LABORATORY SERVICES BRANCH

LABORATORY OPERATIONS and QUALITY ASSURANCE MANUAL

U.S. ENVIRONMENTAL PROTECTION AGENCY
LABORATORY SERVICES AND APPLIED SCIENCE DIVISION
REGION 4
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Laboratory Services Branch

Ethics Policy

"*It shall be the policy of the Region 4 Laboratory to conduct all business with integrity and in an ethical manner. It is a basic and expected responsibility of each staff member and each manager to hold to the highest ethical standard of professional conduct in the performance of all duties and to adhere to EPA's Principles of Scientific Integrity, dated November 24, 1999.*"

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CHAPTER 1

Purpose, Policy, Accreditation and Hierarchy

1.1 Purpose

The purpose of this manual, entitled Laboratory Operations and Quality Assurance Manual (LOQAM), is to document the quality assurance policies and procedures of the EPA, Region 4 Laboratory Services Branch (LSB) laboratory. A defined system of quality assurance practices and operational policies (a quality system) is essential for ensuring that data generated from analytical processes are well-defined and defensible. While the design and development of a quality assurance program is a management function, each individual staff member shares the responsibility for maintaining knowledge of the quality system and for following established quality control (QC) procedures. Meeting the International Organization for Standardization ISO 17025 standard, "General requirements for the competence of testing and calibration laboratories," and continually improving quality system effectiveness is a principle objective of the laboratory.

1.2 Mission of the EPA Regional Laboratory

The mission of LSB is to provide environmental data for decision making in EPA's media programs for protecting the environment and human health. This is achieved by maintaining a fully equipped environmental laboratory and a technically skilled, properly trained and dedicated staff that produces physical, biological, and chemical data of a known and defensible quality. LSB provides environmental data at the request of the customer within the Agency. All requests for analyses must originate with an EPA manager or staff person with the authority to request services from LSB. As an EPA laboratory, LSB is not permitted to operate as a fee-for-service laboratory. Additionally, as a government agency, LSB operations are inherently free from risks to impartiality.

1.3 Operations Policy

It is the policy of LSB to conduct all activities with four guiding principles: **(1) Safety (2) Data Integrity and Laboratory Ethics (3) Quality and (4) Service.** Each of these items must be present for successful operations.

1.3.1 Safety The primary consideration in all laboratory operations must be safety. There is no assignment for which safety should ever be compromised. Safety takes priority over all considerations and it is the responsibility of each staff person to have a clear understanding of the basic safety rules and how to safely perform operations within their area of responsibility. It is the responsibility of each individual to maintain a constant vigilance over safe operations and to notify their supervisor, Safety and Health Manager and the branch Safety Officer of any unsafe conditions. LSB employees must never initiate an action, procedure, or method if they are unsure of the appropriate safety procedures. If unsure of the safety of any method, procedure, or operational activity, it is the responsibility of each employee to contact their supervisor to obtain additional information or instructions on the proper safety procedures. Refer to the Region 4 Laboratory Safety and Chemical Hygiene Plan for safety and health policies and procedures.

1.3.2 Data Integrity and Laboratory Ethics It is the policy of the Region 4 Laboratory to conduct

all business with integrity and in an ethical manner. It is a basic and expected responsibility of each staff member and manager to hold to the highest ethical standard of professional conduct in the performance of all duties and to adhere to EPA's Principles of Scientific Integrity (1999) and the Scientific Integrity Policy (2012). A copy of EPA's Scientific Integrity Policy can be located at the following link:

Policy on EPA Scientific Integrity | Programs of the Office of the Science Advisor (OSA) | US EPA

The quality system has data integrity and ethical behavior at its very foundation. It is essential that every employee of the branch understand and adhere to these ethical standards to preserve the basic integrity of all work products. Data integrity, defined in its most simple terms as "the state of being unimpaired", concerns the ability to define and defend that the entire analytical process has been "unimpaired" and performed in accordance with appropriate practices and procedures. The ability to defend the integrity of the data is through complete documentation of actions and activities which includes, but is not limited to such items as: maintaining chain of custody and security of the samples; clear documentation of the activities performed in the preparation and analysis of the samples according to SOPs and in the final data reduction, review, and reporting; and maintaining complete and clear files of these records.

1.3.3 Quality It is LSB's policy that all data generated is of the quality required to meet or exceed each project's data quality objectives (DQOs) as determined by the customer and communicated at the time of the project request. Branch Managers and analysts share the responsibility of ensuring that analytical methods, instruments, analyte detection and quantitation are such that the data produced is scientifically sound and well-documented. The quality of all LSB data must be well-defined and communicated to the customer. This policy is implemented by:

1.3.3.1 Having in place and following a complete and systematic process of QC activities to assist in defining data quality;

1.3.3.2 Ensuring that data quality is documented and communicated to the customers of the data by assigning appropriate qualifiers according to prescribed procedures; and

1.3.3.3 Having a peer review process to verify that data are generated in accordance with appropriate technical procedures and to ensure that all activities associated with the analyses, calculations and data reduction are complete and accurate. Any modifications and/or deviations must be documented.

1.3.4 Service LSB is a service organization and as such, management and staff must maintain an awareness of customer needs and regulatory requirements as related to satisfaction with work products. Service is built upon the following two important principles.

1.3.4.1 Communication between the laboratory's staff and its clients is required to define a project's measurement and DQOs and to assist the customer in understanding analytical capabilities and limitations. Communications also enhance the ability to learn of emerging needs and to plan accordingly. LSB management and staff must be proactive in initiating these discussions and will inform customers of the advantages and disadvantages of requested methods

and QC procedures. Laboratory management reserves the right to determine the most appropriate analytical methodology and QC procedures based on the DQOs, if provided by the customer.

1.3.4.2 Timeliness Timing of final work products and reports are often critical and are a vital part of the overall service performed. While it is LSB's policy to never compromise safety, data integrity or quality for the sake of timeliness, timeliness is often the most important factor contributing to customer satisfaction. All staff must maintain a high degree of attention toward providing the data in a timely manner as established by project objectives. In the event circumstances result in late reports, the customer must be contacted, kept up-to-date on the issues surrounding the late data, and kept abreast of the progress of project completion.

1.4 Accreditation and Certification

1.4.1 EPA issued a policy directive on February 23, 2004 that all Agency laboratories shall maintain competency by documenting and maintaining a quality system which meets the requirements of EPA Order CIO 2105.0, (formerly 5360.1 A2) May 2000. The policy requires EPA laboratories to participate in an appropriate, recognized laboratory accreditation program when available.

1.4.2 LSB is ISO/IEC 17025 accredited with a Forensics Amplification. Refer to certificate number AT-1644 and scope for specific accreditation information. The laboratory is also Drinking Water certified by the Office of Ground Water and Drinking Water (OGWDW) and is certified to the current Drinking Water methodologies and the Fifth Edition of the Manual for the Certification of Laboratories Analytical Drinking Water (EPA 815-R-05-004, January 2005). Refer to Certificate number AT-2628 and scope for specific drinking water certification information. Drinking Water certification is issued by the Office of Ground Water and Drinking Water (OGWDW) upon concurrence of the findings provided by the ISO accrediting body performing the certification audit.

1.4.3 LSB's objective is to seek and maintain accreditation and certification for the methods and analytes that it performs on a routine basis. A list of the methods for which LSB is currently accredited and certified is available from the Division Quality Assurance Coordinator (QAC). LSB will not use an accrediting organization's logo (such as the ANAB logo) on datareports and does not conduct any advertising which might show an accrediting organization's logo. A statement indicating the accrediting body and the accreditation status of individual tests will be included on all test reports issued by LSB.

1.5 Hierarchy

This manual describes the policies that are the basis of LSB's quality system. Specific technical and procedural details are contained in methods and technical and administrative SOPs. On occasion, an analytical method or procedure may require deviation from some of the policies contained in this manual for specific technical reasons. These deviations will be documented in the individual SOPs. As such, instructions in SOPs and regulatory program requirements take precedence over this manual on those occasions. Drinking water methods for analyzing/reporting regulatory samples are prescriptive and cannot be modified without approval from Office of Water.

CHAPTER 2

Personnel, Facility and Equipment

2.1 Organization

Below is a listing of all LSB Staff and their major area(s) of responsibility. The LSB organizational structure is shown in Figure 2-1. Figure 2-2 depicts LSB as it fits into the total operation of the Laboratory Services and Applied Science Division (LSASD). In the event that the Branch Chief, Section Chief, or Quality Assurance Coordinator (QAC) is absent for a period of a week or more, the appropriate management official within the branch or section shall appoint a deputy to act on behalf of the individual who is absent. Staff signatures and initials are kept on file by the QAC.

Laboratory Services Branch Personnel

Immediate Office

Name	Principal Duties
Sandra Aker	**Branch Chief**
Jeff Hendel	Senior Technical Advisor
Scott Sivertsen	Senior Technical Advisor
Stacie Masters	LSASD Quality Assurance Coordinator , LSB Safety Officer
Mike Beall	Sample Custodian (SEE Employee)
Alva Eisenman	Divisional Document Control (SEE Employee)

Inorganic Chemistry Section

Name	Principal Duties
Floyd Wellborn	**Section Chief-Technical Director Inorganic Analyses**
Daniel Adams	Nutrients, Classicals, Final Data Review/Production
Curtis Callahan	Nutrients, Classicals, TCLP
Anthony Carroll	Mercury, Hexavalent Chromium, Lead Bioavailability
Megan DeJesus	Metals ICP
Blake Snyder	qPCR, Microbiology
Yvette Lane- Walcott	Nutrients
Francine Vancuron	Metals Data Review
Ernest Walton	Metals ICP-MS
Kayle Whiten	Nutrients, Classicals

Organic Chemistry Section

Name	Principal Duties
Vacant	**Section Chief-Technical Director Organic Analyses**
Ian Adams	Semivolatiles, Pesticides/PCBs, LSB Safety Officer
Nazar Ali	Volatiles (water, soil), Monitored Natural Attenuation
Dawn Bowerman	Monitored Natural Attenuation, Volatiles (water, soil)
Sade Brown	Extractions
Diana Burdette	LC-MS/MS
Jason Collum	Pesticides/PCBs, Semivolatiles, LSASD Chemical Hygiene Officer
Sam Dutton	Pesticides/PCBs
John Giles	Extraction, , LC-MS/MS
David Spidle	Volatiles (Air)
Kristin Trapp	Volatiles (Air)
Stephanie Wimpey	Volatiles,

2.2 Educational, Experience and Training Requirements

2.2.1 EPA operates its hiring procedures under the federal government's Office of Personnel Management (OPM) regulations. OPM issues qualification and classification standards for all general schedule (GS) positions. Typically, LSB's professionals and technicians fall within the 1300 – Physical Sciences Group, Job Family Standards for Professional Work and Technical Work. (See https://www.opm.gov/policy-data-oversight/classification-qualifications/classifying-general-schedule-positions/#url=Standards.) The OPM qualification and classification standards describe the educational and experience requirements which a potential employee must meet to satisfy the OPM requirements for a specific job series and grade. Before a laboratory employee is hired, EPA's Shared Service Center for Personnel Management verifies that the applicant meets the OPM education and experience requirements for the appropriate GS series and grade. After the verification process is complete, LSB managers may hire an applicant who meets the OPM requirements from a certificate of eligible candidates.

2.2.2 Prior to hiring a contract employee, an EPA Contracting Officer or Contracting Officer's Representative, in consultation with LSB management, will describe to the contractor in general terms the educational and experience requirements needed to perform the work. Contractor employees' experience and education are verified by the contractor's human resources department.

2.2.3 LSASD has developed a set of required training sessions for each employee; they are specified in the LSASD Employee Training SOP. Training is documented through sign-in forms or certificates, which are maintained by the QAC in LSB's training files. An ongoing goal of LSB's training program is to ensure that personnel are aware of the importance of their activities and how they contribute to the overall mission and goals of the Agency.

2.3 Roles and Responsibilities

2.3.1 Branch Chief

2.3.1.1 Has overall management responsibility, including hiring, budgeting, and policy development for the branch, and mission. The Branch Chief also has ultimate responsibility for the development, implementation, approval, and continued operation of the branch quality assurance system.

2.3.1.2 Assigns the authority and responsibility for day-to-day management of the quality system to the QAC and assures that communication takes place regarding the effectiveness of the quality system.

2.3.1.3 Delegates authority and responsibility for the daily oversight of QC activities in ICS and OCS to the Section Chiefs.

2.3.1.4 Provides leadership promoting a work culture that stresses the importance of safety, integrity, data quality, timeliness and customer service.

2.3.1.5 Assures that qualified analysts and support staff are assigned to the laboratory and that all staff are properly trained to perform their duties.

2.3.1.6 Makes overall decisions relating to staffing, personnel management, work assignments, laboratory capability and capacity in consultation with laboratory supervisors and staff.

2.3.2 LSB Technical Advisor

2.3.2.1 The Senior Technical Advisors provide expert technical advice and support to the Branch Chief and Division Management to evaluate, plan, and oversee future analytical method development in the branch and to serve as a resource for evaluating data quality for new analytical methods.

2.3.2.2 Assess the quality of laboratory data and advise other managers and scientists in the use, quality assurance, and interpretation of data produced by recently developed and applied methods for measuring environmental contaminants. The senior technical advisor has the knowledge and ability to understand analytical processes, interferences, and instrument limitations in producing data of known and documented quality.

2.3.2.3 Advise other managers and scientists in the use, quality assurance, and interpretation of data produced by recently developed and applied methods for measuring environmental contaminants.

2.3.2.4 Provide testimony and serves, as needed, as an expert witness in support of Regional Counsel and the Criminal Investigation Division.

2.3.2.5 Defend the integrity of LSASD scientific products and approaches to the scientific community, industry, academia, special interest groups and the public.

2.3.2.6 The Senior Advisor may serve as primary analyst, technical reviewer and release data to support the overall functions of the laboratory and the LSB Section Chiefs.

2.3.3 LSASD Quality Assurance Coordinator (QAC) – also referred to as the Quality Assurance Manager (QAM)

2.3.3.1 Is independent from all laboratory operations. Reports directly to the LSASD Deputy Director and has the delegated responsibility and authority for the implementation, management, and maintenance of the quality system for the laboratory.

2.3.3.2 Ensures compliance with all laboratory accreditation requirements.

2.3.3.3 Coordinates the branch-wide QA/QC activities.

2.3.3.4 Initiates and leads annual internal audits within the branch.

2.3.3.5 Works with Section Chiefs in determining the adequacy of corrective and preventive actions.

2.3.3.6 Maintains QA files with appropriate documentation to include, but not limited to:

2.3.3.6.1 Managerial and supervisory reports;

2.3.3.6.2 Documents requiring QAC signature/approval;

2.3.3.6.3 Outcomes of internal and external audits (reports, CA/PA/Improvements, etc.);

2.3.3.6.4 Results of inter-laboratory comparisons or proficiency tests;

2.3.3.6.5 Records for measurement traceability of testing equipment (i.e., weights/thermometers/volumetric syringes); and

2.3.3.6.6 Records and Documentation.

2.3.3.6.6.1 Demonstration of Competency (DOC) and Continuing Demonstrations of Proficiency (CDOP)

2.3.3.6.6.2 Method Detection Limits (MDLs)

2.3.3.6.6.3 Instrument Detection Limits (IDLs)

2.3.3.6.6.4 New method/technology validation studies

2.3.3.6.6.5 Special Issue Studies reports

2.3.3.6.6.6 Summaries of updates for acceptance limits in Element® for spikes, replicates, surrogates, and other QC data

2.3.3.6.6.7 Signature and initials of all employees

2.3.3.6.6.8 Training files to include internal and external training, cross-training, certifications, etc.

2.3.3.6.6.9 Corrective Action and Preventive Action reports

2.3.3.7 Reviews and approves all branch SOPs and QA manual updates and submits to Branch Chief for final approval.

2.3.3.8 Initiates and coordinates external proficiency test studies for all branch analytical operations.

2.3.3.9 Advises branch management concerning QA/QC issues.

2.3.3 Section Chief

2.3.3.1 Serves as the Technical Director of the section and oversees its day-to-day activities including analysis of samples within the quality system and production of data within each analytical group.

2.3.3.2 Ensures that a final overview of each work product (e.g., data, written reports) is performed so that all QC information is complete, properly utilized, documented and maintained within the various analytical work units of the section.

2.3.3.3 Reports final data produced by the section to the customer or delegates to authorized staff.

2.3.3.4 Monitors all section work activities.

2.3.3.5 Ensures that appropriate actions are taken as a result of QC indicators. Ensures that appropriate corrective actions are instituted within the analytical work groups as a result of internal and external audits.

2.3.3.6 Reviews and approves all section-specific technical documents, operating procedures and LOQAM updates.

2.3.3.7 Monitors and coordinates section workload and acceptance of work.

2.3.3.8 Ensures that individual project files are generated and maintained in accordance

with branch policies and other appropriate file management requirements.

2.3.3.9 Authorized to offer opinions and interpretations on analyses under their technical direction as well as authorize other qualified individuals under their supervision to offer opinions and interpretations on specific technical areas.

2.3.3.10 Communicates with customers to ensure that needs are met and to solicit feedback on LSB's services.

2.3.3.11 Ensures compliance with all laboratory accreditation requirements.

2.3.4 Analytical Staff

2.3.4.1 General staff are responsible for:

2.3.4.1.1 Having a working knowledge of the branch and divisional policies and procedures including health and safety, data integrity, and waste disposal;

2.3.4.1.2 Having a working knowledge of analytical methodologies used within their work areas;

2.3.4.1.3 Having a working knowledge of all policies, procedures, and QC activities within their respective work areas and ensuring that documentation of work performed is complete, accurate, and that analytical data are properly reported;

2.3.4.1.4 Notifying their immediate supervisor of any issues/problems with any work products; and

2.3.4.1.5 Maintaining and following appropriate SOPs for their work areas.

2.3.4.2 Primary Analyst Defined as the staff analyst performing a test on a given date and time. Typically, the primary analyst performs initial data reduction and transfer of data to the Laboratory Information Management System (LIMS, i.e., Element®) However, this task may also be performed by another analyst competent to perform the analysis. The primary analyst/technician shall ensure that:

2.3.4.2.1 Appropriate analytical methodologies and standard operating procedures are followed;

2.3.4.2.2 Appropriate QC activities are performed as designated by the method, SOPs, and/or the LOQAM;

2.3.4.2.3 Analytical activities are properly documented as specified by the method, SOPs, and/or the LOQAM;

2.3.4.2.4 Appropriate actions are taken when QC indicators do not meet established criteria and assures that necessary corrective action is implemented;

2.3.4.2.5 Individual analytical data points are completely and accurately recorded;

2.3.4.2.6 Data qualifier flags and explanatory footnotes are properly placed;

2.3.4.2.7 All appropriate items on the technical review checklist are properly documented,

2.3.4.2.8 The status of the workorder(s) in Element is set to "Analyzed";

2.3.4.2.9 The data package with the draft report is given to the Technical Data Reviewer within specified time periods required to meet laboratory turnaround time commitments; and

2.3.4.2.10 Communicates all technical issues to the Section Chief.

2.3.4.3 Technical Data Reviewer another staff analyst qualified to perform data review for the analysis being checked. It is the responsibility of the reviewer to perform a thorough technical review of all-important details associated with the data. In some cases, the Technical Data Reviewer may be responsible for final reporting of the data. The Technical Data Reviewer shall ensure that:

2.3.4.3.1 Appropriate analytical methodologies and SOPs were followed;

2.3.4.3.2 Appropriate QC activities were performed as designated by the method, SOP, and/or the LOQAM;

2.3.4.3.3 Analytical activities were properly documented as specified by the method, SOP, and/or the LOQAM;

2.3.4.3.4 Appropriate actions were taken as a result of QC indicators;

2.3.4.3.5 Analytical data qualifiers were accurately recorded and that all qualifier flags and explanatory footnotes are properly placed on the data;

2.3.4.3.6 Data have been entered and verified in Element® and, if qualified, contain the appropriate remarks to show reason(s) for qualification; and

2.3.4.3.7 Traceability of all standards and reagents can be tracked through Element® and all standards and reagents were properly assigned to the bench sheets in Element®.

2.3.4.3.8 Project file contains, or references, location of all necessary information including raw data, calibrations, extraction logs, standards, run logs, and dilutions.

2.3.5 Deputies (Acting Chief or QAC) who are acting on behalf of Chiefs or the QAC assumes the duties and responsibilities of that individual under the quality system. Any deputy will be notified of their temporary assumption of duties and responsibilities and must be familiar with and capable of executing the applicable requirements of the quality system.

2.3.6 Environmental Services Assistance Team (ESAT) Laboratory Support
Analytical support is often obtained through the ESAT contract as funding permits. The ESAT team is located on site within the LSB laboratory areas with space assigned specifically to them. Work is assigned by EPA Contract Officers to ESAT staff through technical direction documents following all contractual rules and regulations. ESAT personnel are expected to be familiar with the LOQAM, follow its policies and practices, and to follow analytical SOPs approved by EPA management.

2.3.7 LSB Staff – ESAT Work Assignments

2.3.7.1 Select LSB staff may submit technical direction requests to ESAT through the ESAT Tracking System. ESAT assignments may require communication with the Section Chief to assure that the ESAT workload is evenly distributed. Under the existing contract, only the EPA Contract Officer to ESAT or Alternate may issue work to ESAT.

2.3.7.2 LSB staff that submit technical direction requests must follow all rules and regulations of the contracting process. These requirements can be located at: https://oamintra.epa.gov/node/429

LSB staff are also responsible for receiving the work products that are generated by ESAT staff and performing an appropriate review of the work performed.

2.3.7.3 Each data package should be reviewed at a minimum to ensure that:

2.3.7.3.1 Appropriate analytical methodologies and SOPs were followed;

2.3.7.3.2 Appropriate QC activities were performed as designated by the method, SOP, and/or the LOQAM;

2.3.7.3.3 Analytical activities were properly documented as specified by the method, SOP, and/or the LOQAM;

2.3.7.3.4 Appropriate actions were taken as a result of QC indicators;

2.3.7.3.5 Recording of all individual analytical data points are complete and accurate and data qualifier flags and explanatory footnotes are properly placed;

2.3.7.3.6 Traceability of all standards and reagents can be tracked through Element® and all standards and reagents were properly assigned to the bench sheets in Element®.

2.3.7.3.7 Project file contains, or references, location of all necessary information including but not limited to raw data, calibrations, extraction logs, standards, run logs, and dilutions; and

2.3.7.3.8 Data have been entered and verified in Element® and, if qualified, contain the appropriate remarks to show reason(s) for qualification.

Note: Divisional Director, Deputy Director and Regional Quality Assurance Manager (RQAM) Roles and Responsibilities are outlined in the LSASD Quality Management Plan (QMP). Please refer to the most recent version of the QMP for more information.

2.4 Facilities

The total facility consists of approximately 55,000 net usable square feet, a little less than a third of which is occupied by LSB. Operation and maintenance of the facility is the responsibility of the lessor through the Government Services Administration (GSA). LSASD has one or more staff members (not within LSB) dedicated to facility issues, coordinating maintenance and operations with GSA and the lessor. The facility has adequate accommodations to perform testing procedures in the laboratory area. The laboratory will ensure the facility and environmental conditions relevant to the procedure will be monitored as required.

2.5 Equipment

2.5.1 Inventory LSB maintains a list of analytical instrumentation on the LAN.

2.5.2 Maintenance/Service Proper maintenance of laboratory instrumentation is a key ingredient to both the longevity of the useful life of the instrument, as well as providing reliable analyses. Maintenance and service requires an alert analytical staff that recognizes the need for equipment maintenance coupled with support services provided either by in-house staff or by vendor technicians. All staff members have the responsibility for ensuring that all primary maintenance is carried out on instrumentation in accordance with manufacturer's recommendations and schedules as practical. Staff are to ensure equipment is clean, free of contamination and operating properly prior to use. Additionally, all staff are required to maintain documentation of all maintenance activities within designated logbooks.

Figure 2-1
Laboratory Services Branch Organization Chart

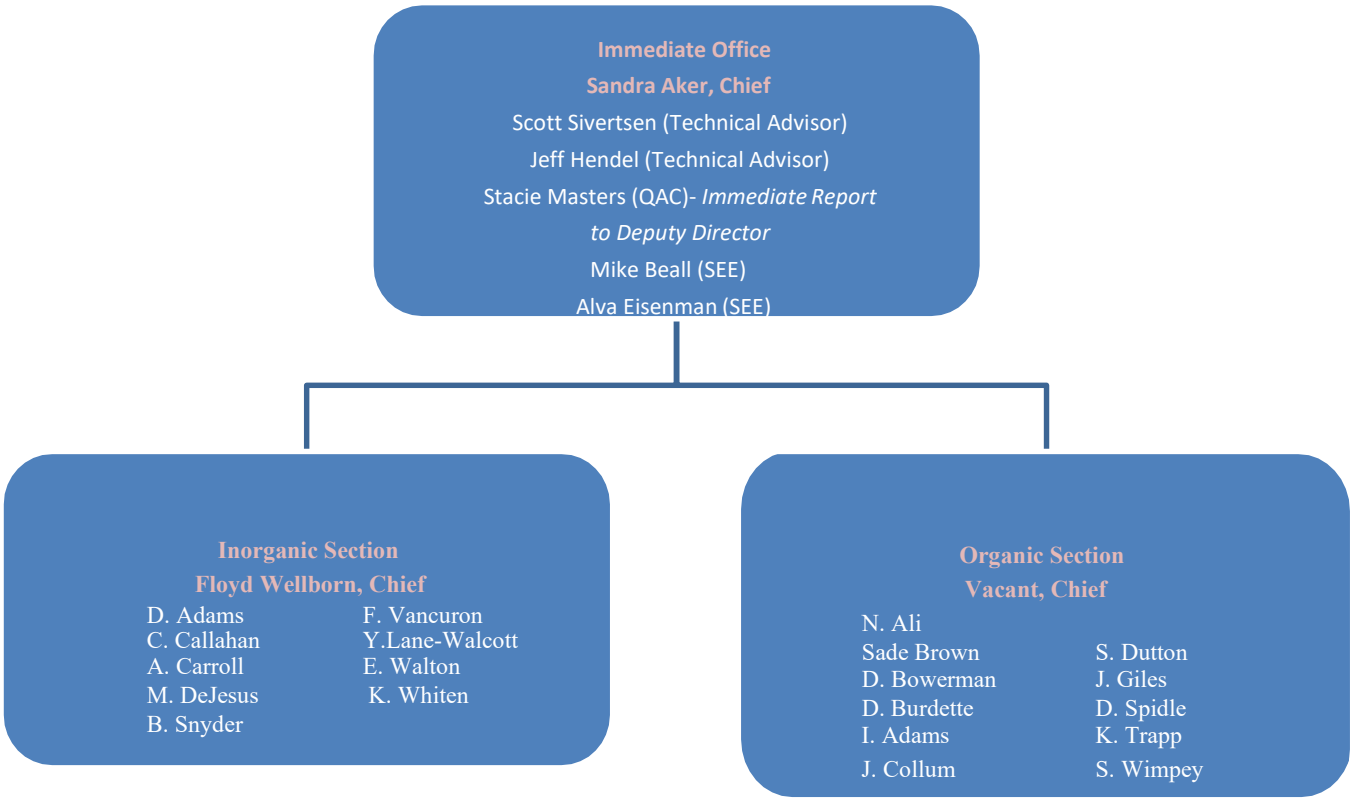
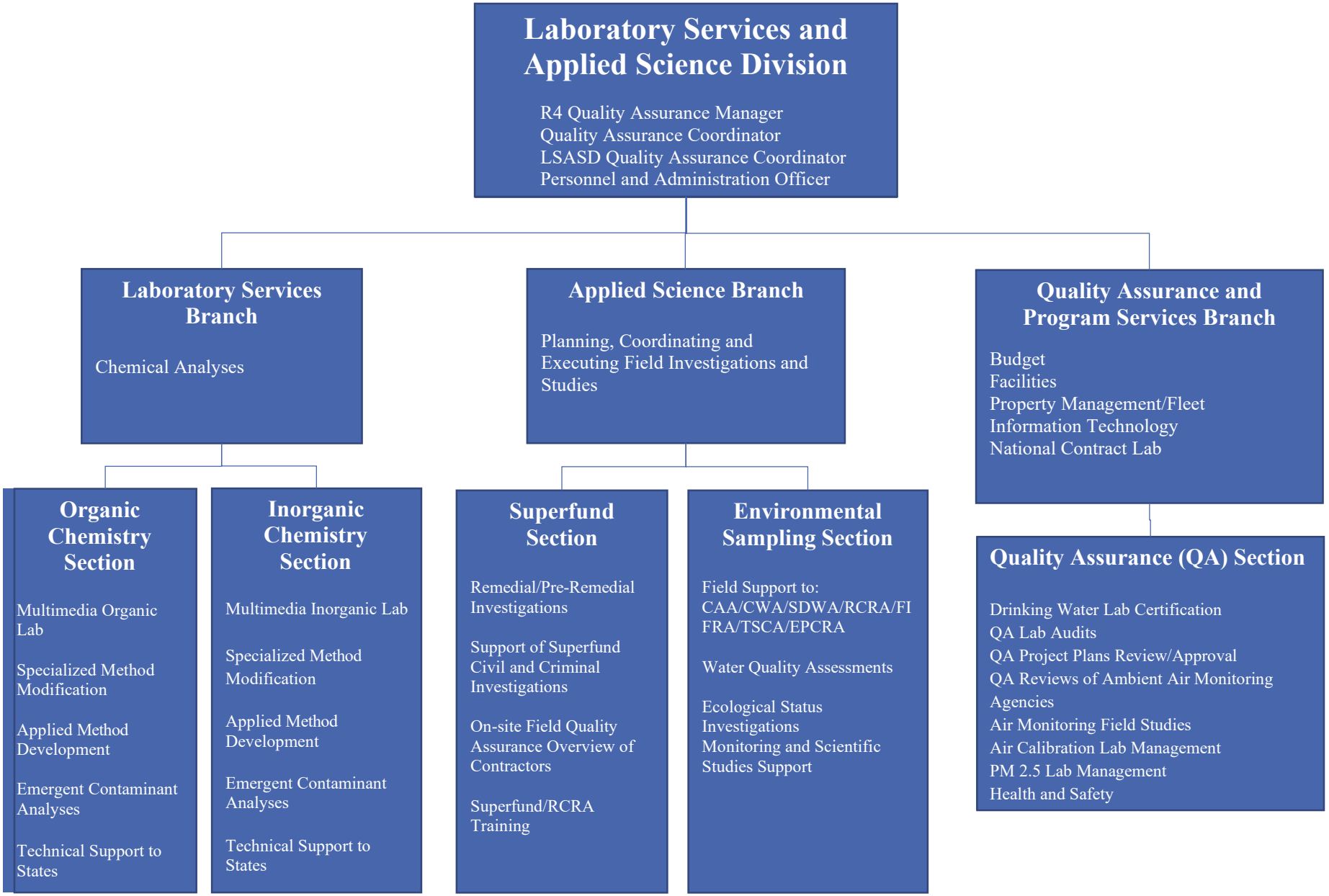


Figure 2-2
Region 4 Laboratory Services and Applied Science Division – Athens, Georgia



CHAPTER 3

Sample Scheduling, Handling, Storage and Disposal

3.1 Introduction

Complete documentation of the sample collection and handling process is an extremely important aspect of producing defensible laboratory data. Chain-of-custody procedures provide a record of sample traceability, accountability, and serve to validate sample integrity. All samples for analysis received by LSB are controlled with documented custody procedures.

