



ANALYTICAL SERVICES BRANCH
LABORATORY OPERATIONS
and
QUALITY ASSURANCE MANUAL

U.S. ENVIRONMENTAL PROTECTION AGENCY
SCIENCE AND ECOSYSTEM SUPPORT DIVISION
REGION 4
980 COLLEGE STATION ROAD
ATHENS, GEORGIA 30605-2700

Concurrences and Approvals:

(1) Name: Danny France Phone: 706-355-8738

Title: Branch Chief, Analytical Services Branch

Signature:  Date: 4-23-18

(2) Name: Jeffrey Hendel Phone: 706-355-8839

Title: Section Chief, Inorganic Chemistry Section

Signature:  Date: 4/23/18

(3) Name: Floyd Wellborn Phone: 706-355-8567

Title: Section Chief, Organic Chemistry Section

Signature:  Date: 4-23-18

(4) Name: Stacie Masters Phone: 706-355-8847

Title: Laboratory Quality Manager

Signature:  Date: 4-19-18

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.

Analytical Services Branch

Ethics Policy

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

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CHAPTER 1

Purpose, Policy, Accreditation and Hierarchy

1.1 Purpose

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

1.2 Mission of the EPA Regional Laboratory

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

1.3 Operations Policy

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

1.3.1 Safety The primary consideration in all laboratory operations must be safety. There is no assignment for which safety should ever be compromised. Safety takes priority over all considerations and it is the responsibility of each staff person to have a clear understanding of the basic safety rules and, in particular, how to safely perform operations within their area of responsibility. It is the responsibility of each individual to maintain a constant vigilance over safe operations and to notify their supervisor, Safety and Health Manager and the branch Safety Officer of any unsafe conditions. ASB 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 Safety, Health and Environmental Management Policy and Procedures Manual (SHEMP) for safety and health policies and procedures.

1.3.2 Data Integrity and Laboratory Ethics It is the policy of the Region 4 Laboratory to conduct all business with integrity and in an ethical manner. It is a basic and expected responsibility of each staff member and manager to hold to the highest ethical standard of professional conduct in the performance of all duties and to adhere to EPA's Principles of Scientific Integrity (1999) and the Scientific Integrity Policy (2012). The quality system of the branch has data integrity and ethical behavior at its very foundation. It is absolutely essential that every employee of the branch understand and adhere to these ethical standards in order to preserve the basic integrity of all work products. Data integrity, defined in its most simple terms as “the state of being unimpaired”, concerns the ability to define and defend that the entire analytical process has been “unimpaired” and performed in accordance with appropriate practices and procedures. The ability to defend the integrity of the data is through complete documentation of actions and activities which includes, but is not limited to such items as: maintaining chain of custody and security of the samples; clear documentation of the activities performed in the preparation and analysis of the samples according to SOPs and in the final data reduction, review, and reporting; and maintaining complete and clear files of these records.

1.3.3 Quality It is the policy of ASB that all data generated is of the quality required to meet or exceed the data quality objectives (DQOs) of each project as determined by the customer. Managers and analysts of the branch share the responsibility of ensuring that analytical methods, instruments, analyte detection and quantitation are such that the data produced is scientifically sound and well documented. It is of utmost importance that the quality of all data produced by ASB be well defined and communicated to the customer. This policy is implemented by:

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

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

1.3.3.3 Having a peer review process to verify that data are generated in accordance with sound and appropriate technical procedures and to ensure that all activities associated with the analyses, calculations and data reduction are complete and accurate.

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

1.3.4.1 Communication between the laboratory's staff and its clients is required to fully 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. ASB management and staff must be proactive in initiating these discussions and will make every effort to inform customers of the advantages and disadvantages of requested methods and quality control procedures. Laboratory management reserves the right to determine the most appropriate analytical methodology and quality control procedures based on the DQOs, if provided by the customer.

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

1.4 Accreditation and Certification

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

1.4.2 ASB is ISO/IEC 17025:2005 accredited. Refer to certificate number AT-1691 and scope for specific accreditation information. The laboratory is also Drinking Water certified by the Office of Ground Water and Drinking Water (OGWDW) and is certified to the current Drinking Water methodologies and the Fifth Edition of the Manual for the Certification of Laboratories Analytical Drinking Water (EPA 815-R-05-004, January 2005).

1.4.3 ASB'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 ASB is currently accredited and certified is available from the Laboratory Quality Manager (LQM). ASB 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 ASB.

1.5 Hierarchy

This manual describes the policies that are the basis of ASB's quality system. Specific technical and procedural details are contained in methods and technical and administrative SOPs. On occasion, an analytical method or procedure may require deviation from some

of the policies contained in this manual for specific technical reasons. These deviations will be documented in the individual SOPs. As such, instructions in SOPs and regulatory program requirements take precedence over this manual on those occasions.

CHAPTER 2

Personnel, Facility and Equipment

2.1 Organization

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

2.1.1 Analytical Services Branch Personnel

Immediate Office

<u>Name</u>	<u>Principal Duties</u>
Danny France	Branch Chief
Scott Sivertsen	Senior Technical Advisor
Stacie Masters	Laboratory Quality Manager
Mike Beall	Sample Custodian (SEE Employee)
Alva Eisenman	Divisional Document Control (SEE Employee)
Marilyn Maycock	Senior Technical Advisor

Inorganic Chemistry Section

<u>Name</u>	<u>Principal Duties</u>
Jeffrey Hendel	Section Chief-Technical Director Inorganic Analyses
Daniel Adams	Nutrients, Classicals, Final Data Review/Production
Pam Betts	Classicals, ASB Safety Officer
Curtis Callahan	Nutrients, Classicals
Anthony Carroll	Mercury, Hexavalent Chromium, Lead Bioavailability
Megan DeJesus	Metals ICP
Earnest Walton	Metals ICP-MS
Terri White	Metals ICP, Final Data review/Production
Kayle Whiten	Nutrients, Classicals

Organic Chemistry Section

<u>Name</u>	<u>Principal Duties</u>
Floyd Wellborn	Section Chief-Technical Director Organic Analyses
Diana Burdette	Semivolatiles, LC-MS/MS

Jason Collum	Pesticides/PCBs, SESD Chemical Hygiene Officer
Sam Dutton	Pesticides/PCBs
John Giles	Extraction, Semivolatiles, LC-MS/MS
Sallie Hale	Volatiles (Air)
David Spidle	Monitored Natural Attenuation
Kristin Trapp	Volatiles (Air)
Stephanie Wimpey	Volatiles, Property Officer, ChemShare Coordinator
Francine Vancuron	LC-MS/MS

2.2 Educational, Experience and Training Requirements

2.2.1 EPA operates its hiring procedures under the federal government's Office of Personnel Management (OPM) regulations. OPM issues qualification and classification standards for all general schedule (GS) positions. Typically, ASB's professionals and technicians fall within the 1300 - Physical Sciences Group, Job Family Standards for Professional Work and Technical Work (See <http://www.opm.gov/qualifications/index.asp> and <https://www.opm.gov/policy-data-oversight/classification-qualifications/classifying-general-schedule-positions/#url=1300>). 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, ASB managers are allowed to hire an applicant who meets the OPM requirements from a certificate of eligible candidates.

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

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

2.3 Roles and Responsibilities

2.3.1 Branch Chief

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

of the branch quality assurance system.

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

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

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

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

2.3.1.6 Is authorized to offer opinions and interpretations.

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

2.3.2 Laboratory Quality Manager (LQM) – also referred to as the Quality Assurance Manager (QAM)

2.3.2.1 Is independent from all laboratory operations, reports directly to the ASB Chief and has the delegated responsibility and authority for the implementation, management, and maintenance of the quality system for the laboratory.

2.3.2.2 Ensures compliance with all laboratory accreditation requirements.

2.3.2.3 Coordinates the branch-wide QA/QC activities.

2.3.2.4 Initiates and leads annual internal audits within the branch.

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

2.3.2.6 Performs periodic spot checks (in addition to or in conjunction with the annual internal audits) of project files to ensure that all proper documentation and QC activities were performed. The spot checks shall be documented and any problems communicated with the appropriate Section Chief and technical staff.

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

2.3.2.7.1 Managerial and supervisory reports;

2.3.2.7.2 Documents requiring LQM signature/approval;

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

2.3.2.7.4 Results of inter-laboratory comparisons or proficiency tests;

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

2.3.2.7.6 Records and Documentation

2.3.2.7.6.1 Demonstration of Competency (DOC) and Continuing Demonstrations of Proficiency (CDOP);

2.3.2.7.6.2 Method Detection Limits (MDLs) and Limits of Detection (LODs);

2.3.2.7.6.3 Instrument Detection Limits (IDLs);

2.3.2.7.6.4 New method/technology validation studies;

2.3.2.7.6.5 Special Issue Studies reports;

2.3.2.7.6.6 Summaries of updates for acceptance limits in Element[®] for spikes, replicates, surrogates, and other QC data;

2.3.2.7.6.7 Trend reports produced by the sections;

2.3.2.7.6.8 Signature and initials of all employees;

2.3.2.7.6.9 Training files to include internal and external training, cross training, certifications, etc.; and

2.3.2.7.6.10 Corrective Action and Preventive Action reports.

2.3.2.8 Performs periodic verification of primary standard prep and external verification of the standards. (As a separate check or in conjunction with the periodic internal audits.)

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

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

2.3.2.11 Advises branch management concerning QA/QC issues.

2.3.2.12 Is authorized to give opinions and interpretations.

2.3.3 Section Chief

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

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

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

2.3.3.4 Monitors all section work activities with the help of Group Leaders where designated.

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

2.3.3.6 Reviews and approves all section specific technical document and LOQAM updates.

2.3.3.7 Monitors and coordinates section workload and acceptance of work.

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

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

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

2.3.3.11 Ensures compliance with all laboratory accreditation requirements.

2.3.4 Analytical Staff

2.3.4.1 General staff are responsible for:

2.3.4.1.1 Having a general knowledge of the branch and divisional policies

and procedures including health and safety, data integrity, and waste disposal;

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

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

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

2.3.4.1.5 Maintaining and following appropriate SOPs for their work areas.

2.3.4.2 Group Leader (if designated by the Section Chief) At the discretion of the Section Chief, the Organic or Inorganic Section may be comprised of work groups. If applicable, each analytical work group may have a Group Leader appointed by the Section Chief. This is not a supervisory position; however, Group Leaders are assigned technical responsibilities to assist the Section Chief and may be called upon to assist the Section Chief with work scheduling issues. The Section Chief will fill the role of the Group Leader during periods for which there is no one so designated.

2.3.4.2.1 Serves as the primary technical contact on analytical issues or questions pertaining to their specific expertise.

2.3.4.2.2 Provides daily direction of the technical activities within their work unit; ensures that QA/QC actions are in accordance with sound technical practices and follows policies and procedures of the LOQAM.

2.3.4.2.3 Ensures that appropriate corrective actions are taken based on quality indicators from the analyses within their work unit.

2.3.4.2.4 Communicates regularly with LQM and other Group Leaders on technical issues and problems.

2.3.4.3 Primary Analyst/Technician Defined as the staff analyst or technician performing a test on a given date and time. Typically, the primary analyst performs initial data reduction and transfer of data to the Element[®]. However, this task may also be performed by another analyst competent to perform the analysis. The primary analyst/technician shall ensure that:

2.3.4.3.1 Appropriate analytical methodologies and standard operating procedures are followed;

2.3.4.3.2 Appropriate QC activities are performed as designated by the

method, SOPs, and/or the LOQAM;

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

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

2.3.4.3.5 Individual analytical data points are completely and accurately recorded;

2.3.4.3.6 Data qualifier flags and explanatory footnotes are properly placed; and

2.3.4.3.7 All appropriate items on the data review/verification checklist are properly documented.

2.3.4.4 Secondary Analyst (Data Verification) may be the Group Leader or another staff analyst that is qualified to perform data review for the analysis being checked. It is the responsibility of the secondary analyst to perform a thorough review of all important details associated with the data being verified. The Secondary Analyst shall ensure that:

2.3.4.4.1 Appropriate analytical methodologies and SOPs were followed;

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

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

2.3.4.4.4 Appropriate actions were taken as a result of QC indicators; and

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

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

2.3.6 Environmental Services Assistance Team (ESAT) Laboratory Support - Analytical support is often obtained through the ESAT contract as funding permits. The ESAT team is located on site within the ASB laboratory areas with space assigned specifically to them. Work is assigned by EPA Contract Officers to ESAT staff through

technical direction documents following all contractual rules and regulations. ESAT personnel are expected to be familiar with the LOQAM, follow its policies and practices, and to follow analytical SOPs approved by EPA management.

2.3.7 ASB Staff - ESAT Work Assignments

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

2.3.7.2 ASB staff that submit technical direction requests must follow all rules and regulations of the contracting process. ASB staff are also responsible for receiving the work products that are generated by ESAT staff and performing an appropriate review of the work performed.

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

2.3.7.3.1 Appropriate analytical methodologies and SOPs were followed;

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

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

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

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

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

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

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

2.4 Facilities

The total facility consists of approximately 55,000 net usable square feet, a little less than a third of which is occupied by ASB. Operation and maintenance of the facility is the

responsibility of the lessor through the Government Services Administration (GSA). SESD (not within ASB) has one or more staff members dedicated to facility issues, coordinating maintenance and operations with GSA and the lessor. The facility has adequate accommodations to perform testing procedures in the laboratory area. The laboratory will ensure the facility and environmental conditions relevant to the procedure will be monitored as required.

2.5 Equipment

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

2.5.2 Maintenance/Service Proper maintenance of laboratory instrumentation is a key ingredient to both the longevity of the useful life of the instrument, as well as providing reliable analyses. Maintenance and service requires an alert analytical staff that recognizes the need for equipment maintenance coupled with support services provided either by in-house staff or by vendor technicians. All staff members have the responsibility for ensuring that all primary maintenance is carried out on instrumentation in accordance with manufacturer's recommendations and schedules as practical.

