from an unknown vendor should be carefully opened in the fume hood (while wearing nitrile gloves and using some form of eye protection) in the Sample Receiving area.

Often a *Safety Data Sheet* (SDS) are packaged with chemicals. The purchaser should forward SDSs to the laboratory OS2 who will file them in the SDS binders located on bookcases in the Administration/Technical Services area. At the time of the writing, planning for a change if this process is underway. After 2013, the manual SDS management system will likely be replaced with an electronic records management system. The new system will be described in an update to this document.

7.5 USE AND CONSUMPTION

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. 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. ELEMENT® LIMS may also be used to document the use of these materials.

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 their manager for the current procedure, which shall include completing the PDR form and moving the equipment to the holding area so that it cannot be put back into service inappropriately.

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.

8 SERVICE TO THE CLIENT (V1M2: 4.7)

The DEQ Director has placed "Excellent service" as one of the a priorities in DEQ's ***Key Goals***. 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 appropriate section manager for information on projects and the Technical Services manager for retrieving data. The public has access to data stored in the agency data repository. 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. In these instances, at the time of log-in, the case is tagged in ELEMENT® to prevent making the data public accessible. LEAD strives for continuous improvement in customer service and to enhance the scope and quality of services provided to our clients. In addition to raw data, the Technical Services Section can provide customized data interpretation, reports and query results, as a few examples of extra value added services.

The LEAD also seeks to obtain customer feedback through the use of surveys using *Survey Monkey* or other survey tools. DEQ staff from HQ and the regions all provide feedback on the laboratory's performance and the results of the survey are reviewed and an action plan for improvement is developed by the Laboratory Mangement Team.

9 COMPLAINTS (V1M2: 4.8)

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 and technical perfomance. The laboratory shall respond to inquiries of data anomalies and complaints of report format or content, response time, and laboratory policy using the control of nonconforming work and corrective action procedures described in Sections 11 and 12. 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 custodian, whereas the receptionist usually receives calls from the public. The receptionist and the sample custodian 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 LEAD 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, or any other section manager, or the LEAD 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 stop any work in progress that is affected by the problem until the problem is corrected and initiate the corrective action process as discussed in **Section 10** and **Section 12** below.

A complaint does not necessarily mean nonconforming work has occurred. However, complaints must be investigated to determine whether or not an error has occurred. Complaints are documented and managed/monitored using the "***Issue Tracker***" database and follow the nonconformance and corrective action process discussed below in **Section 10** and **Section 12**. 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.

10 CONTROL OF NONCONFORMING WORK (V1M2: 4.9)

Nonconforming work is work that does not meet acceptance criteria or requirements. Non-conformances can include, among other things, departures from the policies or procedures within the LQM, monitoring activities, analytical test method SOPs, or unacceptable quality control results (see **Section 22** *Quality of Test Results*). Requests for departures from laboratory procedures are approved by the Section Manager and/or the QAO and documented.

The basic process for the control of non-conforming work is to identify the non-conformance, determine if it will be permitted, and take appropriate action. All employees have the responsibility to stop work when they determine any aspect of the process does not conform to laboratory requirements. SOPs should define most of the corrective action steps to be taken for nonconforming situations. In the cases where the SOP does not provide a corrective action, the section manager, section lead, or QAO should be consulted before proceeding.

The laboratory evaluates the significance of the nonconforming work, and takes corrective action immediately. The client is notified (normally in the form of data qualification) if their data has been impacted. If the report has been previously issued, a formal Report Erratum is generated and issued following procedures outlines in the SOP *Data Correction Process (Analytical Reports)* (**DEQ03-LAB-0002-SOP**). In the absence of instruction in an SOP, resumption of work after a non-conformance is authorized by the Section Manger or the QAO.

Nonconformance investigations are initiated by any staff member upon discovery of a potential issue. The Nonconformance shall be documented using the corrective action (CA) database (called ***Issue Tracker)*** to record the description of the incident, personnel involved in the investigation, deadlines, corrective action taken, and the dates for completing each event. Samples receiving nonconformances may be documented on the sample receipt checklist or in *Issue Tracker*.

The specific procedures for documentation of nonconformance issues are in the SOP "*Nonconformances and Corrective Actions*" **DEQ07-LAB-0053-SOP.** This same documentation/tracking procedure is also used for entering and tracking external audit findings and client complaints.

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 ELEMENT® by creating a unique "Standard Parameter" for the new procedure.

The client (DEQ project manager) will be notified immediately in the case where a non-conforming event will void an entire sample, all samples for a single analyses or entire analyte groups for a sampling event (work order). This will give them the opportunity to resample or make other adjustments if they need to prior to the release of the final report.

Note: Nonsystematic nonconformance's (e.g. most analytical QC failures) are handled as individual instances and are documented using a review form generated from ELEMENT, a *Technical Corrective Action* form or on *Peer Review* forms (and not entered into Issue Tracker). The Technical Corrective Action Form or review forms are maintained in the Case folder of any affected projects.

11 IMPROVEMENT (V1M2: 4.10)

Improvement in the overall effectiveness of the laboratory management system is a result of the implementation of the various aspects of the laboratory's quality/management system: quality policy and objectives (Section 2.1 – "Quality policy"); internal auditing practices (Section 14.1 – "Internal Audits"); the review and analysis of data (Section 22 –"Quality Assurance of Test Results); the corrective action (Section 14 –"Corrective Action") and preventive action (Section 13 – "Preventive Action") process; and the periodic management review of the quality management system (Section 14.4 – "System Audits and Management Review") where the various aspects of the management/quality system are summarized, and evaluated and plans for improvement are developed.

Throughout the year the laboratory monitors PT performance, SOP completeness, corrective actions, report revisions, and turnaround time for improvement with a year-end summarization during the annual management review. Other reports may be included as applicable.

12 CORRECTIVE ACTION (V1M2: 4.11)

Corrective action is the action taken to eliminate the causes of an existing nonconformity, defect, or other undesirable situation in order to prevent recurrence (TNI, 2009).

Sources of deficiencies, as cited above in Section 10, including external assessments, internal quality audits, data reviews, complaints, or managerial reviews are documented and require corrective action. Corrective actions taken are appropriate for the magnitude of the problem and the degree of risk. The specific procedures for documentation of nonconformance issues are in the SOP *Nonconformances and Corrective Actions* **DEQ07-LAB-0053-SOP.**

Note: The discovery of a nonconformance for results that have already been reported to the client must be immediately evaluated for significance of the nonconformance, its acceptability to the client, and determination of the appropriate corrective action.

12.1 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 cannot be determined, the investigation and the corrective action report must still be completed. The root cause investigation should begin with the employees whom appear to be most directly related to the nonconformance.

The section manager immediately brings deviations from the LQM or the QAPP to the attention of the QA team, who shall conduct these nonconformance investigations. The QAO ensures the project coordinator is involved in the corrective action plan. Generally, nonconformance investigations will be handled by the appropriate laboratory sections:

- Technical Services Section investigates and performs corrective actions that relate to ELEMENT® functions, sample receipt, sample integrity, and complaints.
- Analytical sections of the laboratory conduct the investigations and complete the necessary corrective actions for deviations from analytical test methods and failed QC measures within their sections.
- Monitoring sections (Air, Water, Land) investigate nonconformance events related to sample collection, continuous monitoring, and other field operations.

12.2 SELECTION AND IMPLEMENTATION OF CORRECTIVE ACTIONS

Once the root of the problem has been identified through cause analysis, potential solutions are proposed and implemented. Corrective actions shall be appropriate to the magnitude and risk of the problem. The section manager collaborates 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 records these corrective action options into the corrective action database.

Once it has been identified, the appropriate corrective action must be implemented by the affected LEAD Section(s). The appropriate section manager ensures that corrective actions are discharged within the agreed upon time frame. Section managers shall approve changes in analytical data and in SOPs with QAO concurrence. The QAT documents and ensures implementation of any required changes in the LQM or laboratory policy resulting from corrective action investigations.

12.3 MONITORING OF CORRECTIVE ACTIONS

The LQAO or his/her designee shall monitor the effectiveness of corrective actions taken. The corrective action database should provide sufficient information to assess the effectiveness of corrective action procedures. The corrective action must be linked with the appropriate documents through their document control numbers. The success and performance of the implemented corrective action plans is a part of the annual *Quality Systems Review* **(Section 15.4)**.

12.4 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 procedures as soon as possible.

12.5 TECHNICAL CORRECTIVE ACTION

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.

Similarly, routine sample receiving nonconformances are documented on the sample receipt checklist.

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.

Sample data associated with a failed quality control are evaluated for the need to be reanalyzed or qualified (sample acceptance criteria failures are evaluated in a similar fashion.). Unacceptable quality control results are documented, and if the evaluation requires cause analysis, the cause and solution are recorded. The analyst is responsible for initiating or recommending corrective actions and ensuring that exceedances of quality control acceptance criteria are documented using a *Technical Corrective Action Form* (**Appendix C**) or the on the specific analysis data review checklist (or ELEMENT generated review form). The completed form is maintained in every affected case folder.

The second level data reviewer, reviews the corrective action reports and suggest improvements, alternative approaches, and procedures where needed.

If the data reported are affected adversely by the nonconformance, the client is notified in writing. This notification is primarily performed with the use of data qualification and/or narration.

12.6 EXCEPTIONALLY PERMITTING DEPARTURES FROM DOCUMENTED POLICIES AND PROCEDURES

The laboratory allows the release of nonconforming data only with approval by the appropriate section manager or their designee on a case-by-case basis. Planned departures from procedures or policies do not require audits or investigations. Permitted departures for non-conformances, such as QC failures, are fully documented and include the reason for the departure, the affected procedure, and potential impact to the data.

Examples of Departures that would be allowed*: insufficient sample volume for a rerun, where holding time has already been exceeded, or where sample data are not affected by the non-conformance*.

Example of Planned Departures include*: You do not receive the required sample volume, and the client wants you to complete the analysis anyway.*

In both examples, you would document the departure, qualify the data if necessary, but would not perform any investigation

13 PREVENTATIVE MEASURES (V1M2: 4.12)

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 (Section 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.

14 CONTROL OF RECORDS (V1M2: 4.13)

Records include subset of "documents" as described in Section 4, but in this context, records refers to the daily recording of specific information as it applies to the documentation of laboratory activities. Some examples are: data recordings that include annotations, such as instrument logs, sample preparation bench sheets, standard/reagent logs, analytical bench sheets, daily refrigerator temperature and thermometer logs, balance calibration logs, instrument data output records, field logs, etc. Quality records include reports from internal and external audits and management reviews as well as records of corrective and preventative actions. Records may be on any form of media, including electronic and hard copy. Records allow for the historical reconstruction of laboratory activities related to sample-handling and analysis.

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.*

The laboratory retains all original observations, calculations and derived data, calibration records, and a copy of the test report for a minimum of 10 years after last use/issue unless otherwise directed by specific DEQ record retention rules.

14.1 RECORDS MANAGEMENT AND STORAGE

Technical Services is responsible for maintaining the integrity of analytical data generated by the LEAD. The Technical Services Section manages analytical data through the use of ELEMENT® and the archive databases, associated with the LIMs. As data progresses through the system from entry to review to reporting, ELEMENT® adjusts the status code of the result, which restricts what can be done to the data. Chemists or technicians enter data into ELEMENT® and may make corrections to data up until they submit their work for review. Senior analysts review data and may send the data back to the originating chemist for rework. ELEMENT® users cannot alter data after the review process without manager or QA approval.

Records, including electronic records, shall be easy to retrieve, legible, and protected from deterioration or damage; held secure and in confidence; and be available for a minimum of 10 years. The LEAD maintains a record management system for control of laboratory and field notebooks, instrument logbooks, standards logbooks, and records for data reduction, validation, storage, and reporting.

Paper records are sent to the Iron Mountain facility for archival for protection against fire, theft, loss, environmental deterioration, vermin etc. Electronic information (computer drives, ELEMENT® and instrument data) is backed-up to tape incrementally every night and full back-ups are performed monthly and the tape is sent off site and stored in a fireproof safe to protect from fire and electronic or magnetic sources.

14.2 BASIC REQUIREMENTS FOR RECORDS MANAGEMENT

14.2.1 Identification:

Records are uniquely identified. Records are identified through one or a combination of items e.g. their Batch IDs, Method or SOP reference, Work Order number, log book ID, unique sample number, etc.

14.2.2 Collection:

LEAD personnel are responsible to ensure observations, data and calculations are recorded in bound log books, loose leaf binders, or data sheets at the time the observations are made. These logbooks must be labeled and controlled worksheets are maintained according to DEQ SOP *Peer Review and Record Management* **DEQ00-LAB-0004-SOP** .

Record entries in must be legible and shall not be obliterated by methods such as erasures, overwritten files or markings. When mistakes are made in technical records, each mistake is crossed out with a single line (not erased, made illegible, or deleted) and the correct value entered alongside. Corrections are signed or initialed by the person making the correction. When changes are made to technical records for reasons other than for correction of transcription errors, the reason for the change is recorded on the document.

For electronic systems (ELEMENT® and analytical instruments), all changes are tracked by the audit trail or by added notes.

When data is transcribed from an original record to another format (e.g. standard form, ELEMENT® entry, etc), The original record (or copy) must be readily accessible to review the transcription. The original record is still retained according to DEQ record retention requirements.

14.2.3 Storage:

All records stored on electronic media are supported by the hardware and software required for retrieval and have hard-copy or write-protected backup copies.

14.2.4 Filing:

Records are filed in an organized fashion and stored in the individual laboratory areas, the compressed file room and auxillary building, until such a time as they are ready to be moved to offsite archives which are managed by the Technical Services section.

14.2.5 Access:

Completed analytical report files are only accessable to a limited number of staff within LEAD who have key access to the locked compressed files. These files are located in a restricted access storage room accessible only to employees with electronic keys. The files are signed by an authorized tech services staff member to recipient using a sign-out card. As a place holder, the card resides in the original file location in the storage closet and the file is hand-deliver to the requestor.

Access to archived paper records is restricted to designated staff in Technical Services and the records management coordinator through Iron Mountain. Section managers are also authorized to access files from Iron Mountain for their sections.

Access to electronic records (e.g.file servers) is restricted through user privileges and access is monitored with audit trails.

14.2.6 Disposal

Records are disposed of according to agency records retention schedules or after at least five years after the record was archived. Analysis records are stored for ten years.

14.3 LOGBOOKS/NOTEBOOKS

A master log for tracking logbooks and notebooks is maintained in a central QA area. When a need for a new log has been established, the logbook is created by the section needing it. A representative of the section records the logbook into the master log and, following the procedure in the master log, creates a unique tracking ID control number for the new log. The section representative also records pertinent data describing the content, version, author, and date of the initiation of the log. Once the logs and/or notebooks are full, the chemists and technicians must return the records to Technical Services for archiving at Iron Mountain. Analytical records shall be archived for a period of ten years since the last entry in the log.

14.4 RAW INSTRUMENT DATA

Raw instrument data is stored sequentially by instrument and placed in a labeled box in locations designated by the section. When full, these boxes are submitted to Technical Services for archival at Iron Mountain. Electronic instrument files are sent for back up daily by the analysts after they have processed the data. These files are then moved to a server and backed up to tape on the nightly schedule (see **Section 14.1** above)

14.5 DATA REVIEW CHECKLISTS

A copy of Data (peer) Review checklists are kept in every work order file and stored according to requirements for analytical reports. The originals are maintained in the lab section with the raw data files.

14.6 QUALITY RECORDS

In addition to the records discussed above, the LQAO shall maintain procedures for identification, collection, indexing, access, filing, storage, maintenance and disposal of quality 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.

Records of corrective and preventative actions are maintained electronically in the Issue Tracker database.

Reports from Internal and external audits are maintained/filed by the QAO.

Reports from the management review of the quality system are kept as controlled documents and assigned a document control number. They are filed with the other controlled documents (SOPs, QAPPs, LQM, etc) and accessible to staff on **Q-Net**.

14.7 WORK ORDER REPORTS

Once the results are approved and released for all tests performed on a set of samples, an Adobe PDF format report is automatically generated and made available to the client. After the report is finalized it cannot be altered without being reissued as a revision. Subsequent changes in an analytical report must be made through the *Data Correction Process* (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 report revisions 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.

Work order report files are archived sequentially and sent offsite to Iron Mountain for secure storage by the Technical Services Section.

14.8 LEGAL CHAIN OF CUSTODY RECORDS

The procedures for the legal chain of custody records used for evidentiary or higher security cases are found in the *Sample Receiving and Control* SOP **DEQ06-LAB-0054-SOP**. The legal custody case files are labeled as such to prevent premature destruction and archived with the other case files at Iron Mountain.

15 AUDITS AND MANAGEMENT REVIEW (V1M2: 4.14-1.15)

Audits measure laboratory performance and verify compliance with accreditation/certification and project requirements. Audits specifically provide management with an on-going assessment of the quality system. They are also instrumental in identifying areas where improvement in the quality system will increase the reliability of data. Audits are of four main types: internal, external, performance, and system.

Project managers are notified within 24 hours if it is found that an erratum is needed based on discovery of events that cast doubt on the validity of the results

15.1 INTERNAL AUDIT

Internal audits are performed on a regular schedule but may be requested as needed based on findings from corrective actions, issues discovered during data review, complaints, data integrity concerns, etc.

The LQAO shall establish an internal audit schedule, and at least annually, conduct or arrange 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.