3.2 Sample Collection

3.2.1 Procedures LSB staff does not perform field sampling activities, therefore the sampling activities are covered under the project-specific Quality Assurance Projector Sample and Analysis Plans.

3.2.2 Containers and Holding Times Selection of sample container types and preservation techniques are guided by the methods being applied. Guidance is available in such references as Standard Methods for the Examination of Water and Wastewater, ASTM, EPA Methods for Chemical Analyses of Water and Waste, 40 CFR 136, 40 CFR 141 and others. Tables 3-1 and 3-2 include sample containers, analysis, sample matrices, preservatives, and recommended holding times. LSB will accept smaller aliquots of samples than referenced in Table 3-1; however, when reducing sample volumes, the volume of preservative must also be reduced to achieve the same final concentrations of preservative in the sample.

3.3 Sample Scheduling

3.3.1 Initial Scheduling LSB uses an in-house laboratory information management system (LIMS), or R4LIMS also referred to as Project Log, for project scheduling. In the event that a new project log or scheduling system is developed, R4LIMS will remain active until full implementation of the new system has been achieved and all historical data will remain within R4LIMS. Each project that is entered into R4LIMS is assigned a unique project number that is used throughout its life for tracking, reporting, and filing.

3.3.2 Sample Acceptance

3.3.2.1 Review of Requested Analyses LSB Section Chiefs, or designee review requested projects through R4LIMS to determine whether to accept the requested project, or if the project should be contracted outside the laboratory through a national contract such as the Superfund Contract Laboratory Program (CLP). When projects are entered into R4LIMS requesting analysis, LSB management has the first right of refusal of the work. The laboratory will not log in samples that arrive without first being requested and scheduled in R4LIMS prior to receipt. If this situation occurs, laboratory management will contact the project requestor and/or their management to determine if the samples must be analyzed and what priority will be assigned.

Samples in support of criminal investigation projects will be analyzed within the LSB laboratories or sent to the National Enforcement Investigations Center (NEIC) for analysis. Scheduling of the projects must include an estimate of sample numbers, matrices, reporting limits required for meeting regulatory requirements, requested analyses, and turn-around time (TAT) requirements. The standard TAT for the laboratory from the time samples are received until results are reported is 35 calendar days for routine analyses and 45 calendar days for projects with TCLP and lead bioavailability requirements. When a project requires samples to be received by the laboratory over multiple days, the TAT is calculated based on the last day samples are received by the laboratory for the particular project. Communication of special project requirements should be noted in R4LIMS Project Notes.

3.3.2.2 Sample Acceptance Responsibility and Considerations The acceptance of samples into the LSB laboratory is the responsibility of the Section Chiefs, Branch Chief, or designated alternate(s). Factors considered by LSB management when accepting samples for analysis include whether laboratory and staff have the necessary skills, expertise, and instrumental capability to perform the environmental tests requested, a demonstration of competency is on file and the laboratory has accreditation for a specific method/analyte/technology when an accredited test result is requested. If the consideration of the above factors indicates any deficiency, lack of accreditation, or inability to perform the work, laboratory management will notify the data requestor, either verbally or in writing, and resolve any differences in methodology, QC, or scope of work to be performed.

3.3.2.3 Special Project Needs Occasionally, the laboratory receives requests to perform analyses for non-routine analytes or matrices. As a support laboratory for various EPA programs, the laboratory must maintain the flexibility to accept and perform analyses using methods and for analytes for which it is not accredited. The Region's Emergency Response program is an example where the laboratory may be called upon to perform unique analyses to protect public health and the environment. If the laboratory is requested to perform analyses for non-accredited methods, the data requestor will be informed that the laboratory may not have all QC requirements in place to meet accreditation requirements and data will be subject to qualification. Any limitations on data usability will also be explained to the customer.

3.3.2.4 Potable Water On occasion, LSB receives requests for the analysis of potable water samples. Most requests are not in support of the Safe Drinking Water Act (SDWA) found at 40 CFR Part 141. If there is any doubt as to whether the request is in support of SDWA regulations, the Section Chief or designee will contact the requestor to determine the purpose of the analysis.

3.3.2.4.1 If the request is in support of SDWA regulations, analyses must be performed by approved methods found at 40 CFR Part 141. Tables 6-1 and 6-2 list primary drinking water contaminants, including analytical method requirements. As indicated in Table 6-2, LSB does not analyze the full list of primary drinking water contaminants. If the requestor requires the analysis of a primary contaminant which LSB does not analyze, the Regional Sample Control Coordinator (RSCC) will assist the requestor in locating a laboratory that has the capability and proper accreditation.

3.3.2.4.2 If the request is not in support of SDWA regulations, then LSB may choose to use alternate methods which meet the project's data quality objectives.

3.3.2.5 NPDES These analyses requested in support of the National Pollutant Discharge Elimination System (NPDES) regulations at 40 CFR Part 136 require theuse of approved methods.

3.3.2.6 Request for Use of Specific Analytical Methods The procedure for booking samples for analysis includes information from the requestor as to the Minimum Reporting Limits (MRL) required for the project (either routine levels, or special request). LSB chooses an appropriate analytical method to meet the client's needs in consideration of the DQO provided in the Project Notes. On occasion, LSB may receive a request to use a specific analytical method. These requests typically initiate a conversation with the requestor as to the ultimate DQOs and whether the specified method is the most appropriate choice for the requestor's needs.

3.3.2.7 Documenting Communication in R4LIMS and Element® Workorder Notes Communication between the project leader and LSB personnel should be documented. This includes documenting in Element® or the project file any special requests, clarification to requests, or changes to the project. If the customer requests a statement of conformity to a specification or standard for a test or calibration, the decision rule must be communicated and agreed with the customer and documented in the R4LIMS and Element® Notes.

3.3.2.7.1 When negotiating the terms of the initial project request, documentation of verbal or written (email) communication should be included in R4LIMS project notes.

3.3.2.7.2 After samples are received, all changes to the Element® Workorder must be approved by the appropriate LSB manager (or designee) and documented in the Element® Workorder notes. Refer to SOP LSB 105G for more procedures related to sample receipt.

3.3.2.8 Quick Turn-Around Analyses If laboratory capacity allows; a quick turnaround time can be accepted. It is important that the Section Chief monitors R4LIMS for sample receipt and communicates any issues to analysts so that analyses may begin as soon as possible to accommodate the request. If laboratory capacity allows accommodation of the quick-turn requests, distribution of results will be handled in one of two ways.

3.3.2.8.1 Preliminary results will be reported to the customer within the requestedtimeframe, followed by final reporting at a later date.

3.3.2.8.2 Final results that have gone through the necessary QA/QC checks will be reported. Final results supersede all preliminary results previously submitted to a customer.

3.3.3 Canceled Projects/Samples On occasion, whole projects, samples, or analyses can be cancelled due to funding, broken bottles, etc. Element® will be used for documenting the reasons for cancelled projects, samples or analyses. Lost samples or containers will be noted in Element® and the affected analyses will be reported as "Not Analyzed."

3.4 Sample Receipt

3.4.1 Sample Acceptance Policy Samples requested for analysis within LSB are typically from

internal Agency sampling organizations, contractors, or states directly supporting EPA Region 4 Programs. As such, it would be a rare circumstance that a sample directed for analysis within LSB would be refused based on issues related to field sampling (e.g., temperature, improper containers, etc.). Any sampling anomalies for a specific project must be evaluated on individual merit for the impact upon the results and the data quality objectives of the project. If possible, the decision will be to proceed with the analyses with proper documentation and communication of the sampling anomaly and any known or suspected impacts on data quality. Documentation of the issue and the final decision for action shall be included in the project file.

Due to waste handling and sample disposal considerations, LSB's policy is not to provide storage for samples which have been or are to be analyzed by other laboratories. Exceptions to this policy may be made on a case-by-case basis by laboratory management.

3.4.2 Sample Receiving Procedure Samples are received by the LSB Sample Custodian and logged into the laboratory's LIMS system (Element®), where workorder numbers and sample identification numbers are assigned. Detailed sample receiving procedures are documented in the most current revision of LSB SOP for Sample Receiving and Custody.

3.4.3 Sample Receipt Guidelines for Analyses with Short Holding Time Requirements Some organic and inorganic analyses such as waters requiring analysis for semivolatiles, pesticides/PCBs, Total Suspended Solids (TSS), Total Dissolved Solids (TDS), unpreserved Nitrate or Nitrite, Biochemical Oxygen Demand (BOD), and unpreserved volatiles and VOA soil samples require expedited shipping to LSASD due to short holding times. Therefore, the following sample shipping guidelines must be observed by field sampling organizations.

3.4.3.1 Semivolatiles, Pesticides/PCBs, TSS, TDS, and unpreserved VOA waters Water samples collected during the week must be shipped to the lab within 48 hours of collection in order to meet the required holding time. Observation of Federal holidays and weekends should be considered when planning sampling projects.

3.4.3.2 VOA soils These samples must be shipped (or "walked in") daily to meet the 48-hour holding time. However, if the soils are collected in 40-mL vials (with or without water/methanol) and then frozen at -7 to -20°C in the field, quick delivery is not necessary. **Freezing coring devices in the field does not extend the 48-hour holding time.**

Note: Dry ice cannot be used to freeze the samples because the temperature in the cooler may be < -20 ℃.

3.4.3.3 Unpreserved Nitrate or Nitrite and BOD These samples must be shipped(or "walked in") daily to meet the 48-hour holding time.

Microbiology Drinking Water These samples generally must be shipped (or walked in) daily to meet the 30-hour collection and analysis holding time.

3.4.3.4 Short Hold Samples Held in the Field LSB does not guarantee holding times can be met for short-hold samples that are held in the field and not shipped promptly to the laboratory on the aforementioned schedules.

3.4.4 Acceptance of Samples Known to Contain Listed RCRA Dioxin-Containing Waste

3.4.4.1 Environmental samples (biota, soil, sediment, groundwater, and surface water) known or suspected to be contaminated with listed RCRA dioxin-containing hazardous waste will not be accepted by LSB. This policy has been implemented due to the special waste handling and disposal restrictions placed upon listed RCRA dioxin-containing hazardous waste.

3.4.4.2 If capacity is available, LSB will accept other environmental samples including those samples suspected of being contaminated with polychlorinated dibenzo-p-dioxins (PCDDs) and polychlorinated dibenzofurans (PCDFs), if the suspected PCDD and PCDF contamination is not due to listed RCRA dioxin-containing wastes.

3.4.4.3 Depending on a Site's history, environmental samples may contain polychlorinated dibenzo-p-dioxins (PCDDs) and/or polychlorinated dibenzofurans (PCDFs) compounds. LSB does accept and analyze samples containing PCDDs and PCDFs with the exception discussed below.

NOTE: *Any sample that is submitted to the lab for analysis has the potential to contain PCDDs/PCDFs. As a result, especially when handling soils, the analyst should use caution by preventing dust in the breathing zone while using the proper PPE and engineering controls.*

3.4.4.3.1 Scheduling Samples Some environmental media containing PCDD/PCDF may be a hazardous waste when it is excavated or removed from its natural setting due EPA's "contained-in policy" and subject to regulation under RCRA. A solid waste is a hazardous waste if it is specifically listed as a hazardous waste or exhibits one of the characteristics of a hazardous waste as defined in 40 CFR Part 261. Listed hazardous wastes are solid wastes from common manufacturing and industrial processes, specific industries and can be generated from discarded commercial products. For the purpose of this Section the following listed hazardous wastes pertaining to PCDDs/PCDFs are not accepted by LSASD for analysis: F020, F021, F022, F023, F026, F027, F028, F032, and K174, and media subject to EPA's "contained-in policy" resulting from these listed hazardous wastes. This policy has been implemented because of special waste handling disposal restrictions placed upon listed RCRA dioxin-containing hazardous wastes.

The generator is required to determine if a sampling location and resulting unused sample media after analyses would be a listed hazardous waste. (Note: for an inactive site, the generator is the Remedial Project Manager [RPM] or the On-Scene Coordinator [OSC]). Depending on a Site's history when scheduling samples in R4LIMS, the project lead or the Sample Control Coordinator (SCC) are responsible to confer with the generator for determining if specific sampling locations and/or media are determined to be a listed hazardous waste pertaining to PCDDs/PCDFs: F020, F021, F022, F023, F026, F027, F028, F032, and K174, and media subject to EPA's "contained-in policy" resulting from these listed hazardous wastes .

Samples that contain PCDDs/PCDFs can be submitted to LSASD for analysis provided that they do not meet the definition of a listed hazardous waste as described above. For any site samples suspected of containing PCDDs/PCDFs it is requested that the project

lead or SCC notify a laboratory section chief via email or use ProjectNotes about the suspicion. If historical data exists, it is requested that a summary of PCDD/PCDF concentration be provided to LSASD.

3.4.4.4 Mixed Wastes Mixed wastes are hazardous wastes which also contain radioactive material. Mixed waste is regulated under RCRA and the Atomic Energy Act. LSASD **does not** accept mixed waste for analysis.

3.4.5 PCDD/PCDF Containing Sample Disposal LSB will depend on the Project Lead, Sample Control Coordinator, and samplers' knowledge of site conditions concerning listed RCRA dioxin containing wastes. Environmental samples containing dioxin, but which do not contain dioxin-listed hazardous wastes do not require disposal as RCRA hazardous wastes. Such samples will be disposed of as ordinary environmental samples unless they are hazardous by other RCRA characteristics or meet one of the other listed hazardous waste descriptions. At the request of the Site's project manager, the unused samples after laboratory analyses can be returned to the site for disposal.

3.4.6 Sample Disposal LSB will depend on the Project Lead, Sample Control Coordinator, and samplers' knowledge of site conditions concerning listed RCRA dioxin containing wastes. Environmental samples containing dioxin, but which do not contain dioxin-listed wastes do not require disposal as RCRA hazardous wastes. Such samples will be disposed of as ordinary environmental samples unless they are hazardous by other RCRA characteristics or are a listed waste.

3.5 Sample Logging and Storage

3.5.1 Assignment of Numbers Each sample (and container) is assigned a unique identification by Element® based on the following pattern.

3.5.1.1 EYYWWNN-AN-L where EYYWWNN represents a 'Work Order' number, analogous to an R4LIMS project number and -AN-L is a sample number within the work order.

3.5.1.2 The letter E is a non-changing designation for samples analyzed by the LSB lab.

3.5.1.3 YY is a two-number designation for the calendar year.

3.5.1.4 WW is a two-letter designation for the week of the calendar year (01 through 52).

3.5.1.5 NN is a two-number designation (01 through 99) representing an incremental number of the work order received for that week. The sample number -AN- is a two-digit sample number (01 through 99) or alpha character (AA through ZZ) and -L is a unique letter designation assigned to each container received from a sampling location.

3.5.2 Storage of Samples When all numbers are assigned; sample bottles are secured within the custody room walk-in coolers or freezer. Other specifically designated sample storage locations are used by the LSB laboratory such as dedicated refrigerators for VOA samples in the VOA laboratory. Metals do not require storage in a refrigerator. Microbiology samples will be stored in the designated Microbiology laboratory

3.5.2.1 The temperatures of designated storage areas are continuously monitored using a certified wireless temperature sensor (certified annually) which interfaces with a data logger controlled through software located on the DicksonOne website. The system is Cloud-based, and the units are connected via WiFi. The software records and sends e-mail and text message alerts if the temperature falls outside of the specified range.

3.5.2.2 The acceptable temperature range for refrigerators is above freezing to 6°C. Freezer are maintained ≤ -10°C. The DicksonOne temperature monitoring software maintains a list of all temperature excursions. Analyst should acknowledge any excursion and indicate a reason for the excursion within the software system. The Custody Room refrigerator and freezer are also monitored by security personnel outside regular business hours. LSB management receives text message alerts if any of the sample custody refrigerators or freezer experience a temperature excursion during non-business hours.

3.6 Custody

3.6.1 Custody Records for all samples received by LSB are maintained within Element®. Reports can be generated on each Workorder which details the custody for each sample container.

3.6.2 Custody Room Access Key card entry controls access to the main custody room area. Entry is coordinated with the facility representative by each Branch Chief submitting request for all staff authorized for entry. It is the responsibility of the facility representative to ensure that authorized names are properly entered into the computer.

3.6.3 Custody Room Housekeeping The sample custodian or designee monitors all areas of the custody room to ensure it is maintained in a clean, orderly and secure manner. Areas needing attention shall be brought to the attention of the Section Chief (Organic or Inorganic) for which the area is designated for use. Facility cleaning staff do not routinely enter the custody room. The custody room is cleaned only by special coordination and scheduling through the facility representative.

3.6.4 Documentation of Custody Documentation of sample custody from cradle to grave is accomplished by the use of custody seals placed on the sample coolers that are secured by field sampling personnel, a Chain of Custody (COC) form initiated at the time of sample collection, field log books, individual analysis logs, Element® and sample disposal memos and records. The original field custody form, along with a computer printout of the requested analytical tests (workorder printout), is maintained in the LSASD Project files. A copy of the field custody form and a copy of the computer print-out are sent to the project team personnel responsible for sample collection associated with a specific project. It is the project leader's responsibility to check the computer print-out against the COC record for accuracy as it relates to analyses requested for the project, the sampling station identification and other meta data information.

3.6.5 Assuming Custody for Sample Analysis LSB utilizes Element® to perform sample check-out and check-in to/from their storage locations. To receive samples for analysis, an analyst must assume custody of the samples (including those 'aliquoted' in the custody room such as frozen tissue) and designate the location where the container will be relocated for use. When the need for the sample

container is complete, custody is relinquished by setting the location of the container to either the original storage location or marking the container as "Disposed" in Element®.

3.6.6 Tracking Custody of Sample Extracts, Digestates and/or Leachates

3.6.6.1 LSB tracks the custody of sample extracts, digestates and/or leachates throughout the prep and analysis phases of the samples through Element®.

3.6.6.2 The custody of the extracts, digestates and/or leachates are transferred from the preparation personnel to the analytical personnel using the Custody tracking tools in Element®. The new home location of the extract is assigned in Element®, and also documented on the bench sheet. All sample custody transfers are tracked within Element® and Custody logs generated for inclusion in the project file.

3.6.6.3 Batch IDs are assigned automatically by Element® and are in the format 'YYMMnnnn' where:

3.6.6.3.1 'YY' is a two-digit number identifying the year of the batch,

3.6.6.3.2 'MM' is a two-digit number identifying the month and

3.6.6.3.3 'nnnn' is a four-digit number representing the incremental batch created that month.

3.6.6.4 If a batch of samples requires re-extraction or re-digestion, the samples are re-batched within Element®.

3.6.6.5 Batch IDs are also used for tracking QC data associated with a batch of environmental samples. That is, any method blank, Laboratory Control Standard (LCS) data or matrix QC data associated with a particular batch of samples is assigned a unique ID associating it with the batch.

3.6.6.6 Transfer of Custody from LSB

After LSB has assumed custody of the samples, there may be requests for samples to be transferred to other individuals or organizations. Samples shall only be removed from LSB custody by transferring official custody using appropriate COC forms and notations in Element®. All custody transfers of this nature must be coordinated through the sample custodian or designee.

3.7 Review of Custody Records

Review of custody records are performed by the QAC or designee(s) prior to the inclusion of documents in the project file. For a list of the custody records that must be included in the project file, refer to LSB SOP 105G.

Table 3-1
Recommended Preservation & Holding Times

Analytical Group	Soil/Sediment [1]		Water [1, 2] and Waste Water		Waste		Tissue	
	Pres[3] Amt[4] Container Type[5]	Hold[6]	Pres[3] Amt[4] Container Type[5]	Hold[6]	Pres[3] Amt[4] Container Type[5]	Hold[6]	Pres[3] Amt[4] Container Type[5]	Hold[6]
Inorganics								
Acidity	NA	NA	Ice-4ºC 500 mL P, FP Fill completely and cap tightly	14 days	NA	NA	NA	NA
Alkalinity	NA	NA	Ice-4ºC 500-mL P, FP Fill completely and cap tightly	14 days	NA	NA	NA	NA
BOD5	NA	NA	Ice-4ºC 2-L P, FP	48 hrs	NA	NA	NA	NA
BOD – Long Term	NA	NA	Ice-4ºC 1 gal (x2)[15] P, FP	48 hrs	NA	NA	NA	NA
Bromide	NA	NA	Ice-4ºC 500-mL P, FP	28 days	NA	NA	NA	NA
Chloride	None 8-oz G	Not specified	None 500 mL P, FP	28 days	NA	NA	NA	NA
Chlorine - Residual	NA	NA	None 500-mL P	Immediate	NA	NA	NA	NA
Chromium VI (hexavalent)	Ice-4ºC 8-oz G	Extract – 30 days Analysis – 7 days[7]	Filter immed.[9] Ice-4ºC 1L P, FP Buffer to extend HT[3]	24 hrs 28 days if buffered[3]	None 8-oz G	Extract – 30 days Analysis – 7 days[7]	NA	NA
Cyanide	Ice-4ºC 8-oz G	14 days14 days	NaOH to pH>12, ascorbic acid[8], Ice-4ºC 500-mL P, FP	14 days	None 8-oz G	Not specifie d	NA	NA
Dissolved P, total	NA	NA	Filter immed.[9] Ice-4ºC H_2SO_4 to pH<2 500-mL P, FP	28 days	NA	NA	NA	NA
Fluoride	None 8-oz G	Not specified	None 500-mL P	28 days	NA	NA	NA	NA
Hardness (calc)	NA	NA	NA Separate bottle not required, calculated from metals scan	NA	NA	NA	NA	NA
Mercury, Routine	Ice-4ºC 8-oz G	28 days[28]	HNO_3 to pH<2 1-L P, FP	28 days[28]	None 8oz G	180 days	Freeze, 5 g 8-oz G, Al foil or plastic	Not specified
Mercury – TCLP	None 8-oz G	28 days 56 days[10]	None 1-L P, FP or 1-gal. P, FP, G if multiphase (>0.5% and <10%solids)	28 days 56 days[10]	None 8-oz G or 1-gal P, G if multiphase (>0.5% and <10% solids)	28 days 56 days[10]	NA	NA
Mercury – UTL	Ice-4ºC 8-oz G	90 days	HCl to pH<2 1L FP (bottles require prescreening and special	90 days	None 8-oz G	Not specified	Freeze, 5 g 8-oz G, Al foil or plastic	Not specified

			handling)					
Metals, except mercury, includes lead bioavailability	None 8-oz G	6 months	HNO_3 to pH<2 1-L P, FP	180 days 6 months	None 8-oz G	6 months	Freeze, 15 g 8-oz G, Al foil or plastic	Not specified
Dissolved Metals, except mercury	NA	NA	Filter immed[9], HNO_3 to pH<2 1-L P, FP	180 days 6 months	NA	NA	NA	NA
Metals – TCLP (except mercury, see above)	None 8-oz G	180 days 360 days[11]	None 1-L P, FP or 1-gal. P, FP, G if multiphase (>0.5% and <10% solids)	180 days 360 days[11]	None 8 oz G or 1-gal. P, G if multiphase (>0.5% and <10% solids)	360 days[11]	NA	NA
Nitrate (requires two containers: one unpreserved and a 2nd preserved)	Ice-4ºC 8-oz G	Not specified	Ice-4ºC 500 mL P, FP AND 2nd container Ice-4ºC, H_2SO_4 to pH<2 500-mL P	48 hrs	NA	NA	NA	NA
Nitrite	Ice-4ºC 8-oz G	Not specified	Ice-4ºC 500-mL P, FP	48 hrs	NA	NA	NA	NA
Nutrients (ammonia, TKN, NO_3+NO_2-N, total phosphorus)	Ice-4ºC 8-oz G	Not specified	Ice-4ºC, H_2SO_4 to pH<2 500-mL/ parameter or 1-L total P, FP	28 days	NA	NA	NA	NA
Ortho-P	NA	NA	Ice-4ºC 500-mL P, FP	48 hrs	NA	NA	NA	NA
Ortho-P (when equating dissolved with Ortho-P)	NA	NA	Filter immed[9], Ice-4ºC 500-mL P, FP	48 hrs	NA	NA	NA	NA
pH	None 8-oz G	Not specified	None 500-mL P, FP	Immediate except 24 hrs for RCRA[12]	None 8-oz G	24 hrs for aqueous, otherwise not specified	NA	NA
Solids Series (TS, TSS, TDS, TVSS, etc.)	NA	NA	Ice-4ºC 1-L P, FP	7 days	NA	NA	NA	NA
Sulfates	Ice-4ºC 8-oz G	Not specified	Ice-4ºC 500-mL P, FP	28 days	NA	NA	NA	NA
TOC	Ice-4°C 8-oz G	Not Specified	Ice-4°C, H2SO4 to pH<2 500-mL P, FP	28 days	NA	NA	NA	NA
Dissolved TOC	NA	NA	Filter immed[9], Ice 4°C, H_2SO4 to pH<2 500-mL, P, FP	28 days	NA	NA	NA	NA
Organics								
Alcohol - Percent	NA	NA	Ice 1-gal. G/	Not Specified	None 8-oz G	Not Specified	NA	NA
TCLP Extractables (Pesticides, Herbicides, Semivolatiles)	Ice-4ºC 8-oz G	61 days[13]	Ice-4ºC 1-L (x2 per fraction)[15] G/A	61 days[13]	None 8-oz G	61 days[13]	NA	NA
	For multi-phase samples, extra sample volume must be collected for TCLP analysis.							
Extractables (Pesticides/PCBs, SVOAs	Ice-4ºC 8-oz G	54 days[14]	Ice-4ºC 1-L G/A[15]	47 days[16]	None 8 oz G	54 days[14]	Ice & Freeze 30 g Al Foil	Not specified
Extractables-Herbicides	Ice-4ºC 8-oz G	54 days[14]	Ice-4ºC 40-60 mL VOA Vials G/A[15]	47 days[16]	None 8 oz G	54 days[14]	Ice & Freeze 30 g Al Foil	Not specified