Figure 2-1
Analytical Services Branch Organization Chart

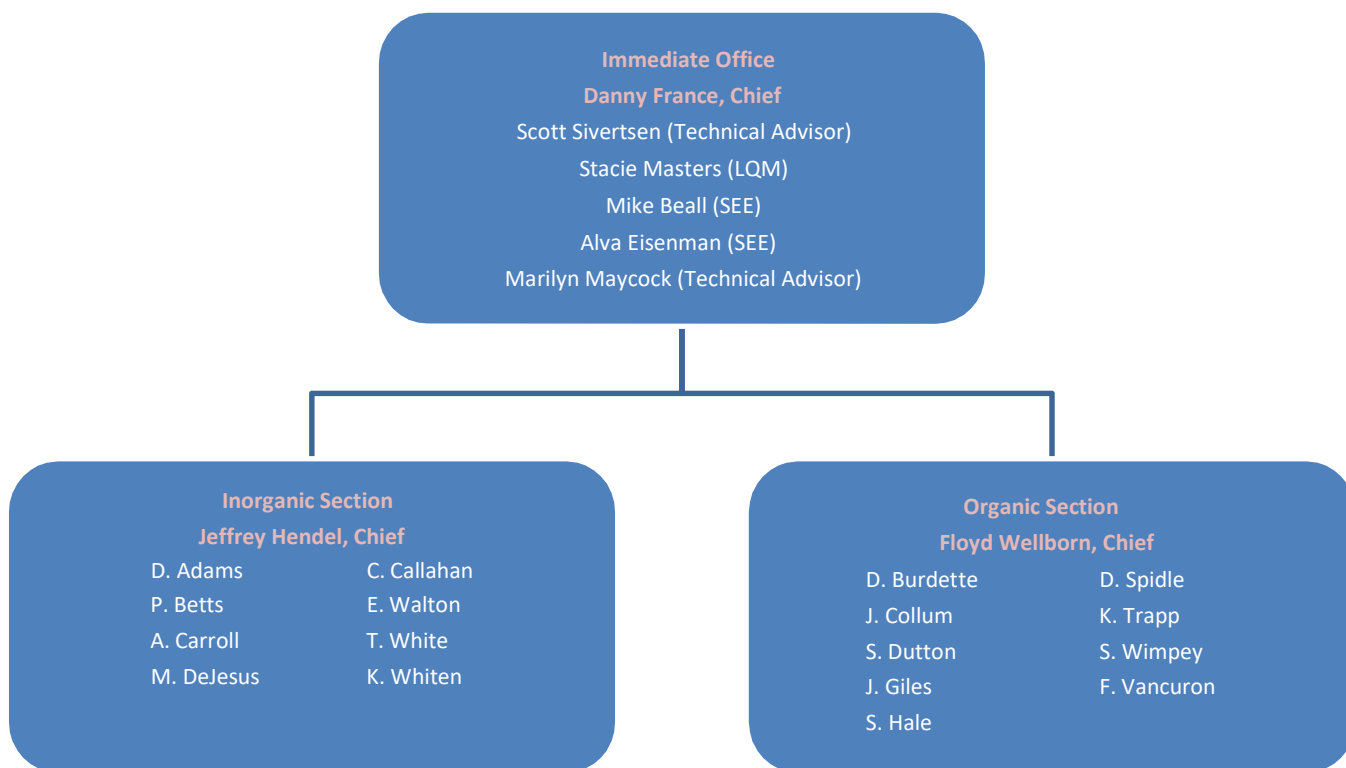
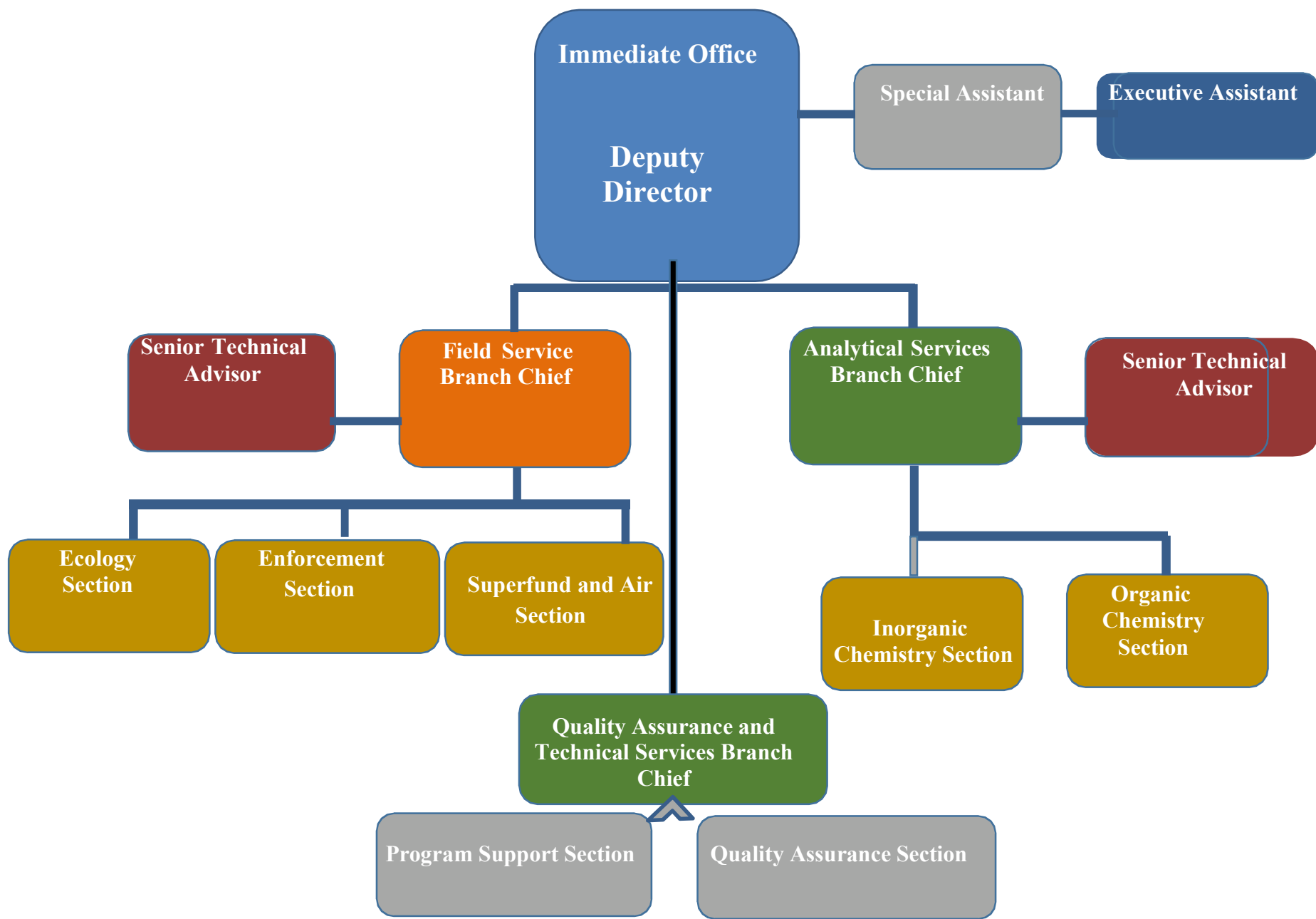


Figure 2-2
Science and Ecosystem Support Division — Athens, Georgia



CHAPTER 3

Sample Scheduling, Handling, Storage and Disposal

3.1 Introduction

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

3.2 Sample Collection

3.2.1 Procedures ASB staff does not perform field sampling activities, therefore the sampling activities are covered under the project specific Quality Assurance Project Plans.

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

3.3 Sample Scheduling

3.3.1 Initial Scheduling ASB uses an in-house laboratory information management system (LIMS), called R4LIMS for project scheduling. Each project that is entered into R4LIMS is assigned a unique project number that is used throughout its life for tracking, reporting, and filing.

3.3.2 Sample Acceptance

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

their management to determine if the samples must be analyzed and what priority will be assigned. Normally all samples in support of criminal investigation projects will be analyzed within the ASB laboratories or sent to the National Enforcement Investigations Center (NEIC) for analysis. R4LIMS shows scheduled projects in grey until they have been officially accepted by ASB. Scheduling of the samples generally includes an estimate of numbers, matrices, requested analyses and turn-around time (TAT) requirements. The standard TAT for the laboratory from the time samples are received until results are reported is 35 calendar days for routine analyses and 45 calendar days for projects with TCLP requirements. When a project requires samples to be received by the laboratory over multiple days, the TAT is calculated based on the last day samples are received by the laboratory for the particular project. Communication of special project requirements are noted in R4LIMS Project Notes.

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

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

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

3.3.2.3.1 If the request is in support of SDWA regulations, analyses must be performed by approved methods found at 40 CFR Part 141. Tables 6-1 and 6-2 list primary drinking water contaminants, including analytical method

requirements. As indicated in Table 6-2, ASB does not analyze the full list of primary drinking water contaminants. If the requestor requires the analysis of a primary contaminant which ASB does not analyze, the Regional Sample Control Coordinator (RSCC) will assist the requestor in locating a laboratory that has the capability and proper accreditation.

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

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

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

3.3.2.6 Documenting Communication in R4LIMS and Element® Workorder Notes Communication between the project leader and ASB personnel should be documented. This includes documenting in Element® or the project file any special requests or clarification to requests, changes to the project.

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

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

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

3.3.2.7.1 Preliminary results will be reported to the customer within the requested timeframe, followed by final reporting at a later date.

3.3.2.7.2 Final results that have gone through the necessary QA/QC checks will be reported.

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

3.4 Sample Receipt

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

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

3.4.2 Sample Receiving Procedure Detailed sample receiving procedures are documented in the most current revision of SOP ASB 105G, Sample Receiving and Custody.

3.4.3 Sample Receipt Guidelines for Analyses with Short Holding Time Requirements Some organic and inorganic analyses such as waters requiring analysis for semivolatiles, pesticides/PCBs TSS, TDS, Unpreserved Nitrate or Nitrite, BOD, and unpreserved volatiles require expedited shipping to SESD due to the short holding times. Therefore, the following sample shipping guidelines should be observed by field sampling organizations.

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

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

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

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

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

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

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

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

3.4.4.2.1 Scheduling Samples When project leaders schedule samples known or suspected to contain dioxin into R4LIMS for routine analyses, the requestors are required to indicate whether the samples are or are not contaminated with listed RCRA dioxin-containing wastes. Because the laboratory has limited, if any, knowledge of site or facility history, it is the project leaders’ responsibility to determine whether samples are contaminated with the listed RCRA-dioxin containing wastes. A waste is determined to be a hazardous waste if it is specifically listed on the F or K lists found in title 40 of the Code of Federal Regulations (CFR) in Section 261. Prior to scheduling samples, review the hazardous waste criteria information at the following link to determine if the samples will be acceptable by ASB:

[Defining Hazardous Waste: Listed, Characteristic and Mixed Radiological Wastes | Hazardous Waste | US EPA](#)

3.4.4.2.2 The documents cited in references 1 and 2 are located on the SESD

LAN and may be consulted for additional information on the management of RCRA wastes.

¹Management of Contaminated Media, EPA Region 4, September 7, 1999

²Management of Remediation Wastes under RCRA, EPA 530-F-98-026, October 1998

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

3.5 Sample Logging and Storage

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

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

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

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

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

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

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

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

3.5.2.2 The acceptable temperature range for the coolers is above freezing to 6°C. The freezer should maintain $\leq -10^{\circ}\text{C}$. A temperature excursion outside acceptance criteria may result in initiating a corrective action to determine if the excursion was a systematic issue. The Custody Room coolers and freezer are additionally monitored by security personnel outside regular business hours.

3.6 Custody

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

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

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

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

3.6.5 Assuming Custody for Sample Analysis ASB utilizes Element[®] to perform sample check-out, check-in to/from their storage locations. To receive samples for analysis, an analyst must assume custody of the samples (including those 'aliquoted' in the custody room such as frozen tissue). When the need for the sample container is complete, custody is relinquished by setting the location of the container to "disposed" in Element[®] and returning it back to the original storage location.

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

3.6.6.1 ASB will track the custody of the extracts, digestates and/or leachates throughout the prep and analysis of the samples.

3.6.6.2 The custody of the extracts, digestates and/or leachates are transferred from the prep area personnel to the analytical personnel by using the bench sheet. There are areas at the end of each bench sheet that track the custody of the samples as they move through the analytical process. The prep analyst signs in the “relinquished by” area and the receiving analyst signs in the “received by” area. If the analyst transfers custody of the samples prior to disposal, the analyst should relinquish custody by signing the bench sheet in the same manner as described above.

3.6.6.3 Batch IDs are assigned automatically by Element[®] and are in the format ‘YYMMnnnn’ where:

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

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

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

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

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

3.6.6.6 Transfer of Custody from ASB: On occasion, after ASB 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 ASB custody by transferring official custody using appropriate COC forms and notations in Element[®]. All custody transfers of this nature must be coordinated through the sample custodian or designee.

3.7 Review of Custody Records

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

Table 3-1 Recommended Preservation & Holding Times								
Analytical Group	Soil/Sediment ¹		Water ^{1,2} and Waste Water		Waste		Tissue	
	Pres³ Amt⁴ Container Type⁵	Hold⁶	Pres³ Amt⁴ Container Type⁵	Hold⁶	Pres³ Amt⁴ Container Type⁵	Hold⁶	Pres³ Amt⁴ Container Type⁵	Hold⁶
Inorganics								
Acidity	NA	NA	Ice-4°C 500 mL P, FP Fill completely and cap tightly	14 days	NA	NA	NA	NA
Alkalinity	NA	NA	Ice-4°C 500 mL P, FP Fill completely and cap tightly	14 days	NA	NA	NA	NA
BOD5	NA	NA	Ice-4°C 2 L P, FP	48 hrs	NA	NA	NA	NA
BOD - Long Term	NA	NA	Ice-4°C 1 gal(x2) ¹⁵ P, FP	48 hrs	NA	NA	NA	NA
Bromide	NA	NA	Ice-4°C 500 mL P, FP	28 days	NA	NA	NA	NA
Chloride	None 8 oz G	Not specified	None 500 mL P, FP	28 days	NA	NA	NA	NA
Chlorine - Residual	NA	NA	None 500 mL P	Immediate	NA	NA	NA	NA
Chromium VI (hexavalent)	Ice-4°C 8 oz G	Extract - 1 month Analysis - 4 days ⁷	Filter immed. ⁹ Ice-4°C 1L P, FP Buffer to extend HT ³	24 hrs 28 days if buffered ³	None 8oz G	Extract - 1 month Analysis - 4 days ⁷	NA	NA
Cyanide	Ice-4°C 8 oz G	14 days	NaOH to pH>12, ascorbic acid ⁸ , Ice-4°C 500 mL P, FP	14 days	None 8oz G	Not specified	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	None 8 oz G	Not specified	None 500 mL P	28 days	NA	NA	NA	NA
Grain Size	None 8oz G	Not Specified	NA	NA	NA	NA	NA	NA
Hardness (calc)	NA	NA	NA Separate bottle not required, calculated from the metals scan	NA	NA	NA	NA	NA
Mercury, Routine	Ice-4°C 8 oz G	28 days ²⁹	HNO ₃ to pH<2 1L P, FP	28 days ²⁹	None 8oz G	180 days	Freeze, 5 g 8 oz G, Al foil or plastic	Not specifie d
Mercury - TCLP	None 8 oz G	28 days 56 days ¹⁰	None 1L P, FP or 1 gal. P, FP, G if multiphase (>0.5% and <10%solids)	28 days 56 days ¹⁰	None 8oz G or 1 gal P, G if multipha se (>0.5%)	28 days 56 days ¹⁰	NA	NA