As a part of the internal audit activities, lead chemists perform technical audits on specific methods and analysts. Findings and associated corrective actions are documented on a spreadsheet maintained on Share Point. These audits are compiled/summarized in the annual quality management review.

During the internal audit of the test method SOP, the auditor shall review the summarized QC data looking for trends. Personnel performing an internal audit shall review test method SOPs ensuring all necessary QC measures listed in Appendix G are accounted for in the procedure. The auditor shall consider the essential QC standards outlined in the current TNI 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 G 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.

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 project coordinators are notified and sent a revised report if findings showing the laboratory results were affected. A record of the notification of the report release is maintained by technical services staff.

The audit team shall record assessment findings in a spreadsheet or similar electronic media, from which an audit report will be printed and submitted to the section manager(s). The section manager shall ensure that a corrective action plan for his/her section is prepared 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 TNI 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.

15.2 EXTERNAL AUDITS

It is DEQ policy to cooperate and assist with all audits performed by external agencies.

All external audits are fully documented and tracked to closure. Any findings related to an external audit follow the corrective action procedures (**See Section 12**) using the Issue Tracker database. Management ensures that all areas of the laboratory are accessible to auditors as applicable and that appropriate personnel are available to assist in conducting the audit.

15.3 PERFORMANCE AUDITS

Performance audits may be Proficiency Test Samples, internal single-blind samples, double-blind samples through a provider, or anything that tests the performance of the analyst and method.

The policy and procedures for Proficiency Test Samples are discussed in **Section 22.3**.

15.4 SYSTEM AUDITS AND MANAGEMENT REVIEWS

A monthly quality system review is performed with the management team (Division administrator and Section managers) and the quality assurance officers. This review evaluates the division's performance in the following areas: report revisions (aka data corrections), corrective action implementation, SOP review status, PT performance, data integrity training, LQM revisions, and performance of an annual review, and completeness of reported data. Corrective actions are taken if any of these indicators are falling significantly short of expectations.

Quarterly, these quality system measures are reviewed along with additional measures that reflect division productivity and other agency goals.

The Management Team (Division administrator and Section managers) reviews the quality management system annually and maintains records of review findings and actions. This review is performed annually within 2 months after the end of the fiscal year.

The LQAO shall schedule the annual quality management review with the LEAD Management. Prior to the meeting, the QAO creates a draft report containing the items below and through a collaborative process with the LMT, creates a final report (except for identifying action items and assigning responsibilities). This report is then provided to the LEAD administrator with this draft-final report prior to the face to face meeting with the LMT. The review 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 quality system, and to introduce necessary changes or improvements. The review shall take account of QA activities and concerns that occurred during the previous fiscal 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.

During the collaborative prepraration of the report, the LEAD administrator and LMT provide input and comment on management review and a corrective action schedule and assignment of responsibilities occur during the face to face meeting.. The LQAO shall maintain a file of the annual QA reports, the LMT'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 LMT shall be monitored as described in the Corrective Action procedure (**Section 12**).

16 PERSONNEL, TRAINING AND DATA INTEGRITY

In addition to the general managerial and organizational descriptions in **Section 2** above, the DEQ LEAD 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 they list their qualifications and certify the information given 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, their application is first reviewed by the agency's Human Resources section to insure they meet the minimum qualifications for the classification as determined by DAS (refer to **http://www.hr.das.state.or.us/JOBS/**).

An applicant is required to meet the established minimum qualifications in order to proceed in the recruiting process. The DEQ HR division prescreens all applicants for the minimum qualifications. 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, TNI standards, and laboratory policy. Training is kept up to date by periodic review of training records and through employee performance reviews. In order to be considered trained to perform an analytical method an analyst must complete a successful initial demonstration of capability (IDOC) and demonstrate on an ongoing basis that they continue to be proficient to perform the analyses. See **Section18.1** *Method Validation* and section 18.1.2 *Continuing Demonstration of Capability.*

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. The laboratory will use a system of routine checks as prescribed in the SOP *Peer Review and Record Management* (**DEQ00-LAB-0004-SOP**). Specific position descriptions are maintained in employee training or HR files and can be found on DEQ's intranet site (Q-Net).

Table 16-1 Minimum Qualification for Laboratory Personnel

Position	Minimum Qualification
Laboratory & Environmental Assessment 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.

Position	Minimum Qualification
Quality Assurance Officer (NRS 4, NRS 3)	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. (Degree or 24 semester hours of college chemistry)
Natural Resource Specialist 4 (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 specialists, project coordinators and chemists frequently 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.

Position	Minimum Qualification
Chemist (Chem 3)	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 degree. 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 (Chem 2 & Chem 1) (Includes Sample Custodian)	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.
Natural Resource Specialist 3 (NRS 3)	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. The sample coordinator also performs QA review on all water quality projects.
Natural Resource Specialist 2 (NRS 2)	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.

Position	Minimum Qualification
Information System Specialist 5 (ISS5) *LIMS Coordinator*	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;
Information System Specialist 4 (ISS4) *Database Specialis*t	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;
Executive Support Specialist (ESS-2)	Skill in performing secretarial or administrative support functions requiring independent judgment, decision-making, and problem resolution. Skill in researching information and composing memos, letters, and other correspondence for own or administrative superior's signature. Ability to participate in top level management meetings to help resolve agency problems and discuss goals and objectives. Ability to maintain confidentiality of agency and/or personnel records. (provides confidential administrative support) Ability to learn agency programs, operations, policies and procedures, affecting assigned work.
Office Specialist 2 (OS-2)	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.

Position	Minimum Qualification
Office Specialist 1 (OS-1)	General knowledge of proper grammar, punctuation, spelling, and capitalization. Basic knowledge of arithmetic (addition, subtraction, multiplication, and division). Skills in: proofing documents, performing clerical duties, communicating orally and in writing, typing (proficiency levels will be based on individual position requirements), and operating typical office equipment.

16.1 DATA INTEGRITY AND ETHICS (V1M2: 4.16)

16.1.1 Data Integrity and Ethics Policy

It is the LEAD policy that appropriate and adequate quality assurance activities shall be implemented to document that all environmental data generated, stored, reported, or used is of known and adequate statistical quantity and quality to fulfill the needs of the primary data user. Data shall be accurate, precise, complete, representative, comparable and, when required, legally defensible. This policy is intended to embrace both internal data, generated by internal LEAD monitoring and testing activities, and external data arising from regulated activities, contracts, interagency agreements, grants, and cooperative agreements.

The DEQ Laboratory is committed to ensuring the integrity of our data, incorporating the highest appropriate standard of quality in all of our analytical programs.

- Personnel shall not condone any accidental or intentional reporting of deceptive or misleading data. If management requests personnel to engage in an activity that compromises data integrity, they have the right to refuse compliance with the request and to appeal the action through the QA officer.
- Management shall not instruct subordinates to perform any practices that would violate this policy, nor will management discourage, intimidate or inhibit a staff member who may choose to appeal instruction under this agreement and will not retaliate against those who do so.
- All work assigned to personnel will be performed in compliance with the quality assurance manual and standard operating procedures. It is the responsibility of staff to be aware of and compliant with current policies and procedure requirements for assigned duties.
- Personnel will only report results or data that match the actual results observed or measured.
- Personnel will not intentionally falsify any data in any manner. Data will not be modified unless the modification is technically justified through a measurable analytical process approved by the QA officer. All such modifications will be clearly documented.
- Recording of dates, times, and initials on data shall accurately reflect who and when the procedure was performed.
- Personnel shall not intentionally make false statements to, or seek to otherwise deceive data users, agency representatives, or auditors.
- Personnel shall not through intentional acts of omission, commission, erasure, or destruction improperly report measurements, standard results, data, test results, or analytical conclusions.

- Personnel shall not destroy, or overwrite records of analyses or original observations. This includes, electronic files and instrument sequences, analytical reports, original recording of observations, etc.
- Personnel are required to understand, through training and review of quality systems documents, that any infractions of the laboratory data integrity procedures will result in a detailed investigation that could lead to very serious consequences such as immediate termination, or civil/criminal prosecution.

16.1.2 Data Integrity and Ethics Procedures and Training

Data integrity and ethics procedures in the laboratory include training, signed, and dated integrity documentation for all laboratory employees, periodic monitoring of data integrity, and documented data integrity procedures. Section managers uphold the spirit and intent by supporting integrity procedures, by enforcing data integrity procedures and ensuring staff participate in annual data integrity training.

Data integrity training is provided for all employees initially upon hire and annually thereafter. Attendance at an initial data integrity training (part of new employee orientation) and the annual refresher training is recorded with a signature attendance sheet and/or documented in iLearn (certificates may be generated from iLearn).

Specific integrity procedures for analyses involving chromatography (IC,GC, GCMS, HPLC, etc.) are identified in DEQ LEAD SOP *Manual Integration Practices* **DEQ09-LAB-0003-SOP**. Training on this SOP is provided to all staff that performs chromatographic analyses.

When contracted technical or support personnel are used, management is responsible for ensuring that they are trained to the laboratory's quality system and data integrity procedures, competent to perform the assigned tasks, and appropriately supervised

Employees shall report all violations to management or quality assurance. Failure to report an integrity violation is an act of condoning the activity and is seen by DEQ as equivalent to having actually committed the violation.

The mechanism for confidential reporting of ethics and data integrity issues is (1) unrestricted access to senior management or QA officers, (2) an assurance that personnel will not be treated unfairly for reporting instances of ethics and data integrity breaches, and (3) anonymous reporting.

Any potential data integrity issue is handled confidentially, to the extent possible, until a follow-up evaluation, full investigation, or other appropriate actions have been completed and the issues clarified. Inappropriate activities are documented, including disciplinary actions, corrective actions, and notifications of clients, if applicable. These documents are maintained for a minimum of 5 years.

Data integrity procedures are reviewed as part of the annual quality systems review (QSR) and periodically monitored through in-depth data review of audit trails or records review as a part of internal audits.

17 ACCOMMODATIONS & ENVIRONMENTAL CONDITIONS (V1M2::5.3)

17.1 LABORATORY FACILITY

The DEQ laboratory (LEAD) is equipped with sufficient power resources to operate instruments and equipment safely for the testing listed in Appendix G**.** Personnel must employ good "house keeping" procedures to accommodate the needs of other personnel and avoid the potential for contamination. When appropriate, the test method SOP should describe "housekeeping" procedures. Personnel must

communicate to management should they recognize limits of the LEAD facility that hamper the progress of producing quality data. As with all other observations and findings, management should ensure that this communication is documented. Issues or concerns regarding the facility are reported to technical services personnel assigned to track and correct facility issues. They then record the information into a ***facility tracker application*** and contact whoever is necessary to correct the problem (e.g. DAS, outside contractor, internal maintenance, etc.).

(The laboratory is structurally organized to isolate similar work to be accomplished in general work areas. Personnel working in these areas are trained and familiar with cleaning procedures, sample handling procedures, and instrument requirements. Special care is taken to ensure that cross contamination won't occur. This division helps to ensure that sample integrity and instrument response won't be compromised. For example, clean VOC work shall be performed in a room isolated (separate with separate air system) from organic extractions that use solvents, which have VOC contaminants,. Special care is also taken for the analysis of metals by ICP/MS, methyl mercury and low level mercury, which are located in a separate clean room to prevent contamination.

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

Desk work areas are located outside of 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-0006-SFTY**), which covers the safety procedures necessary for the work performed at the laboratory. Documentation that personnel have read the CHP will be kept in the employee's training file.

Access to the laboratory is controlled by requiring all visitors to report to the PHL receptionist and sign in. The receptionist shall page the appropriate personnel, who inform the guest of relevant safety information which may include evacuation routes, and location of work areas.

17.2 ENVIRONMENTAL CONTROLS

Where applicable, test method SOPs shall specify requirements for controlling the environment of the laboratory. The laboratory will 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 filter weighing room is monitored and documented, this process is discussed in the DEQ SOP *High Volume Particulate Mass – PM10* (**DEQ04-LAB-0024-SOP**). The records are maintained in the Inorganics Section

Technical Services maintains sample storage refrigerators as well as freezers located in the lab sections for which the temperature is recorded daily.

18 TEST METHODS AND METHOD VALIDATION (V1M2: 5.4)

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 finalized edition of a standard method 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.

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, project coordinators, and management. 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.

18.1 METHOD VALIDATION

Prior to using a procedure, all test methods, whether published or DEQ developed or modified, must be validated through completion of an *Initial Demonstration of Capability* (IDOC) study to ensure it is suitable for its intended use. IDOC method validation studies are documented using **DEQ08-LAB-0001-FORM** and the summary of the IDOC data shall be filed with the original SOP for the method.

18.1.1 Initial Demonstration of Capability

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. For chemical analyses, the initial test method evaluation involves the determination of the limit of detection (LOD), confirmation of the limit of quantitation (LOQ), an evaluation of precision and accuracy, and an evaluation of the selectivity of the method. The precision and accuracy evaluation consists of the preparation and analysis of 4 *Laboratory Control Samples* (LCS) with the average recovery and RPD within the acceptance limits as described in the referenced analytical method and/or analytical SOP. After the initial validation is complete, the suitability for the method's intended use is described in the *Scope and Application* section of the method SOP.

Personnel must also conduct an IDOC study upon completing their training before performing an analytical method.

18.1.2 Continuing Demonstration of Capability

A *Continuing Demonstration of Capability* (CDOC) is required at least annually to demonstrate the analyst's continued proficiency to perform the test methods. A CDOC for an individual primarily consists of an evaluation of control charts for the analytical methods performed by each analyst. This provides an easy to read visual display of quality control performance over time. Any significant trends can be identified and corrective action implemented if needed. If corrective action is needed, the CDOC can be re-established by the successful analysis of a proficiency test sample (PT) or by following the same precision and accuracy check procedure as described for an IDOC. CDOC's are tracked on a spreadsheet to ensure all of the methods and analysts have been reviewed at least annually. For test methods that are not performed frequently, PT studies (blind studies to the analyst) are used for the CDOC or SRMs can be utilized within each batch to ensure analyst and method performance. The CDOC procedures are verified to comply with the current TNI standards during the annual internal audits.

The review of the control charts is accomplished in the ELEMENT® LIMS system by selecting the testcode(s) of interest, the matrix, and a date range to be reviewed. Once the query has run the chart will display. The charts can be reviewed based on specific analysts or extraction chemists or reviewed as an entire section. The charts are analyte specific and to be an acceptable demonstration of capability at least 90% of the LCS (or SRM) analytes within a method must be within control limits 90% of the time unless there are documented circumstances to indicate outliers. Any observed trends are noted and corrective action is initiated. Also see Section 22.2.1 for a discussion on control charts.

The review of the CDOCs (via control charts, PTs, or another IDOC) is documented and tracked in a spreadsheet.

18.1.3 New Methods

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 communication between analytical staff, management, and the QAO. The communications (e.g. email. meeting minutes, etc.) shall become part of the SOP control document file. The communications shall discuss the process and progress of the method under development. 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.

Method validation consists of evaluations the data quality indicators of the sensitivity, precision, accuracy, selectivity and comparability, and measurement uncertainty.

18.2 SENSITIVITY

Sensitivity is the capability of a method or instrument to discriminate between measurement responses representing different levels (e.g. concentrations) of a variable of interest (TNI 2009). Sensitivity is expressed as qualitative identification at the Limit of Detection (LOD) or quantified identification at the Limit of Quantitation (LOQ).

18.2.1 Limit of Detection (LOD)

The LOD is an estimate of the minimum amount of a substance that an analytical process can reliably detect. An LOD is analyte and matrix-specific and may be laboratory-dependent. See technical definition under Method Detection Limit. LODs are determined from a quality system matrix using all sample processing steps, and are verified annually or when a change in the test method or instruments affects sensitivity. The basic process for the determination of the LOD is to take a minimum of seven (7) sample aliquots, containing low levels of target analyte, through the entire analytical process and multiply the standard deviation of the aliquots by the appropriate statistical Student t value. For specific details, see SOP **DEQ06-LAB-0015-SOP** *Determining the LOD &LOQ* for the specifics on the procedure.

If an LOD study is not performed, concentrations less that the Limit of Quantitation are not to be reported.

18.2.2 Limit of Quantitation (LOQ)

The LOQ is an estimate of the minimum amount of a substance that can be reported with a specified degree of confidence. As a general rule where the method utilizes a calibration curve, the LOQ is greater than or equal to the lowest calibration standard. In all cases the LOQ is greater than or equal to the LOD.

The LOQ is verified using a quality systems matrix sample spiked at 1-2 times the determined LOQ that returns a concentration within the acceptance criteria for accuracy, according to the requirements of the method or client data quality objectives. For specific details, see SOP **DEQ06-LAB-0015-SOP** *Determining the LOD &LOQ* for the specifics on the procedure.

18.3 PRECISION AND ACCURACY

Precision is a measure of the scatter of the data when more than one measurement is made on the same sample. Significant differences in precision can be measured depending on when the sample was split.

