



Region 4
U.S. Environmental Protection Agency
Laboratory Services and Applied Science Division
Athens, Georgia

Operating Procedure

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Purpose

The purpose of the 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)

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 17025:2017 standard, “General requirements for the competence of testing and calibration laboratories,” and continually improving quality system effectiveness is a principal objective of the laboratory.

Mission of the EPA Regional Laboratory

The mission of LSB is to provide environmental data for decision making in EPA's multi-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's media programs, States, and Tribes. 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.

DISCLAIMER

The mention of trade names or commercial products in this manual is for illustration purposes only and does not constitute endorsement or recommendation for use by the Environmental Protection Agency.

EPA's Principles of Scientific Integrity

"It is essential that EPA's scientific and technical activities be of the highest quality and credibility if EPA is to carry out its responsibilities to protect human health and the environment. Honesty and integrity in its activities and decision-making processes are vital if the American public is to have trust and confidence in EPA's decisions.", dated February 2012."

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CHAPTER 1 Policy, Accreditation and Hierarchy

1.1 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.

1.2 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 other considerations. 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 everyone to maintain 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 Safety & Occupational Health SharePoint Site at the link below for policies and training requirements.

<https://usepa.sharepoint.com/sites/r4-safety-occup-health/SitePages/Safety-Forms-and-Resources.aspx>

1.3 Data Integrity and Laboratory Ethics

It is the policy of LSB 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 (2016) and the Scientific Integrity Policy (2012). A copy of EPA's Scientific Integrity Policy can be located at the following link:

https://www.epa.gov/sites/production/files/2014-02/documents/scientific_integrity_policy_2012.pdf

1.3.1 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.

1.4 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, and 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:

- 1.4.1** Maintaining and applying a complete and systematic process of QC activities to assist in defining data quality;
- 1.4.2** Ensuring that data quality is documented and communicated to the customer by assigning appropriate qualifiers according to prescribed procedures; and,
- 1.4.3** 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

1.5 Service

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

- 1.5.1 Communication:** Clear communication between the laboratory's staff and its customers 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.
- 1.5.2 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.

- 1.5.3 Accreditation:** As part of the Quality Management System, LSB maintains multiple levels of accreditation and/or certification.
- 1.5.4** 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.
- 1.5.5** The ANSI National Accreditation Board attests that LSB fulfills the requirements of ISO/IEC 17025:2017 ANAB Forensic Testing & Certification AR 3125:2019 in the field of Forensic Testing. Refer to the most current version of the LSASD scope of accreditation for specific accreditation information.
- 1.5.6** The ANSI National Accreditation Board attests under the oversight and approval for EPA's Office of Ground Water and Drinking Water (OGWDW) that LSB fulfills the requirements of ISO/IEC 17025:2017 in the field of testing for drinking water as required by the Fifth Edition of the Manual for the Certification of Laboratories Analytical Drinking Water (EPA 815-R-05-004, January 2005). Refer to the most current LSB scope for specific drinking water certification information. Drinking water certification is issued by OGWDW upon concurrence of the findings provided by the ISO accrediting body performing the certification audit. LSB's certification certificates can be found on the LAN at: <M:\LSB\Current Documents\Accreditation and Certification>
- 1.5.7** 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 data reports 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. For any analyses that LSB subcontracts to a commercial laboratory, the analytical result will show as not accredited under Region 4's quality system on the final report. However, if the laboratory is accredited, the analytical results will show the subcontracts lab certification as applicable.

1.6 Hierarchy

This manual describes the policies that are the basis of LSB's quality system. Specific technical and procedural details are contained in method, 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. If the Branch Supervisor, Section Supervisor, or Quality Assurance Coordinator (QAC) is absent for a more than two days, the appropriate management official within the Branch or Section shall appoint a designee to act on behalf of the individual who is absent. Staff signatures and initials are kept on file by the QAC. For a complete organization chart for LSASD, see the current version of the LSASD Quality Management Plan (QMP).

Laboratory Services Branch Organization

LSB Immediate Office	
Name	Principal Duties
Stacie Masters	Branch Supervisor
Ernest Walton	Senior Technical Advisor
Floyd Wellborn	Senior Technical Advisor

Inorganic Chemistry Section	
Name	Principal Duties
Kristin Trapp	Section Supervisor
Anthony Carroll	Mercury, Hexavalent Chromium, Lead Bioavailability
Megan DeJesus	ICP Metals, Algae
Sally "Yvette" Lane-Walcott	Nutrients, Mercury, Microbiology, Mobile Laboratory (Total Coliforms)
Kayle Whiten	Nutrients, Classicals
Wildelys Colon-Jusino	Nutrients, IC, Hexavalent Chromium, Mercury, Mobile Laboratory (Total Coliforms)
Austin Murray	Nutrients, Classicals
Shannon Ramono	ICP-MS Metals, Microbiology
Blake Snyder	qPCR, Microbiology, Mobile Laboratory (Total Coliforms), LSASD Sample Custodian, Laboratory Quality Coordinator

Organic Chemistry Section	
Name	Principal Duties
David Spidle	Section Supervisor
Brad Acrey	Monitored Natural Attenuation, Volatiles (air)
Coral Ruiz	Volatiles, LSASD Sample Custodian
Justin Schreiner	Semi-Volatiles
Jason Collum	Volatiles , PCBs, LSASD Chemical Hygiene Officer, Laboratory Quality Coordinator, PFAS
John Giles	Organic Extractions, PFAS, Semi-Volatiles, Laboratory Equipment Manager
Tearrany Jackson	Pesticides, Monitored Natural Attenuation, Flashpoint, Mobile Laboratory (Total Coliforms)
Carl Namini	PFAS, Mobile Laboratory (Total Coliforms)
Tomaya Carpenter	LSASD Sample Custodian, Volatiles
Fredrick Allen	Volatiles (air)

2.2 Educational, Experience and Training Requirements

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 fall within the 1300 – Physical Sciences Group, Job Family Standards for Professional Work. (See <https://www.opm.gov/policy-data-oversight/classification-qualifications/classifying-general-schedule-positions/standards/1300/gs1300p.pdf>) 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.

2.2.1 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.

2.2.2 LSASD has developed a set of required training sessions for each employee, which 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, Roles, and Responsibilities. EPA requires ethics training requirements to include: Financial Disclosure, Conflict of Interest, Representation, Misuse of Position, Other Laws/Regulations, and after an employee leaves the agency.

2.3 Branch Supervisor

The LSB Branch Supervisor has overall responsibility for LSB operations. The major duties associated with this position are as follows:

2.3.1 Has overall management responsibility, including hiring, budgeting, and policy development for the branch, and Agency's mission.

2.3.2 Works with the LSASD Deputy Director to ensure the effectiveness of the Quality System through communication with the QAC. The Branch Supervisor provides input for the development, implementation, approval, and continued operation of the branch quality assurance system.

2.3.3 Delegates authority and responsibility for the daily oversight of QC and technical activities in ICS and OCS to the Section Supervisors.

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

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

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

2.4 LSB Technical Advisor

LSB maintains one technical advisor for the entire Branch. The Technical Advisor reports directly to the LSB Branch Supervisor, and the duties of this position are:

2.4.1 The Senior Technical Advisor provides expert technical advice and support to the Branch Supervisor 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 and implementing new analytical methods.

2.4.2 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.

2.4.3 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.

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

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

2.4.6 The Senior Advisor may also serve as primary analyst or technical reviewer as assigned by the Branch Supervisor to support the overall functions of the laboratory and the LSB Section Supervisors.

2.5 Section Supervisor

LSB has two Section Supervisors, one for the Organic Chemistry Section and one for the Inorganic Chemistry Section. Their primary duties are as follows:

2.5.1 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.

2.5.2 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.

2.5.3 Reports final data produced by the section to the customer.

2.5.4 Monitors all section work activities and assignments.

2.5.5 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.

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

2.5.7 Monitors and coordinates section workload and acceptance of work.

2.5.8 Ensures that individual project files are generated and maintained in accordance with branch policies and other appropriate file management requirements.

2.5.9 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.

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

2.5.11 Serve on the Divisional Quality Team

2.5.12 Ensure documents are reviewed and routed for final posting within the specified time periods

2.5.13 Ensures compliance with all laboratory accreditation requirements

2.5.14 Participate in annual Management Reviews

2.5.15 Provide staff for internal audits. Actively engage in audits.

2.5.16 Participate in the corrective action process

2.5.17 Serve as Technical Reviewers for Divisional Quality Management SOPs

2.5.18 Recommend/designate staff for quality support positions

2.6. Laboratory Quality Coordinator

LSB has two designated staff members that serve as Quality Assurance Coordinators in support of the LSASD Quality Management System. The duties of the Branch QAC(s) are as follows:

2.6.1 Perform or assign equipment certifications to ESAT

2.6.2 Manage MDL studies for each Section

2.6.3 Serve as LIMS Administrators

2.6.4 Serve as laboratory lead for internal audits

2.6.5 Serve as laboratory lead for corrective actions

2.6.6 Serve as laboratory SOP technical reviewer

2.6.7 Manage screening of laboratory supplies

2.6.8 Manage DicksonOne Temperature Monitoring System

2.6.9 Serve as Back-up LSASD Sample Custodian

2.7 Analytical Staff

- 2.7.1** Required to have and maintain a working knowledge of the branch and divisional policies and procedures including health and safety, data integrity, and waste disposal.
- 2.7.2** Have a working knowledge of analytical methodologies used within their responsible areas.
- 2.7.3** Have 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.
- 2.7.4** Notify their immediate supervisor of any issues/problems with any work products.
- 2.7.5** Maintain and follow appropriate SOPs for their work areas.

2.8 Primary Analyst

- 2.8.1** 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 deemed competent by the section Supervisor to perform the data reduction.
- 2.8.2** Ensures that the appropriate analytical methodologies and standard operating procedures are followed.
- 2.8.3** Appropriate QC activities are performed as designated by the method, SOPs, and/or the LOQAM.
- 2.8.4** Analytical activities are properly documented as specified by the method, SOPs, and/or the LOQAM.
- 2.8.5** Appropriate actions are taken when QC indicators do not meet established criteria and assures that necessary corrective action is implemented.
- 2.8.6** Individual analytical data points are completely and accurately recorded.
- 2.8.7** Data qualifier flags and explanatory footnotes are properly placed.
- 2.8.8** All appropriate items on the technical review checklist are properly documented.
- 2.8.9** The status of the workorder(s) in Element[®] is set to “Reviewed.”

2.8.10 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.

2.8.11 Communicates all technical and/or QA issues to the Section Supervisor.

2.9 Technical Data Reviewer

2.9.1 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 and completes the required checklist for documentation. In some cases, the Technical Data Reviewer may be responsible for final reporting of the data.

2.9.2 Appropriate analytical methodologies and SOPs were followed.

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

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

2.9.5 Appropriate actions were taken as a result of QC indicators.

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

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

2.9.8 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®.

2.9.9 Project file contains, or references, location of all necessary information including raw data, calibrations, extraction logs, standards, run logs, and dilutions. The status of the data has been updated to Verified upon completion of the Technical Review.

2.10 Acting Supervisor or Designee

Those who are acting on behalf of a Supervisor (also referred to as a designee) assume the duties and responsibilities of that individual under the quality system (except personnel actions). 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. An acting section Supervisor is assigned by the branch Supervisor.

2.11 Environmental Services Assistance Team (ESAT)

- 2.11.1** The EPA has an Agency-wide requirement for technical, analytical, and quality assurance support to the multi-media programs, and uses the ESAT contract for meeting these needs 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 Officer Representative (COR) 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 LSB or LSASD management and leadership.
- 2.11.2** Select LSB staff submit technical direction requests to ESAT through the ESAT Tracking System. ESAT assignments may require communication with the Section Supervisor to assure that the ESAT workload is evenly distributed. Under the existing contract, only the EPA COR to ESAT or Alternate may issue work to ESAT.
- 2.11.3** LSB staff that submit technical direction requests must follow all rules and regulations of the contracting process. These requirements can be located at:
<https://contracts.epa.gov/>. 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. Each data package should be reviewed at a minimum to ensure that:
- Appropriate analytical methodologies and SOPs were followed
 - Appropriate QC activities were performed as designated by the method, SOP, and/or the LOQAM
 - Analytical activities were properly documented as specified by the method, SOP, and/or the LOQAM
 - Appropriate actions were taken as a result of QC indicators
 - Recording of all individual analytical data points are complete and accurate and data qualifier flags and explanatory footNotes are properly placed
 - 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®
 - 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
 - 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, Regional Quality Assurance Manager (RQAM), Divisional Quality Assurance Coordinator and Document Control Coordinator 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.

2.12 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 lessor, LSASD safety officer, and management will ensure the facility and environmental conditions relevant to safety will be monitored as required.

2.13 Equipment

LSB maintains a significant amount of equipment in support of laboratory operations. LSB maintains inventories of all equipment used within the laboratories and documents the maintenance, repairs and required certifications.

2.13.1 Inventory: LSB maintains a list of analytical instrumentation including software and firmware versions as appropriate. The inventory is maintained by the LSB QAC(s) on the LAN in the form of Excel spreadsheets at:

<M:\LSB\Current Documents\Inventories>

2.13.2 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 maintenance logbooks.

2.13.3 Equipment Certifications: LSB maintains records of equipment certifications(thermometers, pipettes, balances, etc,) as well as spreadsheets to track when the certifications are due. These records and spreadsheets are maintained by the LSB QAC(s) and are available for review on the LAN at:

<M:\LSB\Current Documents\Inventories>

CHAPTER 3: Sample Scheduling, Handling, Storage, and Disposal

Complete documentation of the sample collection and handling process is an extremely

important aspect of producing defensible laboratory data. Chain-of-custody (COC) 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.

3.1 Sample Collection

LSB staff does not perform field sampling activities. Sample collection is conducted by LSASD's field branch, contractors, states, and/or tribes following project-specific approved Quality Assurance Project Plans, or Sample and Analysis Plans and SOPs.

3.2 Containers and Holding Times

Selection of sample container types and preservation techniques are guided by the methods being applied. Guidance is available in the applicable reference method, 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, 3-1a, 3-1b, 3-2, and 3-3 below include sample containers, analysis, sample matrices, preservatives, and recommended holding times as observed by LSB. In most cases, LSB can accept smaller aliquots of samples than referenced in the tables below, however, when reducing sample volumes, the volume of preservative must also be reduced to achieve the same final concentrations of preservative in the sample. Before submitting a reduced volume for analysis, the sample requestor shall discuss the requirements with the appropriate Branch or Section Supervisor and document the deviations to the sample volume and preservation in Project Notes.

3.3 Project Scheduling

3.3.1 Initial Scheduling: LSB uses Project Log, an in-house laboratory information management system (LIMS), for project scheduling. Each project entered into Project Log is assigned a unique project number that is used throughout its life for tracking, reporting, and filing. Special Requests and Acceptance of the project will be documented in *Project NOTES* Section of Project Log as detailed below.

3.4 Project Acceptance

3.4.1 LSB Section Supervisors or a designee review requested projects through the Project Log user interface 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 Project Log requesting analysis, LSB management has the first right to refuse the work. If LSB determines there is insufficient capacity or capability to perform the requested work, the customer will be notified in *Project Notes* that the project must be routed to an alternative laboratory. LSB may accept part or all of a requested project depending on capability and capacity. *Project Notes* will clearly indicate which analyses LSB is accepting for the project. LSB does not coordinate CLP services on the customer's behalf. In the event that LSB cannot accept a project, the original requestor will be required to obtain laboratory

services outside of LSB.

3.4.2 Once a project has been accepted, if the project requestor/lead needs to make a change to the request, that change must be documented in *Project Notes* and LSB will again evaluate the ability to accept the project with the updated scope of work. The final acceptance of a project is documented in the *Project Notes* feature in Project Log. *Project Notes* is the official communication and documentation tool for project planning and serves as the official record of what the laboratory and the requestor have agreed upon as the scope of work for the project. Email correspondence, phone calls or verbal conversations do not substitute for acceptance in Project Log.