Extractables/ Pesticides/PCBs – residual chlorine present	NA	NA	Ice-4ºC 3 ml of 10 % sodium thiosulfate per gallon HCL, (pH,2) 1-L (x2 per fraction)[15] G/A	44 days[17]	NA	NA	NA	NA
Flashpoint	N A	NA	NA	NA	None 8-oz G	Not specified	NA	NA
Methane/Ethane/ Ethene	NA	NA	HCL (pH<2), Ice-4ºC 40-mL(x3)[15] G/S	14 days	NA	NA	NA	NA
Org Halide (TOX)	Ice-4ºC 8 oz G	28 days	Ice-4ºC H_2SO_4 to pH<2 1-L G/A	28 days	NA	NA	NA	NA
PFAS, Per- and Polyfluoroalkyl substances	Ice ≤ 10ºC 50 mL polyoxpropylene Digitube™	42 days[30]	Ice ≤ 10°C Pre-weighed 15-mL Polypropylene (x2)	42 days[30]	Ice ≤ 10°C Pre-weighed 15-mL Polypro pylene (x2)	42 days[30]	NA	NA
Carbamates	NA	NA	Ice-4ºC 60-mL G/A (x2)	14 days[27]				
Phenols (analyzed as semivolatile compounds)	Ice 4ºC 8-oz G	54 days[14]	Ice-4ºC 1 L(x2)[15] G/A	47 days[16]	None 8-oz G	54 days[14]	Ice & Freeze 30 g Al Foil	Not specified
Volatile Organics								
Volatile Organics Method 5035A	Ice-4ºC 5 g (x3)[18] E or equivalent[19]	48 hours iced/14 days frozen[20]	NA	NA	Ice-4ºC 8-oz G[26]	14 days[21]	NA	NA
Volatile Organics Method 5035A	Ice-4ºC 5 g (x3)[18] into tared 40-mL VOA vials[19]	48 hours iced/14 days frozen[20]	NA	NA	NA	NA	NA	NA
Volatile Organics Method 5035A	Ice-4ºC 5 g (x3)[18] into tared 40-mL VOA vials containing 5 mL water[19, 22]	48 hours iced/14 days frozen[20]	NA	NA	NA	NA	NA	NA
Volatile Organics Method 5035A	-7 to -20ºC 5 g (x3)[18] into tared 40 mL VOA vials[19,22]	14 days frozen	NA	NA	NA	NA	NA	NA
Volatile Organics Method 5035A	-7 to -20ºC 5 g (x3)[18] into tared 40-mL VOA vials containing 5 mL water[19, 22]	14 days frozen	NA	NA	NA	NA	NA	NA
Volatile Organics Method 5035A	Ice-4ºC 5 g (x3) into tared 40-mL VOA vials containing 5 mL methanol[19]	14 days	NA	NA	NA	NA	NA	NA
Volatile Organics no residual chlorine present	NA	NA	Ice-4ºC 40 mL (x3)[15] G/S	7 days	NA	NA	NA	NA
Volatile Organics no residual chlorine present	NA	NA	0.2 mL 1+1 HCL (pH<2), Ice-4ºC 40-mL (x3)[15] G/S	14 days	NA	NA	NA	NA

Volatile Organics residual chlorine present	NA	NA	3mg $Na_2S_20_3$, 0.2 mL 1+1 HCl (pH<2), Ice-4ºC 40-mL (x3)[15]G/S	14 days[23]	NA	NA	NA	NA
Volatile Organics TCLP	Ice-4ºC 2-oz G	28 days[24]	Ice 4ºC 40-mL (x3)[15]G/S	NA 28 days[24]	Ice-4ºC 8-oz G[26] If <10% solids, 4x 8-oz G	28 days[24]	NA	NA
Volatile Organics in Air	Preservation: closed, leak-free valve with tightened cap. Amount: preferably 10-14 psia Container: passivated 6-liter canister	30 days	NA	NA	NA	NA	NA	NA

Table 3-2
Recommended Preservation & Holding Times-Microbiology

Analytical Group	Non- Potable Water		Regulated Potable Water	
	Pres **Amt** **Container Type**	**Hold**	**Pres** **Amt** **Container Type**	**Hold**
Ecoli	Cool[29] 120 mL ***Sterilized*** plastic, non-corrosive glass bottles or Whirl-pak bags	8 hours from sampled to placement in the incubator	Cool[29] 0.008% $Na_2S_2O_3$[30] 120 mL ***Sterilized*** plastic, non-corrosive glass bottles or Whirl-pak bags	30 hours from sampled to placement in the incubator
Total Coliforms	Cool[29] 120 mL ***Sterilized*** plastic, non-corrosive glass bottles or Whirl-pak bags	8 hours from sampled to placement in the incubator	Cool[29], 0.008% $Na_2S_2O_3$[30] 120 mL ***Sterilized*** plastic, non-corrosive glass bottles or Whirl-pak bags	30 hours from sampled to placement in the incubator

Table 3-3
Recommended Preservation & Holding Times-LSB Certified Drinking Water Methods

Parameter/ Method	Preservative	Suggested Sample Size	Container Type	Sample Holding Time
Metals EPA 200.7/EPA 200.8	HNO3 pH<2	500 mL	Plastic or Glass	6 months
Mercury EPA 245.1	HNO3 pH<2	500 mL	Plastic or Glass	28 Days
Fluoride EPA 300.0	None	100 mL	Plastic or Glass	28 days
Nitrate (chlorinated) EPA 353.2	Cool, 4°C Non-acidified	1000 mL	Plastic or Glass	48 hours
Nitrite EPA 353.2	Cool, 4°C Non-acidified	1000 mL	Plastic or Glass	48 hours
Nitrate+ Nitrite EPA 353.2	H2SO4 pH<2	1000 mL	Plastic or Glass	28 days
Semi-Volatiles/ Pesticides 525.2	Sodium Sulfite, Dark, Cool, 4°C, HCL pH<2	1 Liter*	Amber Glass with PTFE-lined Cap	14 days extraction/30 days analysis
Volatile Organics EPA 524.2	Ascorbic Acid or Sodium Thiosulfate HCL pH,2, Cool 4°C	40 ml X 3 vials*	Glass with PTFE-lined Septum	14 days

* Additional volume necessary for MS/MSD (9 vials total for VOAs, 3 bottles total for extractables).

General Notes:

NA = Not applicable
Pres = Preservation Immed = Immediate

Footnotes:

[1] LSB's policy is that where the sample preservation is specified at 4˚C, the acceptable temperature range for samples during shipping and storage is from above the freezing point of water to 6°C. Where preservation is specified as "Frozen or Freeze", the acceptable temperature range for samples during shipping and storage is -7 to -20ºC.

[2] Consult 40 CFR Part 136 Table II: Required Containers, Preservation Techniques, and Holding Times for latest NPDES requirements.

[3] **Preservatives:**

Ice – Sufficient ice must be placed in the shipping/transport container to ensure ice is still present when the samples are received at the lab.

HCL – Hydrochloric Acid used as a preservative must be present at concentrations ≤ 0.04% by weight (pH ~ ≤ 2.0) as specified in 40 CFR 136.3, Table II, footnote 3. The proper amount of HCl is added to the sample container at the laboratory prior to sampling.

H_2SO_4 – Sulfuric Acid used as a preservative must be present at concentrations ≤ 0.35% by weight (pH ~ ≤ 1.15), as specified in 40 CFR 136.3, Table II, footnote 3.

NaOH – Sodium Hydroxide) used as a preservative must be present at concentrations ≤ 0.080% by weight (pH ~ ≥ 12.30), as specified in 40 CFR 136.3, Table II, footnote 3.

HNO_3 – Nitric Acid used as a preservative must be present at concentrations ≤ 0.15% by weight (pH ~ ≤ 1.62), as specified in 40 CFR 136.3, Table II, footnote 3.

Chromium VI buffer – A concentrated buffer is used to extend the holding time for hexavalent chromium samples from 24 hours to 28 days and uses constituents as allowed by EPA guidance found at: http://water.epa.gov/scitech/methods/cwa/questions-cr6.cfm. The sample preservation buffer is prepared by carefully dissolving 330 g ammonium sulfate and 50 g sodium hydroxide in about 500 mL of deionized water. The solution is allowed to cool and 260 mL of 29% ammonium hydroxide is added and solution is diluted with deionized water to a final volume of 1 L. In-house studies revealed that the equivalent of 1% of buffer volume is needed to preserve samples to attain the pH range (pH 9.3 to 9.7, 10 mL buffer for a 1-L sample) as specified in 40 CFR 136.3 Table II. Adding preservative to sample bottles prior to shipment to the field is recommended to minimize sampler contact with the buffer.

NA - Not Applicable. No sample preservation is required.

[4] Amount: The amounts listed must be considered approximate requirements that are appropriate for most media. If a particular medium to be sampled is very light, more sample volume may be required to obtain the necessary mass for the analysis.

[5] **Container Type:**
G = Glass
P = Polyethylene
FP = Fluoropolymer
E = Coring device
C = Cubitainer
S = Septum Seal
A = Amber
W = Whirl-Pak ™
GF/F = Glass Fiber Filter PP = Polypropylene

[6] Holding Time – Stated in days unless marked otherwise. A holding time of "immed" (immediate) indicates that the sample is to be analyzed within 15 minutes (40 CFR 136 Table II). "Not Specified" indicates no holding time is specified in the method or by the related program.

[7] Chromium VI (hexavalent) – 1 month until extraction, 7 days to analysis of extract. Store at ≤ 6°C until analyzed (SW-846, Table 3-2).

[8] Use ascorbic acid only if the sample contains residual chlorine. To test for residual chlorine, place a drop of sample on potassium iodide-starch test paper. If the test paper turns blue, residual chlorine is present. Add a few crystals of ascorbic acid and re-test until the paper no longer turns blue. Add an additional 0.6 g of ascorbic acid for each liter of sample.

[9] Filter on-site. Use 0.45-µm-filter for dissolved parameters.

[10] TCLP Mercury – 56 days: 28 days to TCLP extraction plus 28 days to analysis of extract (SW-846, Method 1311, Section 8.5).

[11] TCLP Metals – 360 days: 180 days to TCLP extraction plus 180 days to analysis of extract (SW-846, Method 1311, Section 8.5).

[12] pH – Aqueous RCRA samples only – a 24-hour holding time from receipt is allowed.

[13] TCLP Extractables – 61 days: 14 days to TCLP extraction, 7 days to solvent extraction, 40 days to analysis of extract (SW-846, Method 1311, Section 8.5).

[14] Extractables – 54 days: 14 days to extraction, 40 days to analysis of extract (SW-846, Table 4-1).

[15] Collect double volume for MS/MSD analyses at one station per 20 or one per project if < 20 samples in project.

[16] Extractables, water, no residual chlorine present – 47 days: 7 days to solvent extraction, 40 days to analysis of extract (SW-846, Table 4-1).

[17] Extractables – Drinking water, residual chlorine present: 14 days to extraction, 30 days to analysis of extract (EPA 525.2).

[18] Collect triple volume (9 vials) for MS/MSD analyses at one station per 20 samples or one per project if < 20 samples in project or SDG.

[19] Volatile Organics Soil Samples – A separate 2-ounce glass container or 40-mL vial is needed in order to determine percent solids for soil samples. Alternatively, an extra coring device will suffice. Do not freeze percent solidscontainer!

[20] Volatile Organics Soil Samples – Contents of coring device must be analyzed or transferred to VOA vial containing organic-free water and preserved within 48 hours. Preservation is accomplished by sealing and freezing the VOA vial. The sample must be analyzed within 14 days of collection date. Soil samples received in VOA vials must be analyzed within 48 hours or frozen and analyzed within 14 days of collection date. Refer to Method 5035A, July 2002, Table A1 for additional details.

[21] Wastes are dissolved in methanol at the analytical lab.

[22] One 40-mL vial should be empty so that a methanol extraction can be performed if a high-level VOA is needed. Alternately, one tared 40-mL vial may contain 5 mL methanol.

[23] Volatile Organics Waters – 14 days for acid preserved, 7 days if not preserved (40 CFR 136 Table II).

[24] TCLP Volatile Organics – 28 days: 14 days to TCLP extraction plus 14 days to analysis of extract, or 7 days to analysis of extract if not preserved following extraction.

[25] Collect in 50-mL plastic centrifuge tube. Keep sample in the dark. Freeze for up to 24days.

[26] Waste samples collected for volatile analysis are transported in secondary containment.

[27] Sampled to analyzed.

[28] Holding time for routine mercury by Method 200.8, is 28 days from sample collection to digestion and 28 days from digestion to analysis.

[29] Cool = below 10°C but not frozen.

[30] PFAS – 42 days: 28 days to extraction, 14 days to analysis of extract

CHAPTER 4

General Laboratory Practices

4.1 Good Laboratory Practices

4.1.1 Policy Following good laboratory practices in all aspects of the organization's operations is intrinsic to the production of quality analytical data. Recognizing the necessity of maintaining control over laboratory operations, the subsequent sections outline provisions for maintaining quality in all laboratory practices and procedures.

4.1.2 Corrections to Records

4.1.2.1 Corrections to hardcopy records shall be made using a single line-out with the date and the signature or initials of the analyst making the corrections. No changes shall be made with any technique that obliterates the original such as erasures or correction fluid. All records and corrections shall be in ink. Pencil shall not be used on analytical records. When corrections are due to reasons other than transcription errors, the reason for the correction shall be documented. When multiple corrections occur on a single page, all corrections must be signed or initialed and dated.

4.1.2.2 Corrections to final data must be done by reprinting and re-transmitting through Element®, the final data report forms with the corrected results. Corrected results shall be transmitted with a case narrative explanation that the report is to correct data previously reported. The original report name should be included in the case narrative. An official copy of all corrected data, along with the original data, must be retained in the project file and must contain clear documentation as to why the corrections were necessary.

4.1.3 Following SOPs/LOQAM It is the policy of the LSB that the quality system and technical SOPs and LOQAM be followed by all LSB staff, SEE employees, and by the ESAT contractors. Documentation will be maintained in each employee's training file that he/she has read, understood and agreed to follow the latest version of SOPs and LOQAM. Significant deviations from the LOQAM or SOPs shall be coordinated with the appropriate Section Chief, Branch Chief (if necessary) and Laboratory Quality Manager (QAC); the rationale for the deviation shall be clearly documented and included in the project file. When it is determined prior to receipt of samples that standard procedures will need to be modified for a specific project, these proposed deviations will be documented in R4LIMS Project Notes for review by the project leader.

4.1.4 Method Modifications Where allowed by regulation or program, method modifications are encouraged as new technologies are developed which result in analytical efficiencies, lower reporting limits, pollution prevention and increased precision and accuracy. Restrictions apply to the modification of Safe Drinking Water Act (SDWA) and National Pollutant Discharge Elimination System (NPDES) methods. Methodology in support of the Safe Drinking Water Act (40 CFR Part 141) shall not be modified unless specified in the individual method. Permitted modifications are documented in memos from OGWDW or approval as an Alternate Test Procedure (ATP). When these modifications are used, LSB's SOP should state that an allowed modification is being utilized, and reference the specific memo allowing the modification.

4.1.5 Manual Peak Integration Some of the analytical techniques utilized by LSB employ a chromatogram that displays time versus signal, which when integrated, provides a response that is used to calculate concentration.

4.1.5.1 Analysts are required to review the automated electronic data processing (i.e., integration) for accuracy and consistency with appropriate data reduction techniques.

4.1.5.1.1 Some electronic reductions can result in incorrect actions by the system software, and for these instances a manual override and correction of the electronic processing is appropriate. Examples of this may be such items as integration of an incorrect peak or misplacement of the baseline in peak integration due to poor peak shape or interferences. Guidance related to manual integrations is documented in Data Review Guidelines or technical SOPs.

4.1.5.1.2 Manual override actions are appropriate only to correct inaccuracies and shall be done in accordance with sound analytical procedures. When a manual override of the electronic process is performed, most of the current commercially available software packages provide an automated notation on the quantitation report showing that a modification to a peak occurred. When a manual modification of a peak occurs, the analyst shall provide documentation for the file to include a hard-copy representation of the before and after correction, the action taken and why. The action should be concurred by the Technical Data Reviewer or Section Chief and include an initial and date of the review of the modification on the data review form.

4.1.5.1.3 The software option for denoting a manual integration in the quantitation report must always be activated. There shall be no manipulation of the software to conceal an electronic correction that is used to report results.

4.1.6 Checklists-Primary Analyst/ Technical Reviewer Analytical data reduction activities for both the primary analysis and the technical review shall be documented using the appropriate data review checklist. Checklists are designed for the procedure(s) being performed. The individual data review checklists for organic and inorganic analyses are maintained on LSASD's local area network drive (LAN) as controlled documents.

4.2 Document Control/File Management

4.2.1 It is the policy of LSB to maintain complete and accurate records which document all laboratory activities in a readily accessible and understandable manner. These records shall include, but are not limited to: equipment identifier, analytical methods and related activities such as sample receipt, preparation, data review and transfer of custody. Additionally, LSB's policy that all documents issued as part of LSBLSASD's Quality System shall be controlled in the following way.

4.2.1.1 All documents are reviewed and approved by an authorized approving official prior to being issued. Approving officials are Section Chiefs, Branch Chief or QAC. The LSASD Deputy Director is the approving official for all quality system operating procedures.

4.2.1.2 Authorized revisions shall be available to all personnel at the point-of-use.

4.2.1.3 A master list shall be maintained which identifies the current revision (or equivalent) and its distribution status.

4.2.1.4 Documents shall be periodically reviewed for suitability or needed revisions.

4.2.1.5 Obsolete documents shall be removed from the point(s)-of-issue (and marked as obsolete).

4.2.1.6 SOPs that are expired but have not been updated or reviewed will stay in effect until the new version is effective.

4.2.1.7 Archived documents shall be marked as such.

4.2.1.8 LSB procedures for document control are detailed in LSASDPROC-1000-Document Control, most current version.

4.2.2 Internal Chain-of-Custody (COC) LSB analysts check samples in/out of the Custody Room or designated storage location through Element® (see LSB SOP 105G). Custody of extracts and digestates are tracked on Element®-generated bench sheets and custody logs, which are included in the project file.

4.2.3 COC Receipt Form The sample custodian/designee receives a COC record from the field samplers with every shipment of samples (see LSB SOP 105G).

4.2.4 Instrument/Maintenance/Analysis/Preparation Logbooks Each analysis area maintains records using logbooks which are kept within the laboratory work areas when active or in the appropriate records archive. All entries in instrument, sample preparation and other logs are made legibly in ink at the time of the observation or performance of the operation. When full, these logbooks are archived using the Logbook Transfer Form and given to the QAC, or designee. The logbooks are transferred to the LSASD Records Room. If a logbook is discontinued prior to using all the pre-printed pages, a single line is be drawn through the first vacant page and a note added stating that the logbook has been discontinued. This note is dated and initialed by the analyst.

4.2.4.1 Instrument/Analysis Logbooks

4.2.4.1.1 Instrument logs shall indicate the unique instrument ID, date of analysis, analyst and samples which have been analyzed. The logbook shall contain or reference a record of which options or analytical conditions were used for analysis. Where appropriate, instrument acceptance criteria (e.g., tune criteria, sensitivity checks) should be noted in the logbook.

Note: LSB is currently in the process of transitioning to electronic records and project files. This transition is multi-tiered and is targeted for completion in 2021. As the transition progresses, hard-copy logbooks will be replaced with an electronic record. . All documentation requirements for hard copy records will apply to the electronic records, hard copies will be eliminated only when the electronic records have proven to provide the same level of detail as the hard copy records The required elements of this section must be captured within the electronic records prior to full implementation of electronic records and data packages.

4.2.4.1.2 Electronic records, including spreadsheets which contain original measurements, may be used to create logbooks if all the required information can be captured by the instrumental software; however, a sequential analysis log must still be created and maintained. This may be accomplished by printing a copy of the electronic record and including it in a notebook. These sequential logs must also include failed runs, or sequences which were abandoned prior to completion. In the event of a failed or abandoned run, the log should be documented to indicate the reason the run was discontinued (i.e., Initial Calibration Verification (ICV) exceeded method limits, continuing calibration check exceeded method limits, etc.). Any electronic records must accurately reflect actual analytical information.

4.2.4.1.3 When a pre-determined number of pages has been accumulated (e.g., 50 pages), the individual records are combined into a single-bound logbook and retained as specified above.

4.2.4.1.4 For analyses with holding times < 72 hours, or when time-critical or method-specified times are included in the analysis, the time of analysis must also be recorded.

4.2.4.2 Preparation Logbooks Preparation logs shall document all information to reconstruct the preparation of samples, reagents, and standards, and should include, but are not limited to: weights, volumes, lot number of digestion tubes, balance used, weights used, certification dates of balances and weights used, reagents/standards used, preservation checks, units and any cleanup procedures. Electronic traceability via Element® is the accepted option for documenting standard preparation. As Element® is used as the standard prep log, it is subject to all the requirements of this section.

4.2.4.3 Instrument Maintenance Logbooks Each major instrument shall have a maintenance logbook. Maintenance logs are required to be bound and page numbered. At a minimum, instrument serial number, software version, in- service date (if known) and unique name shall be included in the log. Maintenance, service and repair records are maintained in these logbooks. Preventive maintenance schedules should be noted in the log. When a service or maintenance call is completed by the vendor, the analyst is required to place a copy of the documentation or transcribe the details for the work that was performed on the instrument in the logbook. The original work order invoice should be provided to the Quality Assurance and Program Services Branch for payment. Active logbooks are maintained within the laboratory where the instrument is located and should be maintained with the instrument throughout its useful life. At such time the instrument is removed from service the logbook is transferred with the Logbook Transfer Form to the QAC, and then to the LSASD Records Room.

4.2.4.4 Other Some analytical methods are manual and do not use analytical instrumentation to generate a result (e.g., solids). For these methods, LSB relies on spreadsheets or other calculating software for recording/documenting original observations made, such as weights. All spreadsheets or other calculating software used within LSB as logbooks or used in support of data generation will be validated and controlled. All cells, except informational input cells, will be locked to prevent alteration of a formula or essential static information, such as the unique identifier. The entire spreadsheet will be password protected. The password will be assigned by the QAC at the time of posting. Copies of any spreadsheet used must be obtained from the password protected official, posted version on the LAN. Prior to posting and use, all calculations in spreadsheets will be hand-validated by the responsible party and submitted through the Section Chief to the QAC for approval and posting.

4.2.5 LSB Laboratory Operations and Quality Assurance Manual The most current version of the LSB LOQAM is maintained electronically by the QAC. The manual is available to all staff as "read-only" on the LAN at K:\LSB\Current Documents\QA Manual - LOQAM. While copies of the manual may be printed, it is the responsibility of each individual to ensure that they are using the most current version. The LSB LOQAM shall be maintained as described below.

4.2.5.1 The quality manual will be reviewed in totality at least once each year. The Section Chiefs will solicit feedback from their section and incorporate all changes into the proposed version, which is reviewed by management and the QAC. The annual total review of the manual shall be completed as near as possible to the anniversary date of the most recent fully reviewed manual.

4.2.5.2 The annual review and versions of less comprehensive reviews, as described in Section **4.2.5.3** below, that are in use for any given period of time, will be tracked by date. Revisions resulting from less than total review of the manual do not reset the annual review clock. The next full review shall commence at an appropriate date in order to maintain the annual full review schedule described in Section 4.2.5.1 above.

4.2.5.3 To keep the manual as up to date as possible, changes may be made at any time deemed appropriate during the calendar year. When this occurs, the redline strikeout version of the manual will be kept as a record of the changes. The original signed copy will be maintained by the QAC. Signatories for the change authorization will be Organic and Inorganic Section Chiefs, QAC, and the Branch Chief. The effective date of the change will be the signature date of the Branch Chief.

4.2.6 Standard Operations Procedures/Methods SOPs shall be written based on agency guidance EPAQA/G-6 "Guidance for the Preparation of Standard Operating Procedures for Quality Related Documents". Detailed policies and procedures for the preparation, review and change of both administrative and technical SOPs are found in LSASDPROC-1000-Document Control. Access to the technical SOPs for the methods in use in each laboratory shall be available within the laboratory for reference purposes; however, the official copy of each SOP resides on the LAN in the K:\LSB\Current Documents\SOPs folder. Printed copies of the SOPs contain a watermark indicating the copy is an uncontrolled copy.

4.2.7 Project Files

4.2.7.1 A project file is all pertinent information and documentation related to a group of samples that are associated with a unique identifier (project number) assigned by the division's R4LIMS Project Log software. Each analytical project has a "project file" which contains when possible, originals of all the information. In some instances, such as bound logbooks, it will be necessary to make copies; however, it is essential that the copy placed in the file be the exact copy of the original.

4.2.7.2 The project file contains all data (or copies thereof) used to produce the final data report. For example, if an analytical run is not used because of a calibration failure, it need not be retained in the project file, the instrument/analysis log (Section 4.2.4.1) will include this information. However, if the failed run was used to determine the level of dilution required by a sample in the final run, it should be maintained. See SOP LSB 118G for the required elements

needed for a complete project file.

4.2.7.3 If corrections are deemed necessary to original project file documents after the project file has been completed, the primary analyst or data reporter will ensure that a copy of the corrected documents are placed in the file. If the final data reports, either in part or in total, must be corrected or clarified and reported again, a new final report shall be generated for transmittal of the correction, explaining the nature of the correction and placed into the project file along with the corrected data. Additional documents added to the project file must be accounted for on the Project Inventory Form attached to each project file.

4.2.7.4 The analytical information is maintained by analysts while the analyses are in progress. Each completed data packet is transferred to the technical reviewer for review, submitted to the Section Chief or designee for final reporting. Once the project has been reported, all data packets that make up that project file are compiled and inventoried prior to transfer to the LSASD records room for inclusion into the project file. It is essential that the hard copies placed into project files exactly reflect the electronic data produced for the project. LSB includes an inventory checklist that accounts for each page of the project file (See LSASDPROC-1015 Project File Inventory). It is current Division policy that hard copy project files are the official record and e-data files are not required to be maintained. Ultimate retention and disposal of all records will be in accordance with Agency record management rules and regulations as detailed in the "Records Management Standard Operating Procedures, Region 4 Laboratory Services and Applied Science Division."

Note: While data work-up is in progress, raw data may be logged out of the LSASD facility for review at a teleworking location. A log will be maintained in the project file indicating which data package(s) were removed from the LSASD facility, the responsible party and the return date. Under no circumstances will any portion of a project file be removed from the LSASD facility for teleworking purposes after the data has been reported, unless authorized by the Branch Chief.

If any data is maintained in electronic-only format (such as PDF), it shall be stored to allow retrieval of the information for at least five years after completion of the project. Any software supporting electronic-only data must be also available for the same period of time, even if the software/instrumentation has been removed from routine service.

4.2.8 Confidentiality of Data

4.2.8.1 LSB does not, under normal operations, accept samples considered to require the use of Confidential Business Information (CBI) procedures. Therefore, most of the information generated by LSB is accessible under the Freedom of Information Act (FOIA). The exception is data from all criminal investigation projects which will not be reported to anyone other than project managers leading the criminal investigations or to individuals that are authorized by LSB management. Criminal projects are so noted when logged into R4LIMS.

4.2.8.2 Data transmittal memos contain a confidentiality notice stating the data is only for the use of the specific individual addressee(s). LSB does not release data to anyone other than the project manager or those approved by the project manager to receive results.

4.2.9 General Correspondence All general written correspondence (e.g., memos and letters) from LSB technical staff to any party external to LSB, but internal to LSASD, shall be reviewed and approved by the respective Section Chief and shall have the Section Chief as a "THRU" signatory. All correspondence external to the Division shall also include the Branch Chief as a "THRU" signatory. Correspondence related to a specific project shall be filed in the project file. General correspondence shall be forwarded to the QAC for filing. As appropriate.

4.2.10 Training Files A training file shall be maintained for each LSB, SEE employee, and ESAT staff member by the QAC. The file shall contain all training documentation, including conference and seminar participation. Training files may be maintained in hard copy, electronic format, or a combination of both. Refer to LSASDPROC-1003.

4.2.11 QA/QC Records LSB maintains project specific records in the project file. Proficiency records, method development records and managerial reports are examples of QA/QC records maintained by the QAC. Refer to LSASDPROC-1001.

4.2.12 Document/Forms Revisions Many forms and documents (e.g., SOPs, data review check lists, extraction/preparation log forms) are generated within LSB and handled as controlled documents. All forms will be controlled by the QAC, or designee, in the appropriate subdirectory on the LAN at K:\LSB\Current Documents\Forms\. These forms shall be reviewed and revised as necessary at the same frequency detailed in LSASDPROC-1000. Changes to controlled forms are authorized by the Section Chiefs by sending an email to the QAC denoting approval and with a copy of the changes. The QAC also has the authority to approve branch related forms for posting on the LAN. The QAC, or designee is responsible for posting the modified and approved form to the LAN, and to notify all appropriate staff. It is the responsibility of each staff member to ensure the current version as listed on the K: drive is being used. Specific document control procedures are detailed in LSASDPROC-1000.

4.2.13 Records Management/Disposition LSB records will be managed in accordance with the LSASD Procedure for control of Records (SESDPROC-1001) If the LSASD and LSB organizations are eliminated, all records would be maintained as required by U.S. government regulations for records retention in force at the time of the discontinuation.

4.3 Laboratory Apparatus and Instruments

4.3.1 General Policy It is the policy of LSB that all laboratory apparatus and instruments meet or exceed any method-specified tolerances to ensure results are reported within acceptable uncertainty levels. Environmental Management System goals (e.g., reduction in chemical use or more energy efficiency) should be considered when evaluating new equipment for purchase but may not always be the deciding factors. All equipment will be determined to be clean, free of contaminants and operational and will be calibrated prior to use as per manufacturer's instruction and procedures detailed in LSB technical SOPs. If any equipment becomes defective or is suspected of being defective, it will be marked as out-of-service. If possible, the defective equipment will be separated from equipment currently in use. Equipment will be utilized by authorized personnel following manufacturer's user manuals which can be available for review in either electronic or hard copy formats. In general, all LSB laboratory apparatus and instruments remain under the control of LSB at all times. Exceptions may include the sending of equipment to a vendor for calibration. If equipment leaves the direct control of LSB (e.g., loaned to another agency), it shall be verified to be operating

properly prior to being placed back into service at LSB. LSASDPROC-1009 details equipment management procedures.

4.3.2 Incubators Each incubator within LSB will be monitored by an automatic temperature recorder. See Section 4.3.4 for additional information regarding the automatic temperature recorders in use by LSB.