To clarify these differences the DEQ lab has adopted duplicate and replicate terms as defined in the glossary of Standard Methods 20th Edition 1010C:

- *"Duplicate - usually the smallest number of replicates (two) but specifically herein refers to duplicate samples, i.e., two samples taken at the same time from one location."* Within LEAD there are two types of sample duplicates, a laboratory duplicate and a field duplicate
    - *Laboratory Duplicate (DU)*: a second separate sample aliquot from the same sample container processed as a unique sample.
    - *Field Duplicate (FD)*: a second sample taken from the same sampling location simultaneously or within 15 minutes of the original sample.
- *"Replicate - repeated operation occurring within an analytical procedure. Two or more analyses for the same constituent in an extract of a single sample constitute replicate extract analyses."*

Precision is commonly attributed to sampling activities and/or chemical analysis. FD samples are collected in the field to assess precision attributable to sampling activities. DU samples are processed in the laboratory to assess data variability attributed to lab sample handling and analysis. Replicate analyses are performed with some tests to assess data variability attributable to instrumental fluctuations. Matrix spike/matrix spike duplicates (MS/MSD) are used on analyses where contaminants are not routinely detected. Matrix spike/matrix spike duplicates are performed at the same frequency and control criteria as lab duplicate analyses. Precision will be expressed as the relative percent difference.

The routine precision criterion for most methods is ±20% relative percent difference (RPD) between laboratory duplicate and 30% RPD for field duplicate samples (except air FD $\leq$ 20% RPD) however the specific criteria is specified in the analytical SOPs or the project QAPP. These criteria may not be achievable at very low concentrations (i.e. if either sample or duplicate is less than 5X LOQ) and in this case precision is determined by absolute range of duplicate analyses of $\pm$ 1X LOQ difference between sample and DU result and ± 2X LOQ for FD result. Field duplicates are collected at a ten-percent frequency (unless specified otherwise in the project QAPP) and are used as a quality control check on the overall monitoring system. The QA Chemist reviews field duplicate precision and a data qualifier is added to the FD results when poor precision is obtained. The project manager assesses the FD results and will qualify associated samples in the final report if needed.

From replicate analyses the absolute Relative Percent Difference (RPD) is used to assess the precision of the analytical method. It is calculated using the equation:

To calculate the RPD between 2 results (MS and MSD or a sample and sample duplicate):

Equation 1

$$RPD = \frac{|Xs - Xr|}{\frac{(Xs + Xr)}{2}} \times 100$$

Where:

X_s = result for the sample and

X_r = result for the replicate/duplicate sample. The units of X_s must be the same as those of X_r.

Control limits are calculated by multiplying the average RPD by 3.27, which represents the 99% confidence limit. The average RPD is calculated using the previous year's data or a minimum of 20 data points. Outliers are excluded from the data set using *Dixon's Q-test or the Grubbs' test*. A maximum of ten percent of the data can be excluded from the data set using this procedure.

Note: There may be specific instances where a data user may want to see bias reflected in the RPD calculation. In these cases, the RPD calculation can be modified by simply showing the actual difference between (Xs-Xr) and not using the absolute value of the difference. In these cases it is critical to always use the original fraction as the primary reading (Xs) and the duplicate fraction as Xr. (e.g. Air duplicates reported to EPA, comparing field and lab results).

Accuracy is a measure of the difference between observed test results and true sample concentration. Inasmuch as true concentrations are not known, accuracy is inferred from recovery data determined by the analysis of calibration verifications standards, laboratory control samples (LCS), sample matrix spikes (MS) and/or the analyses of reference standards. QC reference standards will be analyzed to determine accuracy for those analyses on which sample spiking cannot be performed (e.g. Turbidity, pH).

Most referenced methods specify control limits for calibration accuracy and laboratory control sample accuracy. The laboratory will use historical limits in the cases where the method does not specify. The limits used for each method are spelled out in each SOP.

Accuracy (Percent Recovery-LCS, CCV, Surrogates) is calculated using the following equation:

Equation 2

$$\%R = \frac{AV}{TV} \times 100$$

Where

R = % Recovery
AV = Analyzed Value
TV = True Value

Accuracy (Percent Recovery - Matrix Spikes) is calculated using the following equation:

The percent recovery calculation for matrix spikes is essentially the same as the calculation shown above except that:

Equation 3

$$AV = S_p - S_a$$

Where:

S_p = Spike result

S_a = Sample result

More accurate control limits are established by averaging the percent recovery from the previous year's data or a minimum of 20 data points. Outliers are excluded from the data set by the use of the *Dixon Q-test or Grubbs' test for outliers (*Appendix F*).* A maximum of ten percent of the data may be excluded from the data set using this procedure. Warning and Control Limits are set as follows:

Warning limits = $A(avg.) \pm 2s_x$

Control limits = $A(avg.) \pm 3s_x$

Where: s_x = standard deviation of the mean of the data set.

18.4 SELECTIVITY

Selectivity is the capability of a test method or instrument to respond to a target substance or constituent in the presence of non-target substances (EPA-QAD). Put another way, the ability of a method to separate and detect target analytes from non-target analytes and the matrix. Some of the common means

for separating and identifying target analytes from non-target constituents are: extractions (separate), digestions (separate), interelement corrections (separate), use of matrix modifiers (separate), retention times (separate and identify), confirmations with different columns or detectors (separate and identify), specific wavelengths (identify), specific mass spectra (identify) and specific electrodes (separate and identify).

18.5 COMPARABILITY

The objective of this parameter is to assure that data generated are either directly comparable, or comparable with defined limitations, to literature data or other applicable criteria.

Method comparability of the data is maintained by using procedures that have been accepted or otherwise approved by EPA, or EPA guidance when no approved procedure is available. Comparability with national standards is demonstrated through the analysis of PT Samples (**Section 22.3**). Analytical methods used by DEQ Laboratories are listed in Appendix G .

18.6 ESTIMATION OF UNCERTAINTY OF MEASUREMENT

With adoption of the 2009 TNI standards, the DEQ Laboratory is required to establish a system for estimating measurement uncertainty where the laboratory must report the uncertainty of the measurement should the client request it. The uncertainty requirements should be spelled out in a QAPP, or in the case where the uncertainty affects compliance to a specification limit (e.g. a permit or regulatory action level), in the program specifications.

When estimating measurement uncertainty, all uncertainty components which are important in a given situation shall be taken into account using appropriate methods of analysis. In most cases, the control limits set on the laboratory control sample will be the estimate for uncertainty attributable to human and analytical errors. The laboratory control sample is used as the basis for analytical uncertainty as it incorporates all of the steps in the analytical process.

Table 18-1 illustrates where sources of error contribute to the total uncertainty of any measurement and how the laboratory addresses some of them. Uncertainty calculations from the laboratory only incorporate the analytical and human error components shown in Table 18-1. However, data may be still be qualified if there are components of field analytical QC which do not meet the criteria. Sample integrity components noted in Table 18-1 are non-quantifiable as to their affect on any analytical results.

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.

Table 18-1 Control Measures for Sources of Error Comprising Overall Uncertainty

Instrument and analytical error	Sampling error	Sample integrity error	Human error
Traceable Reagents Section 20	Record location, time and distance from the surface which the sample was collected	Protocols for Preservation, shipping, and storage Section 21	Define minimum technical competencies Section 16
Method Validation Section 18	Record excursions from plans	System for uniquely identifying samples and the appropriate analyses Section 21	Train Personnel Section 16
Equipment Calibration Section 19	Describe sample collection procedure in SAPs	Accomodation and environmental conditions Section 17	Measure and Monitor Competence Section 15
	Describe sample representativeness in QAPPs		Data Integrity Training Section 16.1

18.7 CONTROL OF DATA

Personnel shall maintain all 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 all analytical and computer systems.

All calculations and all relevant data are subject to appropriate checks in a systematic manner.

- The laboratory assures that computers and software are protected, maintained, and secure. The computer servers are maintained in a locked room with restricted access. All staff have unique passwords and instrumentation software and the ELEMENT® system have activated audit trails to record changes. Tape backups are prepared on a regular basis to ensure no data loss.
- Commercial off-the-shelf software (e.g. word processing, database and statistical programs) used within the designed application range is considered sufficiently validated when in-house programming is not used.

- The laboratory assures that computers, user-developed computer software, automated equipment, or microprocessor based systems used for the acquisition, processing, recording, reporting, storage, or retrieval of environmental test data are:

    a. documented in sufficient detail and validated as being adequate for use

    b. protected for integrity and confidentiality (within the confines of public agency requirements) of data entry or collection, data storage, data transmission and data processing.

c. maintained to ensure proper functioning and are provided with the environmental and operating conditions necessary to maintain the integrity of environmental test data; and

d. held secure; including the prevention of unauthorized access to, and the unauthorized amendment of, computer records

- Transcription and calculation errors are minimized through data review and through periodic review of data reduction processes. Quality control results are reviewed by the analyst and by authorized personnel (such as the supervisor or peer). The results are evaluated for consistency, trend, or feasibility before data are released to the client. See **Section 22.4** *Data Review*
- Manual integrations and any analytical notes are reviewed for integrity and justification prior to the release of data. See **SOP DEQ09-LAB-0003-SOP**, *Manual Integration Practices*.

19 EQUIPMENT AND CALIBRATIONS (V1M2: 5.5)

19.1 GENERAL REQUIREMENTS

The laboratory maintains all of the necessary equipment required for the correct performance of the scope of environmental testing presented in this *Quality Manual.* Table 19-3 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/or by following the manufacturer's troubleshooting and maintenance guidelines, and calibration schedules.

Before being placed into service, equipment (including that used for sampling and measurement of field parameters) shall be calibrated or checked to establish that it meets the specification required for its use. No equipment shall be placed into service without first verifying its suitability and accuracy.

Records of this initial verification and the ongoing maintenance performed on equipment shall be recorded in controlled maintenance/calibration logs (refer to **Section 14** *Control of Records***)**.

Some equipment such as class-A 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 the equipment/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.

Equipment is only operated by authorized personnel that have been trained to use the equipment. The equipment manuals must be available to the staff.

Maintenance of analytical instruments and other equipment may include regularly scheduled preventive maintenance, maintenance on an as-needed basis due to instrument malfunction, or service calls and is documented in Instrument Maintenance Logs, which become part of the laboratory's permanent records. Routine maintenance schedules are included in method SOPs and/or the maintenance logs. See also **Table 19-2** and **Table 19-3**

All equipment that affects the quality of test results are calibrated according to the minimum frequency suggested by the manufacturer, by regulation, by method, or as needed. Calibration procedures must be included in the test method SOPs. Calibration requirements are divided into three parts:

- requirements for analytical support equipment,
- requirements for standardizing reagents used for calculating concentrations, and
- requirements for instrument calibration, which is further divided into:
    - Initial instrument calibration and

- Continuing instrument calibration verification

19.2 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 equipment information, including instrument identification, verifications of performance (calibration data), and Quality Control checks (See Table 19-1) as well as the maintenance activities discussed above shall be recorded in appropriate logs. All of these logs shall be controlled as described in **Section 14** *Control of Records***.**

Support equipment shall be calibrated (or verified) over its entire range of use at least annually, using NIST traceable references when available. 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 support equipment performance.

Prior to use on each working day balances must be calibrated with external working weights. The accuracy of the working standards must be verified annually with NIST certified calibration weights.

The temperature of ovens, refrigerators, freezers, and water baths shall be checked in the expected use range, with calibrated thermometers. Working thermometers must be calibrated annually with NIST traceable thermometers. The acceptability for use or continued use shall depend on the needs of the intended analysis or application. The Technical Services Section performs the function of ensuring that thermometers currently in use are calibrated for use in sample storage refrigerators and freezers. The analyst conducting temperature controlled test methods must ensure their working thermometers are calibrated annually.

Mechanical volumetric dispensing devices including burettes (except Class A glassware) shall be calibrated quarterly. If these devices are not to be used during the quarter it is not necessary to check their accuracy, but they must be tagged as out of service. Refer to SOP **DEQ07-LAB-0011-SOP** for the current calibration procedures of Pipettes, Burettes and Micro-liter Syringes.

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.

Table 19-1 Support Equipment QC Measures

Equipment	QC Measure	QC Frequency
Balances	Verify calibration of balance using secondary mass standards.	Each day of balance use prior to use.
Secondary Mass Standards for support balances	Verify against NIST calibration by service contractor.	Following balance certification.

Equipment	QC Measure	QC Frequency
Secondary Mass Standards for balances used for gravimetric analysis.	Verified against NIST traceable mass standards.	Annually.
Primary Mass Standards	Certified against NIST traceable mass standards by contractor.	Once every five years.
Temperature controlled devices: Drying Ovens Refrigerators Freezers Water baths Hot blocks Incubators Etc.	Verify Temperature of working thermometers (e.g. digital displays, bulb thermometers, dial thermometer) against a primary thermometer.	Daily when in use.
Ovens used for gravimetric analysis TS, TSS, and TDS PFO Oil and Grease	Verify oven temperature display is within analytical method defined control limits.	Each day of oven use.
Primary Thermometers	Recertified as NIST thermometer	Every 2-5 years based on vendor certificate of verification
Working thermometers	Verified against Primary thermometer	Annually
Micro pipettes	Verify volume delivery by weight.	Quarterly
Non-Class A Burettes	Verify volume delivery by weight.	Quarterly

Table 19-2 Support Equipment Routine Maintenance

Instrument	Routine Maintenance	Frequency
Balances	Clean pan and check if level Field service by trained technician	Daily At least annually
Drying Ovens	Temperature adjustments	As required
Refrigerators/ Freezers	Warning system checked Temperature adjustment Defrosting/cleaning	Monthly As required As required
Vacuum Pumps/ Air Compressor	Drained Belts checked Lubricated	Weekly Monthly Semi-annually
BOD Incubator	Cleaning	As needed
Centrifuge	Check brushes and bearings	Every 6 months or as needed
Water Baths	Water replaced	Monthly or as needed

19.3 ANALYTICAL EQUIPMENT

This section of the LQM lists and describes the use of equipment required to perform the analytical methods listed in Appendix G . As stated above, maintenance of analytical instruments may include regularly scheduled preventive maintenance, maintenance on an as-needed basis due to instrument malfunction, or service calls and is documented in instrument maintenance logs. The table below lists some common routine maintenance performed by the laboratory for the common instrumentation. Appendix G lists the major equipment used by LEAD.

Table 19-3 Analytical Equipment Routine Maintenance

Instrument	Routine Maintenance	Frequency
AA (Graphite Furnace)	Clean lens and furnace head Replace windows Check or change cuvette Check & drain compressor drain Clean atomizer cell/furnace hood Nebulizer cleaned/dried Check/change marble stones Clean filters Change graphite tube/platform Empty waste container Remove carbon tube and check wear Check sample introduction probe	Daily As required Daily Daily Daily Weekly or as required Weekly Weekly As required Daily Daily Daily
Mercury Analyzer	Check tubing for wear Fill rinse tank with 10% HCl Insert clean drying tube filled with Magnesium Perchlorate Fill reductant bottle with 10% Stannous Chloride	Daily Daily As needed Daily
ICP	Check pump tubing Check liquid argon supply Check fluid level in waste container Check filters Clean or replace filters Check torch Check sample spray chamber for debris Clean and align nebulizer Check entrance slit for debris Change printer ribbon Replace pump tubing	Daily Daily Daily Weekly As required Daily Monthly Monthly Monthly As required As required
ICP/MS	Check pump tubing Check liquid argon supply Check fluid level in waste container Check filters Clean or replace filters Check torch Check sample spray chamber for debris Clean and align nebulizer Check entrance slit for debris	Daily Daily Daily Weekly As required Daily Monthly Monthly Monthly

Instrument	Routine Maintenance	Frequency
	Change printer ribbon Replace pump tubing Pump oil-level check Pump oil changing Check cones	As required As required Monthly Semi-annually As required
UV-Vis Spectrophotometer	Clean ambient flow cell Precision check/alignment of flow cell Wavelength verification check	As required As required Semi-annually
Auto Analyzers	Clean sampler Check all tubing Clean inside of colorimeter Record manifold temperature Clean pump well and pump rollers Clean wash fluid receptacle Oil rollers/chains/side rails Clean optics and cells	Daily Daily Daily Daily Quarterly Weekly Weekly Quarterly
IR Spectrophotometer	Clean cell Check/adjust cell alignment	Annually As required
GC/MS	Ion gauge tube degassing Pump oil-level check Pump oil changing Analyzer bake-out Analyzer cleaning Resolution adjustment	As required Monthly Semi-annually As required As required As required
Gas Chromatograph	Compare standard response to previous day or since last initial calibration Check carrier gas flow rate in column Check temp. of detector, inlet, column oven Septum replacement Glass wool replacement Check system for gas leaks with SNOOP Check for loose/fray wires and insulation Bake injector/column Change/remove sections of guard column Replace connectors/liners Change/replace column(s)	Daily Daily via use of known compound retention Daily As required As required W/cylinder change as required Monthly As required As required As required As required
Electron Capture Detector (ECD)	Detector wipe test (Ni-63) Detector cleaning	Semi-annually As required

Instrument	Routine Maintenance	Frequency
Flame Ionization Detector (FID)	Detector cleaning	As required
Photoionization Detector (PID)	Change O-rings Clean lamp window	As required As required
HPLC	Change guard columns Change lamps Change pump seals Replace tubing Change fuses in power supply Filter all samples and solvents Change autosampler rotor/stator	As required As required Semi-annually or as required As required As required Daily As required
Conductivity Meter	Conductivity cell cleaning	As required
Turbidimeter	Check light bulb	Daily, when used
Deionized/Distilled Water	Check conductivity Check deionizer light Monitor for VOA's System cleaning Replace cartridge & large mixed bed resins	Daily Daily Daily As required As required
pH/Specific Ion Meter	Clean electrode	As required

19.4 INITIAL & CONTINUING INSTRUMENT CALIBRATION

Initial instrument calibration and continuing instrument calibration verification are an important part of ensuring data of known and documented quality.

