ANALYTICAL SUPPORT BRANCH

LABORATORY OPERATIONS
and
QUALITY ASSURANCE MANUAL

U.S. ENVIRONMENTAL PROTECTION AGENCY
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DISCLAIMER

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Analytical Support 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 Support 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 the ASB is to provide environmental chemistry 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 and chemical data of a known and defensible quality. ASB provides environmental data at the request of data users within the Agency, no data are reported to outside clients. 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 the ASB to conduct all activities with four guiding principles: **(1) Safety (2) Integrity (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 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 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 as well as a Presidential Memorandum dated March 9, 2009 on the same subject. 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 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, by maintaining complete and clear files of these records.

1.3.3 Quality It is the policy of ASB that all data generated by the branch is of the quality required to meet or exceed the data quality objectives of each project. Managers and analysts of the branch share the responsibility of ensuring that analytical methods, instruments, parameter 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 end-user(s) of the data. 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 all users of the data by assigning appropriate qualifiers according to prescribed procedures; and

1.3.3.3 Having an independent 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 Communication between the lab's staff and its clients is required to fully define a project's measurement and data quality objectives 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 data quality objectives 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 and kept up-to-date on the issues surrounding the late data and abreast of the progress of project completion.

1.4 Accreditation and Certification

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.

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

ASB's objective is to seek and maintain accreditation and certification for the methods and analytes which 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 and general procedures that are the basis of ASB's quality system. Specific technical and operational details are contained in 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 take precedence over this manual on those occasions. When there are differences in analytical method, LOQAM and accreditation standard requirements, ASB will follow the strictest, except as specifically noted. When it is unclear which of the three is strictest, method requirements will be followed.

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 Support Branch – Personnel

Immediate Office

Name	Principal Duties
Danny France	**Branch Chief**
Scott Sivertsen	Senior Technical Advisor
Mike Beall	Sample Custodian (SEE Employee)

Inorganic Chemistry Section

Name	Principal Duties
Jeffrey Hendel	**Section Chief-Technical Director Inorganic Analyses**
Daniel Adams	Nutrients
Pam Betts	Nutrients
Curtis Callahan	Nutrients; Classicals; Metals
Anthony Carroll	Mercury; Hexavalent Chromium
Megan DeJesus	Nutrients; Classicals; Metals
Roberta Howes	Metals ICP
Brittany Stuart	Metals ICP; Hexavalent Chromium
Francine VanCuron	Metals ICP-MS
Earnest Walton	Metals ICP-MS
Terri White	Metals ICP-MS

Organic Chemistry Section

Name	Principal Duties
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
Lilia Melendez	Semivolatiles
Janet Muse	Semivolatiles
Brian Shuhler	Pesticides/PCBs
Kristin Trapp	Volatiles
Stephanie Wimpey	Volatiles; Property Officer; ASB Safety Officer; ChemShare Coordinator

2.1.2 Quality System Support– Personnel

ISO Accreditation Team

Name	Principal Duties
Jeannie Williamson	Laboratory Quality Manager

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. Chemistry laboratory professionals and technicians fall within the GS-1300 standard for professional physical science work (see http://www.opm.gov/qualifications/index.asp). 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 an EPA 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 ASB has developed a set of required training sessions for each employee and are specified in ASB's Employee Training SOP. Documentation of training is maintained by sign in forms 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 the overall management responsibility, including hiring, budgeting, and policy development for the branch.

2.3.1.2 Has ultimate responsibility for the development, implementation, approval, and continued operation of the branch quality assurance system. The authority and responsibility for day-to-day management of the quality system is delegated to the branch LQM

2.3.1.3 The authority and responsibility for the daily oversight of quality control activities is delegated to Section Chiefs and Group Leaders.

2.3.1.4 Provides leadership that promotes 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 Assures that communication takes place regarding the effectiveness of the quality system.

2.3.1.7 Is authorized to offer opinions and interpretations.

2.3.1.8 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 SESD Deputy Division Director 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 Serves as the focal point for all QA/QC activities within the branch.

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 appropriate technical staff.

2.3.2.7 Maintains QA files with all appropriate documentation to include:

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, ect.);

2.3.2.7.4 Results of interlaboratory comparisons or proficiency tests;

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

2.3.2.7.6 Records and Documentation:

2.3.2.7.6.1 Demonstration of Capability (DOCs) and Continuing Demonstrations of Proficiency (CDOPs);

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 the LIMS 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;

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 and is responsible for all data produced by the analytical groups within the section.

2.3.3.2 Ensures that a final overview of each work product (e.g., data, written reports, etc.) 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 taken within the analytical work groups as a result of internal and external audits.

2.3.3.6 Reviews and approves all section SOP and QA Manual updates and then submits to LQM and Branch Chief for final approvals.

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 Is 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 Analytical 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 Each section of the branch is comprised of work groups. For each analytical work group there may be 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 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 Defined as the staff analyst performing a test on a given date and time. The primary analyst shall ensure that:

2.3.4.3.1 Appropriate analytical methodologies and standard procedures are followed;

2.3.4.3.2 Appropriate QC activities are performed as designated by the method and/or the LOQAM;

2.3.4.3.3 Analytical activities are properly documented as specified by the method 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.