4.3.3 Water Baths Monitor and record temperature in the preparation and or analysis log at least once each working day while in use or as specified by the published method or technical SOP. Verification of operation within the correct temperature range may be documented in an alternate fashion if it can be demonstrated that the unit did not exceed its minimum or maximum permissible level (e.g., with a min/max temperature record). Drain and clean water baths periodically as recommended by manufacturer, by approved methods or by accepted practice. Be sure to check temperature variations when water baths are loaded to capacity and document this check in the preparation/analysis log or temperature log, whichever is appropriate. Water baths containing glycol should be disposed of properly.

4.3.4 Refrigerators/Freezers/Drying Ovens

4.3.4.1 Refrigerators, freezers and drying ovens are equipped with automatic temperature monitoring systems comprised of a datalogger, a visual display and a temperature monitoring sensor. Sensor types vary by application; however, all sensors are certified annually. The dataloggers allow for data storage both locally on the device and through a Wi-Fi connection to the cloud. The data can be downloaded and reported in a variety of formats. Each monitoring unit contains a visual display that plots temperature readings over time. Additionally, the most current reading is displayed so the user can verify compliance. If a piece of equipment does not require temperature monitoring, a sign will be placed on the unit stating it is not used for maintaining required temperatures.

4.3.4.2 Due to the relatively small volume of refrigerators, freezers and ovens it is expected that the units will go outside of normal operating temperatures for a period of time after loading, unloading or other activities where the door may be open to the ambient environment. Additionally, refrigerators and freezers may undergo defrost cycles where the temperature is above the maximum for a period of time during the cycle. These deviations are unavoidable and will not trigger an out-of-control situation. To account for these normal temperature variations, a recovery time of 45 minutes is allowed for units equipped with automatic temperature recording devices. Exceedances lasting longer than 45 minutes will trigger an alert which will require evaluation and potential corrective action. The evaluation will include consideration of the material under temperature control, as well as the intent of the temperature control. For example, while a method or manufacturer may include instructions for refrigeration of the material, it is recognized that the material is usually shipped at ambient temperature, brought to room temperature before use and/or left on autosamplers at room temperature for several hours before analysis. In these cases, it is obviously the intent of the refrigeration requirement to maintain a colder than ambient temperature for long term storage to prevent degradation over time rather than to maintain a specific temperature for all times. As such, temperature exceedances for these types of materials would be allowed as long as the device returns to normal operating temperature. Temperature exceedances will be monitored for trends to indicate whether a device requires service or replacement.

4.3.4.3 Outdated materials in refrigerators and/or freezers are properly disposed of when no longer needed.

4.3.4.4 Storage of food or drink in any laboratory refrigerator or freezer is prohibited. Drying ovens should never be used to warm food or for drying eating utensils.

4.3.5 Autoclaves

4.3.5.1 Check and document the temperature each time the unit is in use and/or as required in the published methods or technical SOPs.

4.3.5.2 At a minimum, record the date, sterilization time, and temperature for each cycle.

4.3.5.3 The autoclave shall be serviced annually by a certified vendor to ensure proper functionality.

4.3.6 Balances A list of LSB balances and the unique identification assigned to each balance is located on the LAN. All balances are serviced/calibrated annually (± 30 days).

4.3.6.1 Accuracy Balance accuracy shall be validated with NIST-traceable weights at the time of use, or on the same day of use, against the following criteria.

4.3.6.1.1 Method- or SOP-specified criteria take precedence over other criteria.

4.3.6.1.2 If a method specifies the accuracy of a balance to be used in the procedure, (e.g., a balance capable of weighing to the nearest 0.01 g), the accuracy check at the time of use should be within ±1 in the final place. The accuracy check should bracket the targeted weight of the material being weighed.

4.3.6.1.3 In the absence of method-specified accuracy criteria, the accuracy of the balance at the time of use should meet the criteria stated in LSASDPROC-1011, Equipment Certifications.

4.3.6.1.4 The unique identification and certification dates of the balance and the check weight shall be documented for each weighing.

4.3.6.2 Verification The verification should be documented in the appropriate analysis log. Weights are verified annually and should meet the specifications stated in LSASDPROC-1011. This is required on an annual basis (± 30 days) with re-certification coordinated by the QAC.

4.3.6.3 Maintenance Clean and level balances as required and continue annual maintenance services contract and records of the maintenance performed. Analytical balances should be used in areas that are subjected to minimal vibrations or influences from static electricity as appropriate.

4.3.7 Thermometers Unless otherwise specified by regulatory methodology, it is the policy of LSB to use only non-mercury containing thermometers in all laboratory operations. All thermometers used within LSB shall be NIST-traceable. Certification of thermometers is required on an annual

basis(± 30 days) and re-certification is coordinated by the QAC as detailed in LSASDPROC-1011. Analytical equipment with built-in thermometers will have a specific procedure outlined in LSASDPROC-1011 following the manufacturers' instructions for performing the calibration.

4.3.8 Mechanical Dispensing Devices

4.3.8.1 Mechanical volumetric dispensing devices (except Class A glassware) shall be checked for accuracy on an annual basis. Glass µL syringes are exempt from this requirement; however, syringes used for volumetric dispensation must have been demonstrated for accuracy as documented by the manufacturer. Acceptance criteria are located in LSASD-PROC-1011.

4.3.8.2 Autotitrator dispensing accuracy is verified through analytical QC samples (e.g., laboratory control sample) and are not checked as mechanical dispensing devices. The liquid is dispensed in microliter quantities too small to be accurately checked gravimetrically.

4.3.9 Records of NIST Traceability

4.3.9.1 Records of NIST-traceability for thermometers, weights, and mechanical dispensing devices (as applicable) shall be maintained by the QAC. All staff members are responsible for ensuring that they coordinate with the QAC each time new supplies for these items are ordered and/or any time a recertification of any of these items occurs. Staff will ensure that the QAC is furnished originals of any documentation received with new purchases or recertification. The accuracy of check weights and thermometers is verified on an annual basis using NIST-traceable reference standards.

4.3.9.2 Records received from the vendor will be retained for all reference standards to ensure traceability and to keep relevant information intact. These records include the vendor Certificate of Analysis (COA), date of receipt, any recommended storage conditions, expiration date and a cross reference to the Element® ID assigned to the standard. COAs for purchased standards are scanned and uploaded to Element®, while the hardcopies are maintained in individual laboratories for at least five years after the date of last use.

4.3.9.3 LSB will maintain vendor certificates verifying suitability of use (i.e., cleanliness and volume) of products. For example, digestion tubes and GC vial COAs will be maintained in a binder in individual laboratories or similar manner.

4.3.10 Major Instrumentation

4.3.10.1 Major instrumentation includes, but is not limited to, the Inductively Coupled Plasma (ICP); ICP/Mass Spectrometer (ICP/MS); Gas Chromatograph/Mass Spectrometer (GC/MS); Gas Chromatograph (GC); Liquid Chromatograph/Mass Spectrometer/Mass Spectrometer (LC/MS/MS); Ion Chromatograph (IC), Mercury analyzers, Auto-analyzers; Accelerated Solvent Extractors (ASE); Gel Permeation Chromatography (GPC).

4.3.10.2 Major instrumentation shall be maintained in accordance with manufacturers' recommendations and operational guidance. Maintenance records shall be kept updated on each instrument. Additional details on maintenance, calibration and troubleshooting procedures are contained in technical SOPs.

4.3.10.3 A list of all major instrumentation, including unique IDs, is maintained in an electronic file by the QAC on the LAN.

4.4 Laboratory Supplies

4.4.1 General

4.4.1.1 Laboratory supplies shall be maintained in an uncluttered, clean, and organized fashion. Supplies are monitored so that they are ordered before depletion occurs which could cause work stoppages due to lack of supplies routinely kept in the laboratory. Supplies that come in direct contact with samples are pre-screened for suitability of use as detailed in LSB SOP 122G-Screening of Supplies.

4.4.1.2 Contract personnel cannot order supplies with EPA funds, but are still responsible for monitoring supplies that they use. Contractors may fill out an order form and submit it to an EPA staff member or Section Chief. Alternatively, if it is customary in a work area to maintain a list of supplies needing to be purchased (a list that is monitored by EPA personnel) the contractor may use this avenue for ordering supplies as needed.

4.4.1.3 A list of vendors that have furnished acceptable supplies and services is maintained by the QAC, or designee on the LAN. Additional vendors may be added to this list if their supplies and services prove to be acceptable. The approved supplier list is evaluated at a minimum quarterly. Accreditations and, first-time use dates are updated; unacceptable supplies noted, and suppliers removed from the list, etc. Supply vendors that maintain ISO accreditation (and meet ISO 17034 requirements for reference material producers) are placed on the acceptable supply list unless previous experience with the supplier has been unacceptable.

4.4.2 Labware

4.4.2.1 Labware used in laboratory operations must be high quality borosilicate glass, polymethylpentene, or Nalgene™ (Plastic). Volumetric Labware must be Class "A" quality. Certificates accompanying purchase of Class “A” volumetric labware must be maintained within the laboratory.

4.4.2.2 Labware shall be cleaned in accordance with individual SOPs and manufacturer’s instructions.

4.4.2.3 If a new washing compound or cleaning application is used within the laboratory, screening shall be performed to ensure that the labware is free of interferences before placement in service.

4.4.3 Chemicals, Reagents, Solvents, Standards, Gases

4.4.3.1 The quality of chemicals, reagents, solvents and gases is determined by the sensitivity and specificity of the methods being used. Grades of materials for analyses of lesser purity than specified by a method will not be used. When specific grades of materials are not specified by the method, analytical reagent grade materials will be used. LSB will purchase standards from vendors with ISO 17025 and ISO 17034 accreditation, if possible.

4.4.3.1.1 Suitability of routine reagents is documented through method blanks. A clean method blank documents that all reagents used in the associated batch are suitable for use. A contaminated method blank requires technical corrective action to determine whether the contamination is the result of unsuitable reagents, analytical system, or contamination introduced in the sample handling process.

4.4.3.1.2 Records shall be maintained to document the purity of any material requiring additional verification of its suitability for use in a test method (e.g., suitability of acid for ultra-trace mercury analysis). Hard copies of Certificates of Analysis are kept in a binder in the laboratory for five years after the expiration or consumption of the material.

4.4.3.1.3 If any consumables, supplies or services evaluated through the above procedures prove to be unsuitable for use, the personnel making that determination shall document the issue in an email to the QAC. The documentation should include a description of the item, the deficiency and the vendor. Where possible, a copy of the purchase request should be transmitted to the QAC. The QAC, or designee, will compile all occurrences of unsuitable consumables, supplies or services and determine what further action may be necessary.

4.4.3.2 Reagents, chemicals, solvents, and standard reference materials (excluding high-demand items) should be purchased in small quantities to minimize extended shelf- storage past its expiration date.

4.4.3.3 All reagents, chemicals, solvents, and standard reference materials should be labeled with a received, opened and/or prepared date, and discarded when expired, or when evidence of deterioration is detected. All standards received will be entered into Element® for tracking purposes.

4.4.3.3.1 All materials should have an expiration date recorded on the original container. For those materials received without a manufacturer's expiration date, an expiration date of 1 year from the date the container was initially opened will be applied to these materials; however, they should be monitored for deterioration and replaced if evidence of deterioration or contamination is present. Note: For chemicals used to prepare reagents for colorimetric determination, an expiration date is not observed. These chemicals are typically salts and are very stable lasting for many years. The reagents are prepared in excess to assist with color development. QC standards analyzed with each batch are used to validate proper performance of the method and effectiveness of the reagents. In the event the reagents do not perform as required, a new lot of chemicals will be procured.

4.4.3.3.2 Materials prepared and used within the same day (or discarded the same day as prepared), and within control of the analyst are required to have identification of the contents on the container but does not require HMIS labeling. Expiration dates may be documented on the container similar to 'Expires Daily' or 'Expires Today'.

4.4.3.3.3 Intermediate materials that are immediately consumed or promptly added to another labeled container do not need any identification. These intermediate preparations must be labeled if they are not consumed or added to the labeled container within 15 minutes of the preparation of the intermediate. The personnel making these intermediate preparations must

have possession of the material and must label it if he or she leaves the material unattended. The use of an intermediate standard or material to prepare a working standard or material must be documented in the appropriate preparation logbook or Element®.

4.4.3.3.4 Records shall be maintained on reagent, standard and reference material preparation. These records shall indicate traceability to purchased stocks or neat materials, reference to the method of preparation, date of preparation, and expiration and preparer's initials. A unique ID shall be assigned to each prepared reagent and standard. Procedures for achieving traceability are documented either in the individual method SOPs or stand-alone documents for procedures which may apply across a variety of methods. The unique ID and expiration date shall be recorded on each standard, reference material and reagent container. A cross-reference to the Element® ID shall be recorded in standard preparation records and on the Certificate of Analysis.

Note: Reagents which are not deemed critical to the success of the analysis, ones which do not contribute to the quality of the test, do not have to be tracked. For example, acids and solvents used in rinsing glassware prior to use would not require reagent traceability.

4.4.3.4 Expired Stock Standard It is LSB's policy to allow for recertification of analytical standards as described below.

4.4.3.4.1 Certification of an expired material will be performed by comparison with the same material from a second source that is within the original vendor supplied expiration date. Materials should be verified prior to the vendor's original expiration. Certification cannot be performed using a standard that has been previously recertified. Successful certification must be documented on the standard container and certificate of analysis by crossing through the vendor assigned expiration date, assigning a new expiration date one year from the date of recertification and adding the initials of the person who performed the certification. A Recertification of Standards Form (LSBFORM-2001) and the original COA are forwarded to the Section Chief for review and approval. The certificate of analysis (COA) must include the new recertification date, the analyst's initials and the analysis with which the standard was recertified (i.e., the project number or other analysis identification). The Section Chief will verify the proper documentation is in place, scan a copy of the COA into Element and route the form to the QAC for filing. **Standards may only be recertified one time before a new standard must be purchased.**

Note: Reagents, including purchased concentrated acids, may be recertified following the procedure described above.

4.4.3.4.2 Acceptance Criteria for Recertifying Expired Calibration Standards
The stability of the expired calibration standard is verified if:

4.4.3.4.2.1 The ICAL prepared using the expired standard meets method acceptance criteria.

4.4.3.4.2.2 A calibration check standard prepared from a second source that has not exceeded expiration meets the Calibration Verification Check standard (ICV or however named) acceptance criteria in the relevant technical SOP.

4.4.3.4.2.3 If an expired standard material fails the verification test, the expired standard material must be replaced or with the Section Chief's approval, failing analytes must be properly qualified with a Q-3 qualifier until a new standard can be purchased. New standards should be purchased within 30 days of a failed recertification procedure.

4.4.3.5 Storage of large quantities of some chemicals is required in the Hazardous Materials (HAZMAT) Facility. This includes, but is not limited to, concentrated acids and organic solvents. See the SHEMP for chemical storage procedures in the HAZMAT building.

4.4.4 Procurement of Chemicals and Chemical Inventories

4.4.4.1 Chemical inventories within LSASD must be controlled and monitored. These controls are particularly critical for P-Listed hazardous chemicals, which must be tracked from the point of purchase to final disposal. The documentation of the chemical inventories is the responsibility of the LSASD Chemical Hygiene Officer (CHO) who is on the staff of the LSB.

4.4.4.2 Only persons who have been trained in the proper handling of P-Listed chemicals will be authorized to use them. The training will be conducted by the CHO and/or the Safety, Health and Environmental Manager (SHEM) or a designee. Staff taking the training will be required to sign documentation confirming that they have completed the training and that they understand the proper procedures for ordering, use, storage, and disposal. The CHO will coordinate with the QAC on the maintenance of the files for training on P-List chemicals handling.

4.4.4.3 All P-Listed chemicals will be tracked using the "Chemical Tracking Form" that is maintained on the LAN at K:\LSB\Current Documents\Forms\Branch\ and following the procedure as outlined below. The CHO will maintain the files of the Tracking Forms.

4.4.4.4 Ordering of Chemicals See LSASDPROC-1008 for chemical purchasing procedures.

4.4.4.5 Receipt of Chemicals The CHO will be listed on the purchase request as the person to receive all laboratory chemicals delivered to LSASD. If the CHO is not available for an extended period of time, the CHO's designee will serve as an alternate to receive, track and distribute chemicals.

4.4.5 Laboratory Pure Water

4.4.5.1 The laboratory pure water system consists of a pre-treated deionization supply enhanced in individual laboratories by exchange modules and other modules capable of supplying high quality (18 megaohm-cm) water suitable for the application.

4.4.5.2 For laboratories with additional water purification modules, system modules are changed when 18 megaohm-cm water is not achieved or when results of method blanks indicate a water quality issue. Modules should be labelled with the date of installation and tracked within the equipment inventory.

4.4.5.3 Water purity is verified by the analysis of laboratory blanks and is determined acceptable for specific analyses as prescribed in the individual technical SOPs. .

4.4.5.4 HMIS labeling is not required for containers of DI water.

4.5 Laboratory Hazardous and Non-Hazardous Waste Handling and Disposal Procedures

4.5.1 Procedures for Satellite Hazardous Waste Accumulation Many laboratory operations necessitate the generation of hazardous wastes (solvents, acids, etc.) which are required to be near the point of generation. RCRA regulation (40 CFR 262.34(c)(1)) permits satellite accumulation areas of hazardous waste or acutely hazardous wastes at or near the point of generation. In-laboratory "satellite" accumulation of such waste should be carefully controlled by the laboratory analyst(s) working with the SHEM to avoid creating an unsafe situation and also comply with RCRA satellite storage requirements. Laboratory managers or designees shall conduct periodic walk-through inspections to ensure the proper compliance of satellite waste accumulation procedures. The biannual safety inspection by a Safety Officer serves this purpose.

4.5.2 Satellite Storage – Acutely Hazardous Wastes (P-Listed Wastes) Acutely hazardous wastes are those listed in 40 CFR 261.31-261.33 and must be accounted for separately from regular wastes. See the current version of the "SHEMP, Procedures and Policy Manual" for procedures that apply to satellite accumulation of acutely hazardous waste in LSB.

4.5.3 **P-Listed Chemicals** When any unused chemical and/or the empty container(s) for a P-Listed Chemical are ready for disposal, the analyst must notify the SHEM and coordinate transfer of the items to the SHEM. *[Special note: If a P-Listed chemical is transferred as a single component to other containers (and remains as a single component in the new container), then each container becomes "P-Listed" for disposal purposes and must be tracked and accounted for.]*

4.5.4 Disposal of Outdated or Waste Chemicals/Chemical Containers It is the individual analyst's responsibility to ensure that all appropriate procedures are followed when disposing of outdated chemicals, chemicals that are no longer in use, or empty containers of spent chemicals. As a general policy, no chemicals or solvents shall be disposed of by evaporation or by pouring down the sink, with the exception of dilute acid and bases that are accounted for in LSASD's waste stream. The SHEM should be consulted to verify appropriate procedures.

4.5.5 Non-P-Listed Chemicals Follow all Standard Procedures for disposal as specified in the "SHEMP, Procedures and Policy Manual" and LSASDPROC-1010, Maintaining a Chemical Inventory System. Any questions about disposal of unused chemicals should be referred to the appropriate supervisor or the SHEM.

4.5.6 Waste Minimization LSB is an active participant in pollution prevention activities. Each staff member is responsible for monitoring and identifying the waste stream generated by the analyses they perform and for seeking ways to minimize the wastes generated. Ideas to minimize waste generation should be brought to the attention of the employee's supervisor. All appropriate solid wastes are recycled. Currently LSASD has a recycling program for cardboard, aluminum cans, glass, mixed paper, Styrofoam and plastics. This accounts for a large amount of the total waste stream generated by LSB and LSASD.

4.5.6.1 Branch management is responsible for ensuring that staff adhere to all Region 4 recycling, waste handling, and disposal requirements for all laboratory operations. This includes the implementation of procedures (technical and/or management) designed to minimize the

generation of hazardous wastes.

4.5.6.2 Waste minimization should be a prime consideration of initial experimental design and investigation planning. The degree to which waste minimization is achieved ultimately impacts the operation and cost effectiveness of the overall hazardous waste management program.

4.6 Laboratory Cleanliness

Each analyst is responsible for keeping the lab clean and orderly. The work area should be cleaned after each use in a timely manner to prevent the accumulation of used glassware, chemical spillage, or other conditions which may create unsafe working conditions.

CHAPTER 5

Quality Performance and Data Handling

5.1 Introduction

Every component of environmental data acquisition, from sample collection through final data reporting, has associated with it degrees of uncertainty. This laboratory does not attempt to quantify absolute uncertainty, since it includes both sampling and analytical error. The purpose of a laboratory quality assurance program is to determine when the analytical measurement uncertainty has exceeded acceptance limits for precision and bias, and to notify the end user of the exceedances. The operating procedures and QC checks practiced in this laboratory and outlined in this manual are implemented to minimize the analytical error associated with data generation and to identify situations when the acceptance limits for precision and bias data quality indicators are not met. Analyses are performed in support of EPA Programs such as RCRA, NPDES, Drinking Water, Air Toxics, CERCLA, and other initiatives. The methods used for analysis are based primarily on EPA-approved methods, some of which are guidance (e.g., most RCRA methods). Other sources of approved methods come from ASTM and Standard Methods. Modifications analytical methods may have been made to increase quality, efficiency, or to support specific requests of the various programs. Drinking water and NPDES methods will not be modified or altered unless allowed by the method itself or approved by Office of Water or an alternative test procedure, respectively.

5.2 Terminology

5.2.1 Acceptance Criteria/Limits: specified limits placed on characteristics of a QC item as defined in required methods. These limits are either statistically defined by historical method performance or by specific method requirements.

5.2.2 Accuracy: 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. Accuracy is a data quality indicator.

5.2.3 Air and Emissions: whole gas or vapor samples including those contained in flexible or rigid wall containers and the extracted concentrated analytes of interest from a gas or vapor that are collected with a sorbent tube, impinger solution, filter, or another device.

5.2.4 Analyst: designated individual who performs the "hands-on" analytical methods and associated techniques and who is responsible for applying required laboratory practices and other pertinent quality controls to meet the required level of quality.

5.2.5 Analytical Uncertainty: a subset of Uncertainty of Measurement that includes all laboratory activities performed as part of the analysis.

5.2.6 Assessment: evaluation process used to measure or establish the performance, effectiveness, and conformance of an organization and/or its systems to defined criteria.

5.2.7 ASTM Type 1 Water: Type I grade of reagent water; prepared by distillation or other equal

process, followed by polishing with a mixed bed of ion exchange materials and a 0.2-µm membrane filter. Feedwater to the final polishing step must have a maximum conductivity of 20 µS/cm at 298°K (25°C), resistivity >18 MΩ-cm at 25°C, TOC <50 ppb, sodium <1 ppb, chloride <1 ppb, and total silica <3 ppb.

5.2.8 Audit: systematic, independent and documented process for obtaining audit evidence and evaluating it objectively to determine the extent to which the audit criteria are fulfilled.

5.2.9 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 1-20 environmental samples of the same matrix. An analytical batch is composed of prepared environmental samples (extracts, digestates or concentrates) which are analyzed together as a group. An analytical batch, i.e., sequence, can include prepared samples originating from various environmental matrices and can exceed 20 environmental samples. However, all prepared or method-specified QC samples must be analyzed at the correct frequency (e.g., method blank every 20 environmental samples).

5.2.10 Bias: consistent deviation of measured values from the true value caused by systematic errors in a procedure.

5.2.11 Blank: an artificial sample designed to monitor the introduction of artifacts into the analytical process. The blank is subjected to the usual analytical and measurement process to establish a zero baseline or background value.

5.2.11.1 Bottle Blank: empty bottle filled with a volume of analyte-free media in the laboratory and analyzed for contaminants. Results are typically reported as µg/bottle or mg/bottle.

5.2.11.2 Equipment Rinse Blank: sample of analyte-free media which has been used to rinse common sampling equipment to check effectiveness of decontamination procedures.

5.2.11.3 Field Blank: blank prepared in the field, (or in some cases, prepared in the lab and carried to the field) by filling a clean container with analyte-free media and appropriate preservative, if any, for the specific sampling activity.

5.2.11.4 Instrument Blank: analyte-free media processed through the instrumental steps of the measurement process; used to determine the presence of instrument contamination.

5.2.11.5 Method Blank: media similar to the batch of associated environmental samples (when available) in which no target analytes or interferences are present at concentrations that impact the analytical results for sample analyses. Processed simultaneously with and under the same conditions as the environmental samples through all steps of the preparation and analytical procedures.

5.2.12 Blind Sample: 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.

5.2.13 Biological Tissue: any sample of a biological origin such as fish tissue, shellfish, or plant

material. Such samples shall be grouped according to origin.

5.2.14 Calibration: determination, by measurement or comparison with a standard, of the correct value of each scale reading on a meter, instrument, or other device. The levels of the applied calibration standard should bracket the range of planned or expected sample measurements.

5.2.15 Calibration Curve: graphical relationship between the known values, such as concentrations, of a series of calibration standards and their instrument response.

5.2.16 Calibration Method: defined technical procedure for performing a calibration.

5.2.17 Calibration Standard: substance or reference material used to calibrate an instrument.

5.2.18 Certified Reference Material (CRM): 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.

5.2.19 Chain of Custody (COC): 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, mode of collection, collector, time of collection, preservation and requested analyses.

5.2.20 Check Standard: reference standard used to verify the concentration of the calibration standard; obtained from a source independent of the calibration standard.

5.2.21 Confirmation: verification of the identity of a component using 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, alternate detectors or additional cleanup procedures.

5.2.22 Conformance: 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.

5.2.23 Continuing Calibration Verification (CCV): analysis of an analytical standard or reference used to verify the calibration curve.

5.2.24 Corrective Action: action taken to eliminate the causes of an existing nonconformity, defect or other undesirable situation to prevent recurrence.

5.2.25 Formal Corrective Action: higher level corrective action that includes a multi-step process of describing the issue, performing a root cause analysis leading to a proposed action, acceptance and closure.

5.2.26 Data Audit: qualitative and quantitative evaluation of the documentation and procedures associated with environmental measurements to verify that the resulting data are of acceptable quality (i.e., they meet specified acceptance criteria).

5.2.27 Data Quality Objective (DQO): statement of data quality required from an investigation as

established by the end user during the planning phase of a project requiring laboratory support. The DQO is a qualitative and/or quantitative statement of the quality of data required to support specific decisions or regulatory actions.

5.2.28 Data Reduction: process of transforming raw data by arithmetic or statistical calculations, standard curves, concentration factors, collation, etc., into a more useable form.

5.2.29 Deficiency: unauthorized deviation from acceptable procedures or practices, or a defect in an item.

5.2.30 Demonstration of Competency (DOC): procedure to establish the ability of the method and/or analyst to generate acceptable accuracy.

5.2.31 Dissolved: terminology used in analytical reporting referring to an environmental sample that has been filtered prior to preservation and arrival.

5.2.32 Document Control: act of ensuring that documents (and revisions thereto) are proposed, reviewed for accuracy, approved for release by authorized personnel, distributed properly and controlled to ensure use of the correct version at the location where the prescribed activity is performed.

5.2.33 Drinking Water: any aqueous sample that has been designated a potable or potential potable water source and regulated under 40 CFR Part 141.

5.2.34 Estimated Value: calculated value based on a reasonable approximation of the true value.

5.2.35 Holding Time: period of time (usually in hours or days) from sample collection until sample preparation or analysis. Initial time is when a grab sample is collected or the time the last aliquot of a composite is collected; final time is when sample preparation or analysis begins. This time requirement can be expressed in various units (hours, days, weeks, etc.). Holding times are evaluated in the same units as specified. For those analyses with both a preparation and analytical holding time, the LIMS calculates the analytical holding time from the beginning of the sample preparation time.

5.2.36 Initial Calibration Curve (ICAL): calibration curve with concentrations bracketing the range of interest performed at the beginning of the analytical process and again each day prior to sample analysis or at a frequency required by a specific method.

5.2.37 Initial Test Method Evaluation (ITME): procedure for establishing an authorized method in a specific lab through a formal validation study to include an evaluation of a method's precision and bias. The ITME can include an MDL determination and an evaluation of the MRL, where applicable.

5.2.38 Internal Standard: known amount of standard added to a test portion of a sample as a reference for evaluating and controlling precision and bias of the applied analytical method.

5.2.39 Instrument Blanks: a blank aliquot used to assess the analytical system. Instrument blanks are typically analyzed prior to an analytical sequence but can be included within a sequence as

needed. The results of the instrument blank must be less than the analyte reporting limit or corrective action is required.

5.2.40 Laboratory Control Sample (LCS): 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.

5.2.41 Laboratory Control Sample Duplicate (LCSD): replicate LCS prepared in the laboratory and analyzed to obtain a measure of the precision of the recovery for each analyte.

5.2.42 Laboratory Replicate Analyses: measurements of the variable of interest performed identically on two or more sub-samples of the same samples within a short time interval. A laboratory duplicate is a subset of laboratory replicates.

5.2.43 Laboratory/Sample Duplicate: aliquots of a sample taken from the same container under laboratory conditions; processed and analyzed independently.

5.2.44 Management System Review: qualitative assessment of an organization's overall quality system and the effectiveness of its implementation.

5.2.45 Matrix: substrate of a test sample.

5.2.46 Matrix Spike (spiked sample or fortified sample): sample prepared by adding a known mass of target analyte to a specified amount of matrix sample for which an independent estimate of the target analyte concentration is available. Matrix spikes are used to determine the effect of the matrix on a method's recovery efficiency.