It should be noted that typically all procedures the lab performs are developed from reference test methods (EPA, Standard Methods, ASTM, USGS, etc) 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 “zero w/ single point” calibration techniques for the technology and the promulgated test method cites the technique; or

- the method employs standardization with a single standard (e.g. titration techniques, HCID).

When using multipoint calibrations, the analytical results must be calculated from the initial instrument calibration using one of these three approaches (note: some methods incorporate the use of internal standards):

- linear regression (the preferred option);
    - all target compounds have an average response factor RSD < 20%
    - or linear correlation coefficient (r) greater than 0.995
- 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 valid results. Appropriate use of second order regressions must be documented in the SOP. Third order regressions and greater are not allowed.

In each case raw data from the instrument and the knowledge of the standard concentrations are used in the statistical method of least squares (weighted or unweighted) or average response factor to create a calibration curve. The analyst shall record and determine QC control limits for the calibration curve coefficients. Test method SOPs shall provide the control limits for the coefficients.

Unless otherwise stated in the analytical method, for chromatography methods, it is DEQ LEAD policy to use at least five calibration standards (at least six points for non-linear curves) dispersed evenly over the range of analysis for the multipoint calibration curve. The test method SOP shall specify the standard concentrations to be used. In the event that circumstances do not allow for the proper calibration, the analyst must follow the corrective action procedure **(Section 12.5)**. Any results generated from a non-compliant calibration must be clearly qualified on the final report.

For non-chromatographic instrument methods, with the exception of those test methods that allow for a single point calibration, at least three calibration standards (not including the calibration blank or a zero standard) shall be used for the initial calibration. If the calibration range is to extend over an order of magnitude, a minimum of five calibration standards are required and if the calibration range covers two orders of magnitude use seven calibration standards. The lowest calibration standard shall be equal to or less than program required reporting levels when appropriate and the analytical method reporting limit

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;
- analyst's 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, r^2 or other criteria)

Sample results shall be quantified from the initial instrument calibration and may not be calculated from any continuing instrument calibration verification unless otherwise specified in the documented referenced analytical method.

Corrective actions are performed when the initial calibration results are outside acceptance criteria. Calibration points are not dropped from the middle of the curve unless the cause is determined, justified, documented, and approved. If the cause cannot be determined, the calibration curve is re-prepared. If the

low or high calibration point is dropped from the curve, the working curve is adjusted and sample results outside the curve are qualified.

Sample results that fall outside of the calibration range (above or below) shall be flagged as estimated if they are to be reported.

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 is typically determined by using a dilution of the stock standard that is one to five times greater than the *Limit of Detection* (LOD **Section 18.2.1**). Typically the method reporting limit also refered to as *Limit of Quantitation* (LOQ **Section 18.2.2**) is set to this lowest standard, refer to the laboratory's procedure *Determining the Limit of Detection LOD and Limit of Quantitation* (**DEQ06-LAB-0015-SOP**) for additional details. The LOQ cannot be below the lowest calibration standard.

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 LOQ 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 must be verified immediately following the initial calibration with two second source quality control samples. One CCV must be analyzed at the LOQ 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.

Analytical method SOP's shall describe the procedure for determining the LOQ and LOD of single point calibration analytical methods. Some of these methods may appear to have no lower limit, because of the excellent precision measured at low levels. In such cases the laboratory may set the LOQ based on an acceptable background level of contamination rather than on the LOD. The LQAO and section manager must approve the protocol used to determine the LOQ.

Some methods require an initial calibration to be performed each day prior to running samples. However, for many methods, a new initial calibration shall be prepared whenever there is sufficient change in instrument setup or reagents used to cause a quantitative change in system response, or when QC failures initiate the corrective action procedure from which it is determined the system should be recalibrated.

19.5 OTHER TYPES OF CALIBRATIONS

Some analytical methods do not follow the standard calibration protocols discussed above but still have some type of process to "calibrate" the method (e.g. titration and gravimetric methods). Titration methods require a standardization of the titrant against a solution of known composition. Gravimetric analyses require the balances to be calibrated annually (also see section **19.2** *Support Equipment***)**.

19.6 FIELD EQUIPMENT CALIBRATIONS

The concept for calibration of field analytical methods is essentially the same as with the laboratory methods. The calibration and verification requirements for Field methods are found in the DEQ *Water Monitoring and Assessment Mode of Operations Manual (MOMs)* **DEQ03-LAB-0036-SOP**.

19.7 CALIBRATION VERIFICATION

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. Appendix G shows the ICV criteria for most methods; the SOPs have the most current ICV criteria.

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 flagged and reported as estimates with comments explaining the problem.

For analytical methods that do not require an initial calibration with every analytical batch, the validity of the initial calibration is verified at the beginning and end (and in some cases, during) of each day of analysis sample analysis by use of a continuing instrument calibration verification (CCV) standard; except for instances when an internal standard is used. For methods employing internal standards, only one verification is required at the beginning of the analytical batch.

Sufficient raw data records are retained to allow reconstruction of all instrument calibration verifications. Instrument calibration verification records connect the verification date(s) to the initial instrument calibration

Each method SOP shall state the frequency and control limits requirements for the CCV. Appendix G shows the CCV criteria for most methods; the SOPs have the most current CCV criteria..

19.8 UNACCEPTABLE CONTINUING INSTRUMENT CALIBRATION VERIFICATIONS

If routine corrective action for continuing instrument calibration verification fails to produce a second consecutive (immediate) calibration verification within acceptance criteria, then a new calibration is performed or acceptable performance is demonstrated after corrective action with two consecutive calibration verifications.

For any samples analyzed on a system with an unacceptable calibration, some results may be fully useable under the following conditions:

a. If the acceptance criteria are exceeded high (high bias) and the associated samples are below detection, then those sample results that are non-detects may be reported as non-detects.

b. If the acceptance criteria are exceeded low (low bias) and there are samples that exceed the maximum regulatory limit, then those exceeding the regulatory limit may be reported.

In other circumstances, the data may still be useable based on project needs.

IN ALL CASES DATA MUST BE QUALIFIED IN ELEMENT® AND ON REPORTS TO REFLECT THE NON-CONFORMING CALIBRATION.

20 MEASUREMENT TRACEABILITY (V1M2: 5.6)

Measurement quality assurance comes in part from traceability of standards to certified materials. All equipment used that affects the quality of test results are calibrated prior to being put into service and on a

continuing basis. These calibrations are traceable to national standards (e.g. NIST) of measurement where available. **Section 19.2** discusses the traceability requirements for support equipment. In methods where the purity of reagents is not specified, analytical reagent grade is used. If the purity is specified in the referenced method, that is the minimum acceptable grade. The "Standards" module in Element ™ is used to maintain records that ensures standards, reagents, and consumables meet quality objectives and are acceptable for use.

Quality control measures for assuring that standards, reagents, and consumables are of the quality necessary to produce accurate data are described in the test method SOPs.

Standards, reagents, and selected consumables receive identifying numbers (Lot# or ELEMENT® ID), which are transcribed to log books or benchsheets. In addition, primary standards used for batch QC are carried through ELEMENT® to link analytical data to the consumable material used to produce the data.

The procedure for documenting standards and reagents information into ELEMENT® is available in the user guide. Once the data is entered into ELEMENT®, a unique identification number is created for the standard, reagent or consumable. ELEMENT® allows the user to input stock standards, working standards, stock reagents, working reagents, and consumables. Information stored in the database includes:

1. Description of the material
2. Preparation date (either by vendor or lab)
3. Prepared by (either vendor or lab staff)
4. Quantity prepared
5. Received date (as well as opened and disposal dates)
6. Preparation Solvent (where applicable)
7. Concentration units (where applicable)
8. Manufacturer name (aka Vendor)
9. Manufacturer lot number (aka Vendor Lot)
10. Whether the standard was purchased or prepared by lab.
11. Type of material (spike, cal standard, reference material, internal standard, tune solution, reagent, surrogate, or "other" for consumables and other miscellaneous material)
12. Comment field to note where applicable: Preparation information, catalog numbers, and other relevant information.
13. Analyte lists and concentrations

20.1 REFERENCE STANDARDS

Reference Standards, such as ASTM Class 1 weights and NIST traceable thermometers, are used for calibration only and for no other purpose unless it is shown that their performance as reference standards will not be invalidated. Reference Standards are maintained (with certificates analysis) by the LEAD for the exclusive purpose of verifying other standards (daily use thermometers and weights).

Reference standards, such as ASTM Class 1 weights and thermometers, are calibrated by an A2LA/NIST accredited vendor who provides traceability to national or international standards. The reference standards are sent out for calibration or replaced based on the expiration dates on the certificates of analysis provided by the vendors. More discussion is available in **Section 19.2** *Support Equipment*.

20.2 REFERENCE MATERIALS

Reference materials, where commercially available, are traceable to national standards of measurement, or to certified reference materials (CRM), usually by a certificate of analysis (COA). Internal reference materials, such as working standards or intermediate stock solutions, are checked as far as technically and economically possible.

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 documentation 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 **Section 7,** *Purchasing Services and Supplies*.

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 test method signatories. 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.

As mentioned above, the analyst shall document reagent preparation. Information recorded into the ELEMENT® shall include the source of the reagent, the mass or volume used, and dilution information. Each 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.

The quality of reagents are verified during each analytical sequence by measuring QC sample performance.

20.3 GLASSWARE, CHEMICALS, AND GASES

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 upon 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 when opened;
- ensure acids and reagents employed with metals analyses are trace or ultra trace grade.
- ensure analytical reagents or solvents are never stored with samples awaiting analysis.

20.4 TRANSPORT AND STORAGE OF REFERENCE STANDARDS AND MATERIALS

The laboratory handles and transports reference standards and materials in a way that protects their integrity and the safety of the staff. Reference standards and materials are stored according to manufacturer's recommendations and separately from working standards or samples.

20.5 LABELING OF REFERENCE STANDARDS, REAGENTS, AND MATERIALS

Reference standards and materials are tracked from purchase, receipt, and storage through disposal.

Expiration dates can be extended if the reference standard or material's integrity is verified. The procedure for recertifying standards is outlined in the *Recertification of Standards* SOP **DEQ07-LAB-0042-SOP**.

All containers of standards, reagents, or materials, whether original or prepared, are labeled with an expiration date.

All containers of prepared standards and reference materials have a preparation date and unique identifier. The identifier is created by ELEMENT®. This identifier is recorded on the appropriate preparation or analytical bench sheets or logbooks to ensure complete traceability throughout the preparation and analytical process.

21 SAMPLE MANAGEMENT (V1M2: 5.7-5.8)

21.1 COLLECTION OF SAMPLES (V1M2: 5.7)

The DEQ laboratory primarily conducts analytical tests on samples collected by the different regions of the agency and by the laboratory sections of Water Quality Monitoring (WQM) and Air Quality Monitoring (AQM). WQM has written the *Mode of Operations Manual (MOM)* - **DEQ03-LAB-0036-SOP** 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 instruct personnel on how to collect representative samples, record data, and when applicable, submit properly preserved samples (including packing, shipping, and documentation) to the laboratory for further analysis. Data recorded during sampling should include the sampling procedure used; the identification of sampling equipment and personnel, field parameters (flow, temperature, etc.), environmental conditions (if relevant), the time sample collection started and stopped, the date, and the sampling location.

The Agency QAO shall maintain the *Field Sampling Reference Guide*, which also provides guidance to personnel outside the laboratory on collecting and submitting samples to the DEQ laboratory. These documents are stored in the document control database, which is described more fully in SOP **DEQ02-LAB-0004-SOP** *Document Control*. A table of general sample container and preservation requirements can be found in Appendix I of the *Field Sampling Reference Guide*, **DEQ86-LAB-0002-QAG**).

LEAD maintains on file, copies of Quality Assurance Project Plans (QAPPs) or Sampling and Analysis Plans (SAPs) as required by the agency's **QMP**. The QAPP or SAP should describe the methods used to collect samples, ensuring the quality of the data and, if appropriate, the information used to develop the sampling procedures. The QAPP shall provide sampling information or the appropriate template for writing a SAP. SAPs are a subgroup of the larger more encompassing QAPP. There are cases where the QAPP will 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. All appropriate personnel are advised of SAPs based on the SAP Distribution list and all staff shall have access to SAPs through Q-Net.

21.2 SAMPLE HANDLING (V1M2: 5.8)

During the process of sample receipt and storage, uncertainty can potentially be introduced through human error and degradation of sample integrity. DEQ sample handling protocols are designed so as to not invalidate the sample or adversely affect the required quality of any measurement.

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.

All samples received by the laboratory must be accompanied by a completed Chain of Custody Form.

21.2.1 Sample Identification

a) **Water and Land Quality Samples**.

Upon sample receipt, the sample custodian shall assign a sampling event (work order) number, generated by ELEMENT ®, to the group of samples that are part of the same sampling event. These are typically received together in a single shipment. However, in some instances a single work order may not be complete with a single day's shipment, and samples for a single work order may be received over multiple days. Each container is inspected to ensure the container identification information matches the chain of custody form that accompanies the submitted samples. Typically, labels are made of a durable plastic material with pre-printed sample identification. The station identifier and location description along with the sampling date and time uniquely identifies each sample/sub-sample received. In certain instances of legal sample or samples for asbestos, the sample container will be given a unique numeric identifier in the laboratory to provide assurance that there is no possibility of any ambiguity in sample identification. The sample custodian logs the station location and sample date/time and relevant sample information into ELEMENT®, which assigns an item number to each sample linking sample, sub-samples and sample location. This unique combination is used in ELEMENT® in order to eliminate confusion regarding the identity of samples at any time. The combined work order 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.

Sample control maintains a supply of new containers that are used and printed labels are used for sample identification.

b) **Air Quality Samples.**

Air samples (filters, modules, cartridges, canisters, etc.) receive a unique identification number during pre-sampling preparation prior to leaving the lab for transport to the sampling location. This number is used on all field and Chain of Custody forms for this sample. Upon sample receipt, the sample custodian shall assign a sampling event (work order) number, generated by ELEMENT®, to the group of samples received together in a shipment. Each container is inspected to ensure the container identification information matches the Chain of Custody form that accompanies the submitted samples. Typically, labels are made of a durable plastic material with pre-printed numbers and/or bar codes. The container number along with the sampling event number uniquely identifies each sample/sub-sample received. The sample custodian logs the sampling event (work order) number and relevant sample information into ELEMENT, which assigns an item number to each sample linking sample, sub-samples and sample

location. This unique combination of numbers is used in ELEMENT® to eliminate 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.

21.2.2 Sample Receipt

ELEMENT® is used to track who received the samples as well as the date and time the samples are 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 laboratory project manager to account for transportation time from the shipper and the time it takes for the sample custodian to log in the sampling event. The project coordinator discusses these details with the QAO during the development of the QAPP.

Upon receipt of water and land samples, the sample custodian fills 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.

Upon receipt of air quality samples, the air sample custodian (typically AQM staff) fills out the “Shipping Report” that will identify the samples, sampling location, shipping temperature, shipping dates, and time and date of arrival at the lab. For samples collected by LEAD staff and immediately returned to the lab (not shipped), the sample collector completes the “Field Sampling” form and Chain of Custody that identifies the samples, sampling dates, location, and time and date of arrival at the lab.

Any abnormalities or departures from acceptable conditions as described in the QAPP or SOPs must be noted on the checklist or shipping report. This checklist may be used in lieu of the Nonconformance forms, however the general procedures in **Section 12** *Corrective Action* should be followed to ensure the proper information is collected and documented. The sample custodian 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 custodian’s *Sample Control* SOP (**DEQ06-LAB-0054-SOP**) 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 custodian’s 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 sample custodian’s *Sample Contol* SOP. Should the sample custodian discover special sample handling instructions at the time of receipt, the sample custodian shall communicate the sample handling deviation to laboratory managers using the proper Nonconformance documentation procedures.

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 custodian shall record the “Legal” status of the sample in ELEMENT®. The sample custodian, sample coordinator, section managers, and the division administrator are the only personnel that have a key card and password to the locked “Legal” storage areas.

21.2.3 Sample Integrity

The sample custodian shall place all samples in specified storage areas. The sample custodian shall ensure that these areas are maintained and the environmental conditions are monitored, and recorded. The sample custodian’s *Sample Control* SOP (**DEQ06-LAB-0054-SOP**) describes DEQ’s sample

acceptance criteria and 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 > 0°C and ≤ 6°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.

   b) PM2.5 filter air samples require specific handling as described in 40 CFR Part 50 10.13. "After retrieval from the sampler, the exposed filter containing the PM2.5 sample should be transported to the filter conditioning environment as soon as possible, ideally to arrive at the conditioning environment within 24 hours for conditioning and subsequent weighing. During the period between filter retrieval from the sampler and the start of the conditioning, the filter shall be maintained as cool as practical and continuously protected from exposure to temperatures over 25 °C to protect the integrity of the sampling and minimize loss of volatile components during transport and storage."

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 is documented on the Sample Receipt Checklist (**DEQ04-LAB-0043-FORM**). Chemical preservation is identified from the sample container type at the time of receipt and the laboratory chemists further confirm sample preservation prior to analysis. All preservation checks will be documented on a Preservation Check sheet which untimatley is placed in the project file Data not meeting preservation control measures are qualified.

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-0057-SOP**).

4) After logging in samples the sample custodian must notify appropriate personnel of the receipt of samples with rush turnaround times or short holding times (for methods with holding times of less than or equal to 48 hours refer to the sample custodian's *Sample Control SOP* **DEQ06-LAB-0054-SOP**). Once notified, chemists should begin these test methods within the cited holding times.