3.4.3 Scheduling of projects must include an estimate of sample numbers, matrices, requested analyses, turn-around time (TAT) requirements, and reporting limits required for meeting regulatory limits (i.e., decision levels). The standard TAT for the laboratory from the time samples are received until results are reported is 35 calendar days for routine analyses, 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 that project. Communication of special project requirements shall be noted in *Project Notes*, the official record of the project request and acceptance. In the absence of project specific Notes, LSB will assume routine, methods, reporting limits and turnaround time are acceptable for the project.

3.4.4 Project Acceptance Responsibility and Considerations: The acceptance of projects for the LSB laboratory is the responsibility of the Section Supervisors, Branch Supervisor, or designee. Factors considered by LSB management when accepting projects include whether laboratory staff have the necessary skills, expertise, and instrumental capability to perform the environmental tests requested, a demonstration of competency is on file, the laboratory has accreditation for a specific method/analyte/technology when an accredited test result is requested, lab capacity, matrices, reporting limits required for meeting regulatory requirements, requested analyses, and TAT requirements. If the consideration of the above factors indicates any deficiency, or inability to perform the work, laboratory management will notify the project requestor in writing, and resolve any differences in methodology, QC, or scope of work to be performed. The final scope and objectives of the project must be documented in *Project Notes* and accepted by the appropriate LSASD Section Supervisor or designee. *Project Notes* is the official form of communication for LSASD Staff, ESAT and Project Requestor/lead. Any limitations on data usability will also be explained to the customer (e.g., data used for screening purposes only) on the final data report.

3.4.5 Special Requests: 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 new methods and for analyzing analytes for which it is not accredited. Requests for non-routine or new methodologies must be submitted with sufficient lead time (3-6 months) for LSB to establish the method or expand the capabilities to include additional analytes or matrices. The amount of additional time will depend on the scope of the request, availability of supplies for the additional support and availability of staff and instrumentation. Acceptance of these requests will take into account the above-mentioned criteria for acceptance and all correspondence between the Section Supervisor and project requestor will be documented in *Project Notes*. Communication of these limitations will be documented in *Project Notes* during the request and approval process. Any non-accredited data reported by the laboratory will be indicated as such on the final data report.

3.4.6 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. Requests for analytical support for Emergency Response may come from states, local agencies, or other EPA Regions. LSB Section Supervisors will create the request in Project Log in this situation. Acceptance of the project will depend upon LSB's ability to meet the customer's needs in response to the emergency. Acceptance will be documented in the same manner through *Project Notes*. Analysis for which accreditation is not available will be noted in *Project Notes* and the final report will indicate the accreditation status for all analyses.

3.4.7 Potable Water: When LSB receives requests for projects in support of SDWA regulations, analyses must be performed by approved methods found at 40 CFR Part 141. LSB does not analyze the full list of primary drinking water contaminants. See Table 6-3 below for the list of LSB's drinking water certified analytes. For potable water projects, the sample requestor must indicate in *Project Notes* the required method and analytes of interest for the project.

3.4.8 National Pollutant Discharge Elimination System (NPDES): These analyses requested in support of the NPDES regulations at 40 CFR Part 136 require the use of approved methods. Analytical methods being used for regulated NPDES projects cannot be modified without an approved alternate test method, unless they meet conditions of 40CFR136.6.

3.4.9 Request for Use of Specific Analytical Methods or Reporting Limits: When LSB receives a request to use a specific analytical method or provide specific reporting limits, these requests typically initiate a conversation with the requestor as to the ultimate Data Quality Objectives (DQO) and whether the specified method is the most appropriate choice for the requestor's needs. All special requests must be documented in *Project NOTES*. For example, if specific reporting limits are required for comparison to a regulatory limit, the requested reporting limit and the associated analyte should be documented in *Project Notes* when the request is made.

3.4.10 Expedited Turn-Around Projects: If laboratory capacity allows, an expedited turnaround time project can be accepted. It is important that the requested TAT be documented in *Projects Notes* so all analysts are aware of the expedited response needed. Section Supervisors shall also discuss any expedited TAT projects accepted during weekly huddles to ensure staff are aware of the need to be prepared when the project arrives.

3.4.11 Canceled Projects/Samples: Whole projects, individual samples, or analyses may be cancelled due to funding prior to submittal. The project requestor/lead will be responsible for notifying the laboratory in the event a project is cancelled and updating the project status in Project Log.

3.4.12 Statements of Conformity: LSB does not provide statements of conformity. If the customer requests a statement of conformity to a specification or standard for a test or calibration, that request should be included in the *Project Notes*. In the event the Section Supervisor agrees to this request, the decision rule will be communicated and agreed upon with the customer and documented as a PDF attachment to the final report.

3.4.13 Criminal Investigation Requests: LSB may receive project requests in support of criminal investigations. The project request process is the same for these projects. If LSB does not have the capability or capacity to accept the request, the customer will be directed to the National Enforcement Investigations Center (NEIC) for assistance with the request.

3.5 Sample Packing and Sample Receipt

3.5.1 Sample Receipt Policy: LSB will not log in samples that arrive without first being requested and scheduled in Project Log prior to receipt. If this situation occurs, laboratory management will contact the project requestor and/or their management to discuss the unscheduled receipt of samples. LSB may request the project requestor make additional arrangements for laboratory support if LSB does not have the capacity to accept the unscheduled project and will be required to transfer custody of the received samples to the alternate laboratory. LSB does not provide storage for samples which have been or are to be analyzed by other laboratories.

3.5.2 If capacity is available, the project requester will be required to enter the Project in Project Log and the project will be formally accepted prior to sample log-in.

3.5.3 Sample Packing – See Table 3-1 for Recommended Preservation and Hold Times. For samples that are received through carrier service, custody seals are placed on the sample coolers by field sampling personnel. Walk-in samples delivered by EPA Staff or Sample Custodian do not require custody seals. Absence or presence of custody seals is documented on the Sample Receipt Checklist. Due to the nature of environmental

samples, sample containers do not require sealing or re-sealing.

3.5.4 Samples received above method temperature specifications will be affixed with a temperature sticker indicating to all staff the samples were above temperature when received and will require flagging. The project requestor/lead will be notified when samples are received in excess of the temperature requirements. In some instances, the project requestor/lead may direct LSB not to analyze the samples and may schedule an additional sampling event. All communication will be documented in writing and included with the Chain of Custody documentation in the final project file.

3.5.5 Samples received in improper containers or with the incorrect preservation will be brought to the attention of the appropriate Section Supervisor for discussion with the sample requestor/lead. The Section Supervisor will determine if data qualification will be required based on the sample container type received. If qualification will be required, the project requestor will be notified in writing prior to sample log in. Documentation will be included with the Chain of Custody documentation in the final project file.

3.5.6 Sample Receiving Procedure: Samples are received by the LSB LSASD Sample Custodian, or designee 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 version of SOP LSBPROC-105G, Sample Receiving and Custody.

3.5.7 Sample Receipt for Analyses with Short Holding Time Requirements:
Some analyses require expedited receipt to LSASD due to short holding times. The following are the short hold analyses LSB provides that require expedited shipping and receipt:

- Microbiology
- Quantitative Polymerase Chain Reaction (qPCR)
- Semi-volatiles, pesticides/PCBs water samples
- Total Suspended Solids (TSS)
- Total Dissolved Solids (TDS)
- Total Solids (TS)
- Turbidity
- Orthophosphate
- Unpreserved Nitrate or Nitrite,
- Biochemical Oxygen Demand (BOD) Chemical Oxygen demand (CBOD)
- Ethylene oxide water samples
- Unpreserved volatile organic (VOA) water samples,
- VOA soil samples
- pH

NOTE: Observation of Federal holidays and weekends must be considered when planning

sampling projects, especially if any short hold analyses are being requested.

3.5.8 The following sample guidelines must be observed by the field sampling organizations to ensure sufficient time for analysis within the required holding time:

3.5.8.1 Semi-volatiles, Pesticides/PCBs, TSS, TDS, Turbidity, and unpreserved VOA waters: Water samples collected during the week must be received by the lab within 48 hours of collection to meet the required holding time.

3.5.8.2 VOA soils and Ethylene Oxide waters: These samples must be received daily to meet the 48-hour holding time requirement. Freezing of coring devices for VOA soil samples in the field does not extend the 48-hour holding time. VOA soils collected in 40-mL vials and then frozen to -7 to -20°C in the field, do not require expedited receipt.

NOTE: Dry ice cannot be used to freeze VOA soil samples because the temperature in the cooler may fall below -20°C.

3.5.8.3 Unpreserved Nitrate or Nitrite, o-Phosphorous, BOD, and CBOD: These samples must be received daily to meet the 48-hour holding time.

3.5.8.4 Microbiology Potable Water: These samples must be received daily to meet the 30-hour collection to analysis holding time. Non-potable water microbiological samples must be received immediately after sampling to meet the required 6-hour holding times.

3.5.8.5 Unfiltered qPCR samples must be received immediately after sampling to meet the 6-hour collection to filtration holding time. In the event samples are filtered in the field, filters can be shipped on dry ice and received within 48-hours from sampling.

NOTE: LSB does not guarantee holding times can be met for short-hold analyses that are held in the field and not shipped promptly to the laboratory on the aforementioned schedules. When samples are received outside of the holding time, the LSASD Sample Custodian will contact the Project Lead to determine if samples should be analyzed. If requested to analyze samples received outside of the holding time, LSD will qualify all data associated with samples received outside of the required holding times.

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

3.6.1 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. Project requestors must indicate during the

project request process if a project will consist of samples that will be designated RCRA dioxin-containing hazardous waste as part of the sample request process. This policy has been implemented due to the special waste handling and disposal restrictions placed upon listed RCRA dioxin-containing hazardous waste.

3.6.2 LSB will accept environmental 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

3.6.3 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 are advised to use caution and prevent dust in the breathing zone. The use of the proper PPE and engineering controls should be employed

3.6.4 Some environmental media containing PCDD/PCDF may be a hazardous waste when it is excavated or removed from its natural setting due to 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 document, the following listed hazardous wastes pertaining to PCDDs/PCDFs are not accepted by LSASD for analysis: F020, F021, F022, F023, F026, F027, F028, F032, K174, and media subject to EPA's "contained-in policy" resulting from these listed hazardous wastes.

3.6.5 Samples that contain PCDDs/PCDFs will be received by LSB for analysis if they do not meet the definition of a listed hazardous waste as described above. For any site samples suspected of containing PCDDs/PCDFs the project requestor/lead must include this information in Project Notes. If historical data exists, a summary of PCDD/PCDF concentration shall be provided to LSB as part of the sample request.

3.6.6 Disposal of Samples Containing PCDD/PCDF – 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 sites' project manager, the unused samples after laboratory analyses can be returned to the site for disposal.

NOTE: 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.

3.7 Sample Logging and Storage

3.7.1 Assignment of Sample Identification Number : Each sample container is assigned a unique identification number by Element® based on the following pattern:

3.7.2 EYYWWNN-AN-L where EYYWWNN represents a 'Work Order' number, analogous to a Project Log project number. and -AN-L is a sample number within the work order. The letter E is a non-changing designation for samples analyzed by the LSB lab.

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

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

3.7.5 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.

NOTE: See LSBPROC-105G for detailed procedures on logging samples into Element®.

3.7.6 Storage of Samples: All samples are assigned a Home Location in Element® and are stored within the Custody room, in one of three refrigerators or one freezer, which is determined on a project specific basis. All samples must be checked out of and into the Home location when in use. The LSB Sample Custody Laboratory serves as the Home location for all samples following sample login and labeling with the following exceptions:

Samples containers designated for VOA analysis (both water and soil) are assigned a Home Location as the refrigerators/freezers within the VOA analytical laboratory. This is to reduce contamination potential for the VOA analyses.

Microbiology samples are assigned a Home Location of the refrigerator/freezer within either the Microbiology laboratory or Mobile laboratory.

- qPCR samples are assigned a Home location of the freezer in E107.
- VOA air samples canisters are assigned a Home location of the VOA air laboratory and are not subject to temperature requirements for

storage.

- Trace Level Mercury samples are assigned a Home location of E120.

3.7.7 The temperatures of designated storage areas are continuously monitored using an annually certified wireless temperature sensor that interfaces with a data logger controlled via software located on the DicksonOne website.

<https://www.dicksonone.com/>

The system is Cloud-based and the units are connected via WiFi. The software records and sends e-mail and text message alerts to the Divisional Quality Assurance Coordinators, Laboratory Section Supervisors and applicable staff if the temperature falls outside of the specified range.

3.7.8 The acceptable temperature range for refrigerators is from above freezing to 6°C. Freezers are maintained ≤ -10°C and ≤-20°C depending on the method requirements. The DicksonOne temperature monitoring software maintains a list of all temperature excursions. Analysts should acknowledge any excursion and indicate a reason for the excursion within the DicksoneOne website. The LSB Sample Custody Laboratory refrigerator and freezer temperatures are also monitored by security personnel outside regular business hours. LSB management receives text message alerts from the DicksonOne system if the sample custody refrigerators or freezer experience a temperature excursion during non-business hours.

3.8 Sample Custody

3.8.1 Custody Records for all samples received by LSB are maintained within Element®. Reports are generated on each Workorder per analysis, which details the custody of each sample container and is included in the project file.

3.8.2 Custody Room Access – The LSB Sample Custody Laboratory is a secured access area within the LSASD facility. Authorization is restricted to staff that deliver, receive or check samples into or out of the Sample Custody Laboratory, or security that are required to monitor the Sample Custody refrigerators and freezers. The Sample Custody Laboratory contains a card reader that is programmed to restrict access. The LSB Branch Supervisor is responsible for designating access to the LSB Sample Custody Laboratory. Staff access to the Custody Room Access is programmed through the LSASD security system. Staff are required to use their Personal Identity Verification Card(PIV) to gain entry to the Custody Room.

3.8.3 Documentation of Custody – Documentation of sample custody from cradle to grave is accomplished through a COC form initiated at the time of sample collection that documents all transfer of custody, field logbooks, individual analysis logs,

Element® Custody Logs, and sample disposal memos and records. The original Chain of Custody, along with a sample log in or, workorder report, is maintained in the LSB Project files. A copy of the Workorder Report is sent to the project requestor/lead upon completion of sample login. It is the project leader's responsibility to check the Workorder Report against the COC record for accuracy as it relates to analyses requested for the project, sampling station identification, and other meta data information. Any discrepancies between the Chain of Custody and Workorder Report shall be addressed immediately to prevent re-reporting of data.

3.8.4 Assuming Custody for Sample Preparation and Analysis – When assuming custody of samples, staff are required to protect the integrity of all samples by ensuring sample containers remain closed unless in active use. Upon completion of sample preparation or analysis, samples must be returned to the designated storage location, or if consumed, then discarded and recorded in Element®. During preparation if samples are left unattended the sample must remain in the designated secure laboratory. LSB uses Element® to perform sample check-out and check-in to/from the sample Home location. 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 located during use. When the need for the sample container is complete, custody is relinquished by setting the location of the container to either the Home location or marking the container as "Consumed/Disposed" in Element®.

3.8.5 Custody Room Housekeeping – The LSASD 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 LSB Management Team. Facility cleaning staff do not enter the custody room. The custody room is cleaned only by special coordination and scheduling through the facility representative.

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

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

3.9.2 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 documented on the bench sheet. All sample custody transfers are tracked within Element® and custody logs are generated for inclusion in the project file.

3.9.2.1 Extract/Digestion/Leachate Batch IDs are assigned automatically by Element® and are in the format 'YYMMnnnn' where:

3.9.2.2 'YY' is a two-digit number identifying the year of the

batch,

3.9.2.3 ‘MM’ is a two-digit number identifying the month and ‘nnnn’ is a four-digit number representing the incremental batch created that month.

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

3.9.2.5 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.

3.10 Transfer of Custody from LSB

3.10.1 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 LSASD Sample Custodian or designee.

3.11 Review of Custody Records

3.11.1 Review of custody records are performed by the technical reviewer 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 LSBPROC-105G.