5.2.47 Matrix Spike Duplicate (spiked sample or fortified sample duplicate): second replicate matrix spike prepared in the laboratory and analyzed to obtain a measure of the precision of the recovery for each analyte.

5.2.48 May: denotes permitted action, but not required action. The fifth month of the calendar year.

5.2.49 Measurement Quality Objective (MQO): desired sensitivity, range, precision, and bias of a measurement.

5.2.50 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.

5.2.51 Method Detection Limit (MDL): The MDL is defined as the minimum measured concentration of a substance that can be reported with 99% confidence that the measured concentration is distinguishable from method blank results (i.e. background). (40 CFR Part 136 Appendix B). It is determined from the analysis of a series of low-level blank spikes and method blanks. Matrix-specific when possible.

5.2.52 Minimum Reporting Limit (MRL): The MRL is defined by LSB as the smallest concentration of a substance that can be reliably measured by a given analytical method and system. This value represents the low limit for unqualified quantitative data. Establishment of the MRL should account for day-to-day fluctuations in instrument sensitivity, operating factors, analyst performance, Maximum Contaminant Levels (MCLs) for drinking water or other regulatory limits.

LSB establishes the MRL based on the MDL and the limit is set at an amount 2-5 times the calculated MDL.

5.2.53 Minimum Reporting Limit Verification Standard (PS): LSB defines the PS as the standard used to verify the reporting limit (MRL or MDL in special cases). A PS standard is required with each batch of up to 20 environmental samples. The PS standard must be treated identical to environmental samples, spiked at the reporting limit established for the project (if alternative reporting limits have been requested) for each analyte of interest, and carried through all aspects of the preparation and analysis at that level. The PS verifies the MRL (i.e, minimum level at which the sample results will be reported as unqualified, quantitative data); therefore, the low-level calibration standard cannot substitute for the PS. A separate PS aliquot must be included in each analytical sequence. For the mass spectrometry methods within the Organic Section where LSB reports data to the MDL, the reported results between the MRL and MDL will be accompanied by a "J" qualifier to indicate the result is between the MDL and MRL.

5.2.54 Must: denotes required action.

5.2.55 Non-Potable Water: any aqueous sample excluded from the definition of Drinking Water matrix. Includes surface water, groundwater, effluents, water treatment chemicals, and TCLP or other extracts.

5.2.56 Non-Conformance: departure from or absence of a specified requirement.

5.2.57 Non-target Analyte: compound that is detected by an analytical system but is not specifically targeted by the method as a parameter. In this instance, there would not be a calibration standard used to calibrate the analytical system specifically for this analyte. (This most often occurs with analyses for organic parameters.) The identification (qualitative analysis) of the non-target analyte is generally based on a comparison to known or published information (e.g., spectra from published libraries) and is usually considered tentative or provisional. The amounts reported are calculated relative to known concentrations of other reference materials and are reported as estimated or qualified. These analytes are also often referred to as tentatively identified compounds (TICs).

5.2.58 Organic-Free Water: reagent water without organic compounds that might interfere with the extraction or analysis of samples.

5.2.59 Outlier: observation (or subset of observations) which appears to be inconsistent with the remainder of that set of data.

5.2.60 Precision: degree to which a set of observations or measurements of the same property, obtained under similar conditions, conform to themselves; a data quality indicator. Precision is usually expressed as standard deviation, variance or range, in either absolute or relative terms.

5.2.61 Preliminary Data: produced prior to undergoing a complete QA/QC review and may be subject to change as a result of the review process. Upon request, a preliminary draft report of the data requested will be submitted to the project leader via e-mail in PDF format, prior to the data being subject to the complete review process.

5.2.62 Preservation: refrigeration and/or reagents added before (e.g., 50% HCl) or at the time of

sample collection to maintain the chemical and/or biological integrity of the sample. Preservation may also take place after sampling in certain situations.

5.2.63 Preventive Action: proactive process to identify opportunities for improvement rather than a reaction to the identification of problems or complaints.

5.2.64 Proficiency Test Sample (PT): a sample, the composition of which is unknown to the analyst, which is provided to test whether the analyst/laboratory can produce analytical results within specified acceptance criteria.

5.2.65 Pure Reagent Water: water (defined by national or international standard) in which no target analytes or interferences are detected as required by the analytical method.

5.2.66 Quality Assurance: all planned and systematic activities necessary to provide confidence that a product satisfies given acceptance criteria. Quality assurance activities are independent.

5.2.67 Quality Control: operational techniques and activities that are used to fulfill requirements for quality. QC activities are typically performed by staff on a routine basis.

5.2.68 Quality Control Sample: sample used to assess the performance of all or a portion of the measurement system. QC samples may be Certified Reference Materials, quality system matrices fortified by spiking, or actual samples fortified by spiking.

5.2.69 Quality System: defined system of quality assurance practices and operational policies.

5.2.70 Quantitation Limits: levels, concentrations, or quantities of a target variable (e.g., target analyte) that can be reported at a specified degree of confidence.

5.2.71 Range: difference between the minimum and maximum of a set of values.

5.2.72 Raw Data: any original information from a measurement activity or study recorded in laboratory notebooks, worksheets, records, memoranda, notes, or exact copies thereof, necessary for reconstruction and evaluation of the report of activity or study. Raw data may include photography, computer printouts, magnetic/digital media, and recorded data from automated instruments. If exact copies of raw data have been prepared (e.g., tapes transcribed verbatim, data copied and verified accurate by signature), the exact copy or exact transcript may be submitted.

5.2.73 Reference Material: 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.

5.2.74 Reference Method: method of known and documented accuracy and precision issued by an

organization recognized as competent to issue said method.

5.2.75 Reference Standard: standard, generally of the highest metrological quality available at a given location, from which measurements made at that location are traceable.

5.2.76 Reporting Limit: also known as the Minimum Reporting Limit (MRL) in LSB.

5.2.77 Sample: aliquot of a certain matrix (soil/sediment, water, air, etc.) collected at a specific location, date, and time (grab or composite). This aliquot could be distributed over several different

5.2.78 sizes or types of containers depending on the analytical and/or preservation requirements.

5.2.79 Scope of Accreditation: accredited work organized on the certifying statement by category, sub-category and technique.

5.2.80 Second-Source Material: term typically applied to a QC sample used to verify a standard curve. Second-source refers to a stock standard obtained from a different vendor than that used for the calibration standards. Alternatively, if a second vendor is not readily available, a different lot number from the same vendor may be used if the vendor verifies that the lots were prepared independently from different source material.

5.2.81 Selectivity: the capability of a test method or instrument to respond to a target substance or constituent in the presence of non-target substances.

5.2.82 Sensitivity: capability of a method or instrument to discriminate between measurement responses representing different levels (e.g., concentrations) of a variable of interest.

5.2.83 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.

5.2.84 Should: denotes a guideline or recommendation whenever noncompliance with the specification is permissible.

5.2.85 Significant Figures The number of digits in a reported result that are known definitely as justified by the accuracy of the analysis with one additional figure that may have some degree of uncertainty. For example, an analyst would be certain of the “75” in a result reported at “75.6" mg/L but may be uncertain as to whether the “.6 “should be “.5" or “.7" because of unavoidable uncertainty in the analytical procedure. Digits beyond this last figure are not significant. In the example, analysts reporting to 3 significant figures would report “75.6". Only figures justified by the accuracy of the analysis (significant figures) shall be reported. (Based on Standard Methods (SM) for the Examination of Water and Wastewater, 22nd edition)

5.2.86 Solid Material: includes soils, sediments, sludges, products and by-products of an industrial process that results in a matrix not previously defined**.**

5.2.87 Spike: known mass of target analyte added to a blank sample or sub-sample; used to determine recovery efficiency or for other QC purposes.

5.2.88 Standard Reference Material (SRM): 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.

5.2.89 Target Analyte: individual analyte specifically targeted for analysis by using a method designed and validated for the analyte. The method includes calibration standards and other QC

parameters to calibrate and document the ability of the analytical system to successfully analyze for the analyte.

5.2.90 Technical Corrective Action: any action taken to address instrument or QC specifications at the time an exceedance is noted. Technical corrective actions are proactive and preventative in nature and do not require a root cause analysis as they do not impact data quality.

5.2.91 Technical System Review: assessment of analytical procedures, record- keeping, data verification, data management and other technical aspects within an organization.

5.2.92 Tentatively Identified Compound (TIC): see **Non-Target Analyte.**

5.2.93 Traceability: property of a result of a measurement where it can be related to appropriate standards, generally international or national, through an unbroken chain of comparisons.

5.2.94 Uncertainty of Measurement (Uncertainty): parameter, associated with the result of a measurement that characterizes the dispersion of the values that could reasonably be attributed to the measurand (object being measured). Uncertainty differs from error in that it takes the form of a range of values as opposed to error which is the difference from the true value and is represented by a single value.

5.2.95 Verification: confirmation by examination and provision of evidence that specified requirements have been met.

5.2.96 Work Cell: a group of analysts that share responsibility for a specified analysis.

5.3 Essential Quality Control Requirements

5.3.1 Demonstration of Competency (DOC) LSB requires all analysts to demonstrate initial competency (prior to independent analysis of environmental samples) or with method or instrumental changes that could impact method performance. Laboratory analysts may participate in procedures prior to completing a DOC provided they are performing the work with another analyst with an active DOC for that specific method. Analyst DOCs are specific to the method only, demonstrating the analyst's ability to successfully perform the method. Procedures for performing a DOC are detailed in LSBPROC 110G: Standard Operating Procedure for Initial Test Method Evaluations and Establishing Demonstrations of Competency and LSASD PROC-1003 Training and Demonstrations of Competency.

5.3.2 Continuing Demonstrations of Proficiency (CDOP) An analyst's continued proficiency with a test method will be evaluated through the completion of a CDOP. Performance of the CDOP is required every four years at a minimum. Procedures for documenting an analysts' CDOP are detailed in SOP LSB 110G and LSASD PROC-1003.

5.3.3 MDL Studies LSB performs MDL studies as part of the ITME and upon major changes to instrumentation or methods; they are verified on an ongoing basis, in accordance with 40CFR Part 136 Appendix B Revision 2 as detailed in LSB SOP 119G. LSB only reports non-detects at the MDL by special request and approval of the Section Chief.

5.3.4 Instrument Calibration

5.3.4.1 Initial Calibration Curve (ICAL) All instrumentation utilized in the preparation or analysis of environmental samples will be calibrated prior to use. The calibration curve shall bracket the range of expected concentrations for the analytes being evaluated. Calibration frequency and acceptance criteria will follow method and/or technical SOP requirements. Calibration standards shall be prepared using the same, or equivalent, type of acid or solvent and at the expected concentration as the samples following sample preparation. LSB requires purchased standards to be prepared in accordance with ISO 17034 specifications. Traceability shall be to a national standard, when commercially available.

Note: LSB performs some test methods where ISO 17034 standards may not be available. Examples include, but are not limited to, Methods for Determination of pH, Flashpoint, and Natural Attenuation. In those instances, standards will be NIST traceable.

Any data above the calibration range shall be diluted or considered to have an increased quantitative uncertainty and shall be reported with a qualifier, where applicable. For Metals samples analyzed by ICP, a linear range study is performed and verified with each analysis. Samples exceeding the calibration but within the linear range are reported unqualified provided the linear range check standard is within the ±10% acceptance criteria.

5.3.4.2 Initial Calibration Verification (ICV) ICALs shall be verified with a second-source standard, on the frequency prescribed in the published method or technical SOP. Traceability shall be to a national standard, when commercially available. For test methods where a second source is not available (e.g., toxaphene congeners), LSB allows for verification of the calibration through the use of alternative quality controls. In these instances, the technical SOP will describe the verification requirements.

Note: LSB maintains competency for some methods that are not amenable to analysis of an ICV. Examples include, but are not limited to, pH, BOD, natural attenuation, titration methods and determination of solids (total, dissolved, recoverable and volatile). For these methods, QC requirements for verification of the calibration are detailed in the technical SOP.

5.3.4.3 Continuing Calibration Verification (CCV) In addition to the initial calibration verification, LSB requires verification of the calibration over time to assess instrument performance throughout the course of the analysis of samples. A standard solution (either primary or second source is acceptable) will be analyzed prior to analysis of a batch containing environmental samples and at the frequency prescribed in the published method or technical SOP.

5.3.5 Acceptance Criteria All methods in use must have acceptance criteria against which all QC results are evaluated. When acceptance criteria are not prescribed in the method, in-house

acceptance criteria must be developed using a minimum of 20 results. After initial limits are determined, they should be evaluated and updated at a minimum, annually. The following specify how LSB evaluates acceptance of QC results:

- Bias and precision limits are set at three standard deviations from the mean of the dataset.
- Recovery limits will not be set tighter than ± 10% around the mean recovery even if in-house limits calculate tighter.
- Precision limits will not be set tighter than an RPD (or RSD) of 10 even if in-house limits calculate tighter.
- Values supplied with SRMs (such as those purchased from NIST) are supplied with a true value, and an uncertainty range around that true value, which are not meant to be acceptance criteria. In-house acceptance criteria shall be determined for these materials as described above.
- Matrix-specific QC materials are sometimes purchased for use as an LCS (such as nutrients in soil). These materials are typically received with vendor recommended acceptance criteria. Because limits may be dependent on the actual material received, these QC samples will be designated as 'Reference' materials in Element® and vendor-supplied limits will be used for the life of the material rather than attempting to generate specific in-house limits. (The Section Chief and/or QAC may set acceptance criteria tighter than those supplied by the vendor based on experience with the specific analytical method.) Designating these as 'Reference' materials will facilitate segregating results of these samples from LCSs which have been prepared in reagent water or other analyte-free matrix for control charting purposes. Reference method and program acceptance limits supersede vendor supplied limits and will be used as specified in the analytical method.
- When 20 data points are not available, LSB will establish interim limits.

5.3.5.1 Setting Interim Bias (Recovery) Limits LSB allows for the use of interim limits for bias until sufficient data is available to establish limits based on historical data. Interim limits for bias will be calculated using the most recent seven valid spiked results. If seven data points are not available, interim limits for bias will be established based on guidance in the published method or equivalent. If there are no existing guidelines for limits, arbitrary limits will be established and used until such time that seven spike values are generated, and interim limits can be calculated. Limits for inorganic analyses will be set at 85-115% and limits for organic analyses at 70-130%. For complex matrices, acceptance limits may be extended to 50-150% for both Inorganic and Organic analyses at the discretion of the Section Chief/Technical Director or QAC.

5.3.5.2 Setting Interim Precision Limits Interim limits for precision are set at an RPD or Relative Standard Deviation (RSD) of 20. At the discretion of the Section Chief/Technical Director or QAC, interim RPD limits of 50 may be set for complex matrices such as soil, tissue or waste.

5.3.5.4 Control Charting QC Limits When sufficient data is available to establish historical limits, and on an annual basis thereafter, staff will determine the new limits using the Control Chart feature in Element®. Required updates to Element will be submitted to the QAC on an Element Change Request Form. Any changes to acceptance criteria on the data review forms will be completed by the QAC concurrent with the Element® change request. See LSBPROC-119 for

details on control charting.

5.3.6 Method Blank LSB requires one method blank per batch of up to 20 environmental samples per matrix type per sample preparation method, or as specified by the published method. The method blank is utilized to assess potential contamination of the associated sample batch.

5.3.7 Laboratory Control Samples (LCS) For every batch of up to 20 environmental samples per matrix type per sample preparation method, or as specified in the published method, LSB requires an LCS to be carried through the entire analytical process. LSB uses the LCS to assess the performance of all or a portion of the measurement system. LSB also assesses the LCS results as a mechanism for determining if technical corrective action is required through the use of control charts. Control charts can be generated by all laboratory staff through the Control Charting feature in Element® for any QC element loaded into Element®. Control Charts are also used to establish historical limits using three standard deviations from the mean of the dataset.

5.3.8 Matrix Spike (MS) Frequency of the analysis of MS samples shall be determined as part of the systematic planning process (e.g., DQOs) or as specified by the required published method. Unless otherwise allowed by the technical SOP, a minimum of at least one MS should be prepared per batch of up to 20 samples for all methods amenable to performing a MS. If the reference or technical method requires more frequent analysis of matrix spikes (1 for every 10 environmental samples) the method requirements will be observed. The matrix spike analysis is used to assess the performance of the method by measuring the effects of interferences caused by the sample matrix and reflects the bias of the method for the matrix in question. LSB uses the acceptance criteria for evaluating a MS as defined in the published method. If no acceptance criteria are provided, interim QC limits will be established and used until historical data can be generated. LSB does not qualify any batch results based on the MS analysis. Only the sample spiked is qualified if QC results are outside of the MS limits for that sample.

5.3.9 Bias LSB expresses bias as percent recovery (%R). Bias is calculated for both LCS and matrix spikes using the following formulas:

Bias

Spike Reference

$$\%\ RR = \frac{ZZ - XX}{YY}\ (100)$$

Or

Reference Materials

$$\%\ RR = \frac{\ }{TT}\ (100)$$

Where: X = Concentration in unspiked sample.
Y = Measured concentration
Z = Concentration in spiked sample.
T = True concentration of spike added or of analyte in reference material.

5.3.10 Surrogate Spike Recovery of the surrogate standard is used to monitor for matrix effects, gross sample processing errors, etc. and is evaluated by determining whether the measured concentration falls within an established statistical acceptance limit. Surrogate spiking compounds are added, when appropriate, to each sample just prior to preparation, i.e., extraction or purging. Surrogate standards are normally utilized in organic analyses. Sample results with surrogate limits

that fall outside acceptance criteria are qualified appropriately. (Technical SOPs should specify the requirements for sample qualification.) Acceptance limits are defined by the technical SOP. Surrogate recoveries are compared to the method-specified acceptance limits, which are stored within Element®.

5.3.11 Proficiency Test Sample (PT sample)

5.3.11.1 LSB will participate in independent Proficiency Testing Studies as required for accreditation/certification or more often as deemed necessary by LSB management or the QAC.PT samples will be prepared and analyzed identical to environmental samples, with the exception being that PT results are reported to the PT provider to 3 significant figures. For some analyses, the PT Reporting Limit (PTRL) may be less than the routine MRL. If an analyte is present at > PTRL, but < MRL, report the data as less than the MRL as would be done for environmental samples.

5.3.11.2 Performance in these studies further indicates the effectiveness of the laboratory's day-to-day QC procedure. LSB's current Forensic and ISO 17025 accreditation requires the entire scope of accreditation to be covered with a PT every four years. In addition, the laboratory should participate in one PT per calendar year. Drinking Water certification requires participation in a drinking water PT every calendar year. PT samples will be prepared and analyzed identical to environmental samples. The results of the PT studies must be reported to the accrediting body prior to the annual accreditation visit. The laboratory will create and maintain a four-year PT plan that consists of each PT that will be performed during that interim. The accrediting body will review the plan during the annual assessment. When the laboratory receives a performance score of 'not acceptable' a formal corrective action and makeup PT shall be performed for the analytes that were deemed unacceptable prior to the next scheduled PT. PT samples are also used as analyst Demonstrations of Continued Proficiency with a method.

5.3.12 Standard Reference Materials (SRM) and Certified Reference Materials (CRM) These reference materials will be utilized to determine method/analytical performance as deemed appropriate.

Minimum Reporting Limit (MRL) Verification Standard Minimum Reporting Limit Verification Standard (PS): LSB defines the PS as the standard used to verify the reporting limit (MRL or MDL in special cases). A PS standard is required with each batch of up to 20 environmental samples. The PS standard must be treated identical to environmental samples, spiked at the reporting limit established for the project (if alternative reporting limits have been requested) for each analyte of interest, and carried through all aspects of the preparation and analysis at that level. The PS verifies the MRL (i.e, minimum level at which the sample results will be reported as unqualified, quantitative data). For the mass spectrometry methods within the Organic Section where LSB reports data to the MDL, the reported results between the MRL and MDL will be accompanied by a "J" qualifier to indicate the result is between the MDL and MRL.

5.3.13 Precision Refers to the degree to which a set of observations or measurements of the same property, obtained under similar conditions, conform to themselves; a data quality indicator. Precision is usually expressed as standard deviation, variance or range, in either absolute or relative terms. Precision results may be compared to historical LSB limits or the acceptance criteria in the published method. Precision is expressed as relative percent difference (RPD). ASB applies RPD

limits to matrix or laboratory duplicates, matrix spike duplicates and LCS duplicates.

5.3.14 Matrix Duplicate Analyses At a minimum, either a matrix duplicate or MS duplicate (see below) shall be prepared with each batch of up to 20 environmental samples as required by the published method. The results from matrix duplicates are designed to assess the precision of

5.3.15 analytical results in a given matrix. LSB does not qualify any batch results based on the matrix duplicate analysis. Only the sample which was duplicated is flagged if QC results are outside of the matrix duplicate limits for that sample.

5.3.16 Laboratory Control Sample Duplicate (LCSD) LSB does not routinely analyze an LCSD unless mandated by the published method, SOP, project-specific DQOs or if precision of the analysis is not determined through the analysis of a matrix duplicate or spike duplicate. Acceptance criteria for the LCSD results are compared to established limits for that specific matrix if available. If precision results from an LCS/LCSD pair are outside of established acceptance criteria, all results for that analyte in the batch, both detects and non-detects, are qualified as estimated "J" with an appropriate explanatory qualifier.

5.3.17 Matrix Spike Duplicates (MSD) MSDs will be included in each sample batch as specified by the published method where sufficient sample is received for performing the analysis. The results from MSDs are utilized to assess the precision of the analytical results in a given matrix. Results are compared to established limits for that specific matrix if available. LSB does not batch qualify any results based on the MSD analysis. Only the sample which was spiked is flagged if QC results are outside of the matrix spike duplicate limits for that sample.

5.3.18 Internal Standards LSB uses internal standards for the evaluation of instrumental drift as well as suppressions or enhancements of instrument response caused by the sample matrix, as required by the applicable published method. If utilized, internal standards are added to all calibration standards and QC samples (method blank, MS/MSD, LCS/LCSD, MRL verification, matrix duplicates) at the same concentration as the samples following preparation. The Internal Standard acceptance criteria specified in the method will be observed.

5.3.19 Bottle blanks, equipment rinse blanks and other in-house QC analyzed for the Field Branch QA screening and/or LSB supply screening will be performed with a reduced level of QC due to the nature of the matrix which is reagent water.

5.4 Data Handling

5.4.1 Holding Time Sample preparation and analysis will be performed with the recommended holding times specified in the published method or technical SOP. If analyses are performed outside defined recommended maximum holding times, results will be "J"-qualified and an appropriate Element® explanatory qualifier will be added. For analyses that have a preparation/extraction step, holding times for each segment of the analysis must be evaluated. If any segment of the holding time is exceeded (i.e., time elapsed prior to extraction or time elapsed prior to analysis of the extract), LSB will consider the holding time for that sample to have been exceeded.

5.4.2 Reporting data between the MDL and MRL LSB establishes the MRL as detailed in LSBPROC-110 and includes a standard at or below the MRL in the calibration curve. LSB reports data at the MRL on a routine basis unless specific reporting limits are requested by the project leader

or sample requestor. LSB will report data between the MDL and MRL based on the following criteria:

5.4.2.1 Organic Chromatographic/Mass Spectral Data Because chromatographic/mass spectral analyses use both retention time and a spectral match, there is qualitative evidence for presence of target analytes at concentrations between the MDL and MRL. Therefore, reporting between the MDL and MRL is allowed for some organic methods as detailed in the individual technical SOPs.

5.4.2.1.1 Non-detects are reported to the MRL.

5.4.2.1.2 Positive detects between the MDL and the MRL are reported with a 'J' and explanatory qualifier.

5.4.2.1.3 Any requests for non-detects to be reported as < MDL must be approved by the Section Chief or QAC who will verify that a current MDL study is in place that will meet the needs of the data user.

5.4.2.2 All other data

5.4.2.2.1 Non-detects are reported to the MRL.

5.4.2.2.2 Any detects between the MDL and the MRL are reported as < MRL.

5.4.2.2.3 All requests for results to be reported between the MRL and MDL must be approved by the Section Chief or QAC who will verify that a current MDL study is in place that will meet the needs of the data-user.

5.4.3 Units

5.4.3.1 Sediment/Soil All soil/sediment samples shall be reported on a dry-weight basis unless otherwise specified by the published method or technical SOP. Soil/sediment samples are reported in mg/kg, µg/kg or ng/kg units.

5.4.3.2 Waste (aqueous and non-aqueous) Reported on a wet-weight basis unless otherwise specified by the sample requestor.

5.4.3.3 RCRA Wastewater as defined at 40 CFR 268.2(f) analyzed in support of Land Disposal Restrictions constituents (40 CFR 268.48) are reported in mg/L units.

5.4.3.4 Tissue sample Reported on a wet-weight basis unless otherwise specified by the sample requestor. Tissue samples are reported in mg/kg or µg/kg units.

5.4.3.5 Water samples include groundwater, surface water, potable water, etc. and are reported in µg/L, ng/L, pg/L or mg/L units.

5.4.3.6 Air samples including VOA samples are reported in ppbv or µg/m^3 units.

5.4.4 Significant Figures Because the accuracy and/or uncertainty of every procedure is not always precisely known, it is the general practice of LSB to report analytical results to 2 significant figures, with the exception of PT samples which are reported to 3 significant figures.

5.4.5 Rounding Rules

5.4.5.1 Manual Rounding Where manual data entry is performed, entries will be rounded to achieve a final result with 2 significant figures. Round numbers by dropping digits that are not significant. If the digit 6, 7, 8, or 9 is dropped, increase preceding digit by one unit; if the digit 0, l, 2, 3, or 4 is dropped, do not alter preceding digit. If the digit 5 is dropped, round off preceding digit to the nearest even number; thus 2.25 becomes 2.2, and 2.35 becomes 2.4. Use only the digit beyond the last significant figure for rounding. Rounding should be performed only after arriving at the final result in the calculation.

5.4.5.2 Rounding in LIMS Element® follows the above rounding rules when all digits in the preceding example of 2.25 following the 5 are zero. Any numbers transferred to Element® with digits following the 5 that are not zero are interpreted as a result greater than 5 and thus are rounded up.

5.4.5.3 Values that are below the MRL but are equal to the MRL after rounding are reported as detects. For example, if the MRL is 0.5 and the unrounded result is 0.4986, Element® will round the result to 0.50 and report the value as detected at 0.50.

5.4.6 Determination of Outliers – Student *t*-test or Dixon's *Q*-test

5.4.6.1 Data points may not be discarded as outliers without a proper explanation or valid justification. This applies to all data points collected (e.g. LCS, MDL, linear curves, DOC, duplicates, etc.). Justifiable reasons for removing outliers include:

5.4.6.1.1 Known and documented laboratory error and

5.4.6.1.2 Use of an appropriate statistical outlier test.

5.4.6.2 Standard deviation from the mean – typically useful for large data sets

5.4.6.2.1 Calculate the mean and the standard deviation of all the data. Database outliers are established by summarizing all the data in the database and then applying one standard deviation beyond the statistical confidence level required. For example, assuming the statistical confidence level required is 95% (2 standard deviations around the mean), any result greater than 3 standard deviations around the mean would be an outlier.

5.4.6.3 Studentized deviation from the mean – *t*-test

5.4.6.3.1 Including the suspect extreme value (possible outlier), calculate the sample mean ($\bar{x}$) and the standard deviation (s) of the data.

5.4.6.3.2 Calculate the ratio:

$$tt_{cccccccc} = \frac{|ssssssssssstt\ vvvvmssss - \bar{xx}|}{ss}$$

5.4.6.3.3 Apply the following decision rule.

5.4.6.3.3.1 If t_{calc} is greater than the critical value ($t_{critical}$) at a given level of confidence, then the suspect value should be removed.

5.4.6.3.3.2 Critical values of t ($t_{critical}$) as a function of sample size, n, at the 95% level of confidence (level of significance, $\alpha = 0.05$) are given in Table 5-1.

5.4.6.3.4 Example

MDL rep	Lead (µg/L)
1	40.3
2	41.0
3	40.1
4	38.0
5	40.7
6	41.3
7	41.1

For the extreme low value, the calculated value of t is:

$$t_{calc} = \frac{|suspect - \overline{X}|}{s} = \frac{|38.0 - 40.3571|}{1.1252} = 2.09$$

The critical value of t is 2.02 for $\alpha = 0.05$ and n = 7. The calculated value of t, 2.09, is greater than the critical value of t (e.g., $t_{calc} > t_{critical}$). Thus, the suspect value is an outlier and should be removed.

5.4.6.4 Dixon's Q test

5.4.6.4.1 Sort the n data values in ascending order:

$x_1 < x_2 < \ldots < x_{n-1} < x_n$

Where x_1 is the extreme low value (or x_n is the extreme high value) suspected of being an outlier.

5.4.6.4.2 Calculate the absolute difference between the suspect value and the measurement that is nearest in magnitude (e.g., the next higher or lower value.)

5.4.6.4.3 Calculate the range of the entire data set including the suspect value, which is one of the extreme values.

5.4.6.4.4 Calculate the value of Q:

$$Q_{calc} = \frac{|suspect\ value - nearest\ neighbor|}{range\ of\ entire\ data\ set}$$

$\frac{|x1-x2|}{xn-x1}$ $\frac{|xn-xn-1|}{(xn-x1)}$

5.4.6.4.5 Apply the following decision rule:

5.4.6.4.5.1 If the calculated value of Q (Q_{calc}) is greater than the critical value of Q ($Q_{critical}$) at a given level of confidence, then the suspect value is an outlier and shouldbe removed from the data set.