5) Analysts must handle samples in such a manner so as not to compromise the integrity of the remaining sample and their prepared aliquot. Whenever possible the sample must be agitated prior to removing an aliquot to ensure the sample is homogenized. Analysts may encounter special cases where the analysis is to be done on a specific fraction (e.g. the liver of the fish or the solvent layer of a liquid sample). In these cases, the analyst must fully document the process used to prepare their aliquot.

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.

22 QUALITY OF TEST RESULTS (V1M2: 5.9)

In addition to maintaining traceability to NIST (or NIST-like) standards to ensure the production of quality data, the laboratory shall participate in Performance Testing (PT) studies, Inter-laboratory split comparisons, and EPA triennial audits. The QA section has a system in place to track PT sample performance and Technical Services reviews the split data.Performance on audits is tracked with the corrective action database. 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 in **Section 22.4** play 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 laboratory duplicate measurements. The lead or senior chemist/technician shall validate data per sampling event by reviewing sample history, comparing intra-sample results (relationships between associated analyses), and investigating data anomalies. The section manger shall review the comments of the chemists and lead chemist and their decision process when necessary.

Where applicable the chemist shall use statistical techniques to summarize QC data. The chemist shall not rely on other personnel to discover the trends in QC data; chemists shall continually review QC data and document uncovered anomalies or exceeded QC limits.

22.1 ESSENTIAL QUALITY CONTROL PRACTICES

In order to assure the validity of our data, the quality of the analytical process is continuously monitored. Sample handling protocols for storage, preservation and transportation have been developed to preserve the representation of the collected samples. Proper documentation will establish that protocols have been followed and sample identification and integrity are assured. In addition to the instrument calibrations discussed in **Section 19.4**, the analytical process is also monitored with the routine use of both positive and negative quality control measurements (e.g. blanks, laboratory control samples (LCS) or standard reference materials (SRM), matrix spikes (MS), duplicates (DUP), surrogates, and internal standards (IS)). These quality control checks are performed in accordance with the referenced method or regulations to assess precision and accuracy. In addition to the routine process quality control samples, proficiency testing (PT) samples are analyzed 2 times per year (per matrix) to help ensure laboratory performance (exceptions: WS PT studies are analyzed annually, NATTs PTs are analyzed as they are made available). These practices in addition to multiple levels of data review help to ensure the quality of the testing results.

22.2 INTERNAL QUALITY CONTROL PRACTICES

To monitor intra-laboratory performance on a routine basis, the laboratory utilizes both negative control elements (various blanks) and positive control elements (e.g. LCS, SRM, MS, duplicates,). Specific quality control criteria (frequency and acceptance limits) are outlined in each referenced analytical method and/or described in the laboratory method SOP. Quality control needs for water quality field parameters are outlined in MOMs. Transfer blanks, transport blanks and field duplicates will be used to assess field and transport contamination and method variation. Laboratory method blanks will be run on a daily basis. **Appendix G** describes the basic QC practices utilized at DEQ with the type of QC, the frequency it is used and basic corrective actions if it fails. Corrective action for QC failures is to either reanalyze affected samples under acceptable conditions or to ensure the data is appropriately qualified to indicate the failure to the recipient of the final report.

Quality control samples are used to document the validity of data and to control data quality within acceptance limits. Quality control charts are often utilized to assess whether the analytical system is in control; otherwise set limits are used.

22.2.1 Control Charts

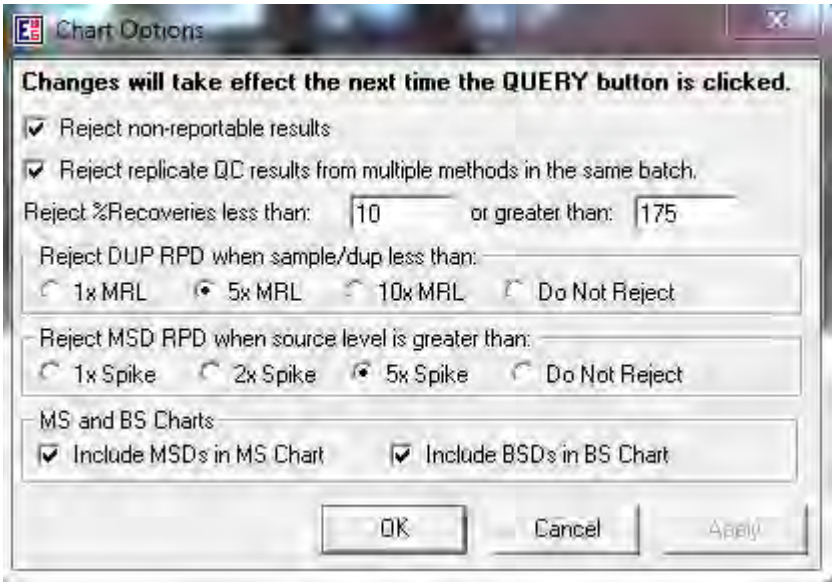
Control charts are a visual means to assess historical laboratory performance of quality control parameters (Blanks, LCS, matrix spikes, duplicates and surrogates) The ELEMENT® LIMs has a control charting function built in that can create a control chart and also calculate limits based on the mean $\pm$ 2, 3, and 4 times the standard deviation. Control limits are usually based on the student-t 99% confidence limit which is 3x the standard deviation though in some cases the 95% confidence limit (2x the standard deviation) may be warranted based on professional judgment may be used.

In ELEMENT®, there is a graphical view as well as a tabular view. One needs to use caution not to over-censor data giving unrealistic narrow control limits as well as not using all possible data points that may lead to unusually wide acceptance limits. In order to group QC results for statistical charting some basic rules must be followed:

- **The spike concentrations and volumes for the QC must be the same.** Method performance is partially a function of concentration and volume so the evaluation of percent recovery must be based on the same concentrations.
- **The sample matrix must be the same for surrogates and matrix spikes.** Method performance is very much matrix dependent so the evaluation of percent recoveries must be based on the same sample matrix.
- **The same sample preparation/extraction method must be used**. Again, method performance is dependent on the preparation methods and only like methods should be combined. In some cases there are more than one preparation method for an analytical technology. Combining data from different preparation methods must be avoided.
- **The instrument must be in proper calibration to statistically evaluate QC samples.** This means that analytes that data points related to calibration abnormalities (represented by CA or CV qualifiers) must be rejected.
- **Internal standard performance affects the performance of target analytes.** If there is indication that the internal standard was not performing correctly, the associated data points must be removed from the statistical data set (rejected). (represented by IS qualifiers)

Additionally, there are many options that are available within ELEMENT® with regards to creating control charts and some basic rules must be followed in order to maintain consistency. Figure 1 shows the option settings that are to be used when creating control charts.

Figure 1 ELEMENT® Control Chart Options



22.3 PROFICIENCY TEST SAMPLES OR INTERLABORATORY COMPARISONS

To demonstrate method performance, the laboratory participates in proficiency test (PT) sample studies 2 times per year or otherwise as required for each method where PT samples are available (exceptions: WS PT studies are analyzed annually, NATTs PTs are analyzed as they are made available). Providers of PT studies must be accredited by A2LA in accordance with the TNI standards. The laboratory participates in the WS, WP, Soil, and NATTs studies. To meet TNI requirements, the laboratory must have two acceptable PT results for each analyte out of the last 3 studies completed. The laboratory institutes corrective action procedures outlined in **Section 12** for any parameter that was outside of the acceptance criteria in a PT sample.

The laboratory does not share PT samples with other laboratories, does not communicate with other laboratories regarding current PT sample results, and does not attempt to obtain the assigned value of any PT sample from the PT provider.

Proficiency testing (PT) samples are treated as typical samples in the normal production process where possible, including the same preparation, calibration, quality control and acceptance criteria, sequence of analytical steps, number of replicates, and sample log-in. PT samples are not analyzed multiple times unless routine environmental samples are analyzed multiple times.

22.4 DATA REVIEW

The laboratory reviews all data generated in the laboratory for compliance with method, laboratory and, where appropriate, client/QAPP requirements. The review is documented using Data Review checklists. A description of the system of routine checks can be found in the LEAD *Peer Review and Record Management* SOP (**DEQ00-LAB-0004-SOP**). Data is qualified if it does not meet method or project requirements. Guidance for lab staff on data qualification can be found in DEQ Guidance document *Data Validation and Qualification* (**DEQ09-LAB-0006-QAG**). Below is the general data review procedure.

1) Chemists/Technicians producing analytical data shall review their work ensuring reporting procedures are followed and sample and batch QC measures meet acceptable limits. They then pass the data packet to another chemist familiar with the test method as a peer review. Chemists

shall follow their SOPs to correct any failed QC and repeat the analysis provided there is sufficient sample. If the analysis cannot 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 due 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:
    1. Matrix Spike
    2. Matrix Spike Duplicate
    3. Duplicate analysis
b. Batch QC measures may include but are not limited to:
    1. Method Blank
    2. Laboratory Control Sample
    3. Continuing Calibration Verification
    4. Calibration coefficients

As noted previously, if a chemist or peer reviewer believes they have identified a questionable procedure or feels the SOP does not meet the policy or procedures detailed in the LQM, they are obligated to bring their concerns forward. They may contact a QAO, their section managers, or the LEAD Administrator.

2) Senior chemist/Lead Chemist/Lead Monitoring Specialists

    a. The lead or senior chemist shall verify data by reviewing sample history, comparing intra-sample contaminants, and investigating data anomalies. The lead chemists shall confer with chemists to evaluate any question they may have about the data and may send the results back to the chemist for rework. This process is controlled through ELEMENT® statuses. The chemist shall verify the transcription of data and calculations from the original source to ELEMENT®, 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.

4) 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. See **Section 15.1** on Internal Audits.

5) The laboratory project mamanger reviews the report as a whole to ensure the project objectives have been met and qualifies sample results where project requirements have not been met and samples are affected (e.g. hits in Field blanks, precision failure in field duplicates). The project coordinator makes the final approval of the report

6) The QAO or designee also audits a percentage of analytical data packets after they have been released for completeness and verifies 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 through the data correction process. If a quality control measure fails, all samples associated with the failed quality control measure shall be re-reported with the appropriate data qualifier(s).

22.5 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 or the QAPP. In the event that data does not meet the requisite QC criteria and samples cannot be reanalyzed, the results will be reported with data qualifiers. By default, data falling outside the default control limits will be qualified to indicate the

failure within ELEMENT® and the QA data quality level of "B" is generally assigned. This is consistent with the usage of the EPA validation "J" qualifier. Data quality levels (DQLs) are used to help the end users of the make decisions regarding the usability of the data for their projects and track the quality of analytical results in DEQs data storage and retrieval online database. This coding system allows the primary and secondary data users to quickly and easily assess data quality using the laboratory's default QC criteria. The DQL of "B" is used to mark data as failing to meet the laboratory's default QC standards or criteria specified in a QA plan. Most data qualifiers in ELEMENT® have pre-established DQLs though some require the analyst or lab project manager to assign a DQL based on the circumstances associated with the qualifier. The data quality level definitions and qualifier usage is discussed in the DEQ guidance document *Data Validation and Qualification* **(DEQ09-LAB-0006-QAG)**. Data users should carefully review data and the associated QC information before using it.

Solids and ion balances are also sometimes used as a data validation tool. Ion balances that do not agree within acceptable control initiate a corrective action process, which may include reanalysis, when holding time and sample quantity permit.

The data user must be able to determine what the data represents. Proper documentation of sampling and analytical protocols will help ensure data users have sufficient information to assess if data are acceptable for their intended use. At the minimum sample collection data will include date, time, latitude, longitude, and elevation with respect to the surface or sea level. Sampling protocols shall define the accuracy to which the time and locations data are to be recorded. Deviations from SOPs must be recorded, transcribed to ELEMENT®, and reported.

The DEQ laboratory has developed QC procedures to ensure the integrity of the sample is maintained during sample handling protocols for transportation, preservation and storage. In the event the QC procedures disclose a potential sample handling problem all associated results will be qualified and the DQL is set to "B" or "C" and the data users will be warned that the data may not be representative of the intended sample. If circumstances warrant, the sample result will be voided.

LEAD does not provide detailed data quality assessment services to the agency by default. Generally, project managers sending samples to the laboratory shall be responsible for the statistical validation of data for their projects. Laboratory and regional project managers submitting samples to the lab should first identify their data quality objectives (e.g. verifying a null hypothesis) and establish the appropriate statistical approach for assessing the data set. Laboratory assistance with data quality assessments can be arranged between the project manager 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. Batch QC data is supplied as part of the analytical report so one can more readily evaluate the effectiveness of the project.

DEQ project managers also evaluate the data quality indicators of representativeness and completeness before the report is released.

22.5.1 Representativeness

Representativeness is a measure of how closely the observed test results on the sample matrix reflects the actual site conditions.

Sampling procedures must be designed so results represent the true nature of the environment from which the sample was collected. Sample handling protocols for storage, preservation and transportation have been developed to preserve the representation of the collected samples. Proper documentation will establish that protocols have been followed and sample identification and integrity assured. Transfer blanks, transport blanks and field duplicates will be used to assess field and transport contamination and method variation. Laboratory method blanks will be run on a daily basis.

Tests that may describe the sample matrix (% solids, density, particle size, and other physical parameters) may be requested for analysis for a specific project. Special sample conditions may be present which effect method accuracy.

22.5.2 Completeness

Completeness is a measure of the amount of valid data obtained from the analytical measurement system for a given project compared to the amount that was expected to be obtained. It is defined as the total number of samples taken for which valid analytical data are obtained divided by the total number of expected samples and multiplied by 100.

23 REPORTING OF RESULTS (V1M2: 5.10)

The laboratory shall report data in useful and comparable formats.

The in-house project coordinators within LEAD have the responsibility of generating final analytical reports. They shall ensure that the projects entered into ELEMENT® and assigned a work order (sampling event) number are generated an official report record that is sent to the DEQ project manager. 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.

23.1 SIGNIFICANT FIGURES AND ROUNDING

As described in the **Section 22.4** Data Review an analyst shall typically review data for transfer and calculation errors prior to completing their work in ELEMENT®. The analyst shall ensure numeric results are transcribed into ELEMENT® with the appropriate significant figures, and sensitivity. The analyst shall round numerical results such that the value does not imply accuracy greater than the limit of quantitation (LOQ) (refer to **DEQ06-LAB-0015-SOP**). The number of digits necessary to express the result of a measurement must be consistent with the measured method precision. During method development the precision measured shall be noted to determine the significant figures. The analytical SOP's method validation section should define the allowable significant figures (normally 3). When the digit immediately following the last digit to be reported is a "5", the analyst shall round significant figure up if it is odd and down if it is even. To illustrate possible scenarios of rounding refer to Table 22-1 and 22-2 below.

Table 23-1 LOQ Rounding Rule

LOQ[1]	Lowest/Highest Cal. Std.	Measured value	Round to:	Report	Flag
0.005	0.005 / 0.4	0.0025	0.002	<0.005	Less than LOQ
0.005	0.005 / 0.4	0.33456	0.334	0.334	
0.005	0.005 / 0.4	0.33450	0.334	0.334	
0.005	0.005 / 0.4	0.33350	0.334	0.334	
0.005	0.005 / 0.4	0.33349	0.333	0.333	

Table 23-2 LOD Rounding Rule

LOD	LOQ	Lowest/Highest Cal. Std.	Measured value	Round to:	Report	Flag
0.0017	0.005	0.002 / 0.4	0.00115	0.0012	<0.0017 (LOD)	
0.0017	0.005	0.002 / 0.4	0.00166	0.0017	0.0017J	
0.0017	0.005	0.002 / 0.4	0.00494	0.0049	0.0049J	

[1] The lowest calibration standard must be at or below the LOQ (TNI 2009 V1:M4 1.7.1.1(f).).

0.0017	0.005	0.002 / 0.4	0.00505	0.0050	0.005	

Note: Rounding rules do not apply to commercially available software. If the software has a built in rounding routine (e.g. always rounds up or always drops the last figure) it will be used as is. The lab staff will not go back and re-round results according to the rules.

Non-detects are generally reported as ND at the LOQ. However there are projects where the results are reported to the LOD, in these casesm non-detects are reported as ND at the LOD. If samples are diluted the reported LOQ and LOD must be adjusted by the dilution factor. If sample preparation volumes or masses are different than those used to determine the LOD/LOQ (method validation procedures: **DEQ07-LAB-0018-FORM**), the reported LOQ and LOD must also be adjusted.

All results will be rounded according to the rules and then compared to the LOQ (or LOD).

Summary
DEQ LEAD generally reports results to 3 significant figures with the following caveats

a. Results are not reported to more decimal places than the LOQ.

b. If the results are to be reported to the LOD and the results are between the LOD and the LOQ, do not exceed the number of decimal places in the LOD (still report 3 sig figs where possible) and are qualified with a "J" (see table 23-3).

23.2 REPORTING DILUTIONS AND DETECTION AND QUANTITATION LIMITS

When diluting samples at the time of analysis, the LOQ and LOD as well as the sample result will be adjusted to reflect the amount of the dilution factor.

Example: LOQ = 20; DF =10; sample = ND: Report: <200 LOQ = 200

When adjustments are made to initial preparation weights or preparation final volumes, the LOD and LOQ will be adjusted to reflect the changes in the preparation factor.
Example: LOQ =20; Prep used ½ normal volume; sample ND: Report <40 LOQ = 40

The dilution factor on ELEMENT® reports will reflect the combination of any preparation factors and post preparation dilution factors. In the case where the LOQ is raised because of matrix effects, the samples are qualified with an SA1 "Limit of quantitation raised due to matrix interference."