Table 3-1 - Recommended Preservation & Holding Times								
Analytical Group	Soil/Sediment ¹		Water ^{1, 2} and Waste Water		Waste		Tissue	
	Preservation ³ , Quantity ⁴ , Container, Type ⁵	Hold ⁶	Preservation ³ , Quantity ⁴ , Container, Type ⁵	Hold ⁶	Preservation ³ , Quantity ⁴ , Container, Type ⁵	Hold ⁶	Preservation ³ , Quantity ⁴ , Container, Type ⁵	Hold ⁶
Inorganics								
Alkalinity	NA	NA	Ice-4°C, 500 mL P, FP, Fill completely and cap tightly	14 days	NA	NA	NA	NA
BOD ₅ / CBOD	NA	NA	Ice-4°C, 2-L P, FP	48 hrs	NA	NA	NA	NA
Bromide	Ice-4°C, 8-ozG	Not specified	Ice-4°C, 500-mL P, FP	28 days	NA	NA	NA	NA
Chloride	Ice-4°C, 8-oz G	Not specified	Ice-4°C, 500-mL P, FP	28 days	NA	NA	NA	NA
Chlorine - Residual	NA	NA	Potable Water Samples	Immediate	NA	NA	NA	NA
Chromium VI (hexavalent)	Ice-4°C 8-oz G	Extract – 30 days Analysis – 7 days ⁷	Filter immed. ⁹ Ice- 4°C, 1L P, FP, Buffer to extend HT ³	24 hrs or 28 days if buffered ³	None 8- oz G	Extract – 30 days Analysis – 7 days ⁷	NA	NA
Dissolved P, total	NA	NA	Filter immed. ⁹ Ice- 4°C, H ₂ SO ₄ to pH<2 500-mL P, FP	28 days	NA	NA	NA	NA
Fluoride	Ice-4°C, 8-oz G	Not specified	Ice-4°C, 500-mL P, FP	28 days	NA	NA	NA	NA

Table 3-1 - Recommended Preservation & Holding Times								
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	Preservation ³ , Quantity ⁴ , Container, Type ⁵	Hold ⁶	Preservation ³ , Quantity ⁴ , Container, Type ⁵	Hold ⁶	Preservation ³ , Quantity ⁴ , Container, Type ⁵	Hold ⁶	Preservation ³ , Quantity ⁴ , Container, Type ⁵	Hold ⁶
Hardness (Calc)	NA	NA	Separate bottle not required, calculated from metals scan	NA	NA	NA	NA	NA
Mercury, Routine	Ice-4°C, 8-oz G	28 days ²⁸	Ice-4°C, 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 8oz 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-4°C 8-oz G	90 days	HCl to pH<2 1L FP (bottles require prescreening and special handling)	90 days	None 8-oz G	Not specified	Freeze, 5 g 8-oz G, Al foil or plastic	Not specified
Metals, except mercury, includes lead bioavailability	None 8-oz G	6 months	HNO ₃ to pH<2 1-L P, FP	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	6 months	NA	NA	NA	NA

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Analytical Group	Soil/Sediment ¹		Water ^{1, 2} and Waste Water		Waste		Tissue	
	Preservation ³ , Quantity ⁴ , Container, Type ⁵	Hold ⁶	Preservation ³ , Quantity ⁴ , Container, Type ⁵	Hold ⁶	Preservation ³ , Quantity ⁴ , Container, Type ⁵	Hold ⁶	Preservation ³ , Quantity ⁴ , Container, Type ⁵	Hold ⁶
Metals – TCLP (except mercury, see above)	None 8-oz G	180 days ¹¹	None, 1-L P, FP or 1-gal. P, FP, G, if multiphase (>0.5% and <10%solids)	180 days ¹¹	None, 8-oz G or 1- gal P, G if multiphase (>0.5% and <10% solids)	180 days ¹¹	NA	NA
Nitrate (requires two containers: one unpreserved and a 2nd preserved. The unpreserved is for nitrite analysis (48hr hold) and the preserved sample is for nitrate+nitrite analysis (28-day hold))	Ice-4°C 8-oz G	Not specified	Ice-4°C, 500 mL P, FP, Fill completely and cap tightly H ₂ SO ₄ to pH<2	48 hrs	NA	NA	NA	NA
Nitrite	Ice-4°C 8-oz G	Not specified	Ice-4°C, 500-mL P, FP	48 hrs	NA	NA	NA	NA
Nutrients (ammonia, TKN, NO ₃ +NO ₂ -N, total phosphorus)	Ice-4°C 8-oz G	Not specified	Ice-4°C, H ₂ SO ₄ to pH<2 500-mL/ parameter or 1-L total P, FP	28 days	NA	NA	NA	NA
Ortho-P	NA	NA	Ice-4°C, 500-mL P, FP	48 hrs	NA	NA	NA	NA
Ortho-P (when equating dissolved with Ortho-P)	NA	NA	Filter immed ⁹ , Ice- 4°C 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
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Table 3-1 - Recommended Preservation & Holding Times

Analytical Group	Soil/Sediment ¹		Water ^{1, 2} and Waste Water		Waste		Tissue	
	Preservation ³ , Quantity ⁴ , Container, Type ⁵	Hold ⁶	Preservation ³ , Quantity ⁴ , Container, Type ⁵	Hold ⁶	Preservation ³ , Quantity ⁴ , Container, Type ⁵	Hold ⁶	Preservation ³ , Quantity ⁴ , Container, Type ⁵	Hold ⁶
Solids Series (TS, TSS, TDS, TVSS)	NA	NA	Ice-4°C, 1-L P, FP	7 days	NA	NA	NA	NA
Sulfate	Ice-4°C 8-oz G	Not specified	Ice-4°C, 500-mL P, FP	28 days	NA	NA	NA	NA
TOC	Ice-4°C 8-oz G	Not Specified	Ice-4°C, H ₂ SO ₄ to pH<2 500-mL P, FP	28 days	NA	NA	NA	NA
Dissolved TOC	NA	NA	Filter immed ⁹ , Ice 4°C, H ₂ SO ₄ to pH<2 500-mL, P, FP	28 days	NA	NA	NA	NA
Turbidity	NA	NA	Ice 4°C, 150 mL, P or G	48 hours	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-4°C 8-oz G ⁴	61 days ¹³	Ice-4°C, 1-L (x2 per fraction) ^{4,15} G/A	61 days ¹³	None 8-oz G ⁴	61 days ¹³	NA	NA

Extractables (Pesticides/PCBs, SVOAs)	Ice-4°C 8-oz G	54 days ¹⁴	Ice-4°C 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-4°C 8-oz G	54 days ¹⁴	Ice-4°C, 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

Table 3-1 - Recommended Preservation & Holding Times

Analytical Group	Soil/Sediment ¹		Water ^{1, 2} and Waste Water		Waste		Tissue	
	Preservation ³ , Quantity ⁴ , Container, Type ⁵	Hold ⁶	Preservation ³ , Quantity ⁴ , Container, Type ⁵	Hold ⁶	Preservation ³ , Quantity ⁴ , Container, Type ⁵	Hold ⁶	Preservation ³ , Quantity ⁴ , Container, Type ⁵	Hold ⁶
Extractables/ Pesticides/PCBs – residual chlorine present	NA	NA	Ice-4°C, 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- 4°C, 40-mL(x3) ¹⁵ G/S	14 days	NA	NA	NA	NA
PFAS, Per- and Polyfluoroalkyl substances (non- drinking water) ASTM	Ice < 6°C, 50 mL, polyoxpropylene DigitubeTM (flat bottom)	42 days ²⁹	Ice ≤6°C, Pre- weighed 15- mL Polypropylene (x2), procured from the lab	42 days ²⁹	Ice ≤10°C, Pre- weighed 15-mL Polypropylene (x2), procured from the lab	42 days ²⁹	NA	NA
PFAS, Per- and Polyfluoroalkyl substances (non- drinking water) 1633	Ice < 6°C, 50 mL, polyoxpropylene DigitubeTM (flat bottom)	90 days	Ice ≤ to 6°C, 250 mL polypropylene bottle (2x)	28 days	NA	NA	NA	NA

PFAS, Per- and Polyfluoroalkyl substances (drinking water)	NA	NA	Ice, above freeze to 6°C, 5gms of Trizma. 250 mL polypropylene bottle, minimum 200 mL of sample	42 days ³⁰	NA	NA	NA	NA
Phenols (analyzed as semivolatile compounds)	Ice 4°C 8-oz G	54 days ¹⁴	Ice-4°C, 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

Table 3-1 - Recommended Preservation & Holding Times

Analytical Group	Soil/Sediment ¹		Water ^{1, 2} and Waste Water		Waste		Tissue	
	Preservation ³ , Quantity ⁴ , Container, Type ⁵	Hold ⁶	Preservation ³ , Quantity ⁴ , Container, Type ⁵	Hold ⁶	Preservation ³ , Quantity ⁴ , Container, Type ⁵	Hold ⁶	Preservation ³ , Quantity ⁴ , Container, Type ⁵	Hold ⁶
Volatile Organics Method 5035A	Ice-4°C, 5 g (x3) ¹⁸ E, or equivalent ¹⁹	48 hours iced/14 days frozen ²⁰	NA	NA	Ice-4°C, 8-oz G ²⁶	14 days ²¹	NA	NA
Volatile Organics Method 5035A	Ice-4°C, 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 -20°C, 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	Ice-4°C 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 Method 5035A	NA	NA	Ice-4°C, 40 mL (x3) ¹⁵ G/S	7 days	NA	NA	NA	NA
Volatile Organics no residual chlorine present Method 5035A	NA	NA	0.2 mL 1+1 HCL (pH<2), Ice-4oC 40-mL (x3)15 G/S	14 days	NA	NA	NA	NA

Table 3-1 - Recommended Preservation & Holding Times

Analytical Group	Soil/Sediment ¹		Water ^{1, 2} and Waste Water		Waste		Tissue	
	Preservation ³ , Quantity ⁴ , Container, Type ⁵	Hold ⁶	Preservation ³ , Quantity ⁴ , Container, Type ⁵	Hold ⁶	Preservation ³ , Quantity ⁴ , Container, Type ⁵	Hold ⁶	Preservation ³ , Quantity ⁴ , Container, Type ⁵	Hold ⁶
Volatile Organics residual chlorine present Method 5035A	NA	NA	3mg Na2S2O3, 0.2mL 1+1 HCl (pH<2), Ice-4°C 40-mL (x3)15 G/S	14 days ²³	NA	NA	NA	NA
Volatile Organics TCLP	Ice-4°C, 2-oz G ⁴	28 days ²⁴	Ice 4°C, 40-mL (x3) ^{4,15} G/S	28 days ²⁴	Ice-4°C, 8-oz G ^{4,26} If solids, 4x 8- oz G	28 days ²⁴	NA	NA

Volatile Organics in Air Method TO-15	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
Ethylene Oxide Method 5035A	NA	NA	Ice 4°C, 40-mL (x3) ¹⁵	48 hours	NA	NA	NA	NA

Table 3-1 - Recommended Preservation & Holding Times- Volatiles in Air

Analytical Group	Air ¹	
	Preservation, ³ Quantity ⁴ , Container, Type ⁵	Hold ⁶
Volatile Organics in Air	Preservation: closed, leak-free valve with tightened cap. Amount: preferably 10-14 psia Container: passivated 6-liter canister	30 days
Methane/Ethane/ Ethene	Preservation: closed, leak-free valve with tightened cap. Amount: preferably 10-14 psia Container: passivated 6-liter canister	30 days

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
<i>E. coli</i> SM 9223B	4°C, 120 mL, Sterilized plastic, non- corrosive glass bottles	8 hours from sampled to placement in the incubator	Cool, 0.008% Na ₂ S ₂ O ₃ , 120 mL Sterilized plastic, non- corrosive glass bottles	30 hours from sampled to placement in the incubator
Total Coliforms SM 9223B	4°C, 120 mL, Sterilized plastic, non- corrosive glass bottles	8 hours from sampled to placement in the incubator	Cool, 0.008% Na ₂ S ₂ O ₃ , 120 mL Sterilized plastic, non- corrosive glass bottles	30 hours from sampled to placement in the incubator
Quantitative Polymerase Chain Reaction (qPCR)	Cool (<10°C), 500 mL, Sterilized plastic or Whirl Pak	Held up to 6 hours before filtration	NA	NA

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	HNO ₃ pH<2	500 mL	Plastic or Glass	6 months
Mercury EPA 245.1	HNO ₃ 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	H ₂ SO ₄ 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 524.4	Ascorbic Acid or Sodium Thiosulfate, HCl pH<2, Cool 4°C	40 ml X 3 vials*	Glass with PTFE-lined Septum	14 days
<i>E. coli</i> (Presence/Absence) SM 9223B	Cool, 0.008% Na ₂ S ₂ O ₃	120 mL	Sterilized plastic, non-corrosive glass bottles	30 hours from sampled to placement in incubator
Total Coliforms (Presence/Absence) SM 9223B	Cool, 0.008% Na ₂ S ₂ O ₃	120 mL	Sterilized plastic, non-corrosive glass bottles	30 hours from sampled to placement in incubator

Tables 3-1, 3-2, 3-3 Notes:

NA = Not applicable

Pres = Preservation

Immed = Immediate

ILSB'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 freezing to 6°C.

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 $\leq 0.04\%$ by weight (pH about 1.96 or greater) 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.

H₂SO₄ - Sulfuric Acid used as a preservative must be present at concentrations $\leq 0.35\%$ by weight (pH about 1.15 or greater), as specified in 40 CFR 136.3, Table II, footnote 3. Approximately 5 mL of the laboratory-prepared preservative is added to the sample.

NaOH - Sodium Hydroxide) used as a preservative must be present at concentrations $\leq 0.080\%$ by weight (pH about 12.30 or less), as specified in 40 CFR 136.3, Table II, footnote 3. Four tablets are added to the sample after collection.

HNO₃ - Nitric Acid used as a preservative must be present at concentrations $\leq 0.15\%$ by weight (pH about 1.62 or greater), as specified in 40 CFR 136.3, Table II, footnote 3. Approximately 5 mL of the laboratory prepared preservative is added to the sample.

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 1L. 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 Sample Quantity: 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. For multi-phase samples, extra sample volume must be collected for TCLP analysis. The TCLP may place requirements on the minimal size of the field sample, depending upon the physical state or states of the waste and the analytes of concern. An aliquot is needed for preliminary evaluation of which extraction fluid is to be used for the nonvolatile analyte extraction procedure. Another aliquot may be needed to actually conduct the nonvolatile extraction.

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, 4 days to analysis of extract. Store at 4±2°C until analyzed (SW-846, Table 3-1).

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 grams 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 solids container!!

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 24 days.

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

27 Perfluorocarbons - Water: 14 days to preparation, 180 days to analysis of extract.

28 From sample collection to analysis.

29PFAS - 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: LSB follows applicable concepts of good laboratory practice standards (GLPS) in all aspects of the organization's operations, and it 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. Good Laboratory Practice Standards are a science management program that specify minimum practices and QA/QC which must be followed in order to ensure the quality and integrity of data submitted to the EPA in support of its everyday operations. GLPS can be found at:

<https://www.epa.gov/compliance/good-laboratory-practices-standards-compliance-monitoring-program>

4.2 Corrections to Records

4.2.1 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.

4.2.2 Corrections to electronic records (e.g., spreadsheets, pdf documents, etc.) shall be made using Adobe Acrobat to preserve the metadata of the changes made. Changes should be documented using a dynamic comment or annotation along with a dynamic stamp that includes the initials of the analyst and date. Element[®] uses an audit trail for tracking modifications made to the database. The audit trail marks the data, time, and analyst's initials of all changes.

4.2.3 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.

4.3 Following SOPs/LOQAM

4.3.1 It is the policy of the LSB's quality system that all technical SOPs, applicable administrative SOPs, and the LOQAM will be followed by all LSB staff, SEE employees, and by the ESAT contract employees. 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 Supervisor, Branch Supervisor (if necessary) and QAC. The rationale for the deviation shall be clearly documented and included and maintained 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 Project Log Notes for review and agreement by the project leader.