5.4.6.4.5.2 Critical values of Q as a function of sample size, n, at the 95% level of confidence (level of significance, $\alpha = 0.05$) are given in Table 5-2.

5.4.6.4.5.3 Example

MDL Rep	Lead (µg/L)
1	40.3
2	41.0
3	40.1
4	38.0
5	40.7
6	41.3
7	41.1

The data sorted in ascending order are:

MDL Rep	Lead (µg/L)
4	38.0
3	40.1
1	40.3
5	40.7
2	41.0
7	41.1
6	41.3

For the extreme low value, the calculated value of Q is:

$$Q_{calc} = \frac{|38.0-40.1|}{(41.3-38.0)} = 0.636$$

The critical value of Q is 0.568 for α=0.05 and for n=7. The calculated value of Q, 0.636, is greater than the critical value of Q (e.g. $Q_{calc} > Q_{critical}$). Thus, the suspect value is an outlier and should be removed.

4.6.1 Uncertainty Where available, LSB utilizes well-recognized test methods which specify limits to major sources of uncertainty (e.g., a balance accurate to ± 0.1 g) and provide data reporting

instructions so that the reported results do not give the wrong impression of the uncertainty. LSB provides customers QC data with each final report. Acceptance requirements for all QC are also included on the report to communicate compliance with the specified limit and provide an estimate of the uncertainty associated with the final results of the dataset. Where applicable, a statement on the estimated uncertainty of measurement will be included, when it is relevant to the validity of the test result, requested by the customer or the uncertainty may affect compliance to a regulatory limit.

4.6.2 If requested to provide a more rigorous estimate of the uncertainty of a test result, the analyst in consultation with the Section Chief and QAC will use one of the following two options.

4.6.2.1 Estimation of Uncertainty using Laboratory Control Samples (adapted from: Georgian, 2000, Environmental Testing and Analysis). This method uses the limits of historical LCS data to estimate results to a 95% confidence interval using the following equation:

$$\text{Uncertainty} = 100\left(\frac{C}{R}\right)\left(1 \pm \frac{L}{R}\right)$$

Where: c = measured concentration of the analyte
L = the half width of the control range, that is, (UCL-LCL)/2
R = mean historical LCS recovery

Because the LCS is a measure of the performance of the entire analytical process, including instrument calibration, this is LSB's preferred method of estimating uncertainty because it can estimate the uncertainty of the entire analytical process with actual analytical results.

4.6.2.2 Standard Methods 1030B Measurement Uncertainty

4.6.2.3 LSB uses the results from External Proficiency Testing Samples to assess bias.

4.7 Data Reporting

All analytical data generated by LSB will be entered into and reported from Element®.

4.7.1 Analytical Data Qualifiers Added to data to best describe the quality of the data to the end-user. These qualifiers, based on the QC criteria specified in the published method or technical SOP, are applied during data reduction by primary analysts.

4.7.2 Report Narrative Additional explanatory remarks about the data can be added by the Section Chief (or designee) in the Report Narrative section of the data report. Analysts will add any necessary explanatory remarks about their analyses in the 'Work Order Notes' section of Element®. The Section Chief (or designee) will summarize any pertinent information that needs to be transmitted to the data user in the final report through the report narrative.

Note: Though the Report Narrative is identified as such on the Final Report, in Element® on the reporting screen, it is called the Work Order Case Narrative.

4.7.3 Chemical Abstract Service (CAS) Registry Numbers and EPA Identifiers (EPA ID) Each analyte reported from Element® is also reported with the analyte's corresponding CAS number. For

some analytes reported by LSB (e.g., BOD), a CAS number does not exist. In these cases, a custom EPA ID number is assigned and reported with the specific analyte. EPA's Substance Registry System (SRS) is the source of CAS numbers and EPA IDs reported with all data. The SRS database is located at: http://www.epa.gov/srs. LSB will assign a unique internal 'R4' code to any analyte for which there is neither a CAS number nor EPA ID available in EPA's SRS.

4.7.4 Opinions and Interpretations LSB rarely, if ever, offers opinions and interpretations of the reported data. However, if included with a laboratory report, the opinions and interpretations shall be based on the results obtained from the tested or calibrated item and shall be clearly identified as such. When opinions and interpretations are directly communicated by dialogue with the customer, a record of the dialogue shall be retained.

4.7.5 Demonstration of MRL or sample calculation LSB will demonstrate one MRL or sample calculation per batch of samples as part of the data review and validation procedure. In the event that there were no analytes detected, a calculation check of another QC element should be performed to verify that the system is calculating final results properly.

4.7.6 Reporting Preliminary Data LSB does not report preliminary data on a routine basis; however, upon request of the project leader, preliminary data may be released by the Section Chief (or designee). All preliminary data released shall be in the form of a Draft report from Element®. The report must contain a narrative indicating that the data presented is preliminary, has not been completely reviewed, and should not be utilized for any decision-making purposes.

4.7.7 Re-Reporting of Data LSB receives requests for re-reporting of data due to corrections to sample locations or stations, etc. In those instances, the request will come through the R4COCCorrections mailbox to the QAC. The QAC will forward the requests to the sample custodian and the appropriate Section Chief. Once corrections are complete, the Section Chief (or designee) will issue a new report. The new report will contain a narrative indicating that the data has been re-reported and the reason, and a statement that the new submission replaces the previous reported results. A copy of the new report along with any additional supporting documentation will be added to the project file.

4.8 Data Management and Data Security

4.8.1 Data is managed using both R4LIMS and Element®. R4LIMS is used for project scheduling and Element® is used for analytical data management. R4LIMS is an in-house developed Sybase PowerBuilder® application. All data is stored in an Oracle database residing on an LSASD Windows 2008 Server. Console-level access to the Oracle Server is limited to the LSASD LAN Administrators, and application developers for application development and database administration.

4.8.1.1 Backups of the Oracle database (and the entire LAN) to magnetic tape are performed Monday through Saturday evenings using a redundant network backup system. One backup is conducted remotely from the ORD computer center and another locally from the LSASD computer center, and then duplicated to a storage device that is replicated to the Regional office in Atlanta. After successful backups, the daily tapes located at LSASD are placed in a fire-proof media safe and a copy of the Friday evening backup is rotated to Iron Mountain for offsite storage. More detailed backup procedures can be found on the Region 4, IT SharePoint site.

The custodian of this site is the Region 4 Information Security Officer in the Atlanta office.

4.8.2 Direct access to the Oracle database table space is restricted to authorized EPA IT staff only. Access is limited and on an as-needed basis.

4.8.2.1 End-user access to the database is controlled through the compiled R4LIMS Powerbuilder application, Element® DataSystem and the Adobe Coldfusion® web server

(currently used for reports, conversion utilities, etc.).

4.8.2.1.1 All R4LIMS and Element® application users are required to login to the system using an R4LIMS or Element® application USERID and PASSWORD. An R4LIMS PUBLIC account and the Coldfusion web server, both with limited access as described later, are the only exceptions to this requirement. Otherwise, access is controlled by USERID, with varying rights assigned to each user.

4.8.2.1.2 Access to the EPA network and an account in R4LIMS or Element® is required for access to data for entry or reporting purposes. Rights are assigned to each R4LIMS or Element® user upon request by their supervisor. Rights are assigned by the ASB R4LIMS/Element coordinator, the SESD LAN Administrator, or the application developer.

4.8.2.2 Users are restricted to certain functions within R4LIMS and Element® based on their need and job function. Immediate supervisors generally have rights equivalent to or greater than their subordinates as deemed appropriate. The SESD LAN Administrator and application developer have the overall responsibility for security and functionality of both databases. The ASB R4LIMS/Element® coordinator has the responsibility of security, accuracy, and integrity of the data in the database.

4.8.2.3 Project log entry in R4LIMS is restricted to the Sample Custodian (or those officially trained as such), Region 4 Superfund Division technical liaison, project leaders and their supervisor, QAC and other project custodians as deemed necessary.

4.8.2.3.1 Modifications to the project log entries are restricted to sample custodians and the QAC after the project has been entered.

4.8.2.3.2 Sample logging in Element® is restricted to the sample custodians (or those officially trained as such), the QAC and Section Chiefs.

4.8.2.3.3 Data entry in Element® is restricted to those users who have been given analyst rights.

4.8.2.3.4 Reporting of final data is restricted to Section Chiefs and their designees.

4.8.3 After data has been reported, it cannot be modified without the status of the data being set from 'Reported' to a lower level by the QAC (or someone with QA Administrator rights in Element®) or designee. A searchable audit trail which tracks any change to the data or analyses in the database is maintained within Element®.

4.8.4 LSASD maintains an Element® service agreement, which provides software updates on a

periodic basis. When new versions of the software are released, the LSASD IT staff review the update notes to determine if the updates will impact the functionality of Element®. A temporary workstation is set-up with the new software version loaded in a Test database to troubleshoot the new software version. Staff are tasked with testing the new revision by performing their typical Element® procedures to determine any potential problems with implementing the new software. Once the revision has been tested thoroughly on the test database. IT staff will install the new software version on the computer of one of the Section Chiefs to test out the final reporting process

(reporting cannot be performed in the test database). If no problems are identified. IT staff will set the new revision to install on all laboratory computers upon the next log-in to the system. If problems do arise, the new software is not installed until the issues are resolved with the manufacturer. A copy of the software revision history is located on the LAN.

4.9 Complaints/Inquiries

All complaints shall be reviewed by management (See LSASDPROC-1006 Complaint Resolution and Control of Non-Conforming Work). If the complaint is determined to represent a departure from LSB's policies or procedures or systemic problem, it will enter the corrective action process. All other complaints will be considered as opportunities for improvement and will be addressed as either a preventative action (risk evaluation) or quality improvement. The customer will be informed of the progress of any actions initiated and the resolution of the complaint. All documentation of complaints and resolution thereof will be maintained by the QAC.

4.10 Formal Corrective Actions

LSB requires resolution of non-conforming work through the formal corrective action process. Formal corrective actions will also be initiated to address all systemic problems identified. The formal corrective action process will include a root cause analysis. Corrective actions can be initiated by any staff member; however, it is the responsibility of the QAC to track, monitor and perform any follow-up action needed in relation to the corrective action. The corrective action procedure is detailed in LSASDPROC-1006-Complaint Resolution and Control of Non-Conforming Work.

4.11 Control of Nonconforming Work

LSB mitigates nonconforming work through the formal corrective action process. Nonconforming work is defined as any work which does not meet stated laboratory standards, either with respect to mode of execution or outcome, i.e., data quality. Nonconforming work can be identified at various times during the analytical process. The procedure for correcting nonconforming work is detailed in LSASDPROC-1006 Complaint Resolution and Control of Non-Conforming Work.

When nonconforming work occurs, project leaders, laboratory analysts, management and the QAC have the authority and responsibility to stop work if appropriate. Depending on the conditions, notification of stop work will be verbally communicated to staff conducting the work, then noted in a logbook or through an email chain to all affected personnel.

4.12 Risks and Opportunities

LSB identifies and mitigates risk through the corrective action process. Risks are identified as

preventative actions or opportunities for improvement. Preventative actions and quality improvements consist of proactive processes to prevent problems or complaints and are used as opportunities for improvement. The preventive action procedure is detailed in LSASDPROC-1006 Complaint Resolution and Control of Non-Conforming Work. Preventative actions are documented identical to formal corrective actions. Preventative actions may not require a root cause analysis prior to implementing corrective action. Opportunities for Improvement are observations which may help improve method performance or prevent non-conforming work from occurring in the future. All improvement actions will be proportional to the potential impact on the validity of final results.

Improvement Actions can be initiated by staff at any level within the Division. The QAC tracks the status of all improvement actions and includes updates in weekly Accreditation Reports to management and annually during Management Review.

4.13 Annual Management Review

LSB conducts an annual Management Review, where the effectiveness and conformance to the accreditation standards of the quality management system are assessed and reported to upper level Divisional management. The review also provides an opportunity to plan for any needed improvements to the quality system. The review is documented and maintained by the QAC and covers the LSB's overall quality objectives, to include at a minimum the items outlined in the ISO 17025 standard.

4.14 Quality System Audits

LSB evaluates adherence to quality system policy and procedures, accreditation and certification requirements through quality systems audits. LSB utilizes audits performed internally as well as audits performed by external assessors to evaluate the quality system. All audits are coordinated by the QAC as detailed in LSASDPROC-1004- Internal Audits. All non-conformances identified as a result of an internal or external audit will be addressed through the formal corrective action process.

4.14.1 External Audits performed by the Accrediting Body/Certification Officers are conducted to evaluate the LSB procedures against the most recent accreditation/certification standard. The accreditation cycle spans 4 years and the scope of the external audits are as follows**:**

- **Year 1 –** On site audit of 100% of the scope of accreditation
- **Year 2 –** Remote Surveillance Audit of a subset of the standard determined by the accrediting body
- **Year 3 –** Onsite audit of 50% of the scope of accreditation
- **Year 4 –** Remote Surveillance Audit of a subset of the standard determined by the accrediting body

Note: The current accreditation body performs drinking water certification every two years for those methods where LSB maintains certification.

4.14.2 Internal Audits of the LSASD quality system are conducted on an annual basis as detailed in LSASDPROC-1004. Internal audits assess all aspects of the quality system for each branch within LSASD. The scope of the audit includes a review of quality system procedures against the ISO standard requirements and LASAD quality system requirements, a review of project files generated by LASAD and also method witnessing of analytical methods. Drinking water methods will be

observed annually. The remaining analytical methods will be reviewed over the course of the 4-year accreditation cycle.

4.14.3 External Audits or Project Specific Audits conducted by Independent Assesors: On occasion, audits of a specific project or procedure may be performed by staff independent of LSB. These audits will be coordinated with the QAC. Any non-conformance with the LSASD quality system requirements will be addressed through the formal corrective action process.

TABLE 5-1	
Critical values of the studentized deviation *t* for testing whether a single point should be rejected as an outlier (a = 0.05, two-sided test).[1]	
Sample Size, n	**Critical Value ($t_{critical}$)**
3	1.15
4	1.48
5	1.71
6	1.89
7	2.02
8	2.13
9	2.21
10	2.29
11	2.36
12	2.41
13	2.46
14	2.51
15	2.55
16	2.59
17	2.62
18	2.65
19	2.68
20	2.71
21	2.73
22	2.76
23	2.78
24	2.80
25	2.82
[1] Pearson, E. S.; Hartley, H.O., Eds, *Biometrika Tables for Statisticians*, Vol. I, 3rd ed., Cambridge University Press, London, 1966.	

TABLE 5-2	
Critical values of the *Q* in Dixon's *Q*-test for testing whether a single point should be rejected as an outlier (a = 0.05, two-sided test).[1]	
Sample Size, n	**Critical Value ($Q_{critical}$)**
3	0.970
4	0.829
5	0.710
6	0.625
7	0.568
8	0.526
9	0.493
10	0.466
11	0.444
12	0.426
13	0.410
14	0.396
15	0.384
16	0.374
17	0.365
18	0.356
19	0.349
20	0.342
21	0.337
22	0.331
23	0.326
24	0.321
25	0.317

[1] Rorabacher, D. B., "Statistical treatment for rejection of deviant values of Dixon's 'Q' parameter and related sub-range ratios at the 95% confidence level," *Anal. Chem*. **1991**, 63, 139-146

CHAPTER 6

Methodology

6.1 General

The analytical methods used by LSB are guided by DQOs of specific projects and by program requirements. Occasionally, matrices and samples present analytical challenges or are not amenable to a standardized methodology. Deviations from SOPs are documented by the analyst, approved by the section chief, and stored in the project files. In Element®, methods are associated with an analysis name. Analysis names include an analyte or group of analytes and Element® identifies a specific analytical method for each analysis name.

6.2 Method Information

Each time an analysis is performed, the appropriate method ID is assigned to analysis logs and bench sheets within Element®. This establishes a definitive record of the technique used to prepare and analyze each sample. Details on method applications and limitations are found within the technical SOPs. (Any reference to an analytical method refers to the version of LSB's SOP that was in place at that time for the specific method.) Acceptance criteria for precision and bias are documented in Element® and stored within the database for all analyses.

6.3 Minimum Reporting Limits

Reporting units and MRL tables for routine target analytes analyzed by LSB are maintained within Element® for each matrix and method. The metals, classical/nutrients, volatiles, semivolatiles, pesticides/PCBs and PFAS (per and polyfluorinated compounds) MRL values are summarized in Tables 6-4 through 6-12 respectively of this chapter. Microbiological analyses do not report an MRL and report results strictly as "Present" or "Absent". Any needs for specific quantitation (reporting) or detection levels should be requested as detailed in the section on 'Scheduling' in Chapter 3 or through direct communication with the LSB Section Chief(s). The MRLs listed in the tables are those which are routinely achievable. However, sample-specific MRLs may be higher or lower. Some factors which may influence MRLs are listed below.

6.3.1 The amount of sample used (either volume or weight) will raise or lower specific MRLs.

6.3.2 Dilutions due to high amounts of target analytes or matrix interferences will raise sample-specific MRLs.

6.3.3 Soil and sediment samples are corrected for percent moisture content and reported on a dry-weight basis which may result in a higher MRL.

6.3.4 Tissue samples with low yields during processing will result in elevated MRLs.

6.4 Land Disposal Restrictions (LDR)

6.4.1 During field investigations for the Resource Conservation and Recovery Act (RCRA) program, samples may be collected, and analyses requested to determine whether the medium being sampled

meets the treatment standards under LDR. The RCRA LDR program is intended to ensure that hazardous waste cannot be placed on the land until the waste meets specific treatment standards to reduce the mobility or toxicity of its hazardous constituents. Requirements are covered in 40 CFR Part 268 and are quite complex. Analyses supporting the LDR regulations must meet certain MRLs to demonstrate whether the sample being tested has met the applicable treatment standard. The levels of concern for LDR regulations are presented in Figure 6-1.

6.4.2 When placing requests for LDR, sufficient lead-time (a minimum of 30 days) will be needed. LDR analyses require special reporting conventions that are not routine for LSB's LIMS. The laboratory needs to prepare for additional analyses required for sample characterization and to ensure that results are reported in accordance with RCRA Land Ban requirements. Project leaders should consult LSB Section Chiefs when planning such projects.

6.4.3 Figure 6-1 is a flowchart which provides a decision tree applicable to LDR samples. In addition to following the flowchart, analysts should consult their Section Chief and/or the QAC when analyzing samples for LDR purposes.

6.4.4 Also, see Section 3.4.4 when planning a LDR project.

Table 6-1 Levels of Concern for Various Programs Inorganics)						
PARAMETER	DRINKING WATER 40 CFR 141.13 and 141.62 MCLs	RCRA TCLP (40CFR 261.24 Table 1) and pH (40CFR 261.22)	RCRA LAND BAN LIMITS 40CFR 268.48 Table UTS		ALTERNATIVE RCRA LAND BAN LIMITS FOR SOIL 40 CFR 268.49	WATER QUALITY STANDARDS*
			Wastewater (<1% TSS & <1% TOC by weight CFR268.2)	Non-wastewater		
Antimony	6 µg/L		1.9 mg/L	1.15 mg/L TCLP	11.5 mg/L TCLP	*See publication at www.epa.gov/ost/pc/revcom.pdf
Arsenic	10 µg/L* as of 1/23/06	5.0 mg/L	1.4 mg/L	5.0 mg/L TCLP	50.0 mg/L TCLP	
Barium	2000 µg/L	100.0 mg/L	1.2 mg/L	21 mg/L TCLP	210 mg/L TCLP	
Beryllium	4 µg/L		0.82 mg/L	1.22 mg/L TCLP	12.2 mg/L TCLP	
Cadmium	5 µg/L	1.0 mg/L	0.69 mg/L	0.11 mg/L TCLP	1.1 mg/L TCLP	
Chromium (total)	100 µg/L	5.0 mg/L	2.77 mg/L	0.60 mg/L TCLP	6.0 mg/L TCLP	
Copper	1300 µg/L* See 40CFR 141.80					
Cyanides (Total)	200 µg/L, as free cyanide		1.2 mg/L	590 mg/kg (by 9010 or 9012)	5900 mg/kg (by 9010 or 9012, inferred)	
Cyanides (Amenable)	NA		0.86 mg/L	30 mg/kg (by 9010 or 9012)	300 mg/kg (by 9010 or 9012, inferred)	
Fluoride	4.0 mg/L (Primary) 2.0mg/L (Secondary)		35 mg/L	NA	NA	
Lead	15 µg/L* See 40CFR 141.80	5.0 mg/L	0.69 mg/L	0.75 mg/L TCLP	7.5 mg/L TCLP	
Mercury (non-wastewater /retort)	NA		NA	0.20 mg/L TCLP	2.0 mg/L TCLP	
Mercury	2 µg/L (inorganic)	0.2 mg/L	0.15 mg/L	0.025 mg/L TCLP	0.25 mg/L TCLP	
Nickel			3.98 mg/L	11 mg/L TCLP	110 mg/L TCLP	
Nitrate, as N	10 mg/L					
Nitrite, as N	1 mg/L					
Nitrate + Nitrite						
pH		< 2 or ≥ 12.5				
Selenium	50 µg/L	1.0 mg/L	0.82 mg/L	5.7 mg/L TCLP	57 mg/L TCLP	
Silver		5.0 mg/L	0.43 mg/L	0.14 mg/L TCLP	1.4 mg/L TCLP	
Sulfide			14 mg/L	NA	NA	
Thallium	2 µg/L		1.4 mg/L	0.20 mg/L TCLP	2.0 mg/L TCLP	
Turbidity	1 NTU					
Vanadium			4.3 mg/L	1.6 mg/L TCLP	16 mg/L TCLP	
Zinc			2.61 mg/L	4.3 mg/L TCLP	43 mg/L TCLP	

Table 6-1 Levels of Concern for Various Programs (Organic)					
Parameter	Drinking Water	RCRA TCLP	RCRA Land Ban Limits 40 CFR 268.48 Universal Treatment Sstandards		Alternative RCRA Land Ban Limits for Soil 40 CFR 268.49 (mg/kg unless noted as "mg/L TCLP") (see Note 1)
	40 CFR 141.13 and 141.62 MCLs (mg/L)	(40 CFR 261.24 Table 1) (mg/L)	Wastewater (mg/L)	Non-Watewater (mg/kg unless noted as "mg/L TCLP")	
Acenaphthylene	-	-	0.059	3.4	34
Acenaphthene	-	-	0.059	3.4	34
Acetone	-	-	0.28	160	1600
Acetonitrile	-	-	5.6	38	380
Acetophenone	-	-	0.01	9.7	97
2-Acetylaminofluorene	-	-	0.059	140	1400
Acrolein	-	-	0.29	-	-
Acrylamide	-	-	19	23	230
Acrylonitrile	-	-	0.24	84	840
Aldrin	-	-	0.021	0.066	0.66
4-Aminobiphenyl	-	-	0.13	-	-
Aniline	-	-	0.81	14	140
o-Anisidine (2-methoxyaniline)	-	-	0.01	0.66	6.6
Anthracene	-	-	0.059	3.4	34
Aramite	-	-	0.36	-	-
alpha-BHC	-	-	0.00014	0.066	0.66
Alachlor	0.002	-	-	-	-
Atrazine	0.003	-	-	-	-
Benzene	0.005	0.5	0.14	10	100
Benz(a)anthracene	-	-	0.059	3.4	34

Benzo(a)pyrene	0.0002	-	0.061	3.4	34
Benzo(b)fluoranthene	-	-	0.11	6.8	68
Benzo(k)fluoranthene	-	-	0.11	6.8	68
Benzo(g,h,i)perylene	-	-	0.0055	1.8	18
Benzal chloride	-	-	0.055	6	60
beta-BHC	-	-	0.00014	0.066	0.66
Bromodichloromethane	-	-	0.35	15	150
Bromoform	-	-	0.63	15	150
Bromomethane/Methyl bromide	-	-	0.11	15	150
4-Bromophenyl phenyl ether	-	-	0.055	15	150
n-Butyl alcohol	-	-	5.6	2.6	26
Butyl benzyl phthalate	-	-	0.017	28	280
Carbon disulfide	-	-	3.8	4.8 mg/L TCLP	(see Note 2)
Carbofuran	0.04	-	-	-	-
Carbon tetrachloride	0.005	0.5	0.057	6	60
Chlordane	0.002	0.03	0.0033	0.26	2.6
p-Chloroaniline	-	-	0.46	16	160
Chlorobenzene	0.1	100	0.057	6	60
Chlorobenzilate	-	-	0.1	-	-
2-Chloro-1,3-butadiene	-	-	0.057	0.28	2.8
Chlorodibromomethane	-	-	0.057	15	150
Chloroethane	-	-	0.27	6	60
bis(2-Chloroethoxy)methane	-	-	0.036	7.2	72
bis(2-Chloroethyl)ether	-	-	0.033	6	60
Chloroform	-	6	0.046	6	60
bis(2-Chloroisopropyl)ether	-	-	0.055	7.2	72
p-Chloro-m-cresol	-	-	0.018	14	140
2-Chloroethyl vinyl ether	-	-	0.062	-	-
Chloromethane/Methyl chloride	-	-	0.19	30	300

2-Chloronaphthalene	-	-	0.055	5.6	56
2-Chloropchenol	-	-	0.044	5.7	57
3-Chloropropylene	-	-	0.036	30	300
Chrysene	-	-	0.059	3.4	34
p-Cresidine	-	-	0.01	0.66	6.6
o-Cresol	-	200	0.11	5.6	56
m-Cresol	-	200	0.77	5.6	56
p-Cresol	-	200	0.77	5.6	56
Cyclohexanone	-	-	0.36	0.75 mg/L TCLP	(see Note 2)
o,p′-DDD	-	-	0.023	0.087	0.87
p,p′-DDD	-	-	0.023	0.087	0.87
o,p′-DDE	-	-	0.031	0.087	0.87
p,p′-DDE	-	-	0.031	0.087	0.87
o,p′-DDT	-	-	0.0039	0.087	0.87
p,p′-DDT	-	-	0.0039	0.087	0.87
Dibenz(a,h)anthracene	-	-	0.055	8.2	82
Dibenz(a,e)pyrene	-	-	0.061	-	-
2,4-D	0.07	10	0.72	10	100
Dalapon	0.2	-	-	-	-
delta-BHC	-	-	0.023	0.066	0.66
1,2-Dibromo-3-chloropropane (DBCP)	0.0002	-	0.11	15	150
Dibromomethane	-	-	0.11	15	150
1,2-Dichlorobenzene	0.6	-	0.088	6	60
1,4-Dichlorobenzene	0.075	7.5	0.09	6	60
1,3-Dichlorobenzene	-	-	0.036	6	60
1,1-Dichloroethane	-	-	0.059	6	60
1,2-Dichloroethane	0.005	0.5	0.21	6	60
1,1-Dichloroethylene	0.007	0.7	0.025	6	60
2,6-Dinitrotoluene	-	-	0.55	28	280

2,4-Dinitrotoluene	-	0.13	0.32	140	1400
cis-1,2-Dichloroethylene	0.07	-	-	-	-
trans-1,2-Dichloroethylene	0.1	-	0.054	30	300
2,4-Dichlorophenol	-	-	0.044	14	140
2,6-Dichlorophenol	-	-	0.044	14	140
Methylene chloride	0.005	-	0.089	30	300
1,2-Dichloropropane	0.005	-	0.85	18	180
cis-1,3-Dichloropropylene	-	-	0.036	18	180
trans-1,3-Dichloropropylene	-	-	0.036	18	180
Dieldrin	-	-	0.017	0.13	1.3
Diethyl phthalate	-	-	0.2	28	280
p-Dimethylaminoazobenzene	-	-	0.13	-	-
2,4-Dimethylaniline	-	-	0.01	0.66	6.6
Di(2-ethylhexyl) adipate	0.4	-	-	-	-
bis(2-ethylhexyl) phthalate	0.006	-	-	-	-
2,4-Dimethyl phenol	-	-	0.036	14	140
Dimethyl phthalate	-	-	0.047	28	280
Di-n-butyl phthalate	-	-	0.057	28	280
Di-n-octyl phthalate	-	-	0.017	28	280
1,4-Dinitrobenzene	-	-	0.32	2.3	23
4,6-Dinitro-o-cresol	-	-	0.28	160	1600
2,4-Dinitrophenol	-	-	0.12	160	1600
Di-n-propylnitrosamine	-	-	0.4	14	140
Dinoseb	0.007	-	0.066	2.5	25
1,4-Dioxane	-	-	12	170	1700
Diphenylamine	-	-	0.92	13	130
Diphenylnitrosamine	-	-	0.92	13	130
1,2-Diphenylhydrazine	-	-	0.087	-	-
Disulfoton	-	-	0.017	6.2	62