Table 3-1 Recommended Preservation & Holding Times								
Analytical Group	Soil/Sediment ¹		Water ^{1,2} and Waste Water		Waste		Tissue	
	Pres³ Amt⁴ Container Type⁵	Hold⁶	Pres³ Amt⁴ Container Type⁵	Hold⁶	Pres³ Amt⁴ Container Type⁵	Hold⁶	Pres³ Amt⁴ Container Type⁵	Hold⁶
					and <10% solids)			
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 8oz G	Not specified	Freeze, 5 g 8 oz G, Al foil or plastic	Not specifie d
Metals, except mercury	None 8 oz G	6 months	HNO ₃ to pH<2 1L P, FP	180 days 6 months	None 8oz G	6 months	Freeze, 15 g 8 oz G, Al foil or plastic	Not specifie d
Dissolved Metals, except mercury	NA	NA	Filter immed ⁹ , HNO ₃ to pH<2 1L P, FP	180 days 6 months	NA	NA	NA	NA
Metals - TCLP (except mercury, see above)	None 8 oz G	180 days 360 days ¹¹	None 1L P, FP or 1 gal., P, FP, G if multiphase (>0.5% and <10% solids)	180 days 360 days ¹¹	None 8 oz G or 1 gal. P, G if multiphase (>0.5% and <10% solids)	360 days ¹¹	NA	NA
Nitrate (requires two containers: one unpreserved and a 2 nd preserved)	Ice-4°C 8 oz G	Not specified	Ice-4°C 500 mL P, FP AND 2 nd container Ice- 4°C, H ₂ SO ₄ to pH<2 500 mL	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 1L 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
Solids Series (TS, TSS, TDS, TVSS, etc.)	NA	NA	Ice-4°C 1L P, FP	7 days	NA	NA	NA	NA
Sulfates	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								
Analytical Group	Soil/Sediment ¹		Water ^{1,2} and Waste Water		Waste		Tissue	
	Pres³ Amt⁴ Container Type⁵	Hold⁶	Pres³ Amt⁴ Container Type⁵	Hold⁶	Pres³ Amt⁴ Container Type⁵	Hold⁶	Pres³ Amt⁴ Container Type⁵	Hold⁶
TOC	Ice-4°C 8 oz G	Not Specified	Ice-4°C, H2SO4 to pH<2 500mL P, FP	28 days	NA	NA	NA	NA
Dissolved TOC	NA	NA	Filter immed ⁹ , Ice 4°C, H2SO4 to pH<2 500mL, P, FP	28 days	NA	NA	NA	NA
Organics								
TCLP Extractables (Pesticides, Herbicides, Semivolatiles)	Ice-4°C 8 oz G	61 days ¹³	Ice-4°C 1 L (x2 per fraction) ¹⁵ G/A	61 days ¹³	None 8 oz G	61 days ¹³	NA	NA
For multi-phase samples, extra sample volume must be collected for TCLP analysis.								
Extractables (Pesticides/PCBs , Herbicides, Semivolatiles)	Ice-4°C 8 oz G	54 days ¹⁴	Ice-4°C 1 L (x2 per fraction) ¹⁵ G/A	47 days ¹⁶	None 8 oz G	54 days ¹⁴	Ice & Freeze 30 g Al Foil	Not specified
Extractables/ Pesticides/PCBs - residual chlorine present	NA	NA	Ice-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	NA	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
Org Halide (TOX)	Ice-4°C 8 oz G	28 days	Ice-4°C H2SO4 to pH<2 1 L G/A	28 days	NA	NA	NA	NA
Perfluorocarbons	NA	NA	Ice-4-15°C lab ambient+ 10-40 % acetonitrile 125-mL Polypropylene (x2)	194 days ²⁷	NA	NA	NA	NA
Carbamates	NA	NA	Ice-4°C 60-mL G/A (x2)	14 days ²⁸				
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								
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

Table 3-1 Recommended Preservation & Holding Times								
Analytical Group	Soil/Sediment ¹		Water ^{1,2} and Waste Water		Waste		Tissue	
	Pres³ Amt⁴ Container Type⁵	Hold⁶	Pres³ Amt⁴ Container Type⁵	Hold⁶	Pres³ Amt⁴ Container Type⁵	Hold⁶	Pres³ Amt⁴ Container Type⁵	Hold⁶
Volatile Organics Method 5035A	Ice-4°C 5 g (x3) ¹⁸ into tared 40- mL VOA vials ¹⁹	48 hours iced/14 days frozen ²⁰	NA	NA	NA	NA	NA	NA
Volatile Organics Method 5035A	Ice-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	-7 to -20°C 5 g (x3) ¹⁸ into tared 40- mL VOA vials containing 5 mL water ^{19,22}	14 days frozen	NA	NA	NA	NA	NA	NA
Volatile Organics Method 5035A	Ice-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	NA	NA	Ice-4°C 40 mL (x3) ¹⁵ G/S	7 days	NA	NA	NA	NA
Volatile Organics no residual chlorine present	NA	NA	0.2 mL 1+1 HCL (pH<2), Ice-4°C 40 mL (x3) ¹⁵ G/S	14 days	NA	NA	NA	NA
Volatile Organics residual chlorine present	NA	NA	3mg Na ₂ S ₂ O ₃ , 0.2 mL 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	28 days ²⁴	NA Ice 4°C 40 mL (x3) ¹⁵ G/S	NA 28 days ²⁴	Ice-4°C 8 oz G ²⁶ If <10% solids, 4x 8oz G	28 days ²⁴	NA	NA
Volatile Organics in Air	Preservation: closed, leak free valve with tightened cap. Amount: preferably 10 to 14 psia Container: passivated 6-liter canister							

General Notes:

NA = Not applicable

Pres = Preservation Immed = Immediate

Footnotes:

¹ ASB's policy is that where the sample preservation is specified at 4°C, the acceptable temperature range for samples during shipping and storage is from above the freezing point of water to 6°C.

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 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.

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.

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.

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

⁵ Container Type:

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

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

⁷ Chromium VI (hexavalent) - 1 month until extraction, 4 days to analysis of extract. Store at 4 \pm 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 μ m-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 or Sample Delivery Group (SDG).
- 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 Sampled to analyzed
- ²⁹ Holding time for routine mercury by Method 200.8, is 28 days from sample collection to digestion and 28 days from digestion to analysis.

CHAPTER 4

General Laboratory Practices

4.1 Good Laboratory Practices

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

4.1.2 Corrections to Records

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

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

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

4.1.4 Method Modifications Method modifications, where allowed by regulation, are encouraged as new technologies are developed which result in analytical efficiencies, pollution prevention and increased precision and accuracy. Restrictions apply to the modification of Safe Drinking Water Act (SDWA) and National Pollutant Discharge Elimination System (NPDES) methods. Methodology in support of the Safe Drinking Water Act (40 CFR Part 141) shall not be modified unless specified in the individual

method. Permitted modifications are documented in memos from OGWDW or approval as an Alternate Test Procedure (ATP). When these modifications are used, ASB's SOP should state that an allowed modification is being utilized, and reference the specific memo allowing the modification.

4.1.5 Manual Peak Integration Some of the analytical techniques utilized by ASB employ a chromatogram that displays time versus signal, which when integrated, provides an area count that is used to calculate concentration of a target analyte.

4.1.5.1 Analysts are required to review the automated electronic data processing (i.e., integration) for accuracy and consistency with appropriate data reduction techniques. Some electronic reductions can result in incorrect actions by the system software, and for these instances a manual override and correction of the electronic processing is appropriate. Examples of this may be such items as integration of an incorrect peak or misplacement of the baseline in peak integration due to poor peak shape or interferences. Guidance related to manual integrations is documented in individual workgroup Data Review Guidelines or technical SOPs. When a manual override of the electronic process is performed, most of the current commercially available software packages provides an automated notation on the quantitation report showing that a modification to a peak occurred. When a manual modification of a peak occurs, the analyst shall provide documentation for the file to include a hard-copy representation of the before and after correction, the action taken and why. The action should be concurred by the Secondary Review Analyst or Section Chief, and include an initial and date of the review of the modification on the data review form. Manual override actions are appropriate only to correct inaccuracies and shall be done in accordance with sound analytical procedures. The software option for denoting a manual integration in the quantitation report must always be activated. There shall be no manipulation of the software to conceal an electronic correction that is used to report results.

4.1.6 Checklists-Primary Analyst/Secondary Analyst Review Analytical data reduction activities for both the primary analysis and the secondary 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 SESD's local area network drive (LAN) as controlled documents.

4.2 Document Control/File Management

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

4.2.1.1 All documents are reviewed and approved by an authorized approving

official prior to being issued. Approving officials are Section Chiefs, Branch Chief or the LQM. The SESD Deputy Director serves as the approving official for all SESD quality system documents.

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

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

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

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

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

4.2.1.7 Archived documents shall be marked as such.

4.2.1.8 ASB's procedures for document control are detailed in SESDPROC-1000- Document Control.

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

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

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

4.2.4.1 Instrument/Analysis Logbooks Instrument logs shall indicate the unique instrument ID, date of analysis, analyst and samples which have been analyzed. The logbook shall contain or reference a record of which options or analytical conditions were used for analysis. Where appropriate, instrument acceptance criteria (e.g., tune criteria, sensitivity checks) should be noted in the logbook. 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 is accomplished by printing a copy of the electronic record and including it in a notebook. These sequential logs must also include failed runs, or sequences which were abandoned prior to completion. When a pre-determined number of pages has been accumulated (e.g., 50 pages), the individual records are combined into a single bound logbook and retained as specified above. Any electronic records must accurately reflect actual analytical information. For analyses with holding times < 72 hours, or when time-critical or method-specified times are included in the analysis, the time of analysis must also be recorded.

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

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

4.2.4.4 Other Some analytical methods are manual and do not use analytical instrumentation to generate a result (e.g., solids). For these methods, ASB relies on spreadsheets or other calculating software for recording/documenting original observations made, such as weights. All spreadsheets or other calculating software used within ASB 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 LQM at the time of posting. Copies of any spreadsheet used must be obtained from the password protected official, posted version on the LAN. Prior to posting and use, all calculations in spreadsheets will be hand-validated by the responsible party and submitted through the Section Chief to the LQM for approval and posting.

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

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

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

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

4.2.6 Standard Operations Procedures/Methods SOPs shall be written based on agency guidance EPAQA/G-6 “Guidance for the Preparation of Standard Operating Procedures for Quality Related Documents”. Detailed policies and procedures for the preparation, review and change of both administrative and technical SOPs are found in SESDPROC-100-Documents Control. Technical SOPs for the various methods in use in each laboratory may be placed within the laboratory for reference purposes; however, the official copy of each SOP resides on the LAN in the K:\ASB\Current Documents\SOPs folder.

4.2.7 Project Files

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

4.2.7.2 The project file contains all data (or copies thereof) used to produce the final

data report. For example, if an analytical run is not used because of a calibration failure, it need not be retained in the project file. However, if the failed run was used to determine the level of dilution required by a sample in the final run, it should be maintained. See SOP ASB 118G for the required elements needed for a complete project file.

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

4.2.7.4 The analytical information is maintained by analysts while the analyses are in progress. Each completed data packet is transferred to the secondary reviewer for review, submitted to the Section Chief or designee for final reporting, and ultimately transferred to the SESD records room for inclusion into the project file. It is absolutely essential that the hard copies placed into project files exactly reflect the electronic data produced for the project. Forensic accreditation policy requires that the complete project file to be paginated, therefore ASB includes an inventory checklist that accounts for each page of the project file. It is agency policy that hard copy project files are the official record and e-data files are not required to be maintained. Ultimate retention and disposal of all records will be in accordance with Agency record management rules and regulations as detailed in the "Records Management Standard Operating Procedures, Science and Ecosystem Support Division."

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

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

4.2.8 Confidentiality of Data

4.2.8.1 ASB 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 ASB is accessible under the Freedom of Information Act (FOIA). The exception is data from all criminal investigation projects; it is not subject to release and will not be reported to anyone other than

project managers leading the criminal investigations or to individuals that are authorized by ASB management. Criminal projects are so noted when logged into R4LIMS.

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

4.2.9 General Correspondence All general written correspondence (e.g., memos, letters) from ASB technical staff to any party external to ASB, but internal to SESD shall be reviewed and approved by the respective section chief and shall have the section chief as a “THRU” signatory. All correspondence external to the Division shall also include the Branch Chief as a “THRU” signatory. Correspondence related to a specific project shall be filed in the project file. General correspondence shall be forwarded to the LQM for filing according to the ASB Divisional File Plan.

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

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

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

4.2.13 Records Management/Disposition ASB records will be managed in accordance with the Records Management Standard Operating Procedures of the Science and Ecosystem Support Division. In the event that the SESD and ASB organizations are eliminated, all records would be maintained as required by U.S. government regulations for records retention in force at the time of the discontinuation.

4.3 Laboratory Apparatus and Instruments

4.3.1 General Policy It is the policy of ASB that all laboratory apparatus and instruments meet or exceed any method-specified tolerances to ensure results are reported within acceptable uncertainty levels. Environmental Management System goals (e.g., reduction in chemical use or more energy efficiency) should be considered when evaluating new equipment for purchase, but may not always be the deciding factors. All equipment will be determined to be clean, free of contaminants and operational and will be calibrated prior to use as per manufacturer's instruction or procedures detailed in the technical SOPs. If any equipment becomes defective or is suspected of being defective, it will either be removed from the work area or marked as out-of-service. The defective equipment will be separated from equipment currently in use. Equipment will be utilized by authorized personnel and user manuals will be available for review either in electronic or hard copy formats. In general, all ASB laboratory apparatus and instruments remain under the control of ASB at all times. However, if equipment leaves the direct control of ASB (e.g., loaned to another agency), it shall be verified to be operating properly prior to being placed back into service at ASB. SESDPROC-1009 details equipment management procedures.

4.3.2 Incubators Each incubator within ASB will be monitored by an automatic temperature recorder.

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

4.3.4 Refrigerators/Freezers/Drying Ovens

4.3.4.1 Check and document the temperature each working day that the refrigerator, freezer or drying oven is "in use". "In use" shall be defined as when the unit contains materials for which a specified temperature is required by method, policy, or procedure. Verification of operation within the correct temperature range may be documented in an alternate fashion if it can be demonstrated that the unit does not exceed its minimum or maximum permissible level (e.g., with a min/max temperature record). If a unit is not being used for this purpose, it should be so noted in the temperature record log and daily checks will not be necessary. If a piece of equipment never requires temperature checks, a sign will be placed on the unit stating it isn't used for maintaining required temperatures. In order to place a unit back into use, a current temperature measurement must be taken for verification that it is at the proper temperature. This must be documented in the temperature

recording log indicating that the unit has been placed back into active service and daily checks must resume. Alternatively, temperature checks for an apparatus which is not used on a daily basis (e.g., drying ovens for percent moisture determinations) may be recorded directly into analysis log(s). When an automatic temperature recorder is used, it is designated as the official record; any other thermometers in use are for convenient quick checks.

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

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

4.3.4.4 Do not store food in any laboratory refrigerator or freezer. Drying ovens should never be used to warm food or for drying eating utensils.

4.3.5 Autoclaves

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

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

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

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

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

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

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

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

4.3.6.2 Verification The verification should be documented in the appropriate analysis log. Weights are verified annually and should meet the specifications stated in SESDPROC-1011. This is required on an annual basis with re-certification coordinated by the LQM.

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

4.3.7 Thermometers Unless otherwise specified by regulatory methodology, it is the policy of ASB to use only non-mercury containing thermometers in all laboratory operations. All thermometers used within ASB will be NIST-traceable. Certification of thermometers is required on an annual basis and re-certification is coordinated by the LQM as detailed in SESDPROC-1011. Analytical equipment with built-in thermometers will have a specific procedure outlined in SESDPROC-1011 following the manufacturers' instructions for performing the calibration.

4.3.8 Mechanical Dispensing Devices

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

4.3.8.2 Autotitrator dispensing accuracy is verified through analytical quality control samples (e.g., laboratory control sample) and are not checked as mechanical

dispensing devices. The liquid is dispensed in microliter quantities and are too small to be accurately checked gravimetrically.