23.3 REPORTING DEFINITIONS

QA project plans may require that results be reported to a value less than the LOQ. When this occurs, the laboratory applies a "J" next to the analytical result so as to qualify the results since they are estimated. The use of "J" has no impact on the data quality level assigned to a sample result. EDL and LOD are also defined in the analytical report; this can be boiler plated to simplify the process. The use of the "J" flag and the other data qualifiers also carry over to the DEQ online database so they are available to the end data user when running a query. Qualifier definitions can be found in the ***Element Qualifier Usage Guidance*** spreadsheet .

Table 23-3 Data Definitions for Reports and online database

Symbol or Acronym	Definition
<	Analyte concentration is less than the concentration shown

>	Analyte concentration is greater than the concentration shown
J	Sample result is an estimated concentration between the laboratory limit of detection (LOD) and the laboratory limit of quantitation (LOQ)
(LOQ)	Concentration shown is the analyte Limit of Quantitation
(LOD)	Concentration shown is the analyte Limit of Detection.
(EDL)	Concentration shown is the analyte Estimated Detection Limit. (Note: Used only in High Resolution GCMS. This is a sample specific detection limit that is used instead of the typical LOD).

23.4 TEST REPORTS

The DEQ report format has been designed to accommodate each type of test performed and to minimize the potential for misunderstanding or misuse.

Analytical reports shall be generated from ELEMENT® 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. See **Section 23.6** for the actual report content/format.

23.5 SUPPLEMENTAL TEST REPORT INFORMATION

During the data review and validation process, data flags/qualifiers are added when there are any deviations from, additions to, or exclusions from:

- Quality control or other analytical requirements in the DEQ test method.
- QAPP Requirements
- Field sampling procedures (including environmental conditions)
- Sample acceptance/integrity requirements

These qualifiers/flags are included in the final report and/or the information regarding deviations is incorporated into the case narrative by the laboratory project manager. The analyst entering data into ELEMENT® shall report the results associated with failed QC: 1) with the addition of a qualifier which will be defined at the end of the report 2) Assign a the DQL; this is automated in Element for most cases (refer to DEQ guidance document *Data Validation and Qualification* (**DEQ09-LAB-0006-QAG**), and 3) enter a comment in a narrative, if needed to better explain the decision to qualify the data if the definition is not sufficient. Through the data review and approval process management shall scrutinize the data and identify which comments will be reported. ELEMENT® will generate definitions for all of the reported qualifiers and insert them onto the last page of the report.

Note: DQL codes from data validation (section 21.5) appear in the analytical report and are also used to filter database queries. Results associated with a QC failure will appear in the analytical report with qualifier describing the QC failure. Even though project managers may only use data stored in an electronic database, an official paper report is generated for document control and to provide additional information that does not appear is a data download.

Changes to data quality levels in the online database must be included in the narrative if the project manager decides that a change in DQL is warranted based on Field QC parameters unless qualifiers are applied to the affected parameters.

Test reports also may include the following additional information where relevant:

- a statement of compliance/non-compliance when requirements of the quality systems are not met;
- a statement on the estimated uncertainty of the measurement (only if required for a specific project and documented in a QAPP);
- where appropriate and needed, opinions and interpretations will be included in the narrative. When opinions and interpretations are included, the basis supporting the opinions and interpretations are documented. Opinions and interpretations are clearly marked as such in the test report.
    a. The section managers may add to 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. (These are most frequently used in potentially legal cases).

23.6 TEST REPORTS THAT CONTAIN THE RESULTS OF SAMPLING

The following is provided when DEQ-LEAD performs the sampling:

a) the date of sampling;
b) unambiguous identification of the material sampled;
c) the locations of the sampling, (when available, diagrams, sketches, or photographs maintained by the LEAD project managers that are part of the project files rather than the case report);
d) a reference to the sampling plan and procedures used;
e) details of any environmental conditions during sampling that may affect the interpretations of the test results;
f) any standard or other specification for the sampling method or procedure, and deviations, additions to or exclusions from the specification concerned.

23.7 ENVIRONMENTAL TESTING OBTAINED FROM SUBCONTRACTORS

Test results obtained from test performed by subcontractors are clearly identified on the test report by subcontractor name and/or accreditation number.

The test results from subcontractors are entered and/or uploaded into ELEMENT® and subsequently, the online database. The results included in the standard DEQ report with the subcontractor are clearly linked to the analyses they performed. A copy of the subcontractors report is available by request.

23.8 REPORT CONTENT/FORMAT

1) Title page w/
    a) Primary report recipient,
    b) Report date,
    c) Laboratory name and address
    d) Approval signatures.
2) Narrative page w/
    a) Sampling event narrative when present,

b) List of report recipients,
c) List of sample collectors, and
d) List of analytical laboratories involved in the analyses.

3) Sampling Event Summary page w/
   a) Project ID linking the sampling event to the QAPP,
   b) List of sampling sites w/
      i) Item number,
      ii) Site (Station) ID,
      iii) Sample description,
      iv) Matrix,
      v) Sample date,
      vi) Sample time, and
      vii) Endnote references
4) Analytical Results page(s) w/
   a) Sample identification w/
      i) Sample ID,
      ii) Sample type
      iii) Site ID,
      iv) Sample description,
      v) Sample date, and
      vi) Sample time
   b) Analytical parameter w/
      i) Analytical laboratory if different than the DEQ laboratory,
      ii) Method reference,
      iii) Limit of quantitation (LOQ),
      iv) Result,
      v) Reporting units,
      vi) Date and time of sample preparation for test methods with holding time less than 72 hours, and
      vii) Data qualifiers
5) Data qualifier definitions
6) Scanned field sample collection forms of initial recordings
7) Scanned chain of custody

The Technical Services section shall include the following original forms and reports in the official analytical report file, although they are not normally included in the electronic data file.

1) The original sample collection form
2) Analytical Section QA Review and Manager Review report, Peer review checklists, preservation check reports, data comparison reports
3) Subcontracted analytical reports

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

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

As noted previously Water Quality Monitoring and Air Quality Monitoring have documented procedures for collecting samples. References to these procedures shall be included in analytical reports where appropriate.

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. ELEMENT® allows the sample custodian 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.

Technical Services shall ensure the integrity of the official report by applying the record management procedures as described in **Section 14.1** Even though project coordinators may only use data stored in an electronic database, Technical Services shall create an official paper report for document control.

APPENDICES

Appendix A Glossary[2]

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. (TNI)
Analytical Uncertainty	A subset of Measurement Uncertainty that includes all laboratory activities performed as part of the analysis.
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 (1) to twenty (20) environmental samples of the same quality systems 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 twenty-four (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 quality system matrices and can exceed twenty (20) samples. (TNI)
Bias	The systematic or persistent distortion of a measurement process, which causes errors in one direction (i.e., the expected sample measurement is different from the sample's true value). (TNI)
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. (TNI)
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.

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

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.
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)
Data Integrity	The result of the processes that together assure valid data of known and documented quality.
Document Control:	The act of ensuring that documents (and revision thereto) are proposed, reviewed for accuracy, approved for release by authorized personnel, distributed properly, and controlled to ensure use of the correct version at the location where the prescribed activity is performed. (ASQC)
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.
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.
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.
Holding Times (Maximum Allowable Holding Times):	The period of time a sample may be stored prior to its required analysis. While exceeding the holding time does not necessarily negate the veracity of analytical results, it causes the qualifying or "flagging" of any data not meeting all of the specified acceptance criteria. (MA DEP) The maximum time that can elapse between two (2) specified activities. (TNI)
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. (TNI)

Method	A body of procedures and techniques for performing an activity (e.g., sampling, chemical analysis, quantification), systematically presented in the order in which they are to be executed. (TNI)
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. (DEQ)
National Air Toxics Trends Station (NATTS)	EPA has established a National Air Toxics Trends Station Network in the contiguous 48 states. The network consists of 22 sampling stations operated by state and/or local agencies. In order to provide an estimate of the quality of data collected in NATTS, EPA has undertaken a Proficiency Testing Program for those laboratories that perform analyses on the collected samples.
National Institute of Standards and Technology (NIST):	An agency of the US Department of Commerce's Technology Administration that is working with EPA, States, TNI, 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)
The NELAC Institute (TNI):	The NELAC Institute (TNI) is a 501(c)(3) non-profit organization whose mission is to foster the generation of environmental data of known and documented quality through an open, inclusive, and transparent process that is responsive to the needs of the community. The organization is managed by a Board of Directors and is governed by organizational **Bylaws** (TNI Website)
National Environmental Laboratory Accreditation Program (NELAP):	The overall National Environmental Laboratory Accreditation Program of which is a part of TNI
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.
Negative Control:	Measures taken to ensure that a test, its components, or the environment do not cause undesired effects, or produce incorrect test results. (TNI)
TNI 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 The NELAC Institute.

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. (TNI)
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. One of any number of samples, such as Certified Reference Materials, a quality system matrix fortified by spiking, or actual samples fortified by spiking, intended to demonstrate that a measurement system or activity is in control. (TNI)
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. (TNI)
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 to be used for purposes of batch and quality control requirements: (TNI)

Air and Emissions:	Whole gas, vapor, or particulate samples including those contained in flexible or rigid wall containers and the extracted concentrated analytes of interest from a gas, vapor, or particulate 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.
Biological Tissue:	Any sample of a biological origin such as fish tissue, shellfish, or plant material. Such samples shall be grouped according to origin.
Chemical Waste:	A product or by-product of an industrial process that results in a matrix not previously defined.
Drinking Water:	Any aqueous sample that has been designated a potable or potential potable water source.
Non-aqueous Liquid	Any organic liquid with <15% settleable solids.
Saline/Estuarine:	Any aqueous sample from an ocean or estuary, or other salt water source such as the Great Salt Lake.
Solids:	Includes soils, sediments, sludges and other matrices with >15% settleable solids.
Reference Materials	Material or substance one or more of whose property values are sufficientlyhomogeneous and well established to be used for the calibration of an apparatus, the assessmentof a measurement method, or for assigning values to materials. (TNI)
Reference Standards	Standard used for the calibration of working measurement standards in a given organization or at a given location (TNI)
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.
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.
Sample – Continuous	The analytical parameters are measured in-situ for a given sample matrix over a period time. The measurements are typically discrete measurements occurring at a set frequency. Data are stored on an electronic medium and transferred to LIMS for processing.
Sample – Grab	The sample matrix is captured in a container or absorbed onto a medium for further analytical analyses. The period of time used to collect the sample shall be defined by the sampling procedure and may range from a few seconds (surface water) to a month (PFO). Grab Samples differ from Temporal Composite Samples in that the Grab Sample is continuously collected.

Sample – Spatial Composite	Fractions of the sample matrix are collected from multiple points within a defined area and are combined together in a single container for further analyses.
Sample – Temporal Composite	Fractions of the sample matrix are collected over a defined period of time and are combined together in a single container for further analyses.
Sampling	Activity related to obtaining a representative sample of the object of conformity assessment, according to a procedure (TNI)
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. (DEQ) equivalent to TNI usage of the term of Technical Manager
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)
Reference Method:	A test method issued by an organization generally recognized as competent to do so. (TNI) ISO uses the term Standard Method to mean the same thing
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.
Traceability:	The ability to trace the history, application, or location of an entity by means of recorded identifications. In a calibration sense, traceability relates measuring equipment to national or international standards, primary standards, basic physical constants or properties, or reference materials. In a data collection sense, it relates calculations and data generated throughout the project back to the requirements for the quality of the project. (TNI)

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.
Verification:	Confirmation by examination and provision of objective evidence that specified requirements have been met. (TNI) NOTE: In connection with the management of measuring equipment, verification provides a means for checking that deviations between values indicated by a measuring instrument of a known quantity (a standard) 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

American Society for Quality Control (ASQC), Definitions of Environmental Quality Assurance Terms, 1996

American National Standards Institute (ANSI), Style Manual for Preparation of Proposed American National Standards, Eighth Edition, March 1991

ANSI/ASQC E4, 1994

International Standards Organization (ISO) Guides 2, 30, 8402

International Vocabulary of Basic and General Terms in Metrology (VIM): 1984. Issued by BIPM, IEC, ISO and OIML

Massachusetts Department of Environmental Protection: *Glossary of Quality Assurance Terminology,* http://www.mass.gov/dep/cleanup/laws/glossa01.htm

National Institute of Standards and Technology (NIST)

The NELAC Institute (TNI), July 2009 Standards

Random House College Dictionary

US EPA Quality Assurance Management Section (QAMS), Glossary of Terms of Quality Assurance Terms, 8/31/92 and 12/6/95

US EPA Quality Assurance Division (QAD)

Webster's New World Dictionary of the American Language

Appendix B Employee Attestation form

By initialing the records listed in the following table, I, _____,

(print employee's name)

attest that I have read and intend to follow the policies and procedures prescribed in the controlled documents to the best of my abilities.

I further attest that I meet the minimum qualification for my current position as defined in Section 15 of the Laboratory Quality Manual.

_____

(employee's signature)

Title	Document Control #	Version	Date completed	Initial	IDOC[i] date
Laboratory Quality Manual	DEQ91-LAB-0006-LQM				NA
Document Control	DEQ02-LAB-0004-SOP				NA
Nonconformance Report-Corrective Actions	DEQ07-LAB-0053-SOP				NA

[i] IDOC: Initial Demonstration of Capability; refer to the Laboratory Quality Manual section18.1.

Appendix C Technical Corrective Action

Technical Corrective Action
SECTION: _____

WA, WQM, 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, data entry, etc. 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 high priority task.**

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

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

		New Result		Data Correction	
Item Number		Date		Bottle	Test Result
Site Name					
Bottle Number					
Alternate Bottle					
Standard Parameter		Initial Result Verified?		Yes	No
Initial Test Result		Reason Code for			
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)

Appendix D Laboratory Audit of Field Measurement Form

Laboratory Audit of Field Measurements

Case No.: _____ **Case Name:** _____

Item Number: _____ **Date/Time Sampled:** _____

Field Data		Laboratory Data					
Analyte, units	Result	Bottle No.	Date	Result	Calculation	Criteria	Pass/Fail
pH, S.U. Meter I.D.: _____	_____					≤ 0.3 Difference	Pass Fail
Alkalinity, mg/L	_____					± 20% RPD ± 2 Difference for values ≤ 10	Pass Fail
Turbidity, NTU Meter I.D.: _____	_____					70-110% recovery ± 3 Difference for values ≤ 10	Pass Fail
Conductivity, µmhos/cm Meter I.D.: _____	_____					± 20% RPD ± 4 Difference for values ≤ 20	Pass Fail
Other, _____ Meter I.D.: _____	_____						Pass Fail

Comments:

Corrective Actions:

When the laboratory audit of a field measurement fails (refer to page 2), all of the samples from that sampling event must be analyzed in the laboratory for the failed parameter, and the sample collectors must be notified as soon as possible of the problem. If the audit sample is associated with more than one sampling event (i.e., more than one case), then all of the associated samples must be analyzed in the laboratory.