Diquat	0.02	-	-	-	-
Endosulfan I	-	-	0.023	0.066	0.66
Endosulfan II	-	-	0.029	0.13	1.3
Endosulfan sulfate	-	-	0.029	0.13	1.3
Endothall	0.1	-	-	-	-
Endrin	0.002	0.02	0.0028	0.13	1.3
Endrin aldehyde	-	-	0.025	0.13	1.3
Ethyl acetate	-	-	0.34	33	330
Ethylbenzene	0.7	-	0.057	10	100
Ethyl cyanide/Propanenitrile	-	-	0.24	360	3600
Ethyl ether	-	-	0.12	160	1600
bis(2-Ethylhexyl)phthalate	-	-	0.28	28	280
Ethyl methacrylate	-	-	0.14	160	1600
Ethylene oxide	-	-	0.12	-	-
Ethylene dibromide (EDB)	0.00005	-	0.028	15	150
gamma_BHC (Lindane)	0.0002	0.4	0.0017	0.066	0.66
Famphur	-	-	0.017	15	150
Fluoranthene	-	-	0.068	3.4	34
Fluorene	-	-	0.059	3.4	34
Glyphosate	0.7	-	-	-	-
Heptachlor	0.0004	0.008	0.0012	0.066	0.66
Heptachlor epoxide	0.0002	0.008	0.016	0.066	0.66
Hexachlorobenzene	0.001	0.13	0.055	10	100
Hexachlorobutadiene	-	0.5	0.055	5.6	56
Hexachlorocyclopentadiene	0.05	-	0.057	2.4	24
Hexachloroethane	-	3	0.055	30	300
Hexachloropropylene	-	-	0.035	30	300
Indeno(1,2,3-c,d) pyrene	-	-	0.0055	3.4	34
Iodomethane	-	-	0.19	65	650

Isobutyl alcohol	-	-	5.6	170	1700
Isodrin	-	-	0.021	0.066	0.66
Isosafrole	-	-	0.081	2.6	26
Kepone	-	-	0.0011	0.13	1.3
Methacrylonitrile	-	-	0.24	84	840
Methanol	-	-	5.6	0.75 mg/L TCLP	(see Note 2)
Methapyrilene	-	-	0.081	1.5	15
Methoxychlor	0.04	10	0.25	0.18	1.8
3-Methylcholanthrene	-	-	0.0055	15	150
4,4-Methylene bis(2-chloroaniline)	-	-	0.5	30	300
Methyl ethyl ketone	-	200	0.28	36	360
Methyl isobutyl ketone	-	-	0.14	0.33	3.3
Methyl methacrylate	-	-	0.14	160	1600
Methyl methanesulfonate	-	-	0.018	-	-
Methyl parathion	-	-	0.014	4.6	46
Naphthalene	-	-	0.059	5.6	56
2-Naphthylamine	-	-	0.52	-	-
o-Nitroaniline	-	-	0.27	14	140
p-Nitroaniline	-	-	0.028	28	280
Nitrobenzene	-	2	0.068	14	140
5-Nitro-o-toluidine	-	-	0.32	28	280
o-Nitrophenol	-	-	0.028	13	130
p-Nitrophenol	-	-	0.12	29	290
N-Nitrosodiethylamine	-	-	0.4	28	280
N-Nitrosodimethylamine	-	-	0.4	2.3	23
N-Nitroso-di-n-butylamine	-	-	0.4	17	170
N-Nitrosomethylethylamine	-	-	0.4	2.3	23
N-Nitrosomorpholine	-	-	0.4	2.3	23
N-Nitrosopiperidine	-	-	0.013	35	350

N-Nitrosopyrrolidine	-	-	0.013	35	350
Oxamyl (Vydate)	0.2	-	-	-	-
Parathion	-	-	0.014	4.6	46
Polychlorinated biphenyls (PCBs)	0.0005	-	0.1	10	100
Pentachlorobenzene	-	-	0.055	10	100
Pentachloroethane	-	-	0.055	6	60
Pentachloronitrobenzene	-	-	0.055	4.8	48
Pentachlorophenol	0.001	100	0.089	7.4	74
Phenacetin	-	-	0.081	16	160
Phenanthrene	-	-	0.059	5.6	56
Phenol	-	-	0.039	6.2	62
1,3-Phenylenediamine	-	-	0.01	0.66	6.6
Phorate	-	-	0.021	4.6	46
Phthalic acid	-	-	0.055	28	280
Phthalic anhydride	-	-	0.055	28	280
Picloram	0.5	-	-	-	-
Pronamide	-	-	0.093	1.5	15
Pyrene	-	-	0.067	8.2	82
Pyridine	-	5	0.014	16	160
Safrole	-	-	0.081	22	220
Simazine	0.004	-	-	-	-
Styrene	0.1	-	-	-	-
Tetrachloroethylene	0.005	0.7	0.056	6	60
Toluene	1	-	0.08	10	100
Toxaphene	0.003	0.5	0.0095	2.6	26
2,4,5-TP (Silvex)	0.05	1	0.72	7.9	79
1,2,4-Trichlorobenzene	0.07	-	0.055	19	190
1,2,4,5-Tetrachlorobenzene	-	-	0.055	14	140
1,1,1,2-Tetrachloroethane	-	-	0.057	6	60

1,1,2,2-Tetrachloroethane	-	-	0.057	6	60
2,3,4,6-Tetrachlorophenol	-	-	0.03	7.4	74
1,1,1-Trichloroethane	0.2	-	0.054	6	60
1,1,2-Trichloroethane	0.005	-	0.054	6	60
Trichloroethylene	0.005	0.5	0.054	6	60
Trichlorofluoromethane	-	-	0.02	30	300
2,4,5-Trichlorophenol	-	400	0.18	7.4	74
2,4,6-Trichlorophenol	-	2	0.035	7.4	74
1,2,3-Trichloropropane	-	-	0.85	30	300
1,1,2-Trichloro-1,2,2-trifluoroethane	-	-	0.057	30	300
tris-(2,3-Dibromopropyl) phosphate	-	-	0.11	0.1	1
Vinyl chloride	0.002	0.2	0.27	6	60
Xylenes (total)	10	-	0.32	6	60
Total Trihalomethanes (TTHMs)	0.08	-	-	-	-
Haloacetic acids (HAA5)	0.06	-	-	-	-
Notes:					

1. See 40 CFR Part 268.49 Alternative LDR treatment standards for contaminated soil Paragraph (c)(1)(C). treatment of any constituent subject to treatment to a 90 percent reduction standard would result in a concentration less than 10 times the Universal Treatment Standard for that constituent, treatment to achieve constituent concentrations less than 10 times the universal treatment standard is not required. Universal Treatment Standards are identified in 40 CFR 268.48 Table UTS

2. Carbon disulfide, cyclohexanone, and methanol, treatment must achieve 90 percent reduction in constituent concentrations as measured in leachate from the treated media (tested according to the TCLP) or 90 percent reduction in total constituent concentrations (when a metal removal treatment technology is used), except as provided by paragraph (c)(1)(C)of this section.

3. For other water quality related support see: https://www.epa.gov/environmental-topics/water-topics

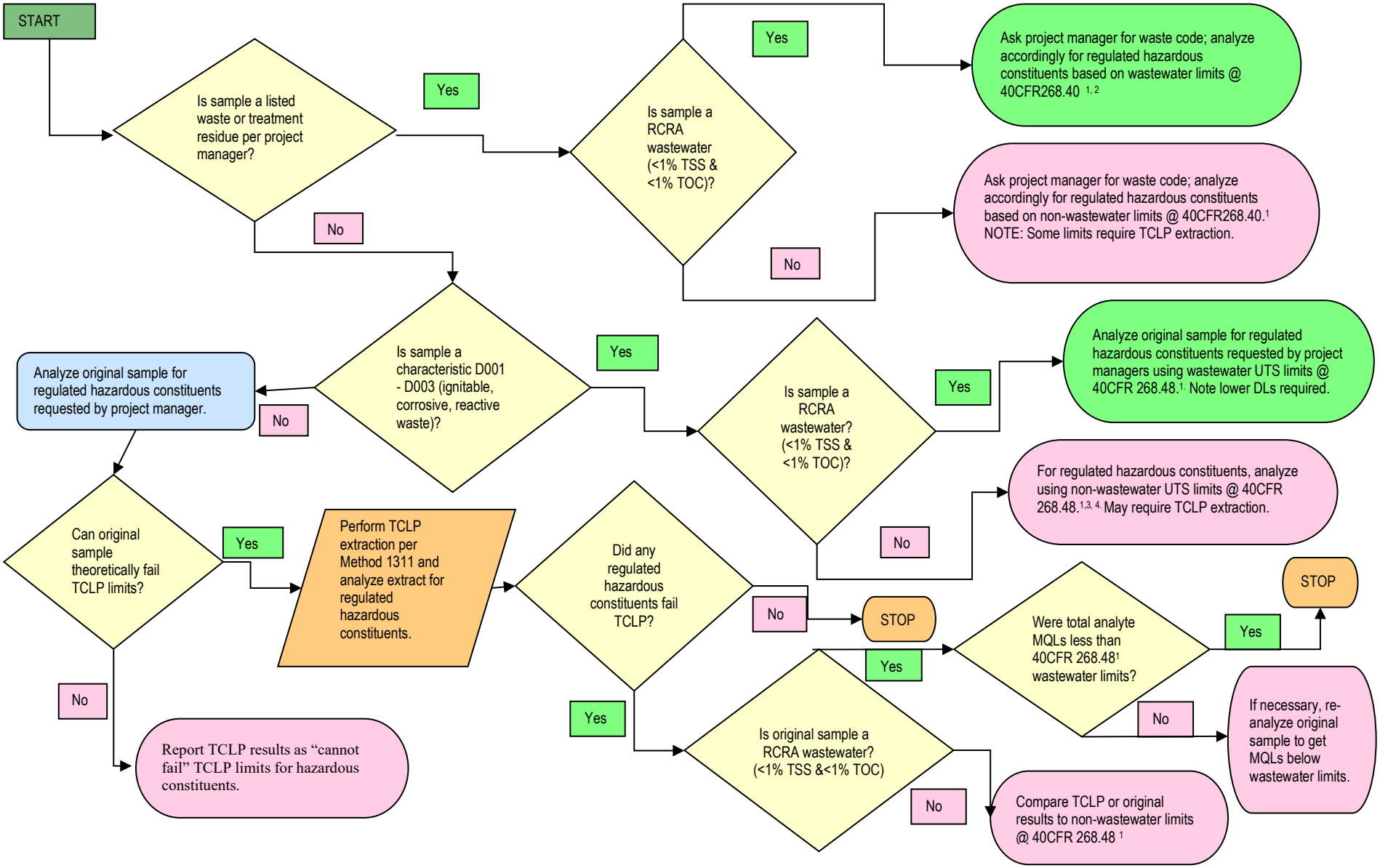


Figure 6-1 Decision Tree for Analysis of Land Disposal Restrictions

Footnotes for Figure 6-1

[1] See LSASD LAN Directory K:\LSB\Current Documents\Miscellaneous Documents for LDR tables contained in 40CFR268.40 and .48.

[2] At 40CFR 268.48 the D009 Wastewater concentration limit requires TCLP extraction for mercury.

[3] A TCLP extraction is required for carbon disulfide, cyclohexanone, methanol, and metals because non-wastewater UTS limits for these analytes are expressed as TCLP extract concentrations.

[4] Non-wastewater cyanide for LDR is performed by special request only. Because the non-wastewater cyanide LDR limits @ 268.48 are expressed in units of mg/kg, do not perform a TCLP extraction for cyanide but instead analyze the original sample for cyanide.

Table 6-2

LSB LOQAM Chapter 6 Table 6-2 Capability for Potable Waters-Inorganics					
SDWA Analyte	**SDWA MCL (mg/L)**	**SDWA Method used by LSB**	**LSB SDWA MRL (mg/L)**	**LSB Routine Method**	**LSB MRL for routine low-level request (mg/L)**
Aluminum (secondary)	0.05-0.2				
Antimony	0.006	200.8	0.0005	200.8	0.0005
Arsenic	0.010	200.8	0.0005	200.8	0.0005
Barium	2	200.7 or 200.8	0.005	200.7 or 200.8	0.005
Beryllium	0.004	200.7 or 200.8	0.003	200.7 or 200.8	0.003
Cadmium	0.005	200.7 or 200.8	0.005	200.7 or 200.8	0.005
Copper (secondary)	1.0	200.7 or 200.8	0.01	200.7 or 200.8	0.01
Chloride (secondary)	250	300.0		300.0	
Chromium (total)	0.1	200.7 or 200.8	0.005	200.7 or 200.8	0.005
Lead	0.0153	200.8	0.0005	200.8	0.0005
Iron (secondary)	0.3				
Manganese (secondary)	0.05				
Mercury (inorganic)	0.002	200.8 or 245.1	0.0004	200.8 or 245.1	0.0001
Selenium	0.05	200.8	0.001	200.8	0.001
Silver (secondary)	0.1				
Thallium	0.002	200.8	0.0005	200.8	0.0005
Zinc (secondary)	5				
Sulfate (secondary)	250				
Asbestos	7MF/L>10u	NA[2]	NA[2]	NA[2]	NA[2]
Bromate	0.010	NA[2]	NA[2]	NA[2]	NA[2]
Chlorite	1.0	NA[2]	NA[2]	NA[2]	NA[2]
Residual Disinfectant	detectable	NA[2]	NA[2]	NA[2]	NA[2]
Fluoride (secondary)	2.0	300.0	0.05	300.0	0.05
Nitrate, as N	10	353.2	0.05	300.0 or 353.2	0.05
Nitrite, as N	1	353.2	0.05	300.0 or 353.2	0.05
Total dissolved solids (secondary)	500				
pH	6.5-8.54	NA[1]	NA[1]	9040C	1.04

Actual MRL may be higher due to variability of analytical instrument conditions or sample interferences.

[1] Not available using SDWA Methods. Please contact Section Chief for more information.

[2] Not available from LSB. Please contact Section Chief for options.

[3] This is an action level, not the MCL. See 40CFR 141.80(c).

[4] The units of the reported numbers are in pH standard units.

NA – Not Available—LSB does not perform this analysis.

Table 6-3

LSB LOQAM Chapter 6 Table 6-3 Capability for Potable Waters - Organics					
SDWA Analyte	**SDWA MCL (mg/L)**	**SDWA Method (special request)**	**LSB SDWA MRL (mg/L)**	**LSB Routine Low-Level Method**	**LSB MRL for routine low-level request (mg/L)**
Benzene	0.005	524.4	0.0005	8260C	0.0005
Carbon Tetrachloride	0.005	524.4	0.0005	8260C	0.0005
Chlorobenzene	0.1	524.4	0.0005	8260C	0.0005
1,2-Dichlorobenzene	0.6	524.4	0.0005	8260C	0.0005
1,4-Dichlorobenzene	0.075	524.4	0.0005	8260C	0.0005
1,2-Dichloroethane	0.005	524.4	0.0005	8260C	0.0005
cis-1,2-Dichloroethylene	0.07	524.4	0.0005	8260C	0.0005
trans-1,2-Dichloroethylene	0.1	524.4	0.0005	8260C	0.0005
Methylene chloride	0.005	524.4	0.0005	8260C	0.0005
1,2-Dichloropropane	0.005	524.4	0.0005	8260C	0.0005
Ethylbenzene	0.7	524.4	0.0005	8260C	0.0005
Styrene	0.1	524.4	0.0005	8260C	0.0005
Tetrachloroethylene	0.005	524.4	0.0005	8260C	0.0005
1,1,1-Trichloroethane	0.2	524.4	0.0005	8260C	0.0005
Trichloroethylene	0.005	524.4	0.0005	8260C	0.0005
Toluene	1	524.4	0.0005	8260C	0.0005
1,2,4-Trichlorobenzene	0.07	524.4	0.0005	8260C	0.0005
1,1-Dichloroethylene	0.007	524.4	0.0005	8260C	0.0005
1,1,2-Trichloroethane	0.005	524.4	0.0005	8260C	0.0005
Vinyl Chloride	0.002	524.4	0.0005	8260C	0.0005
Xylenes (Total)	10	524.4	0.005	8260C	0.0015
Trihalomethanes (Total)	0.08	524.4	0.007	8260C	0.002
2,3,7,8-TCDD (dioxin)	$3x10^{-8}$	NA[2]	NA[2]	NA[2]	NA[2]
Benzo[a]pyrene	0.0002	525.2	0.0002	8270D SIM[3]	0.0001
Carbofuran	0.04	NA[2]	NA[2]	NA[2]	NA[2]
Chlordane	0.002	NA[1]	NA[1]	8081B[3]	0.0015
bis(2-ethylhexyl)adipate	0.4	525.2	0.001	NA	NA
bis(2-ethylhexyl)phthalate	0.006	525.2	0.001	8270D	0.006
Dibromochloropropane (DBCP)	0.0002	NA[1]	NA[1]	8011/8260C[3]	0.00005
Diquat	0.02	NA[2]	NA[2]	NA[2]	NA[2]
Endothall	0.1	NA[2]	NA[2]	NA[2]	NA[2]
Endrin	0.002	525.2	0.002	8081B	0.00005

LSB LOQAM Chapter 6 Table 6-3
Capability for Potable Waters - Organics

SDWA Analyte	SDWA MCL (mg/L)	SDWA Method (special request)	LSB SDWA MRL (mg/L)	LSB Routine Low-Level Method	LSB MRL for routine low-level request (mg/L)
Ethylene dibromide (EDB)	0.00005	NA[1]	NA[1]	8260C[3]	0.00005
Glyphosate	0.7	NA[2]	NA[2]	NA2	NA2
Heptachlor	0.0004	525.2	0.0004	8081B	0.00005
Heptachlor Epoxide	0.0002	525.2	0.0002	8081B	0.00005
Hexachlorobenzene	0.001	525.2	0.001	8270D	0.001
Hexachlorocyclopentadiene	0.05	NA[1]	NA[1]	8270D	0.05
Lindane (gamma-BHC)	0.0002	525.2	0.0002	8081B	0.00005
Methoxychlor	0.04	525.2	0.015	8081B	0.0002
Oxamyl (Vydate)	0.2	NA[2]	NA[2]	NA[2]	NA[2]
PCBs (as Decachlorobiphenyl)	0.0005	NA[2]	NA[2]	8082-Aroclors	0.0005
Pentachlorophenol	0.001	NA[1]	NA[1]	8270D	0.0001
Picloram	0.5	NA[1]	NA[1]	8321B	0.0000125
Simazine	0.004	NA[2]	NA[2]	NA[2]	NA[2]
Toxaphene	0.003	NA[1]	NA[1]	8081B	0.002
HAA5	0.060	NA[2]	NA[2]	NA[2]	NA[2]

Actual MRL may be higher due to variability of analytical instrument conditions or sample interferences.

[1] Not available from LSB using SDWA Method. Please contact Organic Chemistry Section Chief for more information.

[2] Not available from LSB. Please contact Organic Chemistry Section Chief for options.

[3] Analysis available upon request with sufficient lead-time.

NA – Not Available—LSB does not perform this analysis.

Table 6-4

LSB LOQAM Chapter 6 Table 6-4 Metals Analyte List Minimum Reporting Limits by Matrices					
ANALYTE	**LSB Routine Analytical Method[4]**	**Water µg/L (ppb) [3]**	**Soil/Sed mg/kg (ppm) [1, 3]**	**Waste mg/kg (ppm) [1]**	**Tissue mg/kg (ppm) [2, 3]**
Antimony	EPA 200.8	0.5	0.05	0.05	0.01
Arsenic	EPA 200.8	0.5	0.05	0.05	0.01
Aluminum	EPA 6010C	100	10	10	2
Barium	EPA 6010C	5.0	0.5	0.5	0.1
Beryllium	EPA 6010C	3.0	0.3	0.3	0.06
Cadmium	EPA 200.8	0.25	0.025	0.025	0.00025
Calcium	EPA 6010C	250	25	25	5
Cobalt	EPA 6010C	5.0	0.5	0.5	0.1
Chromium	EPA 6010C	5.0	0.5	0.5	0.1
Chrom., Hexavalent	SM 3500 CR B (20th ed)	10	5.0	5.0*	NA
Chrom., Hexavalent, Dissolved	EPA 218.6	1.0, 0.025*	NA	NA	NA
Copper	EPA 6010C	10	1.0	1.0	0.2
Iron	EPA 6010C	100	10	10	2.0
Lead	EPA 200.8	0.5	0.05	0.05	0.01
Magnesium	EPA 6010C	250	25	25	5
Manganese	EPA 6010C	5.0	0.5	0.5	1.0
Mercury	EPA 200.8 or 245.1/7473[5]	0.40	0.05	0.05	0.05
Hg, Ultra-trace	EPA 1631E	0.5 ng/L	0.05 µg/kg	NA	0.05 µg/kg
Molybdenum	EPA 6010C	10	1.0	1.0	0.2
Nickel	EPA 6010C	10	1.0	1.0	0.2
Potassium	EPA 6010C	1000	100	100	20
Selenium	EPA 200.8	1.0	0.10	0.10	0.02
Sodium	EPA 6010C	1000	100	100	20
Strontium	EPA 6010C	5.0	0.5	0.5	0.1
Silver	EPA 6010C	5.0	0.5	0.5	0.1
Tin	EPA 6010C	15	1.5	1.5	NA*

LSB LOQAM Chapter 6 Table 6-4
Metals Analyte List
Minimum Reporting Limits by Matrices

ANALYTE	LSB Routine Analytical Method[4]	Water µg/L (ppb) [3]	Soil/Sed mg/kg (ppm) [1, 3]	Waste mg/kg (ppm) [1]	Tissue mg/kg (ppm) [2, 3]
Titanium	EPA 6010C	5.0	0.5	0.5	0.1
Thallium	EPA 200.8	0.5	0.05	0.05	0.01
Vanadium	EPA 6010C	5.0	0.5	0.5	0.1
Yttrium	EPA 6010C	3.0	0.3	0.3	0.06
Zinc	EPA 6010C	10	1.0	1.0	0.2
Boron **	EPA 6010C	50	5.0	5.0	1.0
Silicon **					
Uranium **					

LSASD routinely performs TCLP extractions and analyses. **MRLs may increase due to variability of interferences that make sample dilutions necessary.** Sample sizes required for achieving the routine quantitation limits are listed below.

[1] Reporting limits are based on 1.0 g of sample (dry-weight basis, % moisture will increase MRLs).

[2] Reporting limits are based on 5.0 g of sample.

[3] Units as specified unless otherwise noted.

[4] Routine methods may be changed at the time of analysis due to sample-specific characteristics. The actual analytical method used will be listed on the final report.

[5] Mercury methods – Water: 245.1/200.8; Soil, Waste, and Tissue: 7473

NA – Not Available—LSB does not perform this analysis.

*This level or matrix is a special request and will need to be discussed with Section Chief on a case by case basis. Consult laboratory for more information.

**These parameters are not usually requested or part of our routine scans. However, if the need arises, please contact LSB personnel.

Table 6-5

LSB LOQAM Chapter 6 Table 6-5
Nutrients and Classicals Analyte List
Minimum Reporting Limits by Matrices

ANALYTE	Analytical Method [5]	Water mg/L (ppm)[1]	Soil/Sed mg/kg (ppm)	Waste mg/kg (ppm)	Tissue mg/kg (ppm)
Acidity	SM 2310	10	NA	NA	NA
Alkalinity	SM 2320B	1.0	NA	NA	NA
Ammonia	EPA 350.1	0.05	2.5[2]	2.5[2]	NA
BOD/C-BOD	SM 5210B	2.0	NA	NA	NA
Bromide	EPA 300.0	0.1	1.0	NA	NA
Chloride	EPA 300.0	0.1	1.0	NA	NA
Cyanide	SM 335.4	0.005	0.25	0.25	NA
Fluoride	EPA 300.0	0.05	0.5	NA	NA
Hardness, Calc	SM 2340B	1.654	NA	NA	NA
Nitrate	EPA 300.0/EPA 353.2	0.05	0.5	0.5	NA
Nitrite	EPA 300.0/EPA 353.2	0.05	0.5	0.5	NA
Nitrate+Nitrite	EPA 353.2	0.05	0.5	0.5	NA
pH	EPA 9040/EPA 9045	1.0 pH units	1.0 pH units	1.0 pH units	1.0 pH units
Phosphorus, Total	EPA 365.1	0.01	1.25[4]	1.25[4]	NA
Phosphorus, Ortho	EPA 365.1	0.01	1.25[4]	1.25[4]	NA
Total Dissolved Solids	USGS I-1750-85	40	NA	NA	NA
Total Solids	SM 2540B-1997	40	NA	NA	NA
Total Suspended Solids	USGS I-3765-85	4.0	NA	NA	NA
Volatile Solids	SM 2540 E	4.0/40[7]	NA	NA	NA
Sulfate	EPA 300.0	0.1	1.0	NA	NA
Total Kjeldahl Nitrogen (TKN)	EPA 351.2	0.05	6.25[4]	6.25[4]	NA
Total Organic Carbon (TOC)	SM5310/LSB 107C	1.0	12,000	NA	NA

MRLs may increase due to variability of interferences that make dilutions of sample necessary. Sample sizes required for achieving the routine quantitation limits are listed below.

[1] Units as specified unless otherwise noted.

[2] Calculated using 1.0 g of sample (dry-weight basis, % moisture will increase MRLs).

[3] Calculated using 5.0 g of sample (dry-weight basis, % moisture will increase MRLs).

[4] Calculated using 0.2 g of sample (dry-weight basis, % moisture will increase MRLs).

[5] Routine methods may be changed at the time of analysis due to sample specific characteristics. The actual analytical method used will be listed on the final report.

[6] Analysis available upon request with sufficient lead-time.

[7] MRL for volatile solids for the TSS method is 4.0 mg/L; if it is derived from the TDS method, then the MRL is 40 mg/L.

NA – Not Available—LSB does not perform this analysis.