4.3.9 Records of NIST Traceability

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

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

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

4.3.10 Major Instrumentation

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

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

4.3.10.3 A list of all major instrumentation, including unique IDs, is maintained by the LQM on the LAN.

4.4 Laboratory Supplies

4.4.1 General

4.4.1.1 Laboratory supplies shall be maintained in an uncluttered, clean, and organized fashion. Supplies are monitored so that they are ordered before depletion

occurs which could cause work stoppages due to lack of supplies routinely kept in the laboratory. Supplies are pre-screened for suitability of use as detailed in ASB SOP 122G-Screening of Supplies.

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

4.4.1.3 A list of suppliers that have furnished acceptable supplies and services is maintained by the LQM, or designee on the LAN. Additional vendors may be added to this list if their supplies and services prove to be acceptable. The approved supplier list is evaluated quarterly, accreditations dates are updated, first-time use updated, unacceptable supplies noted and suppliers removed from the list, etc. Supply vendors that maintain ISO accreditation (and meet Guide 34 requirements for reference material producers) are placed on the acceptable supply list unless previous experience with the supplier has been unacceptable.

4.4.2 Labware

4.4.2.1 Labware used in laboratory operations must be high quality borosilicate glass, polymethylpentene, or Nalgene™ (Plastic). Volumetric Labware must be Class "A" quality.

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

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

4.4.3 Chemicals, Reagents, Solvents, Standards, Gases

4.4.3.1 The quality of chemicals, reagents, solvents and gases is determined by the sensitivity and specificity of the methods being used. Grades of materials for analyses of lesser purity than specified by a method will not be used. When specific grades of materials are not specified by the method, analytical reagent grade materials should be used. ASB will purchase standards from vendors with ISO 17025 and Guide 34 accreditation, if possible.

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

reagents, analytical system, or contamination introduced in the sample handling process.

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

4.4.3.1.3 If any consumables, supplies or services evaluated through the above procedures prove to be unsuitable for use, the personnel making that determination shall document the issue in an email to the LQM. 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 LQM. The LQM, or designee, will compile all occurrences of unsuitable consumables, supplies or services and determine what further action may be necessary.

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

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

4.4.3.3.1 All materials should have an expiration date recorded on the original container. For those materials received without a manufacturer's expiration date, an expiration date of 1 year from the date the container was initially opened will be applied to these materials; however, they should be monitored for deterioration and replaced if evidence of deterioration or contamination is present. Unopened containers will be evaluated based on the date the container is opened.

4.4.3.3.2 Materials prepared and used within the same day (or discarded the same day as prepared) are required to have identification of the contents on the container and HMIS labeling. Expiration dates may be documented on the container similar to 'Expires Daily' or 'Expires Today'.

4.4.3.3.3 Intermediate materials that are immediately consumed or promptly added to another labeled container do not need any identification. These intermediate preparations must be labeled if they are not consumed or added to the labeled container within 15 minutes of the preparation of the intermediate. The personnel making these intermediate preparations must have possession of the material and must label it if he or she leaves the material unattended. The use

of an intermediate standard or material to prepare a working standard or material must be documented in the appropriate preparation logbook or Element[®].

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

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

4.4.3.3.5 Expired Stock Standard It is ASB's policy to allow for reverification of analytical standards as described below. Verification 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 may also be verified prior to expiration.) Successful verification 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 verification, and adding the initials of the person who performed the verification. Depending on the size of the container and label there may not be room to add a new expiration date along with initials. In those cases, it may be necessary to add an additional sticker or label (firmly secured) with the new expiration date clearly marked. It is not necessary for a complete history of expiration dates to be on the container itself. The certificate of analysis (COA) must also include the new recertification date, the analyst's initials, the analysis with which the standard was recertified (i.e., the project number or other analysis identification) and the initials of the Section Chief or LQM to show he or she reviewed the verification information and concurs that the material is still stable. The COA must show a complete history of all recertifications. The revised COA is rescanned into Element[®].

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

4.4.3.3.6 Acceptance Criteria for Verifying Expired Calibration Standards

The stability of the expired calibration standard is considered to be verified if:

4.4.3.3.6.1 The ICAL prepared using the expired standard meets method

acceptance criteria and

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

4.4.3.3.6.3 If an expired standard material fails the verification test, it may be repeated. If it fails a second time, the expired standard material must be replaced or with the Section Chief's approval, failing analytes must be properly qualified.

4.4.3.4 Storage of large quantities of some chemicals is required in the Hazardous Materials (HAZMAT) Facility. This includes such items as concentrated acids and organic solvents. See the SHEMP for chemical storage procedures in the HAZMAT building.

4.4.4 Procurement of Chemicals and Chemical Inventories

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

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

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

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

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

4.4.5 Laboratory Pure Water

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

4.4.5.2 Change system modules annually, as recommended by the manufacturer, or more frequently as indicated by water quality. Modules should be labelled with the date of installation.

4.4.5.3 Water purity is verified by the analysis of laboratory blanks and is determined acceptable for specific analyses as prescribed in the individual technical SOPs. Metals analysis for drinking water requires ASTM Type I water.

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

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

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

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

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

4.5.4 Disposal of Outdated or Waste Chemicals/Chemical Containers It is the individual analyst's responsibility to ensure that all appropriate procedures are followed when disposing of outdated chemicals, chemicals that are no longer in use, or empty

containers of spent chemicals. As a general policy, no chemicals or solvents shall be disposed of by evaporation or by pouring down the sink, with the exception of dilute acid and bases that are accounted for in SESD's waste stream. The SHEM should be consulted to verify appropriate procedures.

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

4.5.6 Waste Minimization ASB 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 SESD has a recycling program for cardboard, aluminum cans, glass, mixed paper, Styrofoam and plastics. This accounts for a large amount of the total waste stream generated by ASB and SESD.

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

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

4.6 Laboratory Cleanliness

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

CHAPTER 5

Performance Quality and Data Handling

5.1 Introduction

Every component of environmental data acquisition from sample collection through final data reporting, has associated with it degrees of uncertainty. This laboratory does not attempt to quantify absolute uncertainty, since it includes both sampling and analytical error. The purpose of a laboratory quality assurance program is to determine when the analytical measurement uncertainty has exceeded acceptance limits for precision and bias, and to notify the end user of the exceedances. The operating procedures and quality control checks practiced in this laboratory and outlined in this manual are implemented to minimize the analytical error associated with data generation and to identify situations when the acceptance limits for precision and bias data quality indicators are not met. Analyses are performed in support of EPA Programs such as RCRA, Superfund, 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). Modifications may have been made to increase quality, efficiency, or to support specific requests of the various programs. Drinking water methods will not be modified or altered unless allowed by the method itself or approved under the alternative test procedure implemented by the Office of Water.

5.2 Terminology

5.2.1 Acceptance Criteria/Limits: specified limits placed on characteristics of a quality control 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; a data quality indicator.

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

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

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

to defined criteria.

5.2.6 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- μ m membrane filter. Feedwater to the final polishing step must have a maximum conductivity of 20 μ S/cm at 298°K (25°C), resistivity >18 M Ω -cm at 25°C, TOC <50 ppb, sodium <1 ppb, chloride <1 ppb, and total silica <3 ppb.

5.2.7 Audit: systematic evaluation to determine the conformance to quantitative and qualitative specifications of some operational function or activity.

5.2.8 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 field samples of the same matrix, meeting the above mentioned criteria. An analytical batch is composed of prepared field 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 field samples).

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

5.2.10 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.

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

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

5.2.10.3 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.

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

5.2.10.5 Method Blank: media similar to the batch of associated field 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 field samples through all steps of the preparation and analytical procedures.

5.2.10.6 Reagent Blank (method reagent blank): analyte-free media consisting of reagent(s), without the target analyte or sample matrix, introduced into the analytical procedure at the appropriate point and carried through all subsequent steps to determine the contribution of the reagents and involved analytical steps.

5.2.11 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.

5.2.12 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.

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

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

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

5.2.16 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.

5.2.17 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, the mode of collection, collector, time of collection, preservation and requested analyses.

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

5.2.19 Confirmation: verification of the identity of a component through the use of

an approach with a different scientific principle from the original method. These may include, but are not limited to: second column confirmation, alternate wavelength, derivatization, mass spectral interpretation, alternate detectors or additional cleanup procedures.

5.2.20 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.

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

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

5.2.23 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.

5.2.24 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., that they meet specified acceptance criteria).

5.2.25 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.

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

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

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

5.2.29 Dissolved: terminology used in analytical reporting referring to a field sample that has been filtered prior to preservation and arrival.

5.2.30 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.

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

5.2.32 Field of Accreditation Matrix: these matrix definitions shall be used when accrediting a laboratory (see Field of Accreditation).

5.2.32.1 Drinking Water: any aqueous sample that has been designated a potable or potential potable water source.

5.2.32.2 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.

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

5.2.32.4 Biological Tissue: any sample of a biological origin such as fish tissue, shellfish, or plant material. Such samples shall be grouped according to origin.

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

5.2.33 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 (i.e., 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.

5.2.34 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.

5.2.35 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 a method detection limit (MDL) determination and an evaluation of the minimum reporting limit (MRL), where applicable.

5.2.36 Internal Standard: known amount of standard added to a test portion of a

sample as a reference for evaluating and controlling the precision and bias of the applied analytical method.

5.2.37 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.

5.2.38 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.

5.2.39 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.

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

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

5.2.42 Marginal Exceedance (ME): term that is used to describe an LCS recovery that is beyond the LCS control limit (three standard deviations), but within ME limits which are between three and four standard deviations from the mean. Data is reviewed for exceedance of the marginal exceedance limits solely for the purpose of determining if the need for technical corrective action exists.

5.2.43 Matrix: substrate of a test sample.

5.2.44 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.

5.2.45 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.

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

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

5.2.48 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.

5.2.49 Method Detection Limit: minimum concentration of a substance (an analyte) that can be measured and reported with a 99% confidence that the analyte concentration is greater than zero and is determined from analysis of a sample in a given matrix containing the analyte.

5.2.50 Minimum Reporting Limit (MRL): concentration level below which the variance of the results for a particular analyte (element or compound) exceeds the acceptable quality control criteria. This value corresponds to the lowest quantitative point on the calibration curve or the lowest demonstrated level of acceptable quantitation. The MRL is sample-specific and accounts for preparation weights and volumes, dilutions, and moisture content of soil/sediments.

5.2.51 Must: denotes required action.

5.2.52 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).

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

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

5.2.55 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.

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

5.2.57 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.

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

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

5.2.60 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.

5.2.61 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, a quality system matrix fortified by spiking, or actual samples fortified by spiking.

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

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

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

5.2.65 Raw Data: any original factual information from a measurement activity or study recorded in a laboratory notebook, 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.

5.2.66 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.

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

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

derived.

5.2.69 Reporting Limit: also known as the Minimum Reporting Limit (MRL) in ASB.

5.2.70 Sample: particular 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 size or type of containers depending on the analytical and/or preservation requirements.

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

5.2.72 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.

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

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

5.2.75 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.

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

5.2.77 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)

5.2.78 Spike: known mass of target analyte added to a blank sample or sub-sample;

used to determine recovery efficiency or for other quality control purposes.

5.2.79 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.

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

5.2.81 Technical Corrective Action: any action taken to address instrument or quality control 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.

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

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

5.2.84 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.

5.2.85 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.

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

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

5.3 Essential Quality Control Requirements

5.3.1 Demonstration of Competency (DOC) ASB requires all analysts to demonstrate initial competency (prior to independent analysis of environmental samples) or with method or instrumental changes that could impact method performance. Extraction Laboratory analysts may participate in extraction procedures prior to completing a DOC provided they are performing the work with

another analyst with an active DOC for that extraction method. Analyst DOCs are specific to the method only, demonstrating the analyst's ability to successfully perform the method. Procedures for performing a DOC are detailed in SOP ASB 110G: Standard Operating Procedure for Initial Test Method Evaluations and Establishing Demonstrations of Competency and SESD 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. Detailed procedures for documenting an analysts' CDOP are detailed in SOP ASB 110G and SESD PROC-1003.

5.3.2 MDL Studies ASB performs MDL studies as part of the ITME and upon change in instrumentation; they are verified on an ongoing basis, in accordance with 40CFR Part 136 Appendix B as detailed in ASB SOP 119G. ASB only reports non-detects at the MDL by special request.

5.3.3 Instrument Calibration

5.3.3.1 Initial Calibration Curve (ICAL) All instrumentation utilized in the preparation or analysis of environmental samples will be calibrated prior to use. The calibration curve shall bracket the range of expected concentrations for the analytes being evaluated. Calibration frequency and acceptance criteria will follow method and/or technical SOP requirements. Calibration standards shall be prepared using the same, or equivalent, type of acid or solvent and at the expected concentration as the samples following sample preparation. ASB requires purchased standards to be prepared in accordance with ISO guide 34 specifications. Traceability shall be to a national standard, when commercially available.

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.

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

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

5.3.4 Acceptance Criteria All methods in use must have acceptance criteria against which all QC results are evaluated. When method-specific acceptance criteria are not specified or available, in-house acceptance criteria must be developed using a minimum of 20 results, but no more than 30 results. After the initial limits are determined, they should be updated again as needed or as soon as practical. It is the policy of ASB that bias and precision limits are set at three standard deviations from the mean of the dataset. Technical SOPs will detail the acceptance criteria for all applicable QC elements required by the published method. Interim limits for bias and precision 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.

5.3.4.1 Setting Interim Bias (Recovery) Limits ASB 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 should be set at 85-115% and limits for organic analyses at 70-130%.

5.3.4.2 Setting Interim Precision Limits Interim limits for precision are set at an RPD or RSD of 20. At the discretion of the Technical Director or LQM, interim RPD limits of 50 may be set for complex matrices such as soil, tissue or waste.

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

5.3.6 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, ASB requires an LCS to be carried through the entire analytical process. ASB uses the LCS to assess the performance of all or a portion of the measurement system. ASB also assesses the LCS results for organic analysis as a mechanism for determining if technical corrective action is through the use of marginal exceedances. A marginal exceedance report is generated from Element[®] and included with each project file. The report identifies any compounds

in the LCS standard that exceed 4 standard deviations of the mean % recovery. Action is required based on the number of exceedances versus the number of compounds spike din the LCS standard as follows:

- *Greater than 90 analytes in LCS, 5 analytes allowed within ME limits
- *From 71-90 analytes within LCS, 4 analytes allowed within the ME limits
- *From 51-70 analytes within LCS, 3 analytes allowed within the ME limits
- *From 31-50 analytes within LCS, 2 analytes allowed within the ME limits
- *From 11-30 analytes within LCS, 1 analytes allowed within the ME limits
- *Less than 11 analytes in LCS, no analytes allowed within ME limits

5.3.7 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 reference 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. 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 particular matrix in question. ASB uses the acceptance criteria for evaluating a MS as defined in the published method. If no acceptance criteria are provided, QC limits will be established using historical data. ASB does not qualify any batch results based on the MS analysis. Only the sample spiked is qualified if QC results are outside of the MS limits for that sample.

5.3.8 ASB expresses bias as percent recovery (%R) and calculated bias for both LCS and matrix spikes using the following formulas:

Bias

Spike Reference

$$\% R = \frac{Z-X}{T} (100)$$

Or

Reference Materials

$$\% R = \frac{Y}{T} (100)$$

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.