4.4 Method Modifications

4.4.1 Where permitted 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 or permitted as a modification and documented in a memo from OGWDW or approved as an Alternate Test Procedure (ATP). When these modifications are used, LSB's SOP must state that an approved modification is being utilized and reference the specific memo or ATP allowing the modification. A copy of all ATPs and/or method change memos must be placed on the specific method SOP reference folder on the LAN.

4.5 Manual Peak Integration

4.5.1 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.

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

4.5.3 In some circumstances, automated electronic integrations can result in incorrect actions by the system software, and for these instances a manual override (i.e., manual integration) and correction of the electronic integration 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.

4.5.4 Manual override actions are appropriate only to correct inaccuracies and shall be done in accordance with sound analytical procedures and analyst's professional judgement. When a manual override of the electronic process is performed, most of the current commercially available software packages provide an automated notation (typically noted by an "M") on the quantitation report showing that a modification to a record has occurred. When a manual modification of a peak occurs, the analyst shall provide documentation for the file to include an electronic hard-copy representation of the before and after correction. The files shall be stamped with a narrative indicating the reason for the integration and include the date and primary analyst's initials. The action must be concurred by the Technical Data Reviewer or Section Supervisor and include an initial and date of the review of the modification on the raw data forms. All documentation of manipulations of a chromatogram must be placed in the final project file as evidence.

4.5.5 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.

4.5.6 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.

4.6 Document Control/File Management

4.6.1 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, analysis, data review and transfer of custody. Additionally, LSB's policy that all documents issued as part of LSASD's Quality System shall be controlled in the following way.

4.6.1.1 All documents are reviewed and approved by an authorized approving official prior to being issued. Approving officials are Section Supervisors, Branch Supervisor or QAC. The LSASD Deputy Director is the approving official for all quality system operating procedures. NOTE: The QAC does not have the authority to release final data reports except for rare occasions as granted by the Branch Supervisor during emergencies.

4.6.1.2 Authorized revisions of controlled documents shall be available to all personnel at the point-of-use.

4.6.1.3 A master list of controlled documents shall be maintained by the Document Control Coordinator which identifies the current revision (or equivalent), control number and its review status.

4.6.1.4 Documents shall be reviewed following the schedule provided in LSASDPROC-1014-Preparation of Standard Operating Procedures for suitability or needed revisions.

4.6.1.5 Obsolete documents shall be removed from the point(s)-of-issue and placed in archive.

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

4.6.1.7 Archived documents shall be marked as such and moved to an Archived folder on the LSASD LAN.

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

4.7 Internal Chain-of-Custody

LSB uses an internal COC procedure for tracking field samples and digestates/extracts as they travel through the lab from the custody room to disposal. LSB analysts check samples in/out of the Custody Room or designated storage location through Element® (see LSB SOP LSBPROC-105G). Custody of extracts and digestates are tracked on Element®-generated bench sheets and custody logs, which are included in the final project file.

4.8 COC Receipt Form

The LSASD Sample Custodian/designee receives a COC record from the field samplers with every shipment of samples (see LSB SOP LSBPROC-105G). The COC is scanned and uploaded to Element for inclusion in the final data package. LSB does maintain the original copy of the COC.

4.9 Instrument/Maintenance/Analysis/Preparation Records

Each analysis area maintains records in electronic format recorded in Element® or if that is not possible a hardcopy logbook will be kept within the laboratory work areas.

4.9.1 All instruments will be accompanied by a hardcopy logbook to document maintenance and troubleshooting. The maintenance log will reside in the laboratory

with the instrument. Maintenance logbooks are specific for one instrument. If the instrument is relocated, the maintenance log will travel with the instrumentation. Maintenance logs must include documentation of all maintenance events including vendor performed maintenance. Maintenance reports provided by vendors will be placed within the maintenance logbooks. All entries within the maintenance logs must be initialed and dated by the analyst completing the maintenance. All troubleshooting activities shall also be documented in the maintenance logbooks.

4.9.2 All hardcopy logbooks maintained for convenience in the laboratory as a duplicate of the information stored within Element® shall be marked “Convenience Copy”. Direct observations shall be preserved in Element® using an electronic logbook form or the Element features such as the bench sheet, sequence, calibration, etc.

4.9.3 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. At the discretion of the analyst, sequential logs may include failed runs or sequences which were abandoned prior to completion but are not required so long as data quality has not been impacted by the failed run. 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.

4.10 Electronic Traceability

4.10.2 Preparation logs or electronic traceability via Element® shall document all information to reconstruct the preparation of samples, reagents, and standards, and shall include, but are not limited to: weights, volumes, lot number of digestion tubes, balance used, weights used, air displacement pipettes used, certification dates of balances and weights used, reagents/standards used, preservation checks, units and any cleanup procedures. As LSB has migrated to the use of Element® for recording all traceable information and/or original observations, the use of hardcopy logbooks except for those marked as “Convenience Copies” will be phased out.

4.11 Other

4.11.2 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 Supervisor to the QAC for approval and posting.

4.12 LSB Laboratory Operations and Quality Assurance Manual

4.12.1 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 M:\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. Printed copies of the LOQAM will contain a watermark indicating the document is uncontrolled when printed. The LSB LOQAM shall be maintained as described below.

4.12.2 The quality manual will be reviewed in totality at least once each year. The Section Supervisors 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.

4.12.3 The annual review and versions of less comprehensive reviews, as described in Section 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.

4.12.4 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 Supervisors, QAC, and the Branch Supervisor. The effective date of the change will be the signature date of the Branch Supervisor.

4.13 Standard Operations Procedures/Methods

4.13.1 SOPs shall be written based on agency guidance EPAQA/G-6 “Guidance for the Preparation of Standard Operating Procedures for Quality Related Documents” and SOP LSASDPROC-1014-Preparation of Standard Operating Procedures. 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 and administrative SOPs in use by the laboratory shall be either available within the laboratory for reference as a hardcopy or accessible via the LAN within the laboratory. The official copy of each SOP resides on the LAN at the following locations:

Divisional SOP: M:\LSASD Quality System Documentation\Divisional Quality System Documentation\Operating Procedures

LSB Branch SOPs: M:\LSB\Current Documents\SOPs\Branch SOPs

LSB Technical SOPs: M:\LSB\Current Documents\SOPs

NOTE: Printed copies of the SOPs contain a watermark indicating the copy is "Uncontrolled When Printed".

4.14 Project Files

4.14.1 A project file contains 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 Project Log software. Each analytical project has a unique project identifier, and each project file contains originals of all the laboratory generated information, notes, and correspondence information.

4.14.2 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 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.

4.14.3 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.

4.14.4 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 Supervisor or designee for final reporting. Once the project has been reported, all data packets that make up that project file are compiled electronically from Element and stored on the LSB Share Point site until transferred to the proper Agency records retention storage location by the LSASD Records Manager. LSB has transitioned to all electronic data file, as such the electronic records serves as the official record for the project. 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."

4.14.5 All data 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.

4.15 Confidentiality of Data

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 leaders of the criminal investigations or to individuals that are authorized by LSB management. Criminal projects are so noted when logged into Project Log. CBI procedures can be employed for any project data when requested by an appropriate project leader.

4.15.1 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 leader or those approved by the project leader to receive results.

4.15.2 General 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 Supervisor and shall have the Section Supervisor as a "THRU" signatory. All correspondence external to the Division shall also include the Branch Supervisor as a "THRU" signatory. Correspondence related to a specific project shall be included in the project file.

4.16 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 DOCs, C-DOP, conference, and seminar participation, etc. Training files may be maintained in hard copy, electronic format, or a combination of both. Refer to LSASDPROC-1003.

4.17 QA/QC Records

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

4.18 Document/Forms Revisions

Many forms and documents (e.g., SOPs, data review check lists,) are generated within LSB and handled as controlled documents. All forms will be controlled by the Document Control Coordinator (DCC), or designee, in the appropriate subdirectory on the LAN at

M:\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 Supervisors by sending an email to the DCC denoting approval and with a copy of the changes. The DCC, 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 LAN is being used. Specific document control procedures are detailed in LSASDPROC-1000.

4.19 Records Management/Disposition

LSB records will be managed in accordance with the LSASD Procedure for Control of Records, LSASDPROC-1001. All records will be maintained as required by U.S. government regulations based on the designated records retention schedule. The LSASD Records Manager is responsible for ensuring records are maintained appropriately.

4.20 Laboratory Apparatus and Instruments

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 (EMS) goals (e.g., reduction in chemical use or energy efficiency) should be considered when evaluating new equipment for purchase but may not always be the deciding factor. All equipment will be clean, free of contaminants, operational, and calibrated prior to use per manufacturer's instruction and procedures detailed in the appropriate LSB technical SOPs. If any equipment becomes defective or is suspected of being defective, it will be labeled as out-of-service and, if possible, will be separated from equipment currently in use. Equipment will be used by authorized personnel following manufacturer's user manuals, which are available for review by hard copy or electronically via the manufacturers' website. In general, all LSB laboratory apparatus and instruments remain under LSB control at all times, with the exceptions of sending equipment to a vendor for repair/calibration. If equipment leaves the direct control of LSB (e.g., loaned to another agency, repair/calibration), it shall be verified, through a certificate from the manufacturer or verification of calibration performed by LSB staff upon receipt of the equipment, to be operating properly prior to being returned to service at LSB. LSASDPROC-1009 details equipment management procedures.

4.20.1 Incubators – When in use, incubator temperature will be monitored by a certified temperature monitor/logger equipped with the appropriate temperature sensor(s). For mobile lab applications that require an incubator, a temperature logger is used as the primary temperature recording device, however, in the event the logger or sensor malfunction, a certified glycol digital or manual thermometer is acceptable. When using these backups, temperature should be manually recorded

twice daily while samples are in the incubator.

4.20.2 Water Baths – Water baths are equipped with a certified thermometer to monitor and record temperature in the preparation and/or analysis log or Element® (i.e., bench sheet) at least once each working day while in use or as specified by the published method or technical SOP. Drain and clean water baths periodically as recommended by manufacturer, via approved methods or accepted practice. Be sure to verify the temperature in multiple locations when water baths are loaded to capacity and document this check in the preparation/analysis log or temperature log, whichever is appropriate.

4.20.3 Refrigerators/Freezers/Drying Ovens-Refrigerators, freezers, and drying ovens are equipped with a certified temperature monitor/logger equipped with the appropriate temperature sensor(s). Sensor types vary by application, however, all sensors are certified annually. The dataloggers allow for data storage locally on the device and via a Wi-Fi connection to the manufacturer's cloud service (www.dicksonone.com), where data can be downloaded. 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.

4.20.3.1 Due to the relatively small volume of refrigerators, freezers, and ovens it is expected that the units will go outside of normal operating temperatures 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 allowable temperature. 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 if the device returns to normal operating temperature. Temperature exceedances will be monitored for trends to indicate whether a device requires service or replacement.

4.20.3.2 Outdated materials in refrigerators and/or freezers are properly disposed once the expiration date is exceeded.

4.20.3.3 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.

4.20.4 Autoclaves: Autoclaves are required to be verified to be operating properly and within the correct temperature and pressure limits. Temperature is documented each time the unit is in use and/or as required in the published methods or technical SOPs. The temperature and pressure (where applicable) are included in the final project data file.

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

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

4.20.5 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).

4.20.5.1 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:

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

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.

4.20.5.3 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.

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

4.20.5.5 Verification: The verification should be documented in the appropriate hardcopy/electronic 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 Laboratory Quality Coordinator.

4.20.5.6 Maintenance – During the annual servicing/calibration visit, each balance is checked for calibration, adjusted as needed, cleaned, and checked for level as required by the manufacturer. The QAC maintains the service records for the work performed. Analytical balances should be used in areas that are subjected to minimal, drafts, vibrations, or influences from static electricity, as appropriate.

4.20.6 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 Laboratory Quality Coordinator 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.

4.20.7 Mechanical Dispensing Devices: 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 in LSASD-PROC-1011.

4.20.8 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.

4.21 Records of NIST Traceability

4.21.1 Records of NIST-traceability for thermometers, weights, and mechanical dispensing devices, as applicable, shall be maintained by the QAC, or designee. 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.

4.21.2 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. All records will be captured and maintained in a digital format in Element by the analyst who creates the Element standard number. COAs for purchased standards are scanned or downloaded from the manufacturers' website and uploaded to Element.

4.21.3 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 for a minimum of 5 years.

4.22 Major Instrumentation

4.22.1 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); Gas Chromatograph/Mass Spectrometer/Mass Spectrometer (GC/MS/MS); Ion Chromatograph (IC), Mercury analyzers, Auto-analyzers; Accelerated Solvent Extractors (ASE); and Gel Permeation Chromatography (GPC).

4.22.2 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.

4.22.3 A list of all major instrumentation, including unique IDs, is maintained electronically by the Laboratory Equipment Manager and is located on the LAN.

4.23 Laboratory Supplies

4.23.1 General

4.23.1.1 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 121G Screening of Supplies.

4.23.1.2 Contract personnel cannot order supplies with EPA funds but are still responsible for monitoring supplies usage. Contractors may fill out an order form and submit it to an EPA staff member or Section Supervisor. 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.

4.23.1.3 A list of vendors that have furnished acceptable supplies and services is maintained by the QAC or designee on the LAN at: M:\LSB\Current Documents\Vendor List. Additional vendors may be added to this list if their supplies and services prove to be acceptable. The approved supplier list is evaluated annually during the Annual Management Review. If any QC issues arise during routine analysis or screening, a detailed evaluation of a vendor may be warranted. 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.

4.23.2 Labware

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

4.23.2.2 Labware shall be cleaned in accordance with individual SOPs and manufacturer's instructions. 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.

4.23.3 Chemicals, Reagents, Solvents, Standards, Gases

4.23.3.1 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, whenever possible.

4.23.3.1.1 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.

4.23.3.1.2 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). Certificates of Analysis are scanned and uploaded to Element®. The hardcopies may also be kept in a binder in the laboratory for five years after the expiration or consumption of the material; however, the scanned version in Element® will serve as the official record.

4.23.3.1.3 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.

4.23.3.2 Reagents, chemicals, solvents, and standard reference materials, excluding high demand items, should be purchased in small quantities to minimize storage past its expiration date.

4.23.3.3 All reagents, chemicals, solvents, and standard reference materials will be entered into Element® for tracking purposes. Materials should be labeled with a

received, opened and/or prepared date, an Element® ID, and discarded when expired, or when evidence of deterioration is detected.

4.23.3.3.1 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, a 1-year expiration date is not observed. An expiration date is assigned in Element® as 35 years from receipt. 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.

4.23.3.3.2 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 as 'Expires Daily' or 'Expires Today'.

4.23.3.3.3 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 they leave 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®.

4.23.3.3.4 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, expiration, and preparer's initials. A unique ID shall be created in Element® for each prepared reagent and standard. The unique Element® 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 deemed not critical to the success or quality of the analysis, do not need to be tracked. For example, acids and solvents used in rinsing glassware prior to use would not require reagent traceability.

4.23.4 Expired Stock Standard

4.23.4.1 It is LSB's policy to allow for recertification of analytical standards upon consultation with the Section Supervisor as described below.

Analysts are required to monitor supplies and initiate purchases prior to expiration dates. Rectification of standards should only be performed in extenuating circumstances, not because of negligence on the part of the analyst.

4.23.4.2 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 shall 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 Supervisor for review and approval. The 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 Supervisor will verify the proper documentation is in place, scan a copy of the COA into Element®, and route the form to the DCC 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 and assigned a 5-year expiration date following the procedure described above.

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

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

1.4.4 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.

- If an expired standard material fails the verification test, the expired standard material must be replaced or, with the Section Supervisor's approval, must be properly qualified with a Q-3 qualifier until a new standard can be purchased.

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 LSASD Divisional Safety Coordinator (DSC) for chemical storage procedures in the HAZMAT building. See the EPA Region 4 Safety and Occupational Health Manual for additional information on proper storage of hazardous materials.

4.24 Procurement of Chemicals and Chemical Inventories

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

4.24.2 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 DSC. 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 DSC will coordinate with the DCC on the maintenance of training files for P-List chemicals.