Analyst: _____ **Date:** _____

DEQ04-LAB-0044-FORM, Version 2.3/clr

Corrective Action: Analyze a confirmation sample if the laboratory results are outside the limits listed on the form. 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.**

Appendix E Major Analytical Equipment

Organic Analytical Section

- ***GC/MS/DS:***
  - Agilent GC/MS 6890/5975 and HP data system #52535 allocated to volatile analyses (TO-15).
  - Agilent GC/MS 6890/5972 and HP data system #50634. Capillary/packed column GC with Tekmar Atomx Purge Trap Concentrator Autosampler. Allocated to volatile analyses (NWTPH-Gx, 524.2, & 8260).
  - Agilent GC/MS 7890A/5975 #52692. Allocated for Semi-Volatiles (525.2 & 8270).
  - Agilent GC/MS 6890N/5973 #52116. Allocated for Semi-Volatiles (TO-13).
- ***High Resolution GCMS***
  - Waters Autospec Premier HRGCMS # 58239A (PCB Congeners, Dioxins/Furans, Pesticides)
- ***GC:***
  - Agilent 6890 series II; FID/FPD. #51154 (Fuels / NMOC) (NWTPH, 8015).
  - Agilent 6890 series II Plus, dual NPD, HP 3365 Chemstation data system. #52184 (Herbicides and Organophosphorus Pesticides) (8151, 515.4.
  - Agilent 6890 series II; dual ECDs; HP 3365 Chemstation data system #51968. (Chlorinated Pesticides and PCBs) (508, 508A, 8081, & 8082).
  - Agilent 5890 series II; TCD & OI PID/FID; HP 3365 Chemstation data system #50298 (Organic Screening).
- ***HPLC:***
  - Agilent Series 1100 equipped with HP Diode Array UV/Vis and Programmable Fluorescence Detectors with an HP Chemstation: #51202. (Air Toxics) (TO-11).
- ***LC/MS/MS***
  - Waters LC/MS/MS #52475. (Pesticides and PPCP's).
- ***Total Organic Carbon (TOC) Analyzer:***
  - Tekmar/Dohrman Apollo 9000 #51343 for TOC & DOC.
- ***COD Reactors:***
  - 3 X HACH COD Reactors #50503.
- ***Spectrophotometers:***
  - HACH DR/3000 #50503. (COD).
- ***Fluorometer:***
  - Turner Designs TD-700 #51081 for Chlorophyll and Pheophytin.
- ***Sample Preparation***
  - Waters Gel Permeation Clean-up apparatus (GPC) #52536
  - Dionex ASE 200 Accelerated Solvent Extractor #52171
  - Labconco RapidVap # 53023, #53024, # 53025
  - Branson Ultrasonic Bath Model 8510 #R53116
  - Branson Ultrasonic Bath Model 3200 #7918
- ***Analytical Balances:***
  - Mettler AT261 semi-micro #50112

- Sartorius CP225D # 52646
- Sartorius CP3202 # 52802
- Mettler PM 4600 #50123

Inorganic Analytical Section

- ***Atomic Absorption Spectrophotometer (AAS):***
  - Varian AA 220Z GFAA. #52172
  - CETAC M6000 Mercury Analyzer. #50722
  - LECO AMA 254 Mercury Analyzer. #52534

Atomic Fluorescence Spectrophotometer (AFS):
  - Brooks Rand Mercury and Methyl Mercury #52980

- ***Autoanalyzer:***
  - Lachat Flow Injection Ion Analyzer Quikchem 8000 #51144 (Nitrate + Nitrite, Nitrite,)
  - Lachat Flow Injection Ion Analyzer Quikchem 8000 #52146 (TK-N, and TPO4-P modules).
- ***Ion Chromatographs:***
  - Dionex Model 120, autosampler and Peaknet software # 50710 (Chloride, Sulfate, and Nitrate).
  - Dionex 600 upgraded with CDM detector, gradient pump, SRS suppresser, AS40 autosampler, and Peaknet software #52211 (Fluoride, Bromide, DBP) (Anions for Air Filters)
  - Dionex Model 2100, autosampler, Chromeleon 7 software #105946(Ammonia) (Cations for Air Filters)
- ***Inductively Coupled Plasma (ICP):***
  - Simultaneous Perkin Elmer Optima 3000 DV #50638.
  - Simultaneous Perkin Elmer Optima 5300 DV #52880.
- ***Inductively Coupled Plasma Mass Spectrometry (ICP/MS):***
  - Thermo Elemental Model VG PQ Excel #52148
  - Agilent Model 7500 #52881
- ***Spectrophotometers:***
  - Perkin Elmer Model Lambda 20 Spectrophotometer #51104 (ortho-Phosphate and Total Phosphate).
- ***X-Ray Fluorescence Spectrometer:***
  - Thermo-Fisher QuantX ARL EDXRF #105944
- ***Titrations:***
  - Digital Buret for Alkalinity
- ***Analytical Meters:***
  1. **pH and/or ISE**
     - Beckman 660 (3) #52877, #52653, #52662
     - Orion Models: 720A #50462; 4-Star #52658; 3-Star #52659
  2. **Dissolved Oxygen**

- Hach – Luminescence #52858
- YSI – Model 58 #51224

3. Conductivity

- YSI Model 3200 #53121

4. Turbidity

- Hach J100 #50504

5. Chlorine

- Hach Pocket Colorimeter #53057

- ***Analytical Balances:***
  - Sartorius A200SFW #8304
  - Mettler AB265 #52661
  - Ohaus Explorer #52652
  - Ohaus Scout II. #52837
  - Sartorius CP225D (2): #52648, 52796
  - Ohaus Balance, E4000D # 52978
  - Mettler AT261 semi-micro # 52663: Solids (Total, Suspended, and Dissolved).
  - Sartorius MC5 (microbalance) #R53094
- ***Optical Microscope:***
  - Zeiss Standard 18, Polarizing Trinocular #3951 with 35-mm camera #52495
  - Olympus Binocular stereo scope #51162
  - Spencer Binocular stereo scope #3043
  - Olympus CH2 Phase Contrast microscope #50186
  - Olympus BX-51, with Smith Detection System # 52888
  - Leitz (monocular) #1662
- ***Sample Preparation Equipment***

1. **Block Digestor (non-metals)**

- Lachat #52145
- Westco #52473

2. **Hot Block Digestors (metals)**

- Digiprep #52173
- Environmental Express (3): #52210; #52628; #52680

3. Microwave Digestor (metals)

- CEM MARS #52449

4. Distillation Unit (non-metals)

- Westco Easy Distillation #52679

5. TCLP Rotator

- Environmental Express #52668

- ***Ovens:***
  - VWR, Model # 1350FM (4): #52665; #52664; #52666; #52846 (Solids).
  - Thermolyne, Model F-A1730 (Muffle) #3927 (TVS).

Appendix F Statistical Outlier Tests

Grubbs Test

This test is based on the difference between the suspected outlier divided by the average and comparing the value to the Grubbs critical value table. The letter N in critical value table represent the total number of points in the data set. If n>25 then the result is just a coarse approximation. If T > than CV then the data point is considered an outlier and should be discarded

A website that has an easy calculator for Grubbs Test outliers is found on GraphPad.com.
http://graphpad.com/quickcalcs/Grubbs1.cfm

$$T = \frac{|Xo - Mean|}{SD}$$

Where T = test value
Xo = value of suspected outlier
Mean = average of all data points
SD = standard deviation

GRUBBS Critical Values

N	CV 0.05	N	CV 0.05
3	1.1543	17	2.6200
4	1.4812	18	2.6516
5	1.7150	19	2.6809
6	1.8871	20	2.7082
7	2.0200	25	2.8217
8	2.1266	30	2.9085
9	2.2150	40	3.0361
10	2.2900	50	3.1282
11	2.3547	60	3.1997
12	2.4116	70	3.2576
13	2.4620	80	3.3061
14	2.5073	90	3.3477
15	2.5483	100	3.3841
16	2.5857		

N = Number of datapoints

CV = Critical Value at 95% confidence level

Dixon Q-Test

This test is based on the difference between the suspected outlier and the next closest data point divided by the total range of the data set and comparing the value to the Q-value to the limits in the table. The letter N in the Q-value table represent the total number of points in the data set. If Q > than the limit in the Q-value table then the data point is considered an outlier and should be discarded

$$Q = \frac{Xo - Xc}{Rh - Rl}$$

Where
Q = Dixon Q-value
Xo = value of suspected outlier
Xc = value of next closest data point
Rh = highest value in data set range
Rl = lowest value in data set range

Dixon Q-Test Limit Values

N	Q 95% conf
3	0.941
4	0.766
5	0.643
6	0.563
7	0.507
8	0.467
9	0.436
10	0.412
15	0.338
20	0.300
25	0.277
30	0.260

N = Number of datapoints

Appendix G Analytical Methods and QC Criteria

Parameter	Units[ii]	Method	Target[iii]	Precision[iv]	Accuracy[v]			
					MS	LCS	CCV	ICV
Field Measurements								
Dissolved Oxygen	mg/L	Hach 10360	0.2	≤ ± 0.3	N/A	N/A	N/A	-0.3 to +0.4
Flow	Cfm	MOMs[vii]	10	N/A	N/A	N/A	N/A	N/A
Percent DO Saturation	%		N/A		N/A	N/A	N/A	N/A
Sample Depth	Ft		1		N/A	N/A	N/A	N/A
Temperature	°C	EPA 170.1	1	± 0.5	N/A	N/A	N/A	≤ ± 0.5[viii]
pH	S.U.	EPA 150.1	Sensitivity to 0.1	± 0.3	N/A	± 0.2[ix]	N/A	≤ ± 0.2[x]
Specific Conductivity (@ 25°C)	µmhos/cm	EPA 120.1	1	± 10%	N/A	N/A	±7%	±7%
Turbidity	NTU	SM 2130 B	1	± 20%	N/A	N/A	±10%	±10%
Redox	Mv	Electrometric probe	1	± 20	N/A	N/A	± 10	
Microbiological Examination								
Escherichia Coli (E.Coli)	CFU / 100mL	SM 9223B	1	0.6 (log)	N/A	Positive Confirmation	N/A	N/A
Enterococcus	CFU / 100mL		10	0.6 (log)	N/A	Positive Confirmation	N/A	N/A
Bacteria - Total Coliform only	CFU / 100mL	TBD	1	0.6 (log)	N/A	Positive Confirmation	N/A	N/A
Physical & Aggregate Properties								
Total Dissolved Solids	mg/L	2540 C	10	± 20%	N/A	± 20%	N/A	N/A
Total Solids	mg/L	2540 B	10	± 20%	N/A	± 20%	N/A	N/A
Total Suspended Solids	mg/L	2540 D	1	± 20%	N/A	± 20%	N/A	N/A
Alkalinity	mg/L	2320 B	1	± 10%	N/A	± 20%	± 0.3 pH	± 0.1 pH
Bicarbonate Alkalinity	mg/L	2320 B	1	± 10%	N/A	± 20%	± 0.3 pH	± 0.1 pH
Conductivity	µmhos/cm	120.1/2510 B	1	± 5%	N/A	N/A	± 2%	± 2%
Salinity	Ppth	2520 B	1		N/A			
Settleable Solids	mL/L	2540 F	0.01		N/A			
Turbidity	NTU	180.1/2130 B	1	± 20%	N/A	± 10%	± 10%	± 10%
pH	SU	150.1/4500-pH B 9040B / 9045C	Sensitivity to 0.1	± 0.2 pH	N/A	± 0.1 pH	± 0.2pH	± 0.1 pH
Color	CU	2120 B	1		N/A			
Metals								
Mercury, Total	µg/L	245.1/7470A	0.02	± 20%	± 20%	± 15%	± 10%	± 10%
Mercury, Total	mg/Kg	7473	0.03	± 10%	± 20%	± 10%	± 20%	± 10%
Mercury, Low Level		1631	LOQ	± 30%	± 20%	± 20%		
Mercury,TCLP	mg/L	1311 / 7470 A	0.0002	± 10%	± 20%	± 15%	± 10%	± 10%

Parameter	Units[ii]	Method	Target[iii]	Precision[iv]	Accuracy[v]			
					MS	LCS	CCV	ICV
Methyl mercury (aqueous)	ng/L	1630	LOQ	± 30%	± 20%	± 20%		
Methyl mercury (sediment)	µg/Kg wet	USGS 5 A-7	LOQ	± 30%	± 20%	± 20%		
Lead by Graphite Furnace, Total Recoverable	µg/L	EPA 200.9	LOQ	± 20%	± 30%	± 15%	± 10%	± 10%
TCLP Percent Solids-Metals	%	1311	LOQ					
Hardness by ICP-AES	mg/L	SM 2340B	LOQ	± 20%	NA	± 20%	± 10%	± 10%
Metals by ICP: Potable and Non-Potable Waters; Total Recoverable or Dissolved								
Aluminum	mg/L	200.7 / 6010C	0.05	± 20%	± 30%	± 15%	± 10%	± 5% / ± 10%
Antimony	mg/L	200.7 / 6010C	0.015	± 20%	± 30%	± 15%	± 10%	± 5% / ± 10%
Arsenic	mg/L	200.7 / 6010C	0.01	± 20%	± 30%	± 15%	± 10%	± 5% / ± 10%
Barium	mg/L	200.7 / 6010C	0.002	± 20%	± 30%	± 15%	± 10%	± 5% / ± 10%
Beryllium	mg/L	200.7 / 6010C	0.0005	± 20%	± 30%	± 15%	± 10%	± 5% / ± 10%
Boron	mg/L	200.7 / 6010C	0.02	± 20%	± 30%	± 15%	± 10%	± 5% / ± 10%
Cadmium	mg/L	200.7 / 6010C	0.005	± 20%	± 30%	± 15%	± 10%	± 5% / ± 10%
Calcium	mg/L	200.7 / 6010C	0.1	± 20%	± 30%	± 15%	± 10%	± 5% / ± 10%
Chromium	mg/L	200.7 / 6010C	0.002	± 20%	± 30%	± 15%	± 10%	± 5% / ± 10%
Cobalt	mg/L	200.7 / 6010C	0.003	± 20%	± 30%	± 15%	± 10%	± 5% / ± 10%
Copper	mg/L	200.7 / 6010C	0.01	± 20%	± 30%	± 15%	± 10%	± 5% / ± 10%
Iron	mg/L	200.7 / 6010C	0.05	± 20%	± 30%	± 15%	± 10%	± 5% / ± 10%
Lead	mg/L	200.7 / 6010C	0.01	± 20%	± 30%	± 15%	± 10%	± 5% / ± 10%
Lithium	mg/L	200.7 / 6010C	0.015	± 20%	± 30%	± 15%	± 10%	± 5% / ± 10%
Magnesium	mg/L	200.7 / 6010C	0.1	± 20%	± 30%	± 15%	± 10%	± 5% / ± 10%
Manganese	mg/L	200.7 / 6010C	0.005	± 20%	± 30%	± 15%	± 10%	± 5% / ± 10%
Molybdenum	mg/L	200.7 / 6010C	0.004	± 20%	± 30%	± 15%	± 10%	± 5% / ± 10%
Nickel	mg/L	200.7 / 6010C	0.004	± 20%	± 30%	± 15%	± 10%	± 5% / ± 10%
Potassium	mg/L	200.7 / 6010C	0.5	± 20%	± 30%	± 15%	± 10%	± 5% / ± 10%
Selenium	mg/L	200.7 / 6010C	0.01	± 20%	± 30%	± 15%	± 10%	± 5% / ± 10%
Silicon (as Silica)	mg/L	200.7 / 6010C	0.5	± 20%	± 30%	± 15%	± 10%	± 5% / ± 10%
Silver	mg/L	200.7 / 6010C	0.002	± 20%	± 30%	± 15%	± 10%	± 5% / ± 10%
Sodium	mg/L	200.7 / 6010C	0.3	± 20%	± 30%	± 15%	± 10%	± 5% / ± 10%
Titanium	mg/L	200.7 / 6010C		± 20%	± 30%	± 15%	± 10%	± 5% / ± 10%
Vanadium	mg/L	200.7 / 6010C	0.002	± 20%	± 30%	± 15%	± 10%	± 5% / ± 10%
Zinc	mg/L	200.7 / 6010C	0.03	± 20%	± 30%	± 15%	± 10%	± 5% / ± 10%
Hardness as $CaCO_3$	mg/L	200.7 / 6010C	0.75	NA	NA	NA	NA	NA
Metals by ICP Scan	mg/L	200.7 / 6010C	LOQ	± 20%	± 30%	± 15%	± 10%	± 5% / ± 10%
TCLP - Toxic Pollutant Metals by ICPMS	mg/L	1311 / 6020A	LOQ (varies)	± 20%	± 25%	± 20%	± 10%	± 10%

Parameter	Units[ii]	Method	Target[iii]	Precision[iv]	Accuracy[v] MS	LCS	CCV	ICV
Metal by ICPMS: Potable and Non-Potable Waters, Solids; Total Recoverable or Dissolved								
Antimony	µg/L / mg/Kg	200.8 / 6020A	0.3 / 0.05	± 20%	± 30% / ±25%	± 15% / ±20%	± 10%	± 10%
Arsenic	µg/L / mg/Kg	200.8 / 6020A	0.25 / 0.1	± 20%	± 30% / ±25%	± 15% / ±20%	± 10%	± 10%
Barium	µg/L / mg/Kg	200.8 / 6020A	2 / 0.1	± 20%	± 30% / ±25%	± 15% / ±20%	± 10%	± 10%
Beryllium	µg/L / mg/Kg	200.8 / 6020A	0.1 / NA	± 20%	± 30% / ±25%	± 15% / ±20%	± 10%	± 10%
Cadmium	µg/L / mg/Kg	200.8 / 6020A	0.1 / 0.05	± 20%	± 30% / ±25%	± 15% / ±20%	± 10%	± 10%
Chromium	µg/L / mg/Kg	200.8 / 6020A	1 / 0.1	± 20%	± 30% / ±25%	± 15% / ±20%	± 10%	± 10%
Copper	µg/L / mg/Kg	200.8 / 6020A	1.5 / 0.05	± 20%	± 30% / ±25%	± 15% / ±20%	± 10%	± 10%
Lead	µg/L / mg/Kg	200.8 / 6020A	0.2 / 0.05	± 20%	± 30% / ±25%	± 15% / ±20%	± 10%	± 10%
Molybdenum	µg/L / mg/Kg	200.8 / 6020A	3 / NA	± 20%	± 30% / ±25%	± 15% / ±20%	± 10%	± 10%
Nickel	µg/L / mg/Kg	200.8 / 6020A	1 / 0.1	± 20%	± 30% / ±25%	± 15% / ±20%	± 10%	± 10%
Selenium	µg/L / mg/Kg	200.8 / 6020A	2 / 0.1	± 20%	± 30% / ±25%	± 15% / ±20%	± 10%	± 10%
Silver	µg/L / mg/Kg	200.8 / 6020A	0.1 / 0.02	± 20%	± 30% / ±25%	± 15% / ±20%	± 10%	± 10%
Thallium	µg/L / mg/Kg	200.8 / 6020A	0.1 / 0.02	± 20%	± 30% / ±25%	± 15% / ±20%	± 10%	± 10%
Uranium	µg/L / mg/Kg	200.8 / 6020A	0.1 / NA	± 20%	± 30% / ±25%	± 15% / ±20%	± 10%	± 10%
Vanadium	µg/L / mg/Kg	200.8 / 6020A	4 / NA	± 20%	± 30% / ±25%	± 15% / ±20%	± 10%	± 10%
Zinc	µg/L / mg/Kg	200.8 / 6020A	5 / 0.2	± 20%	± 30% / ±25%	± 15% / ±20%	± 10%	± 10%
Inorganic Non-Metals								
Ammonia	mg/L	ASTM D6919-09	0.01	± 20%	± 20%	± 10%	± 10%	± 10%
Nitrate by Colorimetry	mg/L	353.2/4500NO_3F	calc	± 10%	± 20%	± 10%	± 10%	± 10%
Nitrate/Nitrite	mg/L	353.2/4500NO_3F	0.005	± 10%	± 20%	± 10%	± 10%	± 10%
Nitrite	mg/L	353.2/4500NO_3F	0.005	± 10%	± 20%	± 10%	± 10%	± 10%
Total Kjeldahl Nitrogen	mg/L	351.2/4500N_{org}D	0.2	± 20%	± 20%	± 20%	± 10%	± 10%
Bromate	µg/L	300.1	5	± 10%	± 20%	± 15%	± 10%	± 10%
Bromide by Ion Chromatography	mg/L	300.0 / 9056A	0.030	± 10%	± 20%	± 10%	± 10%	± 10%
Bromide as disinfection byproduct	µg/L	300.1	30	± 10%	± 20%	± 15%	± 10%	± 10%
Chlorate	µg/L	300.1	30	± 10%	± 20%	± 15%	± 10%	± 10%
Chloride by Ion Chromatography	mg/L	300.0/ 9056A	0.5	± 10%	± 20%	± 10%	± 10%	± 10%
Chlorite	µg/L	300.1	12	± 10%	± 20%	± 15%	± 10%	± 10%
Fluoride by Ion Chromatography	mg/L	300.0/ 9056A	0.1	± 10%	± 20%	± 10%	± 10%	± 10%
Nitrate by IC	mg/L	300.0/ 9056A	0.05	± 10%	± 20%	± 10%	± 10%	± 10%
Sulfate by IC	mg/L	300.0/ 9056A	0.2	± 10%	± 20%	± 10%	± 10%	± 10%
Biochemical Oxygen Demand,5 Day Diluted	mg/L	5210 B	2	± 10%	N/A	± 15%	N/A	N/A