5.3.9 Surrogate Spike Recovery of the surrogate standard is used to monitor for unusual 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. Acceptance limits are defined by the technical SOP. Surrogate recoveries are compared to the method-specified acceptance limits within Element[®].

5.3.10 Proficiency Test Sample (PT sample) ASB will participate in independent Proficiency Testing Studies as required for accreditation or more often as deemed necessary by ASB management or the LQM. Performance in these studies further indicates the effectiveness of the laboratory's day-to-day quality control procedure. ASB'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. 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 that calendar year. 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.

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

5.3.12 Minimum Reporting Limit (MRL) Verification Standard A standard at or near (0.5X- 2X) the MRL that has been processed through all steps (preparation and analysis) of the method used to verify the performance of the measurement system at the lower end of the calibration curve.

5.3.13 Precision Refers to the level of agreement among repeated measurements of the same analyte or property. Results may be compared to historical ASB limits or the acceptance criteria in the published method. Precision is expressed as relative percent difference (RPD).

5.3.14 Matrix Duplicate Analyses At a minimum, either a matrix duplicate or MS duplicate (see below) shall be prepared with each batch of up to 20 environmental samples as required by the published method, where the method is amenable to spiking. The results from matrix duplicates are designed to assess the precision of analytical results in a given matrix. ASB does not qualify any batch results based on the matrix duplicate analysis. Only the sample which was duplicated is flagged if QC results are outside of the matrix duplicate limits for that sample.

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

5.3.16 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. ASB does not qualify any batch results based on the MSD analysis. Only the sample which was spiked is flagged if QC results are outside of the matrix spike duplicate limits for that sample.

5.3.17 Internal Standards ASB 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. Internal standards are added to all calibration standards and QC samples (method blank, MS/MSD, LCS/LCSD, MRL verification) at the same concentration as the samples following preparation. The acceptance criteria in the method will be observed.

5.3.18 Bottle blanks, equipment rinse blanks and other in-house QC analyzed for the field branches and supply screening will be performed with a reduced level of QC due to the nature of the matrix which is reagent water.

5.4 Data Handling

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

5.4.2 Minimum Reporting Limit (MRL) ASB includes a standard at the MRL as the lowest standard in the calibration curve. or, where applicable (i.e., ICP analysis where the calibration curve consists of a blank and a high level standard), analyzes a low level check standard at the MRL following calibration. Results are considered to be within acceptable quantitative accuracy if analyses are performed within the appropriate quantitation range as defined by the calibration curve. Results reported outside these limits will be qualified with the “J”-flag or otherwise as appropriate. A remark describing the reason for the qualifier will be added to the report.

5.4.3 Reporting data between the MDL and MRL As a matter of routine practice, ASB’s reporting level policy is as follows.

5.4.3.1 Organic Chromatographic/Mass Spectral Data Because chromatographic/mass spectral analyses use both retention time and a spectral match, there is qualitative evidence for the presence of target analytes at

concentrations between the MDL and the MRL.

5.4.3.1.1 Non-detects are reported to the MRL.

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

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

5.4.3.2 All other data

5.4.3.2.1 Non-detects are reported to the MRL.

5.4.3.2.2 Any detects between the MDL and the MRL are reported as less than the MRL.

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

5.4.4 Units

5.4.4.1 Sediment/Soil All soil/sediment samples shall be reported on a dry-weight basis unless otherwise specified by the published method or technical SOP. Soil/sediment samples are reported in mg/kg or $\mu\text{g}/\text{kg}$ units.

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

5.4.4.3 RCRA Wastewaters 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.

5.4.4.4 Tissue samples Reported on a wet-weight basis unless otherwise specified by the sample requestor. Tissue samples are reported in mg/kg or $\mu\text{g}/\text{kg}$ units.

5.4.4.5 Water sample Water samples include groundwater, surface water, potable water, etc. and are reported in $\mu\text{g}/\text{L}$ or mg/L.

5.4.4.6 Air sample Air samples including VOA samples are reported in ppbv or $\mu\text{g}/\text{m}^3$.

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

5.4.6 Rounding Rules

5.4.6.1 Manual Rounding Where manual data entry is performed, ASB will round entries 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.

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

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

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

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

5.4.7.1.1 Known and documented laboratory error and

5.4.7.1.2 Use of an appropriate statistical outlier test.

5.4.7.2 Standard deviation from the mean - typically useful for large data sets

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

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

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

5.4.7.3.2 Calculate the ratio

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

5.4.7.3.3 Apply the following decision rule.

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

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

5.4.7.3.4 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.

5.4.7.4 Dixon's Q test

5.4.7.4.1 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.

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

5.4.7.4.3 Calculate the range of the entire data set including the suspect value,

which is one of the extreme values.

5.4.7.4.4 Calculate the value of Q :

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

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

5.4.7.4.5 Apply the following decision rule:

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

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

5.4.7.4.5.3 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:

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

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. $Q_{\text{calc}} > Q_{\text{critical}}$). Thus, the suspect value is an outlier and should be removed.

5.4.8 Uncertainty Where available, ASB utilizes well-recognized test methods which specify limits to major sources of uncertainty (e.g., a balance accurate to ± 0.1 g) and provide data reporting instructions so that the reported results do not give the wrong impression of the uncertainty. ASB provides customers QC data with each final report (both specific batch QC and acceptance criteria) to communicate 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, such as when it is relevant to the validity of the test result, when requested by the customer or when the uncertainty may affect compliance to a regulatory limit.

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

5.4.9.1 Estimation of Uncertainty using Laboratory Control Samples (adapted from: Georgian, 2000, Environmental Testing and Analysis). This method uses the limits of historical LCS data to estimate results to a 99% confidence interval using the following equation:

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

Where: c = measured concentration of the analyte

L = the half width of the control range, that is, $(\text{UCL}-\text{LCL})/2$

\bar{R} = mean historical LCS recovery

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

5.4.9.2 Standard Methods 1030B Measurement Uncertainty

5.5 Data Reporting

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

5.5.1 Analytical Data Qualifiers Added to data in an effort 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.

5.5.2 Report Narrative Additional explanatory remarks about the data can be added

by the Section Chief (or designee) in the Report Narrative section of the data report. Analysts will add any necessary explanatory remarks about their analyses in the ‘Work Order Notes’ section of Element[®] and the Section Chief (or designee) will summarize any pertinent information that needs to be transmitted to the data user in the final report through the report narrative.

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

5.5.3 Chemical Abstract Service (CAS) Registry Numbers and EPA Identifiers (EPA ID) Each analyte reported from Element[®] is also reported with the analyte’s corresponding CAS number. For some analytes reported by ASB (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>. ASB 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.

5.5.4 Opinions and Interpretations ASB rarely, if ever, offers opinions and interpretations of the reported data. However, if included with a laboratory report, the basis upon which the opinion or interpretation was made shall be included in the report. Any opinions or interpretations shall be clearly marked as such.

5.5.5 Demonstration of MRL or sample calculation ASB will demonstrate one MRL or sample calculation per batch of samples as part of the data review and validation procedure.

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

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

5.6 Data Management and Data Security

5.6.1 Data is managed using both R4LIMS and Element[®]. R4LIMS is used for project

scheduling and Element[®] is used for analytical data management. R4LIMS is an in-house developed Sybase PowerBuilder[®] application. All data is stored in an Oracle database residing on an SESD Windows 2003 Server. Console-level access to the Oracle Server is limited to the SESD LAN Administrator, the Region 4 LAN Administrator, and the R4LIMS Database Administrator (DBA) who is an SESD computer specialist responsible for R4LIMS application development and database administration.

5.6.1.1 Backups of the Oracle database (and the entire LAN) to magnetic tape are performed Monday through Saturday evenings using a redundant network backup system. One backup is conducted remotely from the ERD computer center and another locally from the SESD computer center. After successful backups, the daily tapes located at SESD are placed in a fire-proof media safe and a copy of the Friday evening backup is rotated to the Atlanta EPA office for offsite storage. Detailed backup procedures can be Backups of the Oracle database (and the entire LAN) to magnetic tape are performed Monday through Saturday evenings using a redundant network backup system. One backup is conducted remotely from the ERD computer center and another locally from the SESD computer center. After successful backups, the daily tapes located at SESD are placed in a fire-proof media safe and a copy of the Friday evening backup is rotated to the Atlanta EPA office for offsite storage.

5.6.1.2 Detailed backup procedures can be found in the ‘ADP Disaster Recovery Plan for Region 4’ dated June 10, 2004 (and any future updates). The custodian of this document is the Region 4 Information Security Officer in the Atlanta office. An electronic copy is available from the Athens LAN administrator, and a hard copy is located in the safe in room B107.

5.6.2 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. The Contract Programmer only has rights to a development database and not the active R4LIMS database. The SESD LAN Administrator and R4LIMS DBA have unrestricted rights to the database.

5.6.2.1 End-user access to the database is controlled through the compiled R4LIMS Powerbuilder application, Element[®] DataSystem and the Adobe/Macromedia Coldfusion[®] web server (currently limited to read-only access of “public” data).

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

5.6.2.1.2 Access to the EPA network and an account in R4LIMS or Element[®] is required for access to data for entry or reporting purposes. Rights are assigned to

each R4LIMS or Element[®] user upon request by their supervisor. Telephone requests will not be accepted. Rights are assigned by the ASB R4LIMS coordinator, the SESD LAN Administrator, or the R4LIMS DBA.

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

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

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

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

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

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

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

5.7 Annual Analytical Performance Summary

Control charts for each analysis are compiled and reviewed on a quarterly basis, as applicable, and any significant trends are documented in the LQM's Quarterly QA Report. Review of the control charts may initiate either Corrective Actions or Preventive Actions as appropriate. On an annual basis, the LQM will note any highlights from the Quarterly Reports as well as document all methods for which limits and/or MDLs have been updated.

5.8 QC Study Plans

5.8.1 A QC Study Plan is developed when planning a non-routine MDL/DOC study,

developing a new method, evaluating a new/modified analytical method, or addressing a non-routine QC issue/problem for corrective action. The most recent form for documenting this QC Study Plan is located in a subdirectory on the SESD LAN. For routine studies with a limited focus, a QC Study Plan is not required. The decision to develop a QC Study Plan will be made by the project leader in consultation with the LQM and Section Chief.

5.8.2 For studies requiring a QC Study Plan, the appropriate analysts will convene to discuss the issue, define the objective(s), and develop the study procedure. The LQM may be involved in the study planning depending on the nature and complexity of the issue. The final QC Study Plan will be approved by the LQM, Section Chief, and where appropriate, peer-reviewed by other analysts. A report summarizing the study results will be prepared for the LQM's comments as needed. At times, it may be appropriate to update a Study Plan as the study progresses. Changes should be communicated to all participants as needed.

5.9 Complaints/Inquiries

All complaints shall be reviewed by management. Those which are identified as departures from ASB's policies or procedures will enter the corrective action process. All others will be considered as opportunities for improvement. The customer will be informed of the resolution, which shall be maintained by the LQM.

5.10 Corrective Actions

ASB requires resolution of non-conforming work through the corrective action process. The process will include a root cause analysis. Corrective actions can be initiated by any staff member; however, it is the responsibility of the LQM to track, monitor and perform any follow-up action needed in relation to the corrective action. The corrective action procedure is detailed in SESDPROC-1006- Complaint Resolution and Control of Non-Conforming Work.

5.11 Preventive Actions and Improvements

Preventive Actions consist of proactive processes to prevent problems or complaints and are used as opportunities for improvement. The preventive action procedure is detailed in SESDPROC-1006.

5.12 Control of Nonconforming Work

ASB mitigates nonconforming work through the 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 SESDPROC-1006

5.13 Annual Management Review

ASB conducts an annual Management Review, where the effectiveness and conformance to the accreditation standards of the quality management system are assessed and reported to upper level Divisional management. The review also provides an opportunity to plan for any needed improvements to the quality system. The review is documented and maintained by the LQM and covers the ASB's overall quality objectives, to include at a minimum the items outlined in the ISO 17025 standard, Section 4.15.

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

¹ Pearson, E. S.; Hartley, H.O., Eds, *Biometrika Tables for Statisticians*, Vol. I, 3rd ed., Cambridge University Press, London, 1966.

TABLE 5-2	
Critical values of the Q in Dixon's Q -test for testing whether a single point should be rejected as an outlier ($\alpha = 0.05$, 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
¹ Rorabacher, D. B., "Statistical treatment for rejection of deviant values of Dixon's 'Q' parameter and related sub-range ratios at the 95% confidence level," <i>Anal. Chem.</i> 1991 , 63, 139-146	

CHAPTER 6

Methodology

6.1 General

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

6.2 Method Information

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

6.3 Minimum Reporting Limits

Reporting units and MRL tables for routine target analytes analyzed by ASB are maintained within Element[®] for each matrix and method. The metals, classical/nutrients, volatiles, semivolatiles, pesticides/PCBs and perfluorinated compounds MRL values are summarized in Tables 6-3 through 6-11 respectively of this chapter. 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 ASB Section Chief(s). The MRLs listed in the tables are those which are routinely achievable. However, sample-specific MRLs may be higher or lower. Some factors which may influence MRLs are listed below.

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

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

6.3.3 Solid samples corrected for percent moisture content and reported on a dry-weight basis will have higher MRLs.

6.4 Land Disposal Restrictions (LDR)

6.4.1 During field investigations for the Resource Conservation and Recovery Act (RCRA) program, samples may be collected and analyses requested to determine whether the medium being sampled meets the treatment standards under LDR. The RCRA LDR program is intended to ensure that hazardous waste cannot be placed on the land until the waste meets specific treatment standards to reduce the mobility or toxicity of its hazardous constituents. Requirements are covered in 40 CFR Part 268 and are quite complex. Analyses supporting the LDR regulations must meet certain MRLs in order to demonstrate whether the sample being tested has met the applicable treatment standard. The levels of concern for LDR regulations are presented in Figure 6-1.

6.4.2 When placing requests for LDR, sufficient lead-time (a minimum of 30 days) will be needed. LDR analyses require special reporting conventions that are not routine for ASB'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 ASB Section Chiefs when planning such projects.