4.24.3 Ordering of Chemicals- See LSASDPROC-1008 Purchasing Services and Supplies for detailed procedures on procurement of services and supplies.

4.25 Receipt of Chemicals

4.25.1 The CHO will sign off on all chemical purchase requests and will barcode each chemical upon receipt. 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.

4.26 Laboratory Pure Water

4.26.1 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.

4.26.2 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. Depending on the quality of the feed water for the purification units, the cartridges may last longer than the manufacturer's expiration date. Cartridges will be replaced no more than 2 years following installation provided the above criteria are still met.

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

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

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

4.27.1 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 262.15) 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.

4.27.2 Satellite Storage – Acutely Hazardous Wastes (P-Listed Wastes) Acutely hazardous wastes are those listed in 40 CFR 261.31, Subpart D and must be accounted for separately from regular wastes. See the current version of the Region 4 Safety and Occupational Health Manual for procedures that apply to satellite accumulation of acutely hazardous waste in LSB.

4.27.3 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.]

4.27.4 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. 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.

4.27.5 Non-P-Listed Chemicals Follow all Standard Procedures for disposal as specified in the Region 4 Safety and Occupational Health 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.

4.27.6 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 recycles cardboard, aluminum cans, glass, mixed paper, and plastics. This accounts for a large amount of the total waste stream generated by LSB and LSASD.

4.27.6.1 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.

4.27.6.2 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.

4.27.7 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. Eye wash stations shall be unobstructed at all times.

CHAPTER 5 Quality Performance and Data Handling

5.1 Introduction

5.1.1 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.

5.1.2 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 of 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 $\mu\text{S}/\text{cm}$ at 298°K (25°C), resistivity >18 M $\Omega\text{-cm}$ 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 $\mu\text{g}/\text{bottle}$ 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 Designee: A staff member designated by the Section or Branch Supervisor to assume the duties of another position for a limited period of time.

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

5.2.33 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.

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

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

5.2.36 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.

5.2.37 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.

5.2.38 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.

5.2.39 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.

5.2.40 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.

5.2.41 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.

5.2.42 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.

5.2.43 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.

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

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

5.2.46 Matrix: substrate of a test sample.

5.2.47 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.

5.2.48 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.

5.2.49 May: denotes permitted action, but not required action.

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

5.2.51 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.

5.2.52 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.

5.2.53 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.

5.2.54 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.

5.2.55 Must: denotes required action.

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

5.2.57 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.

5.2.58 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).

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

5.2.60 Outlier: observation (or subset of observations) which appears to be

inconsistent with the remainder of that set of data.

5.2.61 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.

5.2.62 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 an Excel or PDF format, prior to the data being subject to the complete review process.

5.2.63 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.

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

5.2.65 Proficiency Test (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.

5.2.66 Project Leader: internal project initiator and coordinator

5.2.67 Project Manager: external project initiator (Remedial Project Manager)

5.2.68 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.

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

5.2.70 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.

5.2.71 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.

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

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

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

5.2.75 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.

5.2.76 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.

5.2.77 Reference Method: method of known and documented accuracy and precision issued by an organization recognized as competent to issue said method.

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

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

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

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

5.2.82 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.

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

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

5.2.85 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.

5.2.86 Should: Denotes a guideline or recommendation whenever noncompliance with the specification is permissible.

5.2.87 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)

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

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

5.2.90 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.

5.2.91 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.

5.2.92 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.

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

5.2.94 Tentatively Identified Compounds (TIC): are another tool used by EPA to characterize hazardous sites. TIC analysis is a useful tool that can aid in clean-up or treatment decisions by identifying compounds that might otherwise be missed at the site. The "Target Compounds List" or parameter is a fixed set of compounds.

5.2.95 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.

5.2.96 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.

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

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

5.2.99 Risk: The laboratory shall consider the risks and opportunities associated with laboratory activities to: give assurance that the management system achieves its intended results; enhance opportunities to achieve the purpose and objectives of the lab; prevent, or reduce, undesired impacts and potential failures in the lab; and, achieve improvements.

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 instrument 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, which demonstrates the analyst's ability to successfully perform the method. Procedures for performing a DOC are detailed in LSBPROC 110 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 110 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 SOP LSBPROC-119-Determining MDLs and MRLs. LSB only reports non-detects at the MDL by special request and approval of the Section Supervisor.

5.4 Instrument Calibration

5.4.1 Initial Calibration Curve (ICAL) – All instrumentation used 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, some PFAS compounds, and Natural Attenuation. In those instances, standards will be NIST traceable.

5.4.2 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 $\pm 10\%$ acceptance criteria.

5.4.3 Initial Calibration Verification (ICV) – ICALs shall be verified with a second-source standard at the frequency 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 using 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.

5.4.4 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 of either a primary or second source will be analyzed prior to analysis of a batch containing environmental samples and at the frequency prescribed in the published method or technical SOP.

5.5 Acceptance Criteria

5.5.1 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 $\pm 10\%$ 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.
- Standard reference materials, such as those purchased from NIST, are supplied with a true value and an uncertainty range. True values for the SRMs will be entered in Element® and used when evaluating QC results. This value is updated when a new SRM is purchased. 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 Supervisor 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.

5.5.2 Quality control sample results are routinely evaluated using quality control limits. In the event that QC limits are exceeded, the QC sample and associated environmental sample are evaluated for potential impacts to data quality, and qualified as appropriate to prevent incorrect results from being reported.

5.5.3 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 Supervisor/Technical Director or QAC.

5.5.4 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 Supervisor/Technical Director or QAC, interim RPD limits of 50 may be set for complex matrices such as soil, tissue, or waste.

5.5.5 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 Laboratory Quality Coordinator 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.

5.5.6 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.

5.5.7 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 and uncertainty. LSB also assesses the LCS results as a mechanism for determining if technical corrective action is required using 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.

5.5.8 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 (i.e. 1 MS 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.

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

Spike Reference		Reference Materials
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$$\%R = \frac{Z-X}{T} (100) \quad \text{OR} \quad \%R = \frac{Y}{T} (100)$$

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.

5.5.9 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®.

5.6 Proficiency Test (PT) Sample

5.6.1 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 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.

5.6.2 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. For Forensically accredited methods, each analyst must participate in proficiency test annually. 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.

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

5.6.4 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.

5.6.5 Precision – Precision is 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). LSB applies RPD limits to matrix or laboratory duplicates, matrix spike duplicates, and LCS duplicates.

5.6.6 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 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.

5.6.7 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.

5.6.8 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.

5.6.9 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.

5.6.10 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.

5.7 Data Handling

5.7.1 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.

5.7.2 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:

5.7.2.1 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.

5.7.2.1.1 Non-detects are reported to the MRL.

5.7.2.1.2 Positive detects between the MDL and the MRL are reported with a ‘J’ and explanatory qualifier.

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

5.7.2.2 All other data

5.7.2.1.4 Non-detects are reported to the MRL.

5.7.2.1.5 As a routine, detects between the MDL and the MRL are reported at the MRL, qualified “U”.

5.7.2.1.6 All requests for results to be reported between the MRL and MDL must be approved by the Section Supervisor or QAC

who will verify that a current MDL study is in place that will meet the needs of the data-user.

5.7.3 Units

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

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

5.7.3.3 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.

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

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

1.4.5 Air samples including VOA samples are reported in ppbv or µg/m³ units. Metals in air samples are reported as ug/filter.

5.7.4 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, except for PT samples and metals in air samples, which are reported to 3 significant figures. QC is reported to 5 significant figures.

5.7.5 Rounding Rules

5.7.5.1 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, 1, 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.

5.7.5.2 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.

5.7.5.3 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.

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

5.7.6.1 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:

5.7.6.1.1 Known and documented laboratory error and

5.7.6.1.2 Use of an appropriate statistical outlier test.

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

5.7.7.1 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.

5.7.8 Studentized deviation from the mean – t-test

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

Calculate the ratio:

$$t_{calc} = \left| \frac{\text{suspect value} - \bar{x}}{s} \right|$$

Apply the following decision rule.

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

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.

Example:

MDL rep	Lead ($\mu\text{g/L}$)
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:

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

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.

Dixon's Q test

Sort the n data values in ascending order:

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

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

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

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

Calculate the value of Q:

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

$$\frac{|x_1 - x_2|}{(x_n - x_1)} \quad \frac{|x_n - x_{n-1}|}{(x_n - x_1)}$$

Apply the following decision rule:

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 should be removed from the data set.

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.

Example:

MDL Rep	Lead ($\mu\text{g/L}$)
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 ($\mu\text{g/L}$)
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:

$$Q_{calc} = \frac{|38.0 - 40.1|}{(41.3 - 38.0)} = \frac{2.1}{3.3} = 0.636$$

The critical value of Q is 0.568 for $\alpha=0.05$ 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.

5.7.9 Uncertainty – Where available, LSB uses well-recognized test methods that 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.

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

5.7.9.2 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:

$$Uncertainty = 100 \left(\frac{c}{\bar{R}} \right) \left(\frac{1 + L}{\bar{R}} \right)$$

Where:

c = measured concentration of the analyte

L = the half width of the control range, that is, (UCL-LCL)/2

\bar{R} = mean historical LCS recovery

5.7.9.3 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. LSB performs verification of the measurement uncertainty limits for the LCS annually. See LSBPROC-123 for details on this procedure.

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

5.8 Data Reporting

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

5.8.2 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.

5.8.3 Report Narrative: Additional explanatory remarks about the data can be added by the Section Supervisor (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 Supervisor (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.

5.8.4 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.

5.8.5 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.

5.8.6 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.

5.8.7 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 Supervisor (or designee). All preliminary data released shall be in the form of a Draft report from Element® or Excel spreadsheet. 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. All preliminary data released by LSB will be sent to the project leader only.

5.8.8 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 R4COC Corrections mailbox to the QAC. The QAC will forward the requests to the LSASD Sample Custodian and the appropriate Section Supervisor. Once corrections are complete, the Section Supervisor (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.

5.9 Data Management and Data Security

5.9.1 Data is managed using both Project Log and Element®. Project Log is used for project scheduling and Element® is used for analytical data management. Project Log is an in-house developed application. All data is stored in an Oracle database residing on an LSASD Windows 2012 Server. Console-level access to the Oracle Server is limited to the LSASD LAN Administrators, and application developers for application development and database administration.

5.9.1.1 Backups of the Oracle database (and other LSASD LAN servers) are performed Monday through Friday evenings. Backups are replicated over the network to the Regional office in Atlanta for disaster recovery. Tape backups are located at LSASD in a fire-proof media safe.

5.9.2 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.

5.9.2.1 End-user access to the database is controlled through the Project Log application, Element® DataSystem and the Adobe Coldfusion® web server (currently used for reports, conversion utilities, etc.).

5.9.2.1.1 All Element® application users are required to login to the system using an Element® application USERID and PASSWORD. A PROJECT LOG 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.

5.9.2.1.2 Access to the EPA network and an account in Project Log or Element® is required for access to data for entry or reporting purposes. Rights are assigned to each Project Log or Element®

user upon request by their supervisor by the QAC, the LSASD LAN Administrator, or the application developer.

5.9.2.2 Users are restricted to certain functions within PROJECT LOG 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 LSASD LAN Administrator and application developer have the overall responsibility for security and functionality of both databases. The LSB PROJECT LOG/Element® coordinator has the responsibility of security, accuracy, and integrity of the data in the database.

5.9.2.3 Project log entry in PROJECT LOG is restricted to the LSASD 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.

5.9.2.3.1 Modifications to the project log entries are restricted to LSASD Sample Custodians and the QAC after the project has been entered.

5.9.2.3.2 Sample logging in Element® is restricted to the LSASD Sample Custodians (or those officially trained as such), the QAC and Section Supervisors.

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

5.9.2.3.4 Reporting of final data is restricted to Section Supervisors and their designees.

5.9.3 After data has been reported, it cannot be modified without the status of the data being set from ‘Reported’ to “Not Analyzed”, then to a lower level by the Section Supervisor or QAC. A searchable audit trail which tracks any change to the data or analyses in the database is maintained within Element®.

5.9.4 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 Supervisors 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.

5.10 Complaints/Inquiries

5.10.1 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.

5.11 Formal Corrective Actions

5.11.1 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.

5.12 Control of Nonconforming Work

5.12.1 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.

5.12.2 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.

5.13 Risks and Opportunities

5.13.1 LSB identifies and mitigates risk through the corrective action process, the opportunities for improvement process, and through employee suggestions. 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 and risk actions are documented identical to formal corrective actions. Preventative actions and risk actions do not require a root cause analysis prior to implementation; however, a corrective action does require a root cause analysis, or equivalent. Opportunities for Improvement are observations which may help improve method or operational performance or to 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. Risk will be identified thru impartiality on an on-going basis. Relationships within the laboratory that threaten impartiality include, but not limited to: governance, management, personnel, shared resources, finances, conflict of interest, and contracts. If risk to impartiality is identified, the laboratory shall be able to demonstrate how it eliminates or minimizes such risk.

5.13.2 LSB shall consider the risks and opportunities associated with the laboratory activities in order to:

- give assurance that the management system achieves its intended results;
- enhance opportunities to achieve the purpose and objectives of the laboratory;
- prevent, or reduce, undesired impacts and potential failures in the laboratory activities; and,
- achieve improvement.

5.13.3 Improvement and Risk Actions can be initiated by staff at any level within the Division. The QAC tracks the status of all improvement actions, corrective actions, and risk evaluations as detailed in SOP LSASDPROC-1005, and reports the results of these actions to management during the annual Management Review meeting

NOTE: Discussion about risk taken from *General requirements for the competence of testing and calibration laboratories*, International Standard ISO/IEC 17025:2017-11 Third edition.

5.14 Annual Management Review

5.14.1 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 risks identified, actions, monitoring, and status in the Annual QA Management Review

meeting. 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/IEC 17025:2017 (E), clause 8.9.

5.15 Quality System Audits

5.15.1 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.

5.15.2 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 for Drinking Water and Forensics
- Year 2 – Remote Surveillance Audit of a subset of the standard for Forensics and Drinking Water as determined by the accrediting body
- Year 3 – On site audit of 100% of the scope of accreditation for Drinking Water and Forensics
- Year 4 – Remote Surveillance Audit of a subset of the standard for Forensics and Drinking Water as determined by the accrediting body

NOTE: The current accreditation body performs a full on-site assessment for drinking water certification every two years for those methods where LSB maintains certification. This is a different requirement than specified in the Certification Manual.

5.15.3 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.

5.15.4 External Audits or Project Specific Audits conducted by Independent Assessors: 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 ($\alpha = 0.05$, 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

¹Pearson, 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 ($\alpha = 0.05$, 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

¹Rorabacher, 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

6 CHAPTER 6 Methodology

6.1 General

6.1.1 The analytical methods used by LSB are guided by DQOs of specific projects and 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 Supervisor, 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

6.2.1 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 (MRL)

6.3.1 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, semi-volatiles, 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 Supervisor(s). The MRLs listed in the tables are those which are routinely achievable. However, sample-specific MRLs may be higher or lower.

6.3.2 Some factors which may influence MRLs are listed below.

- The amount of sample used, either volume or weight, will raise or lower specific MRLs.
- Dilutions due to high amounts of target analytes or matrix interferences will raise sample-specific MRLs.
- Soil and sediment samples are corrected for percent moisture content and reported on a dry-weight basis which may result in a higher MRL.

- 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 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, a minimum of 30 days will need to allow sufficient lead-time. 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 Supervisors 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 Supervisor and/or the QAC when analyzing samples for LDR purposes.