Table 6-6

LSB LOQAM Chapter 6 Table 6-6 Volatile Organics (VOAs) Target Analyte List Minimum Reporting Limits (MRLs) by Matrices					
		Water[1] µg/L (ppb)	Soil/Sed[2] µg/kg (ppb)	Waste[3] mg/kg (ppm)	Air[4,6] ppbv
ANALYTE	**Analytical Method**	**Routine Level**	**Routine Level (Encore®/Tared Vial)**	**Routine Level**	**Routine Level EPA TO-15**
Acetone	EPA 8260C	4.0-10	10-20	1.6-4.0	0.20
Acrolein	EPA 8260C	10-20[5]	NA[5]	4.0[5]	0.20
Acrylonitrile	EPA 8260C	10-20[5]	NA[5]	4.0[5]	NA
Benzene	EPA 8260C	0.50	1.0	0.20	0.040
2,3-Benzofuran	EPA 8260C	0.50[5]	NA[5]	0.2[5]	NA
Benzyl Chloride	EPA 8260C	NA	NA	NA	0.20
Bromobenzene	EPA 8260C	0.50	1.0	0.20	NA
Bromochloromethane	EPA 8260C	0.50	1.0	0.20	NA
Bromodichloromethane	EPA 8260C	0.50	1.0	0.20	0.020
Bromoform	EPA 8260C	1.0-4.0	2.0-10	0.40-1.6	0.20
Bromomethane	EPA 8260C	2.0-5.0	2.0	0.80-2.0	0.20
1,3-Butadiene	EPA 8260C	NA	NA	NA	0.20
n-Butylbenzene	EPA 8260C	0.50	1.0	0.20	NA
sec-Butylbenzene	EPA 8260C	0.50	1.0	0.20	NA
tert-Butylbenzene	EPA 8260C	0.50	1.0-2.0	0.20	NA
Carbon Tetrachloride	EPA 8260C	0.50	1.0	0.20	0.020
Carbon Disulfide	EPA 8260C	2.0	2.0	0.80	0.20

Chlorobenzene	EPA 8260C	0.50	1.0	0.20	0.020
Chloroethane	EPA 8260C	2.0-5.0	2.0	0.80-2.0	0.20
2-Chloroethyl vinyl ether	EPA 8260C	1.0-4.0[5]	NA[5]	0.4[5]	NA
Chloroform	EPA 8260C	0.50	1.0	0.20	0.020
Chloromethane	EPA 8260C	0.50	1.0	0.20	0.044
o-Chlorotoluene	EPA 8260C	0.50	1.0	0.20	NA
p-Chlorotoluene	EPA 8260C	0.50	1.0	0.20	NA
Cyclohexane	EPA 8260C	0.50	1.0	0.20	0.020
Dibromochloromethane	EPA 8260C	0.50	1.0	0.20	0.20
1,2-Dibromo-3-chloropropane[7] (DBCP)	EPA 8260C *EPA 8260C SIM*	1.0-10 *0.20*	2.0-10	0.40-4.0	NA
1,2-Dibromoethane (EDB)[7]	EPA 8260C *EPA 8260C SIM*	1.0 *0.05*	1.0	0.20	0.020
Dibromomethane	EPA 8260C	0.50	1.0	0.20	NA
1,2-Dichlorobenzene	EPA 8260C	0.50	1.0	0.20	0.020
1,3-Dichlorobenzene	EPA 8260C	0.50	1.0	0.20	0.020
1,4-Dichlorobenzene	EPA 8260C	0.50	1.0	0.20	0.020
Dichlorodifluoromethane (R12)	EPA 8260C	0.50	1.0	0.20	0.020
1,1-Dichloroethene[7]	EPA 8260C	0.50	1.0	0.20	0.020
cis-1,2-Dichloroethene	EPA 8260C	0.50	1.0	0.20	0.020
trans-1,2-Dichloroethene[7]	EPA 8260C	0.50	1.0	0.20	0.020
1,1-Dichloroethane[7]	EPA 8260C	0.50	1.0	0.20	0.020
1,2-Dichloroethane[7]	EPA 8260C	0.50	1.0	0.20	0.020
1,2-Dichloropropane	EPA 8260C	0.50	1.0	0.20	0.020
1,3-Dichloropropane	EPA 8260C	0.50	1.0	0.20	NA
2,2-Dichloropropane	EPA 8260C	0.50	1.0	0.20	NA
1,1-Dichloropropene	EPA 8260C	0.50	1.0	0.20	NA
cis-1,3-Dichloropropene	EPA 8260C	0.50	1.0	0.20	0.020

Dichlorotetrafluoroethane (R114)	EPA TO-15	NA	NA	NA	0.020
trans-1,3-Dichloropropene	EPA 8260C	0.50	1.0	0.20	0.020
1,4-Dioxane	EPA TO-15	NA	NA	NA	0.20
Ethyl acetate	EPA TO-15	NA	NA	NA	0.020
Ethyl benzene	EPA 8260C	0.50	1.0	0.20	0.020
4-Ethyltoluene (1-Ethyl-4-methyl benzene)	EPA TO-15	NA	NA	NA	0.020
Heptane	EPA TO-15	NA	NA	NA	0.040
Hexachlorobutadiene	EPA 8260C	0.50	1.0	0.20	0.020
Hexane	EPA TO-15	NA	NA	NA	0.040
Isopropanol	EPA TO-15	NA	NA	NA	0.20
Isopropylbenzene	EPA 8260C	0.50	1.0	0.20	NA
p-Isopropyltoluene	EPA 8260C	0.50	1.0-2.0	0.20	NA
Methyl acetate	EPA 8260C	0.50	2.0	0.40	NA
Methyl cyclohexane	EPA 8260C	0.50	1.0	0.20	NA
Methylene chloride (Dichloromethane)	EPA 8260C EPA 8260C SIM	0.50 0.050	10	0.20	0.20
Methyl butyl ketone	EPA 8260C	1.0	5.0-10	0.40	0.20
Methyl ethyl ketone	EPA 8260C	4.0-10	5.0-10	1.6-4.0	0.20
Methyl isobutyl ketone	EPA 8260C	1.0	5.0-10	0.40	0.20
Methyl Methacrylate	EPA 8260C	0.50[5]	NA[5]	0.2[5]	0.020
Methyl-t-butyl ether	EPA 8260C	0.50	1.0	0.20	0.020
Naphthalene	EPA 8260C	0.50-5.0	1.0-10	0.20-2.0	0.020
n-Propylbenzene	EPA 8260C	0.50	1.0	0.20	NA
Styrene	EPA 8260C	0.50	1.0	0.20	0.020
1,1,1,2-Tetrachloroethane[7]	EPA 8260C	0.50	1.0	0.20	NA
1,1,2,2-Tetrachloroethane[7]	EPA 8260C	0.50	1.0	0.20	0.020
Tetrachloroethene	EPA 8260C	0.50	1.0	0.20	0.020
Tetrahydrofuran	EPA TO-15	NA	NA	NA	0.20

Toluene	EPA 8260C	0.50	1.0	0.20	0.20
1,2,3-Trichlorobenzene	EPA 8260C	0.50	1.0	0.20	NA
1,2,4-Trichlorobenzene	EPA 8260C	0.50	1.0	0.20	0.020
1,1,1-Trichloroethane[7]	EPA 8260C	0.50	1.0	0.20	0.020
1,1,2-Trichloroethane	EPA 8260C	0.50	1.0	0.20	0.020
Trichloroethene	EPA 8260C	0.50	1.0	0.20	0.020
Trichlorofluoromethane (R11)	EPA 8260C	0.50	1.0	0.20	0.020
1,2,3-Trichloropropane	EPA 8260C	0.50	1.0-2.0	0.20	NA
Trichlorotrifluoroethane (R113)	EPA 8260C	0.50	1.0	0.20	0.020
1,2,3-Trimethylbenzene	EPA 8260C	0.50[5]	NA[5]	0.2[5]	NA
1,2,4-Trimethylbenzene	EPA 8260C	0.50	1.0	0.20	0.020
1,3,5-Trimethylbenzene	EPA 8260C	0.50	1.0	0.20	0.020
Vinyl acetate	EPA TO-15	NA	NA	NA	0.20
Vinyl chloride[7]	EPA 8260C *EPA 8260C SIM*	0.50 *0.015*	1.0	0.20	0.020
o-Xylene	EPA 8260C	0.50	1.0	0.20	0.020
(m- and/or p-) Xylene	EPA 8260C	1.0	2.0	0.40	0.040

MRLs may increase due to variability of interferences necessitating sample dilutions.

[1] Water – 5 mL from septum-sealed vial.

[2] Routine Level Soil – 5 g in water (reported on dry-weight basis).

[3] Waste – 1 g dissolved in 5-mL methanol and 62.5 uL of resulting extract purged.

[4] Air – 250 cc from 6-L passivated canister – nominal values. MRLs in µg/m^3 units depend on molecular weight and vary depending on the analyte and the standard lot.

[5] Not routinely reported but available upon request.

[6] MRLs don't account for the ~2x pressurization dilution of canisters after arrival at the lab.

[7] SIM MRLs available for waters upon special request.

[8] NATTS SIM MRLs are 10X lower than routine MRLs.

NA – Not Available—LSB does not perform this analysis.

Table 6-7

LSB LOQAM Chapter 6 Table 6-7 Semivolatile Organics Target Analyte List Minimum Reporting Limits by Matrices					
		Water[1] µg/L (ppb)	Soil/Sed[2] µg/kg (ppb)	Waste[3] mg/kg (ppm)	Tissue[4] mg/kg (ppm)
ANALYTE	**Analytical Method**	**Routine Level**	**Routine Level**	**Routine Level**	**Routine Level**
1-Methylnaphthalene	EPA 8270D	2.0	66	20	0.066
1,1'-Biphenyl	EPA 8270D	2.0	66	20	0.066
1,4-Dioxane	EPA 8270D	2.0	66	NA	NA
1,2,4-Trichlorobenzene	EPA 8270D	10	330	100	0.33
2-Nitrophenol	EPA 8270D	10	330	100	0.33
2-Methyl-4,6-dinitrophenol	EPA 8270D	10	330	100	0.33
2,4-Dimethylphenol	EPA 8270D	10	330	100	0.33
2,4-Dinitrotoluene	EPA 8270D	10	330	100	0.33
2,4-Dinitrophenol	EPA 8270D	20	660	200	0.66
2-Methylphenol	EPA 8270D	10	330	100	0.33
2-Nitroaniline	EPA 8270D	10	330	100	0.33
2-Chlorophenol	EPA 8270D	10	330	100	0.33
2-Methylnaphthalene	EPA 8270D	2.0	66	20	0.066
2,3,4,6-Tetrachlorophenol	EPA 8270D	10	330	100	0.33
2,4,5-Trichlorophenol	EPA 8270D	10	330	100	0.33
2-Chloronaphthalene	EPA 8270D	10	330	100	0.33
2,6-Dinitrotoluene	EPA 8270D	10	330	100	0.33
2,4-Dichlorophenol	EPA 8270D	10	330	100	0.33
2,4,6-Trichlorophenol	EPA 8270D	10	330	100	0.33
3,3'-Dichlorobenzidine	EPA 8270D	10	330	100	0.33
(3- and/or 4-) Methylphenol	EPA 8270D	10	330	100	0.33
3-Nitroaniline	EPA 8270D	10	330	100	0.33
4-Chlorophenyl phenyl ether	EPA 8270D	10	330	100	0.33
4-Chloroaniline	EPA 8270D	10	330	100	0.33
4-Nitroaniline	EPA 8270D	10	330	100	0.33
4-Nitrophenol	EPA 8270D	10	330	100	0.33

LSB LOQAM Chapter 6 Table 6-7 Semivolatile Organics Target Analyte List Minimum Reporting Limits by Matrices					
		Water[1] µg/L (ppb)	Soil/Sed[2] µg/kg (ppb)	Waste[3] mg/kg (ppm)	Tissue[4] mg/kg (ppm)
ANALYTE	**Analytical Method**	**Routine Level**	**Routine Level**	**Routine Level**	**Routine Level**
4-Chloro-3-methylphenol	EPA 8270D	10	330	100	0.33
4-Bromophenyl phenyl ether	EPA 8270D	10	330	100	0.33
Acenaphthene	EPA 8270D	2.0	66	20	0.066
Acenaphthylene	EPA 8270D	2.0	66	20	0.066
Acetophenone	EPA 8270D	10	330	100	0.33
Anthracene	EPA 8270D	2.0	66	20	0.066
Atrazine	EPA 8270D	10	330	100	0.33
Benzo[a]anthracene	EPA 8270D	2.0	66	20	0.066
Benzo[a]pyrene	EPA 8270D	2.0	66	20	0.066
Benzo[b]fluoranthene	EPA 8270D	2.0	66	20	0.066
Benzo[k]fluoranthene	EPA 8270D	2.0	66	20	0.066
Benzo[g,h,i]perylene	EPA 8270D	2.0	66	20	0.066
Benzaldehyde	EPA 8270D	10	330	100	0.33
Benzyl butyl phthalate	EPA 8270D	10	330	100	0.33
Bis(2-ethylhexyl) phthalate	EPA 8270D	10	330	100	0.33
Bis(2-chloroethyl) ether	EPA 8270D	10	330	100	0.33
Bis(chloroethoxy)methane	EPA 8270D	10	330	100	0.33
Bis(chloroisopropyl) ether	EPA 8270D	10	330	100	0.33
Caprolactam	EPA 8270D	10	330	100	0.33
Carbazole	EPA 8270D	2.0	66	20	0.066
Chrysene	EPA 8270D	2.0	66	20	0.066
Di-n-butyl phthalate	EPA 8270D	10	330	100	0.33
Di-n-octyl phthalate	EPA 8270D	10	330	100	0.33
Dibenz(a,h)anthracene	EPA 8270D	2.0	66	20	0.066
Dibenzofuran	EPA 8270D	2.0	66	20	0.066
Diethyl phthalate	EPA 8270D	10	330	100	0.33

LSB LOQAM Chapter 6 Table 6-7
Semivolatile Organics Target Analyte List
Minimum Reporting Limits by Matrices

		Water[1] µg/L (ppb)	Soil/Sed[2] µg/kg (ppb)	Waste[3] mg/kg (ppm)	Tissue[4] mg/kg (ppm)
ANALYTE	**Analytical Method**	**Routine Level**	**Routine Level**	**Routine Level**	**Routine Level**
Dimethyl phthalate	EPA 8270D	10	330	100	0.33
Fluoranthene	EPA 8270D	2.0	66	20	0.066
Fluorene	EPA 8270D	2.0	66	20	0.066
Hexachlorobenzene (HCB)	EPA 8270D	10	330	100	0.33
Hexachlorobutadiene	EPA 8270D	10	330	100	0.33
Hexachlorocyclopentadiene (HCCP)	EPA 8270D	10	330	100	0.33
Hexachloroethane	EPA 8270D	10	330	100	0.33
Indeno[1,2,3-cd]pyrene	EPA 8270D	2.0	66	20	0.066
Isophorone	EPA 8270D	10	330	100	0.33
N-Nitrosodiphenylamine	EPA 8270D	10	330	100	0.33
Naphthalene	EPA 8270D	2.0	66	20	0.066
Nitrobenzene	EPA 8270D	10	330	100	0.33
Nitroso-di-n-propylamine	EPA 8270D	10	330	100	0.33
Pentachlorophenol	EPA 8270D	10	330	100	0.33
Phenanthrene	EPA 8270D	2.0	66	20	0.066
Phenol	EPA 8270D	10	330	100	0.33
Pyrene	EPA 8270D	2.0	66	20	0.066

MRLs may increase due to possible interferences necessitating sample dilutions and moisture content of soil samples.

[1] Water – 1000 mL; final extract volume 1 mL.

[2] Soil – 30 g extracted (reported as dry-weight); final extract volume 1 mL.

[3] Waste – 1 g extracted (reported as wet-weight); final extract volume 10 mL.

[4] Fish or biological tissue – Same as soil.

[5] SA = Special Analysis requiring additional QC currently not in place. Contact OCS Section Chief. Tentative MRL.

NA – Not Available—LSB does not perform analysis for this compound.

Table 6-8

LSB LOQAM Chapter 6 Table 6-8 Semivolatile Organics Full Scan – Low Level Minimum Reporting Limits by Matrices					
		Water[1] µg/L (ppb)	Soil/Sed[2] µg/kg (ppb)	Waste mg/kg (ppm)	Tissue mg/kg (ppm)
ANALYTE	**Analytical Method**	**Low Level**	**Low Level**	**Low Level**	**Low Level**
1-Methylnaphthalene	EPA 8270D	0.1	3.33	NA	NA
2-Methylnaphthalene	EPA 8270D	0.1	3.33	NA	NA
Acenaphthene	EPA 8270D	0.1	3.33	NA	NA
Acenaphthylene	EPA 8270D	0.1	3.33	NA	NA
Anthracene	EPA 8270D	0.1	3.33	NA	NA
Benzo[a]anthracene	EPA 8270D	0.1	3.33	NA	NA
Benzo[a]pyrene	EPA 8270D	0.1	3.33	NA	NA
Benzo[b]fluoranthene	EPA 8270D	0.1	3.33	NA	NA
Benzo[k]fluoranthene	EPA 8270D	0.1	3.33	NA	NA
Benzo[g,h,i]perylene	EPA 8270D	0.1	3.33	NA	NA
Carbazole	EPA 8270D	0.1	3.33	NA	NA
Chrysene	EPA 8270D	0.1	3.33	NA	NA
Dibenz(a,h)anthracene	EPA 8270D	0.1	3.33	NA	NA
Fluoranthene	EPA 8270D	0.1	3.33	NA	NA
Fluorene	EPA 8270D	0.1	3.33	NA	NA
Indeno[1,2,3-cd]pyrene	EPA 8270D	0.1	3.33	NA	NA
Naphthalene	EPA 8270D	0.1	3.33	NA	NA
Pentachlorophenol	EPA 8270D	1.0	33.3	NA	NA
Phenanthrene	EPA 8270D	0.1	3.33	NA	NA
Pyrene	EPA 8270D	0.1	3.33	NA	NA

MRLs may increase due to interferences necessitating smaller extraction amounts, dilutions and moisture content of soil samples. The above analytes can also be analyzed by full scan GC/MS at the stated MRLs.

[1] Water – 1000 ml; final extract 1 mL[1] Water – 1000 mL; final extract volume 1 mL.

[2] Soil – 30 g extracted (reported as dry-weight); final extract 1 mL

[3] 0.2 ug/L can be reported if specifically requested.

NA – Not Available—LSB does not perform this analysis.

Table 6-9

LSB LOQAM Chapter 6 Table 6-9 Routine Pesticide/PCB Target Analyte List Minimum Reporting Limits (MRLs)* by Matrices					
		Water[1] µg/L (ppb)	Soil/Sed[2] µg/kg (ppb)	Waste[3] mg/kg (ppm)	Tissue[4] mg/kg (ppm)
ANALYTE	**Analytical Method(s)**	**Routine Level**	**Routine Level**	**Routine Level**	**Routine Level**
Aldrin	EPA 8081B	0.04	1.3	SA[5]-0.50	0.020
Heptachlor	EPA 8081B	0.04	1.3	SA[5]-0.50	0.020
Heptachlor epoxide	EPA 8081B	0.04	1.3	SA[5]-0.50	0.020
α-BHC	EPA 8081B	0.04	1.3	SA[5]-0.50	0.020
β-BHC	EPA 8081B	0.04	1.3	SA[5]-0.50	0.020
γ-BHC	EPA 8081B	0.04	1.3	SA[5]-0.50	0.020
δ-BHC	EPA 8081B	0.04	1.3	SA[5]-0.50	0.020
Endosulfan I	EPA 8081B	0.04	1.3	SA[5]-0.50	0.020
Dieldrin	EPA 8081B	0.04	1.3	SA[5]-0.50	0.020
p,p'-DDT	EPA 8081B	0.04	1.3	SA[5]-0.50	0.020
p,p'-DDE	EPA 8081B	0.04	1.3	SA[5]-0.50	0.020
p,p'-DDD	EPA 8081B	0.04	1.3	SA[5]-0.50	0.020
Endrin	EPA 8081B	0.04	1.3	SA[5]-0.50	0.020
Endosulfan II	EPA 8081B	0.04	1.3	SA[5]-0.50	0.020
Endosulfan sulfate	EPA 8081B	0.04	1.3	SA[5]-0.50	0.020
Endrin aldehyde	EPA 8081B	0.04	1.3	SA[5]-0.50	0.020
Endrin ketone	EPA 8081B	0.04	1.3	SA[5]-0.50	0.020
Methoxychlor	EPA 8081B	0.04	1.3	SA[5]-1.0	0.050
γ-Chlordane	EPA 8081B	0.04	1.3	SA[5]-0.50	0.020
α-Chlordane	EPA 8081B	0.04	1.3	SA[5]-0.50	0.020
Aroclor 1221	EPA 8082A	1.0	33	SA[5]-5.0	0.20
Aroclor 1232	EPA 8082A	1.0	33	SA[5]-2.5	0.10
Aroclor 1242	EPA 8082A	1.0	33	SA[5]-2.5	0.10
Aroclor 1016	EPA 8082A	1.0	33	SA[5]-2.5	0.10
Aroclor 1248	EPA 8082A	1.0	33	SA[5]-2.5	0.10
Aroclor 1254	EPA 8082A	1.0	33	SA[5]-2.5	0.10
Aroclor 1260	EPA 8082A	1.0	33	SA[5]-2.5	0.10

LSB LOQAM Chapter 6 Table 6-9
Routine Pesticide/PCB Target Analyte List
Minimum Reporting Limits (MRLs)* by Matrices

		Water[1] µg/L (ppb)	Soil/Sed[2] µg/kg (ppb)	Waste[3] mg/kg (ppm)	Tissue[4] mg/kg (ppm)
ANALYTE	**Analytical Method(s)**	**Routine Level**	**Routine Level**	**Routine Level**	**Routine Level**
Aroclor 1262	EPA 8082A	1.0	33	SA[5]-2.5	0.10
Aroclor 1268	EPA 8082A	1.0	33	SA[5]-2.5	0.10
Toxaphene	EPA 8081B	5.0	170	SA[5]-20	SA[5]-1.0

MRLs may increase due to possible interferences necessitating sample dilutions and moisture content of soil samples.

[1] Water – 1000 mL extracted; 8081/8082A, final extract volume 10 mL.

[2] Soil – 30 g extracted (reported as dry-weight); 8081/8082A, final extract volume 10 mL.

[3] Waste – 1 g extracted (reported as wet-weight); final extract volume 10 mL.

[4] Fish or biological tissue – 10 g extracted (reported as wet-weight); final extract volume 10 mL.

[5] SA = Special Analysis requiring additional QC currently not in place. Contact OCS Chief. Tentative MRL.

***Pesticide/PCB water and soil MRLs set by LSB at CLP reporting levels.**

Table 6-10

LSB LOQAM Chapter 6 Table 6-10 Pesticide/PCB Analyte List Performed by SPECIAL REQUEST ONLY Minimum Reporting Limits (MRLs) by Matrices					
		Water[1] µg/L (ppb)	Soil/Sed[2] µg/kg (ppb)	Waste[3] mg/kg (ppm)	Tissue[4] mg/kg (ppm)
ANALYTE	**Analytical Method(s)**	**Routine Level**	**Routine Level**	**Routine Level**	**Routine Level**
Technical Chlordane[6]	EPA 8081B	SA[5]-1.5	SA[5]-50	SA[5]-1.5	SA[5]-0.050
β-Chlordene	Modified 8270	SA[5]-0.50	SA[5]-20	SA[5]-0.50	SA[5]-0.020
Chlordene	Modified 8270	SA[5]-0.50	SA[5]-20	SA[5]-0.50	SA[5]-0.020
α-Chlordene	Modified 8270	SA[5]-0.50	SA[5]-20	SA[5]-0.50	SA[5]-0.020
trans-Nonachlor	Modified 8270	SA[5]-0.50	SA[5]-20	SA[5]-0.50	SA[5]-0.020
cis-Nonachlor	Modified 8270	SA[5]-0.50	SA[5]-20	SA[5]-0.50	SA[5]-0.020
Dicofol	Modified 8270	0.080	5.0	NA	NA
4,4'-Dichlorobenzophenone	Modified 8270	0.080	5.0	NA	NA
Chlorobenzilate	Modified 8270	SA[5]-0.020	SA[5]-0.67	NA	NA
2,4'-DDT	Modified 8270	SA[5]-0.040	SA[5]-1.3	NA	SA[5]-0.0013
2,4'-DDE	Modified 8270	SA[5]-0.020	SA[5]-0.67	NA	SA[5]-0.0067
2,4'-DDD	Modified 8270	SA[5]-0.040	SA[5]-1.3	NA	SA[5]-0.0013
PCB (as Congeners) – Green List	EPA 8082A	0.020	1.0	SA[5]-0.20	SA[5]-0.0010
Diazinon	EPA 8270D	SA[5]-1.0	SA[5]-33	SA[5]-2.5	SA[5]-0.050
Methyl Parathion	EPA 8270D	SA[5]-1.0	SA[5]-33	SA[5]-2.5	SA[5]-0.050
Trithion (Carbophenothion)	EPA 8270D	SA[5]-1.0	SA[5]-33	SA[5]-2.5	SA[5]-0.050
Malathion	EPA 8270D	SA[5]-1.0	SA[5]-33	SA[5]-2.5	SA[5]-0.050
Guthion	EPA 8270D	SA[5]-1.0	SA[5]-33	SA[5]-2.5	SA[5]-0.050
Dichlorvos (DDVP)	EPA 8270D	SA[5]-1.0	SA[5]-33	SA[5]-2.5	SA[5]-0.050
Vernolate	EPA 8270D	SA[5]-1.0	SA[5]-33	SA[5]-2.5	SA[5]-0.050
Dimethoate	EPA 8270D	SA[5]-1.0	SA[5]-33	SA[5]-2.5	SA[5]-0.050
Dursban (Chlorpyrifos)	EPA 8270D	SA[5]-1.0	SA[5]-33	SA[5]-2.5	SA[5]-0.050
Phorate	EPA 8270D	SA[5]-1.0	SA[5]-33	SA[5]-2.5	SA[5]-0.050
Ronnel	EPA 8270D	SA[5]-1.0	SA[5]-33	SA[5]-2.5	SA[5]-0.050
Atrazine	EPA 8270D	SA[5]-1.0	SA[5]-33	SA[5]-2.5	SA[5]-0.050
Alachlor	EPA 8270D	SA[5]-1.0	SA[5]-33	SA[5]-12.5	SA[5]-0.25
Stirofos	EPA 8270D	SA[5]-1.0	SA[5]-33	SA[5]-2.5	SA[5]-0.050

LSB LOQAM Chapter 6 Table 6-10
Pesticide/PCB Analyte List Performed by SPECIAL REQUEST ONLY
Minimum Reporting Limits (MRLs) by Matrices

		Water[1] µg/L (ppb)	Soil/Sed[2] µg/kg (ppb)	Waste[3] mg/kg (ppm)	Tissue[4] mg/kg (ppm)
ANALYTE	**Analytical Method(s)**	**Routine Level**	**Routine Level**	**Routine Level**	**Routine Level**
Toxaphene (as congeners except Parlar 62)	EPA 8276	0.0010	0.033	SA[5]-0.005	0.0001
Toxaphene Parlar 62	EPA 8276	0.0050	0.17	SA[5]-0.0250	0.0005

MRLs may increase due to interferences necessitating smaller extraction amounts and dilutions. Percent moisture content of soil samples also affects MRLs.

[1] Water – 1000 mL extracted: 8081A/8082, final extract volume 10 mL; 8276, final extract volume 1 mL; 35 mL extracted: 8011, final extract volume 2 mL.

[2] Soil – 30 g extracted (reported on dry-weight basis); 8081A/8082, final extract volume 10 mL.

[3] Waste – 1 g extracted (reported on wet-weight basis); final extract volume 10 mL.

[4] Fish or biological tissue – 10 g extracted (reported on wet-weight basis); final extract volume 10 mL. Toxaphene congeners: 10 g extracted (reported on wet-weight basis); final extract volume 1.0 mL.

[5] SA = Special Analysis requiring additional QC currently not in place. Contact OCS Section Chief. Tentative MRL.

[6] For TCLP samples, Chlordane must be specifically requested if it is an analyte of interest.

[7] See Appendix 3 VOA MRLs – 8260 SIM method

NA – Not Available—LSB does not perform this analysis.

Table 6-11

LSB LOQAM Chapter 6 Table 6-11 Herbicides Target Analyte List Minimum Reporting Limits (MRLs) by Matrices					
		Water[1] µg/L (ppb)	**Soil/Sed µg/kg (ppb)**	**Waste mg/kg (ppm)**	**Tissue mg/kg (ppm)**
ANALYTE	**Analytical Method**	**Routine Level**	**Routine Level**	**Routine Level**	**Routine Level**
2,4,5-T	EPA 8321B	1.0	NA	NA	NA
2,4-D	EPA 8321B	1.0	NA	NA	NA
2,4-DB	EPA 8321B	2.0	NA	NA	NA
Silvex (2,4,5-TP)	EPA 8321B	1.0	NA	NA	NA
Dalapon	EPA 8321B	2.0	NA	NA	NA
Dicamba	EPA 8321B	1.0	NA	NA	NA
Dichlorprop	EPA 8321B	1.0	NA	NA	NA
Dinoseb	EPA 8321B	4.0	NA	NA	NA
MCPA	EPA 8321B	5.0	NA	NA	NA
MCPP	EPA 8321B	5.0	NA	NA	NA

MRLs may increase due to interferences necessitating smaller sample amounts and dilutions.

NA – Not Available—LSB does not perform this analysis.

Table 6-12

LSB LOQAM Chapter 6 Table 6-12
Per- and Polyfluoroalkyl Substances (PFAS) Target Analyte List
Minimum Reporting Limits (MRLs) by Matrices

ANALYTE	Water µg/L (ppb)	Soil/Sed µg/kg (ppb)	Waste µg/kg (ppb)	Tissue mg/kg (ppm)
Perfluorotetradecanoic acid (PFTeDA)	0.020	NA	NA	NA
Perfluorotridecanoic acid (PFTrDA)	0.010	0.10	0.040	NA
Perfluorododecanoic acid (PFDoDA)	0.010	0.10	0.040	NA
Perfluoroundecanoic acid (PFUDA)	0.010	0.10	0.040	NA
Perfluorodecanoic acid (PFDA)	0.010	0.10	0.040	NA
Perfluorononanoic acid (PFNA)	0.010	0.10	0.040	NA
Perfluorooctanoic acid (PFOA)	0.010	0.10	0.040	NA
Perfluoroheptanoic acid (PFHpA)	0.010	0.10	0.040	NA
Perfluorohexanoic acid (PFHxA)	0.020	0.10	0.040	NA
Perfluoropentanoic acid (PFPeA)	0.010	0.10	0.040	NA
Perfluorobutyric acid (PFBA)	0.010	0.10	0.040	NA
Perfluorodecanesulfonate (PFDS)	0.0097	0.097	0.039	NA
Perfluorononanesulfonate (PFNS)	0.0096	0.096	0.038	NA
Perfluorooctanesulfonate (PFOS)	0.0093	0.093	0.037	NA
Perfluoroheptanesulfonate (PFHpS)	0.0095	0.095	0.038	NA
Perfluorohexanesulfonate (PFHxS)	0.0091	0.091	0.036	NA
Perfluoropentanesulfonate (PFPeS)	0.0094	0.094	0.038	NA
Perfluorobutanesulfonate (PFBS)	0.0089	0.089	0.036	NA
Perfluorooctanesulfonamide (FOSA)	0.010	0.10	0.040	NA
Fluorotelomer sulfonate 8:2 (8:2 FTS)	0.0096	0.096	0.038	NA
Fluorotelomer sulfonate 6:2 (6:2 FTS)	0.0095	0.095	0.038	NA
Fluorotelomer sulfonate 4:2 (4:2 FTS)	0.0094	0.094	0.038	NA
N-ethyl-N-((heptadecafluorooctyl)sulfonyl)glycine (N-EtFOSAA)	0.010	0.10	NA	NA
N-(Heptadecafluorooctylsulfonyl)-N- methylglycine (N-MeFOSAA)	0.010	0.10	0.056	NA
Hexafluoropropylene oxide–dimer acid (HFPO-DA)*	0.020	0.10	0.040	NA
Perfluor-1-butanesulfonamide (FBSA)**	0.010	NA	NA	NA
1,1,2,2,3,3,4,4,4-Nonafluoro-N,N-bis(2-hydroxyethyl)butane-1-sulphonamide (FBSEE-diol)**	0.010	NA	NA	NA

MRLs may increase due to interferences necessitating smaller sample amounts and dilutions.
NA – Not Available—LSB does not perform this analysis.
* HFPO-DA is single lab in-house validated
** Analyte not routinely reported. Special requests with advanced notice required for reporting of these analytes.
Note: This table reflects Region 4 Laboratory Services and Applied Science Division, Laboratory Services Branch (LSB) capability for PFAS analysis using ASTM standards D7979 (water) and D7968 (solids). Lab capacity is 40 samples per week with a 35-day turnaround time. Water matrices is for non-drinking water samples.