Parameter	Units[ii]	Method	Target[iii]	Precision[iv]	Accuracy[v]			
					MS	LCS	CCV	ICV
Biochemical Oxygen Demand,5 Day Un-Diluted Stream	mg/L	5210 B	0.1					
Biochemical Oxygen Demand,Carbonaceous 5 Day Diluted	mg/L	5210 B	2	± 10%	N/A	± 15%	N/A	N/A
Chlorine, Total Residual	mg/L	4500CL G	0.05	± 20%	NA	± 10%	NA	± 10%
Orthophosphate	mg/L	4500P E	0.005	± 10%	± 20%	± 10%	± 10%	± 10%
Total Phosphorus	mg/L	4500P B,E	0.01	± 10%	± 20%	± 10%	± 10%	± 10%
Organic GC, GCMS and HPLC								
Drinking Water - Chlorinated Pesticides by GC/ECD Method 508	µg/L	508 / 8081B	LOQ	≤ 30%	± 35% / CC[xiii]	± 30%	± 20%	± 20%
Chlorinated Pesticides by GC/ECD	µg/L	508 / 608 / 8081B	LOQ	≤ 30%	± 35% / CC[xiii]	± 30%	/± 20%	± 20%
PCBs as Arochlors	µg/L	508/ 508A / 8082A	LOQ	≤ 30% / 20% / 30%	35% / 30% / CC[1]	± 30% / ± 20%	/± 20%	± 20%
EDB/DBCP/TCP by GC/ECD	µg/L	504.1	LOQ	≤ 30%	± 35%	± 30%	/± 20%	± 20%
Nitrogen/Phosphorous Pesticides by GC/NPD	µg/L	8141B	LOQ	≤ 30%	CC[xiii]	± 30%	/± 20%	± 20%
Volatile Organic Compounds by GC/MS	µg/L	524.2 / 8260C	LOQ	≤ 30%	± 30%	± 30%	± 30%	± 30%
Drinking Water Semi-volatile Organic Compounds by GC/MS	µg/L	525.2	LOQ	< 30%	± 30%	± 30%	± 30%	± 30%
Oragnic Disinfection By-Products GC-ECD (HAAs)	µg/L	552.2	LOQ	< 30%	± 30%	NA	± 30%	± 30%
Semi-volatile Organic Compounds by GC/MS	ηg/L	EPA 625 / 8270D	LOQ	≤ 30%	CC[xi]	± 30%	± 20%	± 20%
Formaldehyde by HPLC	µg/L	8315A	LOQ	≤ 30%	CC[xiii]	± 30%	± 20%	± 20%
Non-Volatile Compounds by HPLC/TS/MS	ηg/L	8321	LOQ	≤ 30%	CC[xiii]	± 30%	± 20%	± 20%
Polynuclear Aromatic Hydrocarbons by GC/MS SIM	µg/L	8270D SIM	LOQ	≤ 30%	CC[xiii]	± 30%	± 20%	± 20%

Parameter	Units[ii]	Method	Target[iii]	Precision[iv]	Accuracy[v]			
					MS	LCS	CCV	ICV
Phenoxy Herbicides by GC/ECD	µg/L	6640B / 515.4	LOQ	≤ 30%	CC[xiii]	± 30%	± 20%	± 20%
Dioxins and Furans by Isotope Dilution HR GC/MS	ng/L	EPA 1613B	LOQ	≤ 30%	CC[xiii]	Approx ± 30% (varies)	Approx ± 20% (varies)	Approx ± 20% (varies)
Polybrominated Diphenyl Ethers (PBDE) by HR GC/MS	ng/L	EPA 1614	LOQ	≤ 30%	50-150	50-150	70-130%	70-130%
Chorinated Biphenyl Congeners by HR GC/MS	µg/L	EPA 1668A	LOQ	≤ 30%	50-150	50-150	70-130%	70-130%
Pharmaceuticals and Personal Care Products by LC/MS/MS	ng/L	1694	LOQ	≤ 30%	CC[xiii]	± 30%	± 20%	± 20%
Steroids and Hormones by HRGC/MS	ng/L	1698	LOQ	≤ 30%	CC[xiii]	± 30%	± 20%	± 20%
NW Total Petroleum Hydrocarbons - Diesel Range	mg/L	NWTPH-D	LOQ	≤ 30%	± 50%	± 30%	± 20%	± 20%
NW Total Petroleum Hydrocarbons - Gasoline Range	mg/L	NWTPH-G	LOQ	≤ 30%	± 50%	± 30%	± 20%	± 20%
NW Total Petroleum Hydrocarbons Identification	mg/L	NWTPH	LOQ	N/A	N/A	N/A	± 20%	± 20%
Aldehydes and Ketones in Air by HPLC	ppbv	TO-11A	LOQ	≤ 20%	>80% ±10%		± 10% / ± 15%	± 15%
SVOCs in air by GC/MS	ppbv	TO-13A			60%-120%		± 30%	± 30%
VOCs in Air by GC/MS	ppbv	TO-15	LOQ	≤ 25%			± 30%	± 30%
Algal Toxins by LC/MS-MS Anantoxin and Microcystin	µg/L	MOM grab	LOQ					
Microcystin by ELISA	µg/L	MOM grab	LOQ					
TCLP - Phenoxy Herbicides by GC/MS	mg/L	1311/8270D	LOQ	≤ 30%	CC[xiii]	± 30%	± 20%	± 20%

Parameter	Units[ii]	Method	Target[iii]	Precision[iv]	Accuracy[v] MS	LCS	CCV	ICV
TCLP - Semivolatile Organic Compounds by GC/MS	mg/L	1311/8270D	LOQ	≤ 30%	CC[xiii]	± 30%	± 20%	± 20%
TCLP - Volatile Organic Compounds by GC/MS	mg/L	1311/8260C	LOQ	≤ 30%	± 30%	± 30%	± 30%	± 30%
TCLP - Chlorinated Pesticides by GC/ECD	mg/L	1311/8081B	LOQ	≤ 30%	CC[xiii]	± 30%	/± 20%	± 20%
Organic: Aggregate Constituents & Properties								
Oil & Grease (Hexane Extractable Material)	mg/L	1664	5	± 18%	78-114	78-114%	N/A	N/A
Chemical Oxygen Demand	mg/L	5220D	5 or 10	± 20%	± 25%	± 15%	± 10%	± 10%
Dissolved Organic Carbon	mg/L	415.1/5310B	LOQ	± 20%	± 25%	± 15%	± 10%	± 10%
Total Organic Carbon	mg/L	415.1/5310B / 9060	LOQ	± 20%	± 25%	± 15%	± 10%	± 10%
Flash Point	°F	1020B / ASTM D3278	LOQ		N/A			
Chlorophyll	µg/L	SM10200 H	0.1		N/A	N/A	± 10%	± 10%
Chlorophyll by Area	mg/m²	Calculation	LOQ		N/A	N/A	N/A	N/A
Total Cyanide	mg/L mg/Kg	9014/4500CN E	0.01 / 0.5	± 20%	± 25%	± 15%	± 10%	± 10%
Total Cyanide (SDWA)	mg/L	4500CN E	0.01	± 20%	± 10%	± 10%	± 10%	± 10%
Cyanide Weak Acid Dissociable	mg/L	4500CN C,G	0.01	± 20%	± 25%	± 15%	± 10%	± 10%
Lignin and Tannin	mg/L	5550 B	1	± 20%	± 25%	± 25%	± 10%	± 10%
NCASI Color	CU	NCASI 71.01	5	± 15%	N/A	± 10%	N/A	N/A

[ii] The units of the QC (Target, Precision, and Accuracy) limits are listed in this column. If the QC limit is reported with a "%" sign it is unit-less.
[iii] The target level is the anticipated reporting level for this project. A target level of "LOQ" means the laboratory will use its current LOQ. LOQs may be found in the specific ELEMENT analysis codes. If the requested target level is less than the laboratories LOQ, the laboratory will estimate the result down to the laboratory's LOD. The laboratory will not report values less than its LOD.
[iv] The precision control limit is to be used to evaluate both field duplicate and laboratory duplicate samples. Use the laboratory's current duplicate control limits, unless specified otherwise.
[v] Actual laboratory control limits may vary, since laboratories are expected to revise control limits over time. Some QC measures are not applicable (NA) to the test method. Use the laboratory's current accuracy control limits, unless specified otherwise.
[vii] Stream flow measurements will be conducted according to the ODEQ methodology derived from USGS stream flow protocols.
[viii] Thermometer Accuracy checked with NIST standards.
[ix] Low ionic control sample.
[x] Low ionic control sample.
[xi] Limits are based on laboratory historical data obtained from Control Charts

Appendix H QC Definitions

QC Class	QC	LIMS	Frequency	Description/Corrective Action
Analytical	Instrument Blank	IB	1/analytical 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 cannot 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	Method/Reagent Blank	RB	1/ preparation batch	Method 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 cannot 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 cannot be corrected all reported results bracketed between the RB and CCB or between CCBs shall be flagged with the appropriate data qualifier.

QC Class	QC	LIMS	Frequency	Description/Corrective Action
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 is normally near the midpoint of the calibration range. Some inorganic methods utilize an additional CCV at or near the LOQ. 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 cannot be corrected, all reported results bracketed between the ICV & CCV or between CCV's shall be flagged with the appropriate data qualifier.
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 cannot 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 cannot 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)

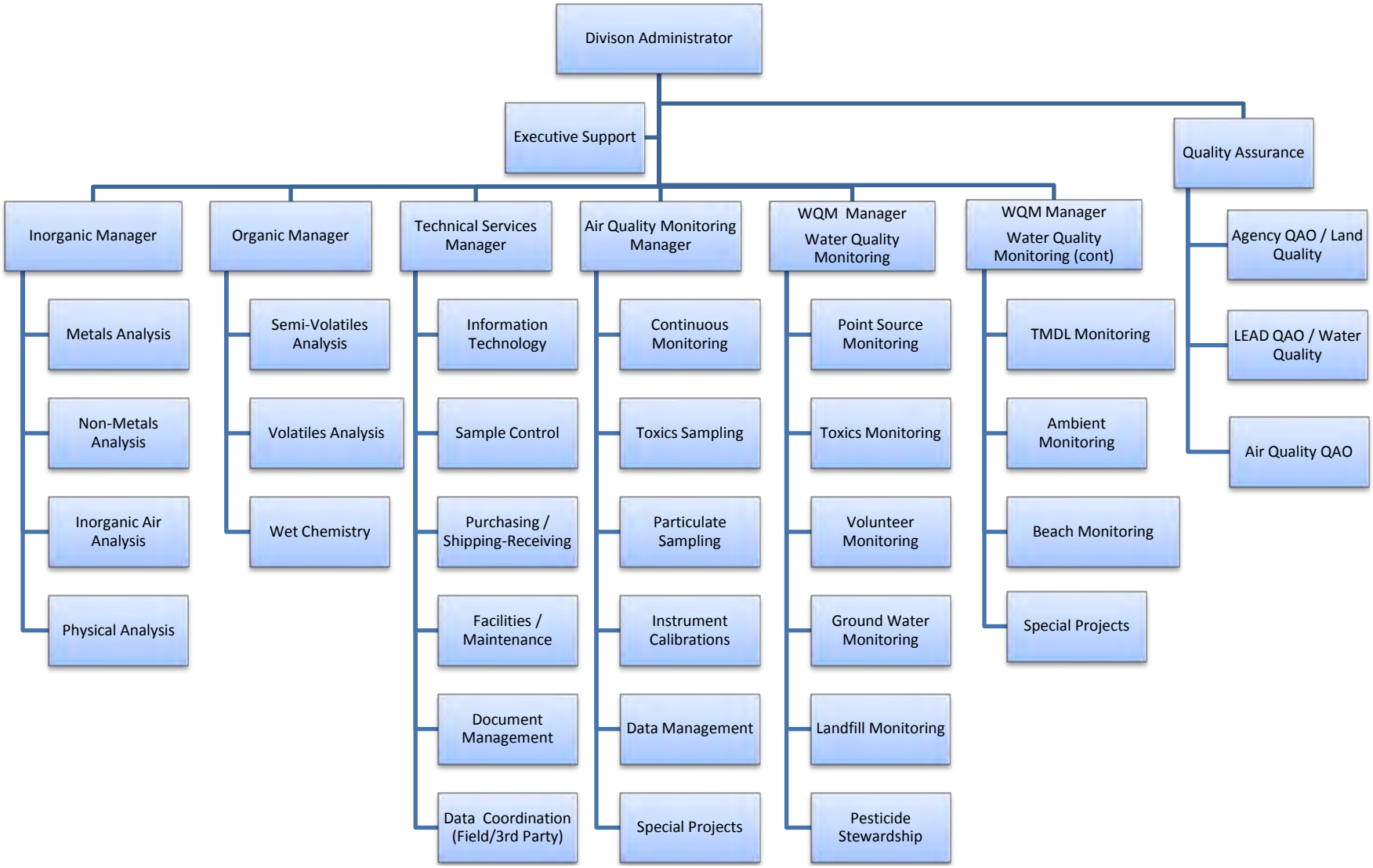
QC Class	QC	LIMS	Frequency	Description/Corrective Action
Field	Field Duplicate	FD	10% samples collected during a sampling expedition	Field Duplicate: discrete samples taken from the same field location and processed and analyzed independently by the laboratory. The original sample is identified by space 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)
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)

QC Class	QC	LIMS	Frequency	Description/Corrective Action
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)
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)"

QC Class	QC	LIMS	Frequency	Description/Corrective Action
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 individual LCS is compared to the acceptance criteria as published in the referenced test method, or where there are no established criteria, the laboratory established limits as noted in the SOP. 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)
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)

QC Class	QC	LIMS	Frequency	Description/Corrective Action
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 I LEAD Organizational Chart



Appendix J Revision History

Revision	Description	Section	Date	Author
7.0	Reorganization of LQM to flow with NELAC Standard 2003 and ISO 17025.		10/14/09	CLR, SCH
	Inserted Revision History table			
	Updated tables and org chart.			
	Added Ethics and Data Integrity Section			
	Added Employee Attestation form			
	Revised policy for reporting Uncertainty			
	Appendix E Major Analytical Equipment			
8.0	Updated all references to the 2003 NELAC standard to the 2009 TNI Standard	Throughout the LQM	5/2/2011	SCH
	Clarifications and edits	Section 5		
	Added Section on “Improvement” as required by TNI 2009 standard	Section 11		
	Updated Procedure for accessing Report files	Section 14.2.5		
	Management Review updated	Section 15.4		
	Added J flag reporting and additional detail for reporting results and qualifying data	Section 23		
	Updated Definitions in Glossary to be consistent with TNI 2009 standard	Appendix A		
	Updated equipment list	Appendix G		
	Added Mercury methods (low level, methyl) to methods table	Appendix H		

Revision	Description	Section	Date	Author
8.1	Editorial corrections throughout. Updated tables to reflect new Inorganic instrumentation. Updated Purchasing section to reflect current practices, Added LOD/LOQ reporting from earlier memorandum.	7.0 , 23.1-23.2	7/6/2012	SCH
8.2	Updated throughout to reflect new processes based on ELEMENT.	All	8/15/2013	SCH
	Added in review of monthly and quarterly metrics	15.4		
	Added in section on control charting.	22.2.1		
	Edited CDOC requirements to incorporate review of control charts. Removed sample container and preservation appendices and refered to *Field Sampling Reference Guide*. Added Appendix for Outlier tests	18.2.1 Appendix F		