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

Table 6-1 Levels of Concern for Various Programs

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

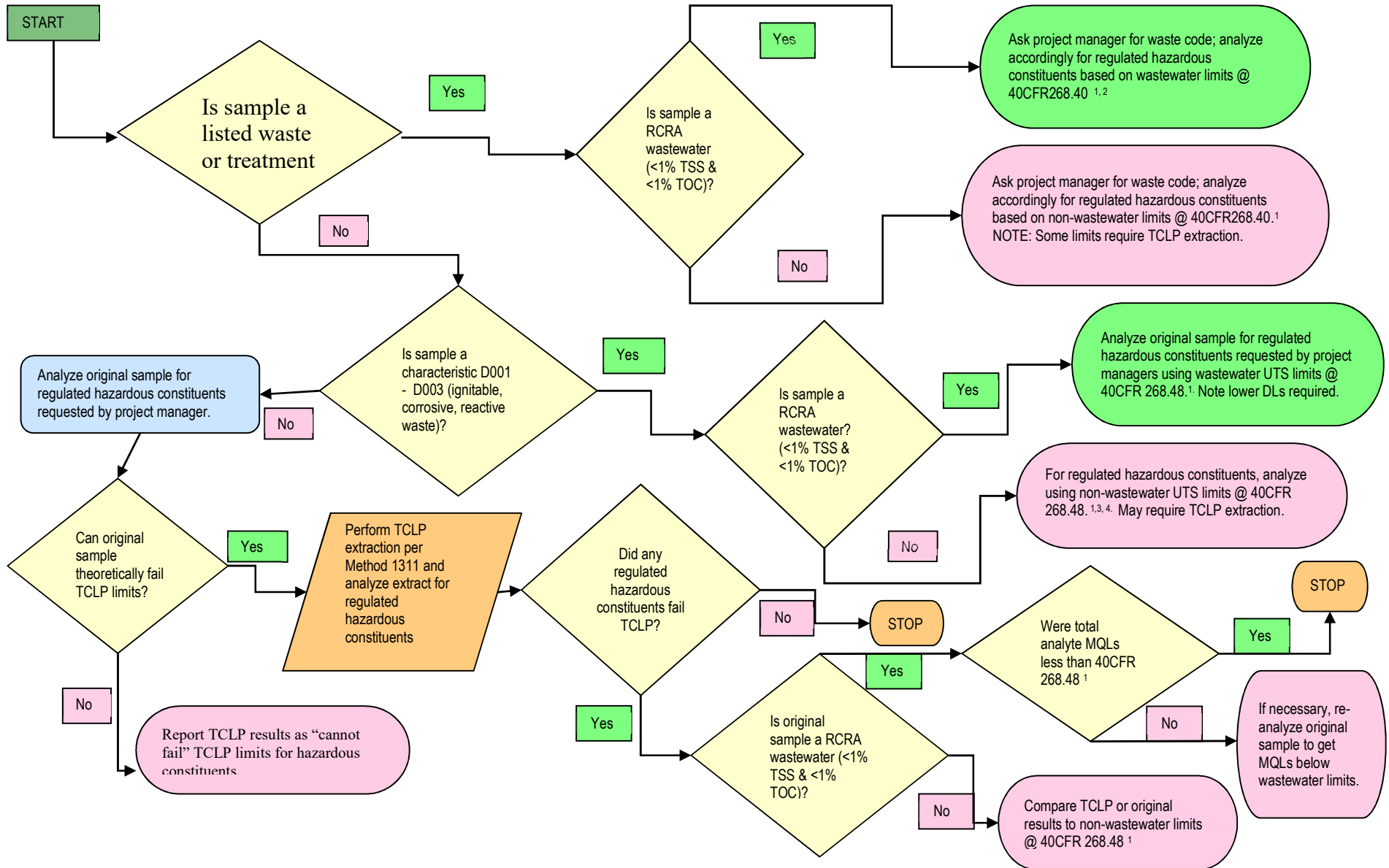


Figure 6-1 Decision Tree for Analysis of Land Disposal Restrictions

Footnotes for Figure 6-1

¹ See SESD LAN Directory K:\ASB\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

ASB LOQAM Chapter 6 Table 6-2 Capability for Potable Waters-Inorganics					
SDWA Analyte	SDWA MCL (mg/L)	SDWA Method used by ASB	ASB SDWA MRL (mg/L)	ASB Routine Method	ASB MRL for routine low level request (mg/L)
Aluminum (secondary)	0.05 – 0.2				
Antimony	0.006	200.8	0.0005	200.8	0.0005
Arsenic	0.010	200.8	0.0005	200.8	0.0005
Barium	2	200.7 or 200.8	0.005	200.7 or 200.8	0.005
Beryllium	0.004	200.7 or 200.8	0.003	200.7 or 200.8	0.003
Cadmium	0.005	200.7 or 200.8	0.005	200.7 or 200.8	0.005
Copper (secondary)	1.0	200.7 or 200.8	0.01	200.7 or 200.8	0.01
Chloride (secondary)	250	300.0		300.0	
Chromium (total)	0.1	200.7 or 200.8	0.005	200.7 or 200.8	0.005
Lead	0.015 ³	200.8	0.0005	200.8	0.0005
Iron (secondary)	0.3				
Manganese (secondary)	0.05				
Mercury (inorganic)	0.002	200.8 or 245.1	0.0004	200.8 or 245.1	0.0001
Selenium	0.05	200.8	0.001	200.8	0.001
Silver (secondary)	0.1				
Thallium	0.002	200.8	0.0005	200.8	0.0005
Zinc (secondary)	5				
Sulfate (secondary)	250				
Asbestos	7MF/L>10u	NA ²	NA ²	NA ²	NA ²
Bromate	0.010	NA ²	NA ²	NA ²	NA ²
Chlorite	1.0	NA ²	NA ²	NA ²	NA ²
Residual Disinfectant	detectable	NA ²	NA ²	NA ²	NA ²
Fluoride (secondary)	2.0	300.0	0.05	300.0	0.05
Nitrate, as N	10	353.2	0.05	300.0 or 353.2	0.05
Nitrite, as N	1	353.2	0.05	300.0 or 353.2	0.05
Total dissolved solids (secondary)	500				
pH	6.5-8.5 ⁴	NA ¹	NA ¹	9040C	1.0 ⁴
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 ASB. 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-ASB does not perform this analysis.					
⁴ The units of the reported numbers are in pH standard units. NA- Not Available-ASB does not perform this analysis.					

Table 6-3

ASB LOQAM Chapter 6 Table 6-3 Capability for Potable Waters - Organics					
SDWA Analyte	SDWA MCL (mg/L)	SDWA Method (special request)	ASB SDWA MRL (mg/L)	ASB Routine Low-Level Method	ASB 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
2,4-D	0.07	NA ¹	NA ¹	8321B	0.000025
Benzo[a]pyrene	0.0002	525.2	0.0002	8270D SIM ³	0.0001
Carbofuran	0.04	NA ²	NA ²	NA ²	NA ²
Chlordane	0.002	NA ¹	NA ¹	8081B ³	0.0015
Dalapon	0.2	NA ¹	NA ¹	8321B	0.0000125
bis(2-ethylhexyl)adipate	0.4	525.2	0.001	NA	NA
bis(2-ethylhexyl)phthalate	0.006	525.2	0.001	8270D	0.006
Dibromochloropropane (DBCP)	0.0002	NA ¹	NA ¹	8011/8260C ³	0.00005
Dinoseb	0.007	NA ¹	NA ¹	8321B	0.0000125
Diquat	0.02	NA ²	NA ²	NA ²	NA ²
Endothall	0.1	NA ²	NA ²	NA ²	NA ²
Endrin	0.002	525.2	0.002	8081B	0.00005

ASB LOQAM Chapter 6 Table 6-3 Capability for Potable Waters - Organics					
SDWA Analyte	SDWA MCL (mg/L)	SDWA Method (special request)	ASB SDWA MRL (mg/L)	ASB Routine Low-Level Method	ASB MRL for routine low-level request (mg/L)
Ethylene dibromide (EDB)	0.00005	NA ¹	NA ¹	8260C ³	0.00005
Glyphosate	0.7	NA ²	NA ²	NA ²	NA ²
Heptachlor	0.0004	525.2	0.0004	8081B	0.00005
Heptachlor Epoxide	0.0002	525.2	0.0002	8081B	0.00005
Hexachlorobenzene	0.001	525.2	0.001	8270D	0.001
Hexachlorocyclopentadiene	0.05	NA ¹	NA ¹	8270D	0.05
Lindane (gamma-BHC)	0.0002	525.2	0.0002	8081B	0.00005
Methoxychlor	0.04	525.2	0.015	8081B	0.0002
Oxamyl (Vydate)	0.2	NA ²	NA ²	NA ²	NA ²
PCBs (as Decachlorobiphenyl)	0.0005	NA ²	NA ²	8082-Aroclors	0.0005
Pentachlorophenol	0.001	NA ¹	NA ¹	8270D	0.0001
Picloram	0.5	NA ¹	NA ¹	8321B	0.0000125
Simazine	0.004	NA ²	NA ²	NA ²	NA ²
2,4,5-TP (Silvex)	0.05	NA ¹	NA ¹	8321B	0.0000125
Toxaphene	0.003	NA ¹	NA ¹	8081B	0.002
HAA5	0.060	NA ²	NA ²	NA ²	NA ²
Actual MRL may be higher due to variability of analytical instrument conditions or sample interferences.					
¹ Not available from ASB using SDWA Method. Please contact Organic Chemistry Section Chief for more information.					
² Not available from ASB. Please contact Organic Chemistry Section Chief for options.					
³ Analysis available upon request with sufficient lead-time.					
NA - Not Available-ASB does not perform this analysis.					

Table 6-4

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

ASB LOQAM Chapter 6 Table 6-4 Metals Analyte List Minimum Reporting Limits by Matrices					
ANALYTE	ASB Routine Analytical Method ⁴	Water µg/L (ppb) ³	Soil/Sed mg/kg (ppm) ^{1,3}	Waste mg/kg (ppm) ¹	Tissue mg/kg (ppm) ^{2,3}
Titanium	EPA 6010C	5.0	0.5	0.5	0.1
Thallium	EPA 200.8	0.5	0.05	0.05	0.01
Vanadium	EPA 6010C	5.0	0.5	0.5	0.1
Yttrium	EPA 6010C	3.0	0.3	0.3	0.06
Zinc	EPA 6010C	10	1.0	1.0	0.2
Boron **	EPA 6010C	50	5.0	5.0	1.0
Silicon **					
Uranium **					

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

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

² Reporting limits are based on 5.0 g of sample.

³ Units as specified unless otherwise noted.

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

⁵ Mercury methods - Water: 245.1/200.8; Soil, Waste, and Tissue: 7473

NA-Not Available-ASB 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 ASB personnel.

Table 6-5

ASB LOQAM Chapter 6 Table 6-5 Nutrients and Classical Analyte List Minimum Reporting Limits by Matrices					
ANALYTE	Analytical Method ⁵	Water mg/L (ppm) ¹	Soil/Sed mg/kg (ppm)	Waste mg/kg (ppm)	Tissue mg/kg (ppm)
Acidity	SM 2310	10	NA	NA	NA
Alkalinity	SM 2320B	1.0	NA	NA	NA
Ammonia	EPA 350.1	0.05	2.5 ²	2.5 ²	NA
BOD/C-BOD	SM 5210B	2.0	NA	NA	NA
Bromide	EPA 300.0	0.1	1.0	NA	NA
Chloride	EPA 300.0	0.1	1.0	NA	NA
Cyanide	SM 335.4	0.015	0.75	0.75	NA
Fluoride	EPA 300.0	0.05	0.5	NA	NA
Hardness, Calc	SM 2340B	1.654	NA	NA	NA
Nitrate	EPA 300.0/EPA 353.2	0.05	0.5	0.5	NA
Nitrite	EPA 300.0/EPA 353.2	0.05	0.5	0.5	NA
Nitrate+Nitrite	EPA 353.2	0.05	0.5	0.5	NA
pH	EPA 9040/EPA 9045	1.0 pH units	1.0 pH units	1.0 pH units	1.0 pH units
Phosphorus, Total	EPA 365.1	0.01	1.25 ⁴	1.25 ⁴	NA
Phosphorus, Ortho	EPA 365.1	0.01	1.25 ⁴	1.25 ⁴	NA
Total Dissolved Solids	USGS I-1750-85	40	NA	NA	NA
Total Solids	SM 2540B-1997	40	NA	NA	NA
Total Suspended Solids	USGS I-3765-85	4.0	NA	NA	NA
Volatile Solids	SM 2540 E	4.0/40 ⁷	NA	NA	NA
Sulfate	EPA 300.0	0.1	1.0	NA	NA
Total Kjeldahl Nitrogen (TKN)	EPA 351.2	0.05	6.25 ⁴	6.25 ⁴	NA
Total Organic Carbon (TOC)	SM5310/ASB 107C	1.0	12,000	NA	NA
<p>MRLs may increase due to variability of interferences that make dilutions of sample necessary. Sample sizes required for achieving the routine quantitation limits are listed below.</p> <p>¹ Units as specified unless otherwise noted.</p> <p>² Calculated using 1.0 g of sample (dry-weight basis, % moisture will increase MRLs).</p> <p>³ Calculated using 5.0 g of sample (dry-weight basis, % moisture will increase MRLs).</p> <p>⁴ Calculated using 0.2 g of sample (dry-weight basis, % moisture will increase MRLs).</p> <p>⁵ Routine methods may be changed at the time of analysis due to sample specific characteristics. The actual analytical method used will be listed on the final report.</p> <p>⁶ Analysis available upon request with sufficient lead-time.</p> <p>⁷ MRL for volatile solids for the TSS method is 4.0 mg/L; if it is derived from the TDS method, then the MRL is 40 mg/L.</p> <p>NA- Not Available-ASB does not perform this analysis.</p>					

Table 6-6

ASB LOQAM Chapter 6 Table 6-6					
Volatile Organics (VOAs) Target Analyte List					
Minimum Reporting Limits (MRLs) by Matrices					
		Water ¹ µg/L (ppb)	Soil/Sed ² µg/kg (ppb)	Waste ³ mg/kg (ppm)	Air ^{4,8} ppbv (µg/m ³)
ANALYTE	Analytical Method	Routine Level	Routine Level (Encore®/Tared Vial)	Routine Level	Routine Level EPA TO-15 ⁶
Acetone	EPA 8260C	4.0-10	10-20	1.6-4.0	0.22
Acrylonitrile	EPA 8260C	NA	NA	NA	0.0184
Benzene	EPA 8260C EPA 8260C SIM	0.50 0.05	1.0	0.20	0.044
Benzyl Chloride	EPA 8260C	NA	NA	NA	0.22
Bromobenzene	EPA 8260C EPA 8260C SIM	0.50 0.050	1.0	0.20	NA
Bromochloromethane	EPA 8260C EPA 8260C SIM	0.50 0.015	1.0	0.20	NA
Bromodichloromethane	EPA 8260C EPA 8260C SIM	0.50 0.015	1.0	0.20	0.0101
Bromoform	EPA 8260C EPA 8260C SIM	1.0-4.0 0.10	2.0-10	0.40-1.6	0.011
Bromomethane	EPA 8260C	2.0-5.0	2.0	0.80-2.0	0.0220
1,3-Butadiene	EPA 8260C	NA	NA	NA	0.0440
n-Butylbenzene	EPA 8260C EPA 8260C SIM	0.50 0.05	1.0	0.20	NA
sec-Butylbenzene	EPA 8260C EPA 8260C SIM	0.50 0.05	1.0	0.20	NA
tert-Butylbenzene	EPA 8260C EPA 8260C SIM	0.50 0.05	1.0-2.0	0.20	NA
Carbon Tetrachloride	EPA 8260C EPA 8260C SIM	0.50 0.05	1.0	0.20	0.0220
Carbon Disulfide	EPA 8260C	2.0	2.0	0.80	0.22
Chlorobenzene	EPA 8260C EPA 8260C SIM	0.50 0.05	1.0	0.20	0.022