PARAMETER	DRINKING WATER 40 CFR 141.13 and 141.62 (MCLs) ¹	RCRA TCLP (40CFR 261.24 Table 1) and pH (40CFR 261.22)	RCRA LAND BAN LIMITS 40CFR 268.48 Table UTS ²		ALTERNATIVE RCRA LAND BAN LIMITS FOR SOIL 40 CFR 268.49 ³
			Wastewater (<1% TSS & <1% TOC by weight CFR268.2)	Non-wastewater (mg/kg unless noted as "mg/L TCLP")	
Antimony	6 µg/L	-	1.9 mg/L	1.15 mg/L TCLP	11.5 mg/L TCLP
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	-	-	-	-
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)	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	-	-	-	-

Table 6-1 Levels of Concern for Various Programs (Inorganics, Cont'd)

PARAMETER	DRINKING WATER 40 CFR 141.13 and 141.62 (MCLs) ¹	RCRA TCLP (40CFR 261.24 Table 1) and pH (40CFR 261.22)	RCRA LAND BAN LIMITS 40CFR 268.48 Table UTS ²	ALTERNATIVE RCRA LAND BAN LIMITS FOR SOIL 40 CFR 268.49 ³
Nitrate + Nitrite	-	-	-	-
pH	-	< 2 or > 12.5	-	-
Selenium	50 µg/L	1.0 mg/L	0.82 mg/L	5.7 mg/L TCLP
Silver	-	5.0 mg/L	0.43 mg/L	0.14 mg/L TCLP
Sulfide	-	-	14 mg/L	NA
Thallium	2 µg/L	-	1.4 mg/L	0.20 mg/L TCLP
Turbidity	1 NTU	-	-	-
Vanadium	-	-	4.3 mg/L	1.6 mg/L TCLP
Zinc	-	-	2.61 mg/L	4.3 mg/L TCLP

Table 6-1 Levels of Concern for Various Programs (Organics)

PARAMETER	DRINKING WATER 40 CFR 141.13 and 141.62 (MCLs) ¹	RCRA TCLP (40CFR 261.24 Table 1) and pH (40CFR 261.22)	RCRA LAND BAN LIMITS 40CFR 268.48 Table UTS ²		ALTERNATIVE RCRA LAND BAN LIMITS FOR SOIL 40 CFR 268.49 ³
			Wastewater (<1% TSS & <1% TOC by weight CFR268.2)	Non-wastewater (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

PARAMETER	DRINKING WATER 40 CFR 141.13 and 141.62 (MCLs) ¹	RCRA TCLP (40CFR 261.24 Table 1) and pH (40CFR 261.22)	RCRA LAND BAN LIMITS 40CFR 268.48 Table UTS ²		ALTERNATIVE RCRA LAND BAN LIMITS FOR SOIL 40 CFR 268.49 ³
			Wastewater (<1% TSS & <1% TOC by weight CFR268.2)	Non-wastewater (mg/kg unless noted as "mg/L TCLP")	
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

Table 6-1 Levels of Concern for Various Programs (Organics, Cont'd)

PARAMETER	DRINKING WATER 40 CFR 141.13 and 141.62 (MCLs) ¹	RCRA TCLP (40CFR 261.24 Table 1) and pH (40CFR 261.22)	RCRA LAND BAN LIMITS 40CFR 268.48 Table UTS ²		ALTERNATIVE RCRA LAND BAN LIMITS FOR SOIL 40 CFR 268.49 ³
			Wastewater (<1% TSS & <1% TOC by weight CFR268.2)	Non- wastewater (mg/kg unless noted as "mg/L TCLP")	
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 4)
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

Table 6-1 Levels of Concern for Various Programs (Organics, Cont'd)

PARAMETER	DRINKING WATER 40 CFR 141.13 and 141.62 (MCLs) ¹	RCRA TCLP (40CFR 261.24 Table 1) and pH (40CFR 261.22)	RCRA LAND BAN LIMITS 40CFR 268.48 Table UTS ²		ALTERNATIVE RCRA LAND BAN LIMITS FOR SOIL 40 CFR 268.49 ³
			Wastewater (<1% TSS & <1% TOC by weight CFR268.2)	Non- wastewater (mg/kg unless noted as "mg/L TCLP")	
2-Chloronaphthalene	-	-	0.055	5.6	56
2-Chlorophenol	-	-	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 4)
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

Table 6-1 Levels of Concern for Various Programs (Organics, Cont'd)

PARAMETER	DRINKING WATER 40 CFR 141.13 and 141.62 (MCLs) ¹	RCRA TCLP (40CFR 261.24 Table 1) and pH (40CFR 261.22)	RCRA LAND BAN LIMITS 40CFR 268.48 Table UTS ²		ALTERNATIVE RCRA LAND BAN LIMITS FOR SOIL 40 CFR 268.49 ³
			Wastewater (<1% TSS & <1% TOC by weight CFR268.2)	Non- wastewater (mg/kg unless noted as "mg/L TCLP")	
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	-	-

Table 6-1 Levels of Concern for Various Programs (Organics, Cont'd)

PARAMETER	DRINKING WATER 40 CFR 141.13 and 141.62 (MCLs) ¹	RCRA TCLP (40CFR 261.24 Table 1) and pH (40CFR 261.22)	RCRA LAND BAN LIMITS 40CFR 268.48 Table UTS ²		ALTERNATIVE RCRA LAND BAN LIMITS FOR SOIL 40 CFR 268.49 ³
			Wastewater (<1% TSS & <1% TOC by weight CFR268.2)	Non-wastewater (mg/kg unless noted as "mg/L TCLP")	
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

Table 6-1 Levels of Concern for Various Programs (Organics, Cont'd)

PARAMETER	DRINKING WATER 40 CFR 141.13 and 141.62 (MCLs) ¹	RCRA TCLP (40CFR 261.24 Table 1) and pH (40CFR 261.22)	RCRA LAND BAN LIMITS 40CFR 268.48 Table UTS ²		ALTERNATIVE RCRA LAND BAN LIMITS FOR SOIL 40 CFR 268.49 ³
			Wastewater (<1% TSS & <1% TOC by weight CFR268.2)	Non-wastewater (mg/kg unless noted as "mg/L TCLP")	
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

Table 6-1 Levels of Concern for Various Programs (Organics, Cont'd)

PARAMETER	DRINKING WATER 40 CFR 141.13 and 141.62 (MCLs) ¹	RCRA TCLP (40CFR 261.24 Table 1) and pH (40CFR 261.22)	RCRA LAND BAN LIMITS 40CFR 268.48 Table UTS ²		ALTERNATIVE RCRA LAND BAN LIMITS FOR SOIL 40 CFR 268.49 ³
			Wastewater (<1% TSS & <1% TOC by weight CFR268.2)	Non-wastewater (mg/kg unless noted as "mg/L TCLP")	
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 4)
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

PARAMETER	DRINKING WATER 40 CFR 141.13 and 141.62 (MCLs) ¹	RCRA TCLP (40CFR 261.24 Table 1) and pH (40CFR 261.22)	RCRA LAND BAN LIMITS 40CFR 268.48 Table UTS ²		ALTERNATIVE RCRA LAND BAN LIMITS FOR SOIL 40 CFR 268.49 ³
			Wastewater (<1% TSS & <1% TOC by weight CFR268.2)	Non-wastewater (mg/kg unless noted as "mg/L TCLP")	
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-Nitrosomethylalkylamine	-	-	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

Table 6-1 Levels of Concern for Various Programs (Organics, Cont'd)

PARAMETER	DRINKING WATER 40 CFR 141.13 and 141.62 (MCLs) ¹	RCRA TCLP (40CFR 261.24 Table 1) and pH (40CFR 261.22)	RCRA LAND BAN LIMITS 40CFR 268.48 Table UTS ²		ALTERNATIVE RCRA LAND BAN LIMITS FOR SOIL 40 CFR 268.49 ³
			Wastewater (<1% TSS & <1% TOC by weight CFR268.2)	Non-wastewater (mg/kg unless noted as "mg/L TCLP")	
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

Table 6-1 Levels of Concern for Various Programs (Organics, Cont'd)

PARAMETER	DRINKING WATER 40 CFR 141.13 and 141.62 (MCLs) ¹	RCRA TCLP (40CFR 261.24 Table 1) and pH (40CFR 261.22)	RCRA LAND BAN LIMITS 40CFR 268.48 Table UTS ²		ALTERNATIVE RCRA LAND BAN LIMITS FOR SOIL 40 CFR 268.49 ³
			Wastewater (<1% TSS & <1% TOC by weight CFR268.2)	Non-wastewater (mg/kg unless noted as "mg/L TCLP")	
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	-	-	-	-

Table 6-1 Levels of Concern for Various Programs (Organics, Cont'd)

PARAMETER	DRINKING WATER 40 CFR 141.13 and 141.62 (MCLs) ¹	RCRA TCLP (40CFR 261.24 Table 1) and pH (40CFR 261.22)	RCRA LAND BAN LIMITS 40CFR 268.48 Table UTS ² Wastewater (<1% TSS & <1% TOC by weight CFR268.2)	Non-wastewater (mg/kg unless noted as "mg/L TCLP")	ALTERNATIVE RCRA LAND BAN LIMITS FOR SOIL 40 CFR 268.49 ³
Haloacetic acids (HAA5)	0.06	-	-	-	-

Notes: For specifics on Water Quality Standards, See <https://www.epa.gov/wqs-tech>

1 National Primary Drinking Water Regulations, <https://www.epa.gov/ground-water-and-drinking-water/national-primary-drinking-water-regulations>

2 Land Disposal Restriction for Hazardous Waste

3 Demonstrating Compliance with LDR Alternative Soil Treatment Standards, <https://www.epa.gov/hw/guidance-demonstrating-compliance-land-disposal-restrictions-ldr-alternative-soil-treatment>

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

4. 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.

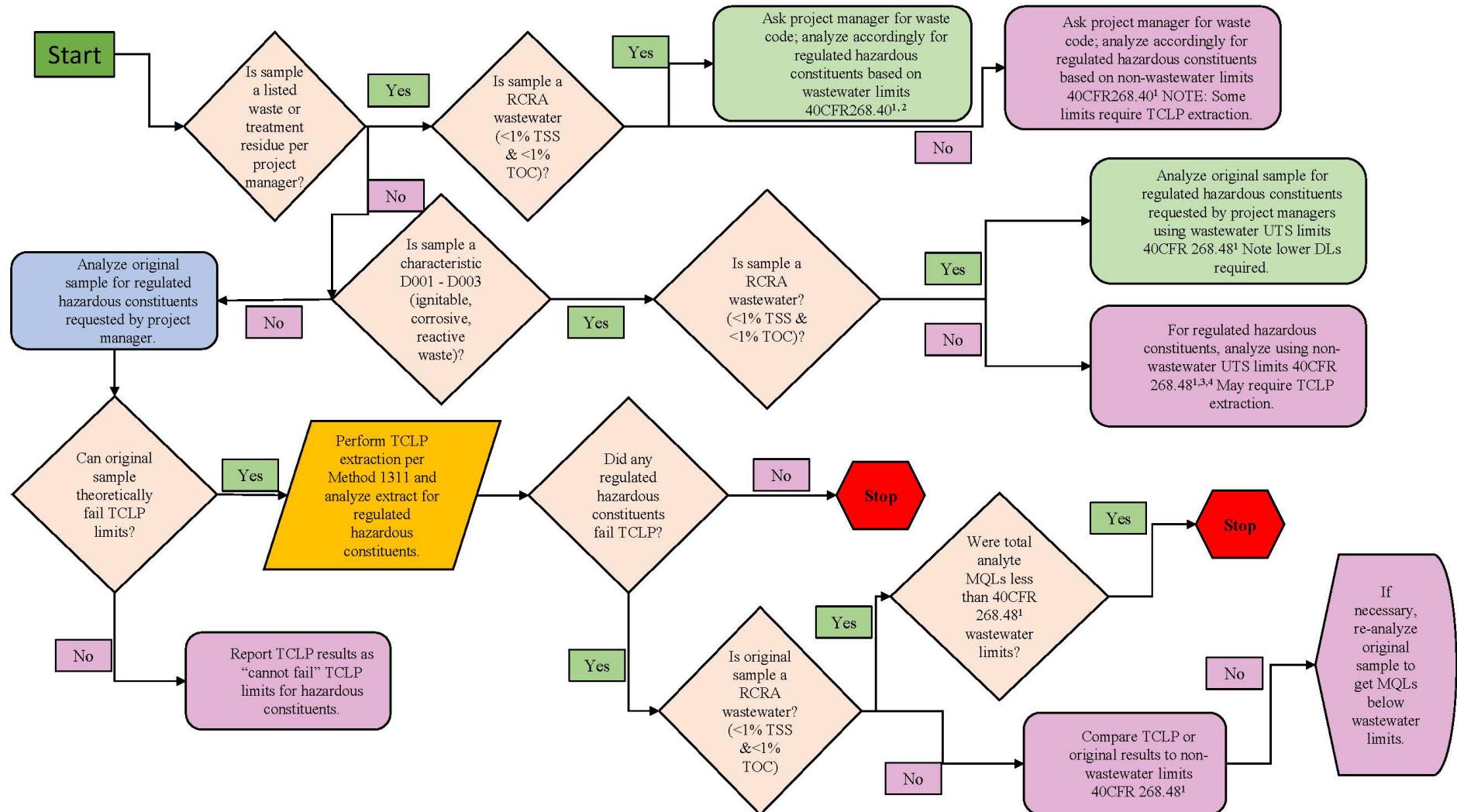
**Figure 6-1 Decision Tree for Analysis of Land Disposal Restrictions**¹ See LSASD LAN Directory M:\LSB\Current Documents\Miscellaneous Documents for LDR tables contained in 40CFR268.40 and .48.² At 40CFR 268.48 the D009 Wastewater concentration limit requires TCLP extraction for mercury.³ A TCLP extraction 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 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	0.01	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	200.7 or 200.8	0.01	200.7 or 200.8	0.01
Chloride (secondary)	250	300	-	300	-
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	245.1	0.0004	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	0.01	NA ²	NA ²	NA ²	NA ²

Table 6-2 Capability for Potable Waters-Inorganics (Cont'd)					
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)
Chlorite	1	NA ²	NA ²	NA ²	NA ²
Residual Disinfectant	detectable	NA ²	NA ²	NA ²	NA ²
Fluoride (secondary)	2	300	0.05	300	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 ⁴	6.5-8.54	NA ¹	NA ¹	9040C	1.04

Notes:

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 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	8270E (Mod) SIM3	0.0001
Carbofuran	0.04	NA ²	NA ²	NA ²	NA ²

Table 6-3 Capability for Potable Waters – Organics (Cont'd)					
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)
bis(2-ethylhexyl)adipate	0.4	525.2	0.001	NA	NA
bis(2-ethylhexyl)phthalate	0.006	525.2	0.001	8270E (Mod)	0.006
Dibromochloropropane (DBCP)	0.0002	NA ¹	NA ¹	8011/8260C ³	0.00005
Endrin	0.002	525.2	0.00004	8270E (Mod)	0.00005
Ethylene dibromide (EDB)	0.00005	NA ¹	NA ¹	8260C ³	0.00005
Heptachlor	0.0004	525.2	0.00004	8270E (Mod)	0.00005
Heptachlor Epoxide	0.0002	525.2	0.00004	8270E (Mod)	0.00005
Hexachlorobenzene	0.001	525.2	0.001	8270E (Mod)	0.001
Hexachlorocyclopentadiene	0.05	NA ¹	NA ¹	8270E (Mod)	0.05
Lindane (gamma-BHC)	0.0002	525.2	0.00004	8270E (Mod)	0.00005
Methoxychlor	0.04	525.2	0.00004	8270E (Mod)	0.0002
PCBs (as Decachlorobiphenyl)	0.0005	NA ²	NA ²	8082-Aroclors	0.0005
Pentachlorophenol	0.001	NA ¹	NA ¹	8270E (Mod)	0.0001
Picloram	0.5	NA ¹	NA ¹	8321B	0.0000125
Simazine	0.004	NA ²	NA ²	NA ²	NA ²
Technical Toxaphene	0.003	NA ¹	NA ¹	8081B	0.002

Notes:

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 Metals Analyte List Minimum Reporting Limits by Matrix					
ANALYTE	LSB Routine Analytical Method⁴	Water µg/L (ppb)³	Soil/Sed mg/kg^{1,3}	Waste mg/kg	Tissue mg/kg^{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 6010D	100	10	10	2
Barium	EPA 6010D	5	0.5	0.5	0.1
Beryllium	EPA 6010D	3	0.3	0.3	0.06
Cadmium	EPA 200.8	0.25	0.025	0.025	0.005
Calcium	EPA 6010D	250	25	25	5
Cobalt	EPA 6010D	5	0.5	0.5	0.1
Chromium	EPA 6010D	5	0.5	0.5	0.1
Chrom., Hexavalent	SW-846 3060A / EPA 218.6	5.0 ug/L	5	5.0*	NA
Chrom., Hexavalent, Dissolved	EPA 218.6	5.0 , 0.025*	NA	NA	NA
Copper	EPA 6010D	10	1	1	0.2
Iron	EPA 6010D	100	10	10	2
Lead	EPA 200.8	0.5	0.05	0.05	0.01
Magnesium	EPA 6010D	250	25	25	5
Manganese	EPA 6010D	5	0.5	0.5	1
Mercury	245.1/7473 ⁵	0.1	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 6010D	10	1	1	0.2
Nickel	EPA 6010D	10	1	1	0.2
Potassium	EPA 6010D	1000	100	100	20
Selenium	EPA 200.8	1	0.1	0.1	0.02

Table 6-4 Metals Analyte List Minimum Reporting Limits by Matrix (Cont'd)					
ANALYTE	LSB Routine Analytical Method ⁴	Water µg/L (ppb) ³	Soil/Sed mg/kg ^{1,3}	Waste mg/kg	Tissue mg/kg ^{2,3}
Sodium	EPA 6010D	1000	100	100	20
Strontium	EPA 6010D	5	0.5	0.5	0.1
Silver	EPA 6010D	5	0.5	0.5	0.1
Tin	EPA 6010D	15	1.5	1.5	NA*
Titanium	EPA 6010D	5	0.5	0.5	0.1
Thallium	EPA 200.8	0.5	0.05	0.05	0.01
Vanadium	EPA 6010D	5	0.5	0.5	0.1
Yttrium	EPA 6010D	3	0.3	0.3	0.06
Zinc	EPA 6010D	10	1	1	0.2
Boron **	EPA 6010D	50	5	5	1
Uranium **	EPA 200.8	0.5	NA	NA	NA

Notes:

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; 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 Nutrients and Classicals Analyte List Minimum Reporting Limits by Matrices					
ANALYTE	Analytical Method	Water mg/L	Soil/Sed mg/kg	Waste mg/kg	Tissue mg/kg
Acidity	SM 2310	10	NA	NA	NA
Alkalinity	SM 2320B	1	NA	NA	NA
Ammonia, distilled	EPA 350.1	0.05	2.5	2.5	NA
Ammonia, undistilled	EPA 350.1	0.05	2.5	NA	NA
BOD/C-BOD	SM 5210B	2	NA	NA	NA
Bromide	EPA 300.0	0.1	1	NA	NA
Chloride	EPA 300.0	0.1	1	NA	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	NA	NA
Nitrite	EPA 300.0/EPA 353.2	0.05	0.5	NA	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	1.25	NA
Phosphorus, Ortho	EPA 365.1	0.01	NA	NA	NA
Total Dissolved Solids	USGS I-1750-85	50	NA	NA	NA
Total Solids	SM 2540B-1997	55	NA	NA	NA
Total Suspended Solids	USGS I-3765-85	5	NA	NA	NA
Total Volatile Solids	SM 2540 E	40	NA	NA	NA
Sulfate	EPA 300.0	0.1	1	NA	NA
Total Kjeldahl Nitrogen (TKN)	EPA 351.2	0.05	6.2	6.2	NA
Total Organic Carbon (TOC)	SM5310/LSB 107C	1	12,000	NA	NA

Notes:

MRLs may change due to variability of interferences that make dilutions necessary, initial sample weight/volume, and/or percent moisture for soil/sed.

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

ANALYTE	Water ¹ µg/L	Soil/Sed ² µg/kg	Waste ³ mg/kg
	Routine Level	Routine Level (Encore®/Tared Vial)	Routine Level
(m- and/or p-) Xylene	1	2	0.005
1,1,1,2-Tetrachloroethane	0.5	1	0.0025
1,1,1-Trichloroethane	0.5	1	0.0025
1,1,2,2-Tetrachloroethane	0.5	1	0.0025
1,1,2-Trichloroethane	0.5	1	0.0025
1,1-Dichloroethane	0.5	1	0.0025
1,1-Dichloroethene	0.5	1	0.0025
1,1-Dichloropropene	0.5	1	0.0025
1,2,3-Trichlorobenzene	0.5	1	0.0025
1,2,3-Trichloropropane	0.5	1	0.0025
1,2,3-Trimethylbenzene	0.5*	NA	0.0025
1,2,4-Trichlorobenzene	0.5	1	0.0025
1,2,4-Trimethylbenzene	0.5	1	0.0025
1,2-Dibromo-3-chloropropane (DBCP)	1	2	0.0025
1,2-Dibromo-3-chloropropane (DBCP)	0.1 (SIM) ⁴	NA	NA
1,2-Dibromoethane (EDB)	0.5	1	0.0025
1,2-Dibromoethane (EDB)	0.05 (SIM) ⁴	NA	NA
1,2-Dichlorobenzene	0.5	1	0.0025
1,2-Dichloroethane	0.5	1	0.0025
1,2-Dichloropropane	0.5	1	0.0025
1,3,5-Trimethylbenzene	0.5	1	0.0025
1,3-Dichlorobenzene	0.5	1	0.0025
1,3-Dichloropropane	0.5	1	0.0025
1,4-Dichlorobenzene	0.5	1	0.0025
2,2-Dichloropropane	0.5	1	0.0025
2,3-Benzofuran	0.5*	NA	0.0025*
Acetone	4	10	0.005

Table 6-6 Volatile Organics Target Analyte List MRLs by Matrix (Cont'd)

ANALYTE	Water ¹ µg/L	Soil/Sed ² µg/kg	Waste ³ mg/kg
	Routine Level	Routine Level (Encore®/Tared Vial)	Routine Level
Acrolein	10*	NA	0.05*
Acrylonitrile	10*	NA	0.05*
Benzene	0.5	1	0.0025
Bromobenzene	0.5	1	0.0025
Bromochloromethane	0.5	1	0.0025
Bromodichloromethane	0.5	1	0.0025
Bromoform	1	2	0.005
Bromomethane	2	2	0.01
Carbon Disulfide	2	2	0.01
Carbon Tetrachloride	0.5	1	0.0025
Chlorobenzene	0.5	1	0.0025
Chloroethane	2	2	0.01
Chloroform	0.5	1	0.0025
Chloromethane	0.5	1	0.0025
cis-1,2-Dichloroethene	0.5	1	0.0025
cis-1,3-Dichloropropene	0.5	1	0.0025
Cyclohexane	0.5	1	0.0025
Dibromochloromethane	0.5	1	0.0025
Dibromomethane	0.5	1	0.0025
Dichlorodifluoromethane (R12)	0.5	1	0.0025
Ethyl benzene	0.5	1	0.0025
Ethylene Oxide ⁵	10*	NA	NA
Hexachlorobutadiene	0.5	1	0.0025
Isopropylbenzene	0.5	1	0.0025
Methyl acetate	1	2	0.005
Methyl butyl ketone	1	5	0.005
Methyl cyclohexane	0.5	1	0.0025

ANALYTE	Water ¹ µg/L	Soil/Sed ² µg/kg	Waste ³ mg/kg
	Routine Level	Routine Level (Encore®/Tared Vial)	Routine Level
Methyl ethyl ketone	4	5	0.02
Methyl isobutyl ketone	1	5	0.005
Methylene chloride (Dichloromethane)	0.5	10	0.0025
Methyl-t-butyl ether	0.5	1	0.0025
n-Butylbenzene	0.5	1	0.0025
n-Propylbenzene	0.5	1	0.0025
o-Chlorotoluene	0.5	1	0.0025
o-Xylene	0.5	1	0.0025
p-Chlorotoluene	0.5	1	0.0025
p-Isopropyltoluene	0.5	1	0.0025
sec-Butylbenzene	0.5	1	0.0025
Styrene	0.5	1	0.0025
tert-Butylbenzene	0.5	1	0.0025
Tetrachloroethene	0.5	1	0.0025
Toluene	0.5	1	0.0025
trans-1,2-Dichloroethene	0.5	1	0.0025
trans-1,3-Dichloropropene	0.5	1	0.0025
Trichloroethene	0.5	1	0.0025
Trichlorofluoromethane (R11)	0.5	1	0.0025
Trichlorotrifluoroethane (R113)	0.5	1	0.0025
Vinyl chloride	0.5	1	0.0025

Notes:

MRLs may change due to variability of interferences that make dilutions necessary, initial sample weight/volume, and/or percent moisture for soil/sed.

Reference Method 8260C.

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 SIM MRLs available for waters upon special request.

5 Ethylene Oxide requires a separate analysis and bottle requirements (Table 3-1).

*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.

Table 6-6 Volatile Organics Target Analyte List MRLs by Matrix

ANALYTE	Air ^{1,2}	Air ^{1,2}
	(ppbv)	(ug/m ³)
(m- and/or p-) Xylene	0.04	0.18
1,1,1-Trichloroethane	0.02	0.11
1,1,2,2-Tetrachloroethane ⁷	0.02	0.15
1,1,2-Trichloro-1,2,2-Trifluoroethane (Freon 113)	0.02	0.16
1,1,2-Trichloroethane	0.21	1.2
1,1-Dichloroethane ⁷	0.02	0.084
1,1-Dichloroethene ⁷	0.02	0.086
1,2,4-Trichlorobenzene	0.21	1.6
1,2,4-Trimethylbenzene	0.02	0.1
1,2-Dibromoethane (EDB) ⁷	0.02	0.17
1,2-Dichlorobenzene	0.02	0.12
1,2-Dichloroethane ⁷	0.02	0.084
1,2-Dichloropropane	0.02	0.1
1,3,5-Trimethylbenzene	0.02	0.1
1,3-Butadiene	0.2	0.49
1,3-Dichlorobenzene	0.02	0.13
1,4-Dichlorobenzene	0.02	0.13
1,4-Dioxane	0.2	0.77
4-Ethyltoluene (1-Ethyl-4-methyl benzene)	0.02	0.1
Acetone	1	2.5
Acrolein	0.2	0.5
Benzene	0.04	0.14
Benzyl Chloride	0.2	1.1
Bromodichloromethane	0.02	0.14
Bromoform	0.2	2.2
Bromomethane	0.2	0.81
Carbon Disulfide	0.2	0.62
Carbon Tetrachloride	0.02	0.14

ANALYTE	Air ^{1,2}	Air ^{1,2}
	(ppbv)	($\mu\text{g}/\text{m}^3$)
Chlorobenzene	0.02	0.099
Chloroethane	0.2	0.53
Chloroform	0.02	0.1
Chloromethane	0.2	0.43
cis-1,2-Dichloroethylene	0.02	0.086
cis-1,3-Dichloropropene	0.02	0.094
Cyclohexane	0.02	0.074
Dibromochloromethane	0.2	1.8
Dichlorodifluoromethane (R12)	0.2	0.99
1,2-Dichlorotetrafluoroethane (R114)	0.02	0.14
Ethanol	1	2
Ethyl acetate	0.2	0.78
Ethyl benzene	0.02	0.95
Ethylene Oxide ³	0.080 (SIM)	0.14 (SIM)
Heptane	0.2	0.18
Hexachlorobutadiene	0.02	0.23
Hexane	0.04	0.77
Isopropanol	0.2	0.54
Methyl butyl ketone	0.2	0.88
Methyl ethyl ketone	0.2	0.62
Methyl isobutyl ketone	0.2	0.85
Methyl Methacrylate	0.02	0.09
Methylene chloride (Dichloromethane)	0.2	0.75
Methyl-t-butyl ether	0.02	0.078
Naphthalene	0.2	1.1
o-Xylene	0.02	0.093
Styrene	0.02	0.09
Tetrachloroethylene	0.02	0.14
Tetrahydrofuran	0.2	0.62

Table 6-6 Volatile Organics Target Analyte List MRLs by Matrix (Cont'd)		
ANALYTE	Air ^{1,2}	Air ^{1,2}
	(ppbv)	($\mu\text{g}/\text{m}^3$)
Toluene	0.2	0.83
trans-1,2-Dichloroethene ⁷	0.02	0.085
trans-1,3-Dichloropropene	0.2	0.93
Trichloroethene	0.02	0.11
Trichlorofluoromethane (R11)	0.02	0.12
Vinyl acetate	0.2	0.77
Vinyl chloride	0.02	0.053

Notes:

Reference Method TO-15

SIM - Selective Ion Monitoring

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

1 Air – 250 cc from 6-L passivated canister – nominal values.

MRLs in $\mu\text{g}/\text{m}^3$ units depend on molecular weight and vary depending on the analyte and the standard lot.

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

3 Ethylene Oxide requires a separate analysis and bottle requirements (Table 3-1).

Table 6-7 Routine Level Semivolatile Organics Target Analyte List MRLs by Matrix

ANALYTE	Water ¹ µg/L	Soil/Sed ² µg/kg	Waste ³ mg/kg	Tissue ⁴ mg/kg
(3- and/or 4-) Methylphenol	10	330	100	0.33
1,1'-Biphenyl	2	66	20	0.066
1,2,4-Trichlorobenzene	10	330	100	0.33
1,4-Dioxane	2	66	NA	NA
1-Methylnaphthalene	2	66	20	0.066
2,3,4,6-Tetrachlorophenol	10	330	100	0.33
2,4,5-Trichlorophenol	10	330	100	0.33
2,4,6-Trichlorophenol	10	330	100	0.33
2,4-Dichlorophenol	10	330	100	0.33
2,4-Dimethylphenol	10	330	100	0.33
2,4-Dinitrophenol	20	660	200	0.66
2,4-Dinitrotoluene	10	330	100	0.33
2,6-Dinitrotoluene	10	330	100	0.33
2-Chloronaphthalene	10	330	100	0.33
2-Chlorophenol	10	330	100	0.33
2-Methyl-4,6-dinitrophenol	10	330	100	0.33
2-Methylnaphthalene	2	66	20	0.066
2-Methylphenol	10	330	100	0.33
2-Nitroaniline	10	330	100	0.33
2-Nitrophenol	10	330	100	0.33
3,3'-Dichlorobenzidine	10	330	100	0.33
3-Nitroaniline	10	330	100	0.33
4-Bromophenyl phenyl ether	10	330	100	0.33
4-Chloro-3-methylphenol	10	330	100	0.33
4-Chloroaniline	10	330	100	0.33
4-Chlorophenyl phenyl ether	10	330	100	0.33
4-Nitroaniline	10	330	100	0.33
4-Nitrophenol	10	330	100	0.33
Acenaphthene	2	66	20	0.066
Acenaphthylene	2	66	20	0.066

Table 6-7 Routine Level Semivolatile Organics Target Analyte List MRLs by Matrix (Cont'd)

ANALYTE	Water ¹ µg/L	Soil/Sed ² µg/kg	Waste ³ mg/kg	Tissue ⁴ mg/kg
Acetophenone	10	330	100	0.33
Anthracene	2	66	20	0.066
Atrazine	10	330	100	0.33
Benzaldehyde	10	330	100	0.33
Benzo[a]anthracene	2	66	20	0.066
Benzo[a]pyrene	2	66	20	0.066
Benzo[b]fluoranthene	2	66	20	0.066
Benzo[g,h,i]perylene	2	66	20	0.066
Benzo[k]fluoranthene	2	66	20	0.066
Benzyl butyl phthalate	10	330	100	0.33
Bis(2-chloroethyl) ether	10	330	100	0.33
Bis(2-ethylhexyl) phthalate	10	330	100	0.33
Bis(chloroethoxy)methane	10	330	100	0.33
Bis(chloroisopropyl) ether	10	330	100	0.33
Caprolactam	10	330	100	0.33
Carbazole	2	66	20	0.066
Chrysene	2	66	20	0.066
Dibenz(a,h)anthracene	2	66	20	0.066
Dibenzofuran	2	66	20	0.066
Diethyl phthalate	10	330	100	0.33
Dimethyl phthalate	10	330	100	0.33
Di-n-butyl phthalate	10	330	100	0.33
Di-n-octyl phthalate	10	330	100	0.33
Fluoranthene	2	66	20	0.066
Fluorene	2	66	20	0.066
Hexachlorobenzene (HCB)	10	330	100	0.33
Hexachlorobutadiene	10	330	100	0.33
Hexachlorocyclopentadiene (HCCP)	10	330	100	0.33
Hexachloroethane	10	330	100	0.33
Indeno[1,2,3-cd]pyrene	2	66	20	0.066

Table 6-7 Routine Level Semivolatile Organics Target Analyte List MRLs by Matrix (Cont'd)

ANALYTE	Water ¹ µg/L	Soil/Sed ² µg/kg	Waste ³ mg/kg	Tissue ⁴ mg/kg
Isophorone	10	330	100	0.33
Naphthalene	2	66	20	0.066
Nitrobenzene	10	330	100	0.33
Nitroso-di-n-propylamine	10	330	100	0.33
N-Nitrosodiphenylamine	10	330	100	0.33
Pentachlorophenol	10	330	100	0.33
Phenanthrene	2	66	20	0.066
Phenol	10	330	100	0.33
Pyrene	2	66	20	0.066

Notes:

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

Method Reference SW-846 Method 8270D

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.