ASB LOQAM Chapter 6 Table 6-6					
Volatile Organics (VOAs) Target Analyte List					
Minimum Reporting Limits (MRLs) by Matrices					
		Water¹ µg/L (ppb)	Soil/Sed² µg/kg (ppb)	Waste³ mg/kg (ppm)	Air^{4, 8} ppbv (µg/m³)
ANALYTE	Analytical Method	Routine Level	Routine Level (Encore®/Tared Vial)	Routine Level	Routine Level EPA TO-15⁶
Chloroethane	EPA 8260C EPA 8260C SIM	2.0-5.0 0.05	2.0	0.80-2.0	0.022
Chloroform	EPA 8260C EPA 8260C SIM	0.50 0.015	1.0	0.20	0.022
Chloromethane	EPA 8260C	0.50	1.0	0.20	0.044
3-Chloroprene (2-Chloro- 1,3-butadiene)	EPA 8260C	NA	NA	NA	0.044
o-Chlorotoluene	EPA 8260C EPA 8260C SIM	0.50 0.05	1.0	0.20	NA
p-Chlorotoluene	EPA 8260C EPA 8260C SIM	0.50 0.05	1.0	0.20	NA
Cyclohexane	EPA 8260C EPA 8260C SIM	0.50 0.05	1.0	0.20	0.20
Dibromochloromethane	EPA 8260C EPA 8260C SIM	0.50 0.015	1.0	0.20	0.20
1,2-Dibromo-3- chloropropane ⁷ (DBCP)	EPA 8260C EPA 8260C SIM	1.0-10 0.10	2.0-10	0.40-4.0	NA
1,2-Dibromoethane (EDB) ⁷	EPA 8260C EPA 8260C SIM	1.0 0.025	1.0	0.20	0.022
Dibromomethane	EPA 8260C EPA 8260C SIM	0.50 0.05	1.0	0.20	NA
1,2-Dichlorobenzene	EPA 8260C EPA 8260C SIM	0.50 0.05	1.0	0.20	0.020
1,3-Dichlorobenzene	EPA 8260C EPA 8260C SIM	0.50 0.05	1.0	0.20	0.022
1,4-Dichlorobenzene	EPA 8260C EPA 8260C SIM	0.50 0.05	1.0	0.20	0.020

ASB LOQAM Chapter 6 Table 6-6					
Volatile Organics (VOAs) Target Analyte List					
Minimum Reporting Limits (MRLs) by Matrices					
		Water¹ µg/L (ppb)	Soil/Sed² µg/kg (ppb)	Waste³ mg/kg (ppm)	Air^{4, 8} ppbv (µg/m³)
ANALYTE	Analytical Method	Routine Level	Routine Level (Encore®/Tared Vial)	Routine Level	Routine Level EPA TO-15⁶
Dichlorodifluoromethane (R12)	EPA 8260C EPA 8260C SIM	0.50 0.015	1.0	0.20	0.022
1,1-Dichloroethene ⁷	EPA 8260C EPA 8260C SIM	0.50 0.015	1.0	0.20	0.022
cis-1,2-Dichloroethene	EPA 8260C EPA 8260C SIM	0.50 0.015	1.0	0.20	0.022
trans-1,2-Dichloroethene ⁷	EPA 8260C EPA 8260C SIM	0.50 0.015	1.0	0.20	0.020
1,1-Dichloroethane ⁷	EPA 8260C EPA 8260C SIM	0.50 0.015	1.0	0.20	0.022
1,2-Dichloroethane ⁷	EPA 8260C EPA 8260C SIM	0.50 0.015	1.0	0.20	0.022
1,2-Dichloropropane	EPA 8260C EPA 8260C SIM	0.50 0.015	1.0	0.20	0.022
1,3-Dichloropropane	EPA 8260C EPA 8260C SIM	0.50 0.015	1.0	0.20	NA
2,2-Dichloropropane	EPA 8260C EPA 8260C SIM	0.50 0.050	1.0	0.20	NA
1,1-Dichloropropene	EPA 8260C EPA 8260C SIM	0.50 0.015	1.0	0.20	NA
cis-1,3-Dichloropropene	EPA 8260C EPA 8260C SIM	0.50 0.05	1.0	0.20	0.020
Dichlorotetrafluoroethane (R114)	EPA TO-15	NA	NA	NA	0.022
trans-1,3-Dichloropropene	EPA 8260C EPA 8260C SIM	0.50 0.050	1.0	0.20	0.020
1,4-Dioxane	EPA TO-15	NA	NA	NA	0.24
Ethyl acetate	EPA TO-15	NA	NA	NA	0.022
Ethyl benzene	EPA 8260C EPA 8260C SIM	0.50 0.05	1.0	0.20	0.022

ASB LOQAM Chapter 6 Table 6-6					
Volatile Organics (VOAs) Target Analyte List					
Minimum Reporting Limits (MRLs) by Matrices					
		Water¹ µg/L (ppb)	Soil/Sed² µg/kg (ppb)	Waste³ mg/kg (ppm)	Air^{4, 8} ppbv (µg/m³)
ANALYTE	Analytical Method	Routine Level	Routine Level (Encore®/Tared Vial)	Routine Level	Routine Level EPA TO-15⁶
4-Ethyltoluene (1-Ethyl-4-methyl benzene)	EPA TO-15	NA	NA	NA	0.042
Heptane	EPA TO-15	NA	NA	NA	0.040
Hexachlorobutadiene	EPA 8260C EPA 8260C SIM	0.50 0.05	1.0	0.20	0.022
Hexane	EPA TO-15	NA	NA	NA	0.044
Isooctane (2,2,4- Trimethylpentane)	EPA TO-15	NA	NA	NA	0.020
Isopropanol	EPA TO-15	NA	NA	NA	0.22
Isopropylbenzene	EPA 8260C EPA 8260C SIM	0.50 0.05	1.0	0.20	NA
p-Isopropyltoluene	EPA 8260C EPA 8260C SIM	0.50 0.05	1.0-2.0	0.20	NA
Methyl acetate	EPA 8260C	0.50	2.0	0.40	NA
Methyl cyclohexane	EPA 8260C EPA 8260C SIM	0.50 0.05	1.0	0.20	NA
Methylene chloride (Dichloromethane)	EPA 8260C EPA 8260C SIM	0.50 0.050	10	0.20	0.20
Methyl butyl ketone	EPA 8260C EPA 8260C SIM	1.0 0.10	5.0-10	0.40	0.22
Methyl ethyl ketone	EPA 8260C	4.0-10	5.0-10	1.6-4.0	0.20
Methyl isobutyl ketone	EPA 8260C EPA 8260C SIM	1.0 0.10	5.0-10	0.40	0.22
Methyl-t-butyl ether	EPA 8260C EPA 8260C SIM	0.50 0.015	1.0	0.20	0.022
Naphthalene ⁵	EPA 8260C EPA 8260C SIM	0.50-5.0 0.05	1.0-10	0.20-2.0	0.20
n-Propylbenzene	EPA 8260C EPA 8260C SIM	0.50 0.05	1.0	0.20	NA

ASB LOQAM Chapter 6 Table 6-6					
Volatile Organics (VOAs) Target Analyte List					
Minimum Reporting Limits (MRLs) by Matrices					
		Water¹ µg/L (ppb)	Soil/Sed² µg/kg (ppb)	Waste³ mg/kg (ppm)	Air^{4, 8} ppbv (µg/m³)
ANALYTE	Analytical Method	Routine Level	Routine Level (Encore[®]/Tared Vial)	Routine Level	Routine Level EPA TO-15⁶
Styrene	EPA 8260C EPA 8260C SIM	0.50 0.05	1.0	0.20	0.020
1,1,1,2-Tetrachloroethane ⁷	EPA 8260C EPA 8260C SIM	0.50 0.05	1.0	0.20	NA
1,1,2,2-Tetrachloroethane ⁷	EPA 8260C EPA 8260C SIM	0.50 0.05	1.0	0.20	0.020
Tetrachloroethene	EPA 8260C EPA 8260C SIM	0.50 0.05	1.0	0.20	0.022
Tetrahydrofuran	EPA TO-15	NA	NA	NA	0.20
Toluene	EPA 8260C EPA 8260C SIM	0.50 0.05	1.0	0.20	0.044
1,2,3-Trichlorobenzene	EPA 8260C EPA 8260C SIM	0.50 0.05	1.0	0.20	NA
1,2,4-Trichlorobenzene	EPA 8260C EPA 8260C SIM	0.50 0.05	1.0	0.20	0.044
1,1,1-Trichloroethane ⁷	EPA 8260C EPA 8260C SIM	0.50 0.05	1.0	0.20	0.022
1,1,2-Trichloroethane	EPA 8260C EPA 8260C SIM	0.50 0.015	1.0	0.20	0.022
Trichloroethene	EPA 8260C EPA 8260C SIM	0.50 0.015	1.0	0.20	0.022
Trichlorofluoromethane (R11)	EPA 8260C EPA 8260C SIM	0.50 0.015	1.0	0.20	0.022
1,2,3-Trichloropropane	EPA 8260C EPA 8260C SIM	0.50 0.05	1.0-2.0	0.20	NA
Trichlorotrifluoroethane (R113)	EPA 8260C EPA 8260C SIM	0.50 0.015	1.0	0.20	0.022
1,2,4-Trimethylbenzene	EPA 8260C EPA 8260C SIM	0.50 0.05	1.0	0.20	0.020

ASB LOQAM Chapter 6 Table 6-6					
Volatile Organics (VOAs) Target Analyte List					
Minimum Reporting Limits (MRLs) by Matrices					
		Water¹ µg/L (ppb)	Soil/Sed² µg/kg (ppb)	Waste³ mg/kg (ppm)	Air^{4, 8} ppbv (µg/m³)
ANALYTE	Analytical Method	Routine Level	Routine Level (Encore®/Tared Vial)	Routine Level	Routine Level EPA TO-15⁶
1,3,5-Trimethylbenzene	EPA 8260C EPA 8260C SIM	0.50 0.05	1.0	0.20	0.022
Vinyl acetate	EPA TO-15	NA	NA	NA	0.22
Vinyl bromide (Bromoethene)	EPA TO-15	NA	NA	NA	0.022
Vinyl chloride ⁷	EPA 8260C EPA 8260C SIM	0.50 0.015	1.0	0.20	0.022
o-Xylene	EPA 8260C EPA 8260C SIM	0.50 0.05	1.0	0.20	0.02
(m- and/or p-) Xylene	EPA 8260C EPA 8260C SIM	1.0 0.10	2.0	0.40	0.04
Acrolein	EPA 8260C	10-20	NA ⁵	4.0 ⁵	0.048 ⁵
Acrylonitrile	EPA 8260C	10-20	NA ⁵	4.0 ⁵	0.018 ⁵
Methyl Methacrylate	EPA 8260C	0.50	NA ⁵	0.2 ⁵	NA ⁵
2-Chloroethyl vinyl ether	EPA 8260C	1.0-4.0	NA ⁵	0.4 ⁵	NA ⁵
2,3-Benzofuran	EPA 8260C	0.50	NA ⁵	0.2 ⁵	NA ⁵
1,2,3-Trimethylbenzene	EPA 8260C	0.50	NA ⁵	0.2 ⁵	NA ⁵
MRLs may increase due to variability of interferences necessitating sample dilutions.					
¹ Water - 5 mL from septum-sealed vial.					
² Routine Level Soil - 5 g in water (reported on dry-weight basis).					
³ Waste - 1 g dissolved in 5-mL methanol and 62.5 uL of resulting extract purged.					
⁴ Air - 250 cc from 6-L passivated canister - <u>nominal</u> values. MRLs in µg/m ³ units depend on molecular weight and vary depending on the analyte and the standard lot.					
⁵ Not routinely reported but available upon request.					
⁶ MRLs don't account for the ~2x pressurization dilution of canisters after arrival at the lab.					
⁷ SIM MRLs available for waters upon special request.					
⁸ NATTS SIM MRLs are 10X lower than routine MRLs.					
NA – Not Available- ASB does not perform this analysis.					

Table 6-7

ASB LOQAM Chapter 6 Table 6-7 Semivolatile Organics Target Analyte List Minimum Reporting Limits by Matrices					
		Water¹ µg/L (ppb)	Soil/Sed² µg/kg (ppb)	Waste³ mg/kg (ppm)	Tissue⁴ mg/kg (ppm)
ANALYTE	Analytical Method	Routine Level	Routine Level	Routine Level	Routine Level
1-Methylnaphthalene	EPA 8270D	2.0	66	20	0.066
1,1'-Biphenyl	EPA 8270D	2.0	66	20	0.066
1,4-Dioxane	EPA 8270D	2.0	66	NA	NA
1,2,4-Trichlorobenzene	EPA 8270D	10	330	100	0.33
2-Nitrophenol	EPA 8270D	10	330	100	0.33
2-Methyl-4,6-dinitrophenol	EPA 8270D	10	330	100	0.33
2,4-Dimethylphenol	EPA 8270D	10	330	100	0.33
2,4-Dinitrotoluene	EPA 8270D	10	330	100	0.33
2,4-Dinitrophenol	EPA 8270D	20	660	200	0.66
2-Methylphenol	EPA 8270D	10	330	100	0.33
2-Nitroaniline	EPA 8270D	10	330	100	0.33
2-Chlorophenol	EPA 8270D	10	330	100	0.33
2-Methylnaphthalene	EPA 8270D	2.0	66	20	0.066
2,3,4,6-Tetrachlorophenol	EPA 8270D	10	330	100	0.33
2,4,5-Trichlorophenol	EPA 8270D	10	330	100	0.33
2-Chloronaphthalene	EPA 8270D	10	330	100	0.33
2,6-Dinitrotoluene	EPA 8270D	10	330	100	0.33
2,4-Dichlorophenol	EPA 8270D	10	330	100	0.33
2,4,6-Trichlorophenol	EPA 8270D	10	330	100	0.33
3,3'-Dichlorobenzidine	EPA 8270D	10	330	100	0.33
(3- and/or 4-) Methylphenol	EPA 8270D	10	330	100	0.33
3-Nitroaniline	EPA 8270D	10	330	100	0.33
4-Chlorophenyl phenyl ether	EPA 8270D	10	330	100	0.33
4-Chloroaniline	EPA 8270D	10	330	100	0.33
4-Nitroaniline	EPA 8270D	10	330	100	0.33
4-Nitrophenol	EPA 8270D	10	330	100	0.33