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

Table 6-8 Routine Pesticide/PCB Target Analyte List - MRLs by Matrix					
ANALYTE	Analytical Method	Water¹ µg/L	Soil/Sed² µg/kg	Waste³ mg/kg	Tissue⁴ mg/kg
Aldrin	EPA 8270E	0.04	1.3	0.33	0.02
Heptachlor	EPA 8270E	0.04	1.3	0.33	0.02
Heptachlor epoxide	EPA 8270E	0.04	1.3	0.33	0.02
α-BHC	EPA 8270E	0.04	1.3	0.33	0.02
β-BHC	EPA 8270E	0.04	1.3	0.33	0.02
γ-BHC	EPA 8270E	0.04	1.3	0.33	0.02
δ-BHC	EPA 8270E	0.04	1.3	0.33	0.02
Endosulfan I	EPA 8270E	0.04	1.3	0.33	0.02
Dieldrin	EPA 8270E	0.04	1.3	0.33	0.02
p,p'-DDT	EPA 8270E	0.04	1.3	0.33	0.02
p,p'-DDE	EPA 8270E	0.04	1.3	0.33	0.02
p,p'-DDD	EPA 8270E	0.04	1.3	0.33	0.02
Endrin	EPA 8270E	0.04	1.3	0.33	0.02
Endosulfan II	EPA 8270E	0.04	1.3	0.33	0.02
Endosulfan sulfate	EPA 8270E	0.04	1.3	0.33	0.02
Endrin aldehyde	EPA 8270E	0.04	1.3	0.33	0.02
Endrin ketone	EPA 8270E	0.04	1.3	0.33	0.02
Methoxychlor	EPA 8270E	0.04	1.3	0.33	0.02
γ-Chlordane	EPA 8270E	0.04	1.3	0.33	0.02
α-Chlordane	EPA 8270E	0.04	1.3	0.33	0.02
Aroclor 1221	EPA 8082A	0.5	16	0.5	0.05
Aroclor 1232	EPA 8082A	0.25	8.3	0.25	0.025
Aroclor 1242	EPA 8082A	0.25	8.3	0.25	0.025
Aroclor 1016	EPA 8082A	0.25	8.3	0.25	0.025
Aroclor 1248	EPA 8082A	0.25	8.3	0.25	0.025
Aroclor 1254	EPA 8082A	0.25	8.3	0.25	0.025

Table 6-8 Routine Pesticide/PCB Target Analyte List - MRLs by Matrix (Cont'd)

ANALYTE	Analytical Method	Water¹ µg/L	Soil/Sed² µg/kg	Waste³ mg/kg	Tissue⁴ mg/kg
Aroclor 1260	EPA 8082A	0.25	8.3	0.25	0.025
Aroclor 1262	EPA 8082A	0.25	8.3	0.25	0.025
Aroclor 1268	EPA 8082A	0.25	8.3	0.25	0.025
Toxaphene	EPA 8270E (Mod)	2	67	NA	1

Notes:

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.

Table 6-9 Pesticide/PCB Analyte List Performed by SPECIAL REQUEST MRLs by Matrix					
ANALYTE	Analytical Method	Water^{1,5} µg/L	Soil/Sed^{2,5} µg/kg	Waste^{3,5} mg/kg	Tissue^{4,5} mg/kg
Technical Chlordane ⁶	EPA 8081B	1.5	50	1.5	0.05
trans-Nonachlor	Modified 8270	0.5	20	0.5	0.02
cis-Nonachlor	Modified 8270	0.5	20	0.5	0.02
Dicofol	Modified 8081B	0.08	5	NA	NA
4,4'-Dichlorobenzophenone	Modified 8081B	0.08	5	NA	NA
Chlorobenzilate	Modified 8270	0.02	0.67	NA	NA
2,4'-DDT	Modified 8270	0.04	1.3	NA	0.0013
2,4'-DDE	Modified 8270	0.02	0.67	NA	0.0067
2,4'-DDD	Modified 8270	0.04	1.3	NA	0.0013
PCB (as Congeners) – Green List	EPA 8082A	0.02	1	0.2	0.001
Toxaphene (as congeners except Parlar 62)	EPA 8276	0.001	0.033	0.005	0.0001
Toxaphene Parlar 62	EPA 8276	0.005	0.17	0.025	0.0005

Notes:

MRLs may increase due to interferences necessitating smaller extraction amounts, percent moisture, and dilutions.

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.

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

**Table 6-10 Herbicides Target Analyte List MRLs by SPECIAL REQUEST
by Matrix**

ANALYTE	Water ($\mu\text{g}/\text{L}$)	Soil/Sed	Waste	Tissue
2,4,5-T	1	NA	NA	NA
2,4-D	1	NA	NA	NA
2,4-DB	2	NA	NA	NA
Silvex (2,4,5-TP)	1	NA	NA	NA
Dalapon	4	NA	NA	NA
Dicamba	5	NA	NA	NA
Dichlorprop	1	NA	NA	NA
Dinoseb	4	NA	NA	NA
MCPA	5	NA	NA	NA
MCPP	5	NA	NA	NA

Notes:

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

Method Reference SW-846 Method 8321B (LC/MS/MS)

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

Table 6-11 Per- and Polyfluoroalkyl Substances (PFAS) Target Analyte List MRLs by Matrix				
ANALYTE	Water ng/L	Soil/Sed ng/kg	Waste ng/kg	Tissue
Perfluorotetradecanoic acid (PFTeDA)	20	400	NA	NA
Perfluorotridecanoic acid (PFTrDA)	10	100	40	NA
Perfluorododecanoic acid (PFDoA)	10	100	40	NA
Perfluoroundecanoic acid (PFUDA)	10	100	40	NA
Perfluorodecanoic acid (PFDA)	10	100	40	NA
Perfluorononanoic acid (PFNA)	10	100	40	NA
Perfluorooctanoic acid (PFOA)	10	100	40	NA
Perfluoroheptanoic acid (PFHpA)	10	100	40	NA
Perfluorohexanoic acid (PFHxA)	20	100	47	NA
Perfluoropentanoic acid (PFPeA)	10	100	40	NA
Perfluorobutyric acid (PFBA)	10	100	40	NA
Perfluorodecanesulfonate (PFDS)	9.7	97	40	NA
Perfluorononanesulfonate (PFNS)	9.6	96	40	NA
Perfluorooctanesulfonate (PFOS)	9.3	93	40	NA
Perfluoroheptanesulfonate (PFHpS)	9.5	95	40	NA
Perfluorohexanesulfonate (PFHxS)	9.1	91	40	NA
Perfluoropentanesulfonate (PFPeS)	9.4	94	40	NA
Perfluorobutanesulfonate (PFBS)	8.9	89	40	NA
Perfluorooctanesulfonamide (FOSA)	10	100	40	NA
Fluorotelomer sulfonate 8:2 (8:2 FTS)	9.6	96	40	NA
Fluorotelomer sulfonate 6:2 (6:2 FTS)	9.5	95	40	NA
Fluorotelomer sulfonate 4:2 (4:2 FTS)	9.4	9.4	40	NA
N-ethyl-N- ((heptadecafluoroctyl)sulfonyl)glycine (N-EtFOSAA)	10	NA	NA	NA
N-(Heptadecafluoroctylsulfonyl)-N- methylglycine (N-MeFOSAA)	10	100	56	NA

Table 6-11 Per- and Polyfluoroalkyl Substances (PFAS) Target Analyte List MRLs by Matrix (Cont'd)				
ANALYTE	Water ng/L	Soil/Sed ng/kg	Waste ng/kg	Tissue
Hexafluoropropylene oxide–dimer acid (HFPO-DA)*	20	100	40	NA
Perfluor-1-butanesulfonamide (FBSA)**	10	NA	NA	NA
1,1,2,2,3,3,4,4,4-Nonafluoro-N,N-bis(2-hydroxyethyl)butane-1-sulphonamide (FBSEE-diol)**	10	NA	NA	NA

Notes:

MRLs may increase due to interferences necessitating smaller sample amounts, dilutions and moisture content for soils.

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

Method Reference ASTM standards D7979-17 (water) and D7968-14 (solids).

Lab capacity is 40 samples per week with a 35-day turnaround time. Water matrices is for non-drinking water samples.

* HFPO-DA is single lab in-house validated

** Analyte not routinely reported. Special requests with advanced notice required for reporting of these analytes.

Table 6-12 Per- and Polyfluoralkyl Substances Target Analyte List MRLS for Drinking Water¹

Analyte	Potable Water ng/L
Hexafluoropropylene oxide dimer acid (HFPO-DA)	8
N-ethyl perfluoroctanesulfonamidoacetic acid (NEtFOSAA)	8
N-methyl perfluoroctanesulfonamidoacetic acid (NMeFOSAA)	8
Perfluorobutanesulfonic acid (PFBS)	7.08
Perfluorodecanoic acid (PFDA)	8
Perfluorododecanoic acid (PFDoA)	8
Perfluoroheptanoic acid (PFHpA)	8
Perfluorohexanesulfonic acid (PFHxS)	7.3
Perfluorohexanoic acid (PFHxA)	8
Perfluorononanoic acid (PFNA)	8
Perfluorooctanesulfonic acid (PFOS)	7.4
Perfluorooctanoic acid (PFOA)	8
Perfluorotetradecanoic acid (PFTeDA, also known as PFTA)	8
Perfluorotridecanoic acid (PFTrDA)	8
Perfluoroundecanoic acid (PFUDA)	8
11-chloroeicosfluoro-3-oxaundecane-1-sulfonic acid (11Cl-PF3OUdS)	7.52
9-chlorohexadecafluoro-3-oxanone-1-sulfonic acid (9Cl-PF3ONS)	7.44
4,8-dioxa-3H-perfluorononanoic acid (ADONA)	7.56

Notes:

Method Reference EPA 537.1

1 PFAS is not a regulated DW parameter, therefore not certified by OGWDW.

Some PFAS compounds are commercially available as ammonium, sodium and potassium salts.

This method measures all forms of the analytes as anions while the counterion is inconsequential. (see Method 537.1 for specifics).

Table 6-13 Per-and Polyfluoroalkyl Substances (PFAS) 1633 Target Analyte List MRLs by Matrix				
Analyte	Water ng/L	Soil/Sed ng/kg	Waste ng/kg	Tissue
Perfluoroalkyl carboxylic acids				
Perfluorobutanoic acid (PFBA)	16.00	800	NA	NA
Perfluoropentanoic acid (PFPeA)	8.00	400	NA	NA
Perfluorohexanoic acid (PFHxA)	4.00	200	NA	NA
Perfluoroheptanoic acid (PFHpA)	4.00	200	NA	NA
Perfluorooctanoic acid (PFOA)	4.00	200	NA	NA
Perfluorononanoic acid (PFNA)	4.00	200	NA	NA
Perfluorodecanoic acid (PFDA)	4.00	200	NA	NA
Perfluoroundecanoic acid (PFUnA)	4.00	200	NA	NA
Perfluorododecanoic acid (PFDoA)	4.00	200	NA	NA
Perfluorotridecanoic acid (PFTrDA)	4.00	200	NA	NA
Perfluorotetradecanoic acid (PFTeDA)	4.00	200	NA	NA
Perfluoroalkyl sulfonic acids				
Acid Form				
Perfluorobutanesulfonic acid (PFBS)	3.55	177	NA	NA
Perfluoropentansulfonic acid (PFPeS)	3.76	188	NA	NA
Perfluorohexanesulfonic acid (PFHxS)	3.66	183	NA	NA
Perfluoroheptanesulfonic acid (PFHpS)	3.81	191	NA	NA
Perfluorooctanesulfonic acid (PFOS)	3.71	186	NA	NA
Perfluorononanesulfonic acid (PFNS)	3.85	192	NA	NA
Perfluorodecanesulfonic acid (PFDS)	3.86	193	NA	NA
Perfluorododecanesulfonic acid (PFDoS)	3.88	194	NA	NA
Fluorotelomer sulfonic acids				
1H,1H, 2H, 2H-Perfluorohexane sulfonic acid (4:2 FTS)	15.00	750	NA	NA
1H,1H, 2H, 2H-Perfluorooctane sulfonic acid (6:2 FTS)	15.20	760	NA	NA
1H,1H, 2H, 2H-Perfluorodecane sulfonic acid (8:2 FTS)	15.36	768	NA	NA
Perfluorooctane sulfonamides *				
Perfluorooctanesulfonamide (PFOSA)	4.00	200	NA	NA
N-methyl perfluorooctanesulfonamide (NMeFOSA)	4.00	200	NA	NA

N-ethyl perfluorooctanesulfonamide (NEtFOSA)	4.00	200	NA	NA
Perfluorooctane sulfonamidoacetic acids *				
N-methyl perfluorooctanesulfonamidoacetic acid	4.00	200.00	NA	NA
N-ethyl perfluorooctanesulfonamidoacetic acid	4.00	200.00	NA	NA
Perfluorooctane sulfonamide ethanols *				
N-methyl perfluorooctanesulfonamidoethanol (NMeFOSE)	40.00	2000.00	NA	NA
N-ethyl perfluorooctanesulfonamidoethanol (NEtFOSE)	40.00	2000.00	NA	NA
Per- and Polyfluoroether carboxylic acids				
Hexafluoropropylene oxide dimer acid (HFPO-DA)	16.00	800	NA	NA
4,8-Dioxa-3H-perfluorononanoic acid (ADONA)	15.12	756	NA	NA
Perfluoro-3-methoxypropanoic acid (PFMPA) or (PF5OHxA)	8.00	400	NA	NA
Perfluoro-4-methoxybutanoic acid (PFMBA) or (PF4OPeA)	8.00	400	NA	NA
Nonafluoro-3,6-dioxaheptanoic acid (NFDHA) or (3,6-OPFHpA)	8.00	400	NA	NA
Ether sulfonic acids				
9-Chlorohexadecafluoro-3-oxanonane-1-sulfonic acid (9Cl-PF3ONS)	14.96	756	NA	NA
11-Chloroeicosafauro-3-oxaundecane-1-sulfonic acid (11Cl-PF3OUdS)	15.12	756	NA	NA
Perfluoro(2-ethoxyethane)sulfonic acid (PFEESA)	7.12	356	NA	NA
Fluorotelomer carboxylic acids				
3-Perfluoropropyl propanoic acid (3:3FTCA) or (FPrPA)	20.00	1000	NA	NA
2H,2H,3H,3H-Perfluorooctanoic acid (5:3FTCA) or (FPePA)	100.00	5000	NA	NA
3-Perfluoroheptyl propanoic acid (7:3FTCA) or (FHpPA)	100.00	5000	NA	NA

* Analytes in this class may not perform as well as others (LSBPROC-811 Determination of PFAS via Isotope Dilution, current version)