ASB LOQAM Chapter 6 Table 6-7 Semivolatile Organics Target Analyte List Minimum Reporting Limits by Matrices					
		Water¹ µg/L (ppb)	Soil/Sed² µg/kg (ppb)	Waste³ mg/kg (ppm)	Tissue⁴ mg/kg (ppm)
ANALYTE	Analytical Method	Routine Level	Routine Level	Routine Level	Routine Level
4-Chloro-3-methylphenol	EPA 8270D	10	330	100	0.33
4-Bromophenyl phenyl ether	EPA 8270D	10	330	100	0.33
Acenaphthene	EPA 8270D	2.0	66	20	0.066
Acenaphthylene	EPA 8270D	2.0	66	20	0.066
Acetophenone	EPA 8270D	10	330	100	0.33
Anthracene	EPA 8270D	2.0	66	20	0.066
Atrazine	EPA 8270D	10	330	100	0.33
Benzo[a]anthracene	EPA 8270D	2.0	66	20	0.066
Benzo[a]pyrene	EPA 8270D	2.0	66	20	0.066
Benzo[b]fluoranthene	EPA 8270D	2.0	66	20	0.066
Benzo[k]fluoranthene	EPA 8270D	2.0	66	20	0.066
Benzo[g,h,i]perylene	EPA 8270D	2.0	66	20	0.066
Benzaldehyde	EPA 8270D	10	330	100	0.33
Benzyl butyl phthalate	EPA 8270D	10	330	100	0.33
Bis(2-ethylhexyl) phthalate	EPA 8270D	10	330	100	0.33
Bis(2-chloroethyl) ether	EPA 8270D	10	330	100	0.33
Bis(chloroethoxy)methane	EPA 8270D	10	330	100	0.33
Bis(chloroisopropyl) ether	EPA 8270D	10	330	100	0.33
Caprolactam	EPA 8270D	10	330	100	0.33
Carbazole	EPA 8270D	2.0	66	20	0.066
Chrysene	EPA 8270D	2.0	66	20	0.066
Di-n-butyl phthalate	EPA 8270D	10	330	100	0.33
Di-n-octyl phthalate	EPA 8270D	10	330	100	0.33
Dibenz(a,h)anthracene	EPA 8270D	2.0	66	20	0.066
Dibenzofuran	EPA 8270D	2.0	66	20	0.066
Diethyl phthalate	EPA 8270D	10	330	100	0.33

ASB LOQAM Chapter 6 Table 6-7 Semivolatile Organics Target Analyte List Minimum Reporting Limits by Matrices					
		Water¹ µg/L (ppb)	Soil/Sed² µg/kg (ppb)	Waste³ mg/kg (ppm)	Tissue⁴ mg/kg (ppm)
ANALYTE	Analytical Method	Routine Level	Routine Level	Routine Level	Routine Level
Dimethyl phthalate	EPA 8270D	10	330	100	0.33
Fluoranthene	EPA 8270D	2.0	66	20	0.066
Fluorene	EPA 8270D	2.0	66	20	0.066
Hexachlorobenzene (HCB)	EPA 8270D	10	330	100	0.33
Hexachlorobutadiene	EPA 8270D	10	330	100	0.33
Hexachlorocyclopentadiene (HCCP)	EPA 8270D	10	330	100	0.33
Hexachloroethane	EPA 8270D	10	330	100	0.33
Indeno[1,2,3-cd]pyrene	EPA 8270D	2.0	66	20	0.066
Isophorone	EPA 8270D	10	330	100	0.33
N-Nitrosodiphenylamine	EPA 8270D	10	330	100	0.33
Naphthalene	EPA 8270D	2.0	66	20	0.066
Nitrobenzene	EPA 8270D	10	330	100	0.33
Nitroso-di-n-propylamine	EPA 8270D	10	330	100	0.33
Pentachlorophenol	EPA 8270D	10	330	100	0.33
Phenanthrene	EPA 8270D	2.0	66	20	0.066
Phenol	EPA 8270D	10	330	100	0.33
Pyrene	EPA 8270D	2.0	66	20	0.066
MRLs may increase due to possible interferences necessitating sample dilutions and moisture content of soil samples.					
¹ Water - 1000 mL; final extract volume 1 mL.					
² Soil - 30 g extracted (reported as dry-weight); final extract volume 1 mL.					
³ Waste - 1 g extracted (reported as wet-weight); final extract volume 10 mL.					
⁴ Fish or biological tissue - Same as soil.					
⁵ SA = Special Analysis requiring additional QC currently not in place. Contact OCS Section Chief. Tentative MRL.					
NA-Not Applicable -ASB does not perform analysis for this compound.					

Table 6-8

ASB LOQAM Chapter 6 Table 6-8 Semivolatile Organics Full Scan - Low Level Minimum Reporting Limits by Matrices					
		Water¹ µg/L (ppb)	Soil/Sed² µg/kg (ppb)	Waste mg/kg (ppm)	Tissue mg/kg (ppm)
ANALYTE	Analytical Method	Low Level	Low Level	Low Level	Low Level
1-Methylnaphthalene	EPA 8270D	0.1	3.33	NA	NA
2-Methylnaphthalene	EPA 8270D	0.1	3.33	NA	NA
Acenaphthene	EPA 8270D	0.1	3.33	NA	NA
Acenaphthylene	EPA 8270D	0.1	3.33	NA	NA
Anthracene	EPA 8270D	0.1	3.33	NA	NA
Benzo[a]anthracene	EPA 8270D	0.1	3.33	NA	NA
Benzo[a]pyrene	EPA 8270D	0.1	3.33	NA	NA
Benzo[b]fluoranthene	EPA 8270D	0.1	3.33	NA	NA
Benzo[k]fluoranthene	EPA 8270D	0.1	3.33	NA	NA
Benzo[g,h,i]perylene	EPA 8270D	0.1	3.33	NA	NA
Carbazole	EPA 8270D	0.1	3.33	NA	NA
Chrysene	EPA 8270D	0.1	3.33	NA	NA
Dibenz(a,h)anthracene	EPA 8270D	0.1	3.33	NA	NA
Fluoranthene	EPA 8270D	0.1	3.33	NA	NA
Fluorene	EPA 8270D	0.1	3.33	NA	NA
Indeno[1,2,3-cd]pyrene	EPA 8270D	0.1	3.33	NA	NA
Naphthalene	EPA 8270D	0.1	3.33	NA	NA
Pentachlorophenol	EPA 8270D	1.03	33.3	NA	NA
Phenanthrene	EPA 8270D	0.1	3.33	NA	NA
Pyrene	EPA 8270D	0.1	3.33	NA	NA
<p>MRLs may increase due to interferences necessitating smaller extraction amounts, dilutions and moisture content of soil samples. The above analytes can also be analyzed by full scan GC/MS at the stated MRLs.</p> <p>¹ Water – 1000 ml; final extract 1 mL¹ Water - 1000 mL; final extract volume 1 mL.</p> <p>² Soil – 30 g extracted (reported as dry-weight); final extract 1 mL</p> <p>³ 0.2 ug/L can be reported if specifically requested.</p> <p>NA- Not available- ASB does not perform this analysis.</p>					

Table 6-9

ASB LOQAM Chapter 6 Table 6-9 Routine Pesticide/PCB Target Analyte List Minimum Reporting Limits (MRLs)* by Matrices					
		Water¹ µg/L (ppb)	Soil/Sed² µg/kg (ppb)	Waste³ mg/kg (ppm)	Tissue⁴ mg/kg (ppm)
ANALYTE	Analytical Method(s)	Routine Level	Routine Level	Routine Level	Routine Level
Aldrin	EPA 8081B	0.04	1.3	SA ⁵ -0.50	0.020
Heptachlor	EPA 8081B	0.04	1.3	SA ⁵ -0.50	0.020
Heptachlor epoxide	EPA 8081B	0.04	1.3	SA ⁵ -0.50	0.020
α-BHC	EPA 8081B	0.04	1.3	SA ⁵ -0.50	0.020
β-BHC	EPA 8081B	0.04	1.3	SA ⁵ -0.50	0.020
γ-BHC	EPA 8081B	0.04	1.3	SA ⁵ -0.50	0.020
δ-BHC	EPA 8081B	0.04	1.3	SA ⁵ -0.50	0.020
Endosulfan I	EPA 8081B	0.04	1.3	SA ⁵ -0.50	0.020
Dieldrin	EPA 8081B	0.04	1.3	SA ⁵ -0.50	0.020
p,p'-DDT	EPA 8081B	0.04	1.3	SA ⁵ -0.50	0.020
p,p'-DDE	EPA 8081B	0.04	1.3	SA ⁵ -0.50	0.020
p,p'-DDD	EPA 8081B	0.04	1.3	SA ⁵ -0.50	0.020
Endrin	EPA 8081B	0.04	1.3	SA ⁵ -0.50	0.020
Endosulfan II	EPA 8081B	0.04	1.3	SA ⁵ -0.50	0.020
Endosulfan sulfate	EPA 8081B	0.04	1.3	SA ⁵ -0.50	0.020
Endrin aldehyde	EPA 8081B	0.04	1.3	SA ⁵ -0.50	0.020
Endrin ketone	EPA 8081B	0.04	1.3	SA ⁵ -0.50	0.020
Methoxychlor	EPA 8081B	0.04	1.3	SA ⁵ -1.0	0.050
γ-Chlordane	EPA 8081B	0.04	1.3	SA ⁵ -0.50	0.020
α-Chlordane	EPA 8081B	0.04	1.3	SA ⁵ -0.50	0.020
Aroclor 1221	EPA 8082A	1.0	33	SA ⁵ -5.0	0.20
Aroclor 1232	EPA 8082A	1.0	33	SA ⁵ -2.5	0.10
Aroclor 1242	EPA 8082A	1.0	33	SA ⁵ -2.5	0.10
Aroclor 1016	EPA 8082A	1.0	33	SA ⁵ -2.5	0.10
Aroclor 1248	EPA 8082A	1.0	33	SA ⁵ -2.5	0.10
Aroclor 1254	EPA 8082A	1.0	33	SA ⁵ -2.5	0.10
Aroclor 1260	EPA 8082A	1.0	33	SA ⁵ -2.5	0.10

ASB LOQAM Chapter 6 Table 6-9 Routine Pesticide/PCB Target Analyte List Minimum Reporting Limits (MRLs)* by Matrices					
		Water¹ µg/L (ppb)	Soil/Sed² µg/kg (ppb)	Waste³ mg/kg (ppm)	Tissue⁴ mg/kg (ppm)
ANALYTE	Analytical Method(s)	Routine Level	Routine Level	Routine Level	Routine Level
Aroclor 1262	EPA 8082A	1.0	33	SA ⁵ -2.5	0.10
Aroclor 1268	EPA 8082A	1.0	33	SA ⁵ -2.5	0.10
Toxaphene	EPA 8081B	5.0	170	SA ⁵ -20	SA ⁵ -1.0
<p>MRLs may increase due to possible interferences necessitating sample dilutions and moisture content of soil samples.</p> <p>¹ Water - 1000 mL extracted; 8081/8082A, final extract volume 10 mL.</p> <p>² Soil - 30 g extracted (reported as dry-weight); 8081/8082A, final extract volume 10 mL.</p> <p>³ Waste - 1 g extracted (reported as wet-weight); final extract volume 10 mL.</p> <p>⁴ Fish or biological tissue - 10 g extracted (reported as wet-weight); final extract volume 10 mL.</p> <p>⁵ SA = Special Analysis requiring additional QC currently not in place. Contact OCS Chief. Tentative MRL.</p> <p>*Pesticide/PCB water and soil MRLs set by ASB at CLP reporting levels.</p>					

Table 6-10

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

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

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

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

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

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

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

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

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

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

⁷ See Appendix 3 VOA MRLs – 8260 SIM method

NA - Not Available- ASB does not perform this analysis.

Table 6-11

ASB LOQAM Chapter 6 Table 6-11 Herbicides Target Analyte List Minimum Reporting Limits (MRLs) by Matrices					
		Water¹ µg/L (ppb)	Soil/Sed µg/kg (ppb)	Waste mg/kg (ppm)	Tissue mg/kg (ppm)
ANALYTE	Analytical Method	Routine Level	Routine Level	Routine Level	Routine Level
2,4,5-T	EPA 8321B	1.0	NA	NA	NA
2,4-D	EPA 8321B	1.0	NA	NA	NA
2,4-DB	EPA 8321B	2.0	NA	NA	NA
Silvex (2,4,5-TP)	EPA 8321B	1.0	NA	NA	NA
Dalapon	EPA 8321B	2.0	NA	NA	NA
Dicamba	EPA 8321B	1.0	NA	NA	NA
Dichlorprop	EPA 8321B	1.0	NA	NA	NA
Dinoseb	EPA 8321B	4.0	NA	NA	NA
MCPA	EPA 8321B	5.0	NA	NA	NA
MCPP	EPA 8321B	5.0	NA	NA	NA
MRLs may increase due to interferences necessitating smaller sample amounts and dilutions.					
NA - Not Available-ASB does not perform this analysis.					

Table 6-12

ASB LOQAM Chapter 6 Table 6-12 Per - and Polyfluoroalkyl Substances (PFAS) Target Analyte List Minimum Reporting Limits (MRLs) by Matrices					
		Water µg/L (ppb)	Soil/Sed µg/kg (ppb)	Waste µg/kg (ppb)	Tissue mg/kg (ppm)
ANALYTE	Analytical Method	Routine Level	Routine Level	Routine Level	Routine Level
Perfluorotetradecanoic acid (PFTeDA)	ASBPROC-800	0.080	NA	0.047	NA
Perfluorotridecanoic acid (PFTrDA)	ASBPROC-800	0.040	NA	0.040	NA
Perfluorododecanoic acid (PFDoA)	ASBPROC-800	0.040	NA	0.040	NA
Perfluoroundecanoic acid (PFUDA)	ASBPROC-800	0.040	NA	0.040	NA
Perfluorodecanoic acid (PFDA)	ASBPROC-800	0.040	NA	0.040	NA
Perfluorononanoic acid (PFNA)	ASBPROC-800	0.040	NA	0.040	NA
Perfluorooctanoic acid (PFOA)	ASBPROC-800	0.040	NA	0.040	NA
Perfluoroheptanoic acid (PFHpA)	ASBPROC-800	0.040	NA	0.040	NA
Perfluorohexanoic acid (PFHxA)	ASBPROC-800	0.040	NA	0.047	NA
Perfluoropentanoic acid (PFPeA)	ASBPROC-800	0.040	NA	0.040	NA
Perfluorobutyric acid (PFBA)	ASBPROC-800	0.040	NA	0.040	NA
Perfluorodecanesulfonate (PFDS)	ASBPROC-800	0.040	NA	0.040	NA
Perfluorononanesulfonate (PFNS)	ASBPROC-800	0.040	NA	0.040	NA
Perfluorooctanesulfonate (PFOS)	ASBPROC-800	0.040	NA	0.040	NA
Perfluoroheptanesulfonate (PFHpS)	ASBPROC-800	0.040	NA	0.040	NA
Perfluorohexanesulfonate (PFHxS)	ASBPROC-800	0.040	NA	0.040	NA
Perfluoropentanesulfonate (PFPeS)	ASBPROC-800	0.040	NA	0.040	NA
Perfluorobutanesulfonate (PFBS)	ASBPROC-800	0.040	NA	0.040	NA
Perfluorooctanesulfonamide (FOSA)	ASBPROC-800	0.040	NA	0.040	NA
Fluorotelomer sulfonate 8:2 (8:2 FTS)	ASBPROC-800	0.040	NA	0.040	NA
Fluorotelomer sulfonate 6:2 (6:2 FTS)	ASBPROC-800	0.040	NA	0.040	NA
Fluorotelomer sulfonate 4:2 (4:2 FTS)	ASBPROC-800	0.040	NA	0.040	NA
N-ethyl-N-((heptadecafluorooctyl)sulfonyl)glycine (N-EtFOSAA)	ASBPROC-800	0.040	NA	0.040	NA
N-(Heptadecafluorooctylsulfonyl)-N-methylglycine (N-MeFOSAA)	ASBPROC-800	0.040	NA	0.056	NA
Hexafluoropropylene oxide-dimer acid (HFPO-DA)	ASBPROC-800	0.040	NA	0.040	NA
MRLs may increase due to interferences necessitating smaller sample amounts and dilutions.					
NA -Not Available- ASB does not perform this analysis.					