2.3.4.3.8 Typically, the primary analyst performs initial data reduction and transfer of data to the LIMS. However, this task may also be performed by another analyst competent to perform the analysis.

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 standard procedures were followed;

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

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

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

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; and

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

2.3.5 Deputies (Acting Chief or LQM) who are acting on behalf of a management official within the branch or section assume the duties and responsibilities of that individual under the quality system. Specific duties and responsibilities may be assumed by different individuals, either a member of ASB management or a staff member, as appropriate. 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 Work Assignment Managers 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 Section Chiefs may designate certain ASB staff to assign work to ESAT. As needed, these staff members will direct work to ESAT via the ESAT Tracking System on the LAN. ESAT assignments will normally require communication with the Section Chief to assure that the EPA/ESAT workload is evenly distributed. Under the existing contract, only the EPA ESAT Project Officer or Alternate Project Officer(s) may issue work to ESAT.

2.3.7.2 ASB staff that assign work to ESAT must follow all rules and regulations of the contracting process. They are also responsible for receiving the work products from the ESAT staff, performing an appropriate review of the work performed and insuring that it is appropriately entered into the LIMS and subsequently reported. In those cases where secondary review is performed by ESAT a reduced level of final overview by EPA may be appropriate.

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 standard procedures were followed;

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

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

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

2.3.7.3.5 Complete and accurate recording of all individual analytical data points and that 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 LIMS 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 resent 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 the Analytical Support Branch. Operation and maintenance of the facility is the responsibility of the lessor through 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 lab area. The lab will ensure the facility and environmental conditions relevant to the procedure will be monitored as required.

2.5 Equipment

2.5.1 Inventory ASB has maintains a list of major 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 primary maintenance is carried out on instrumentation.

Figure 2-1
Analytical Support Branch Organization Chart

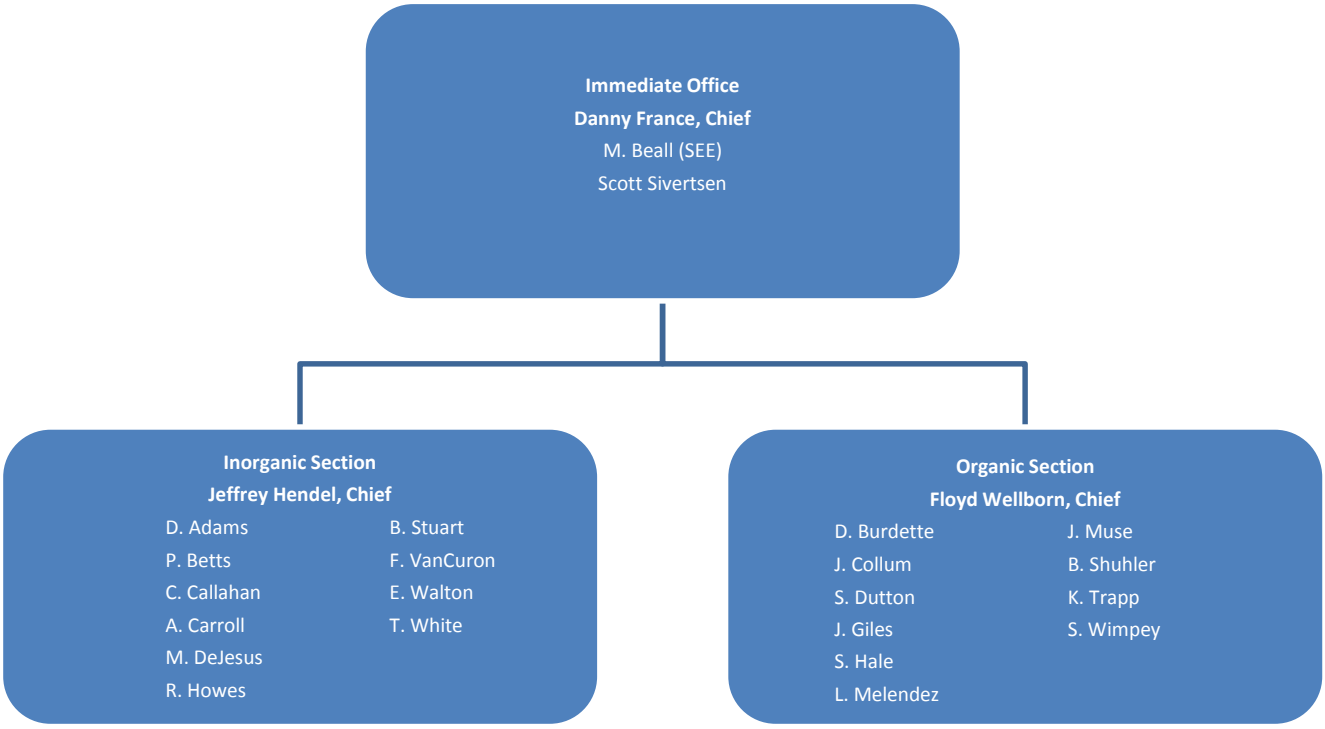
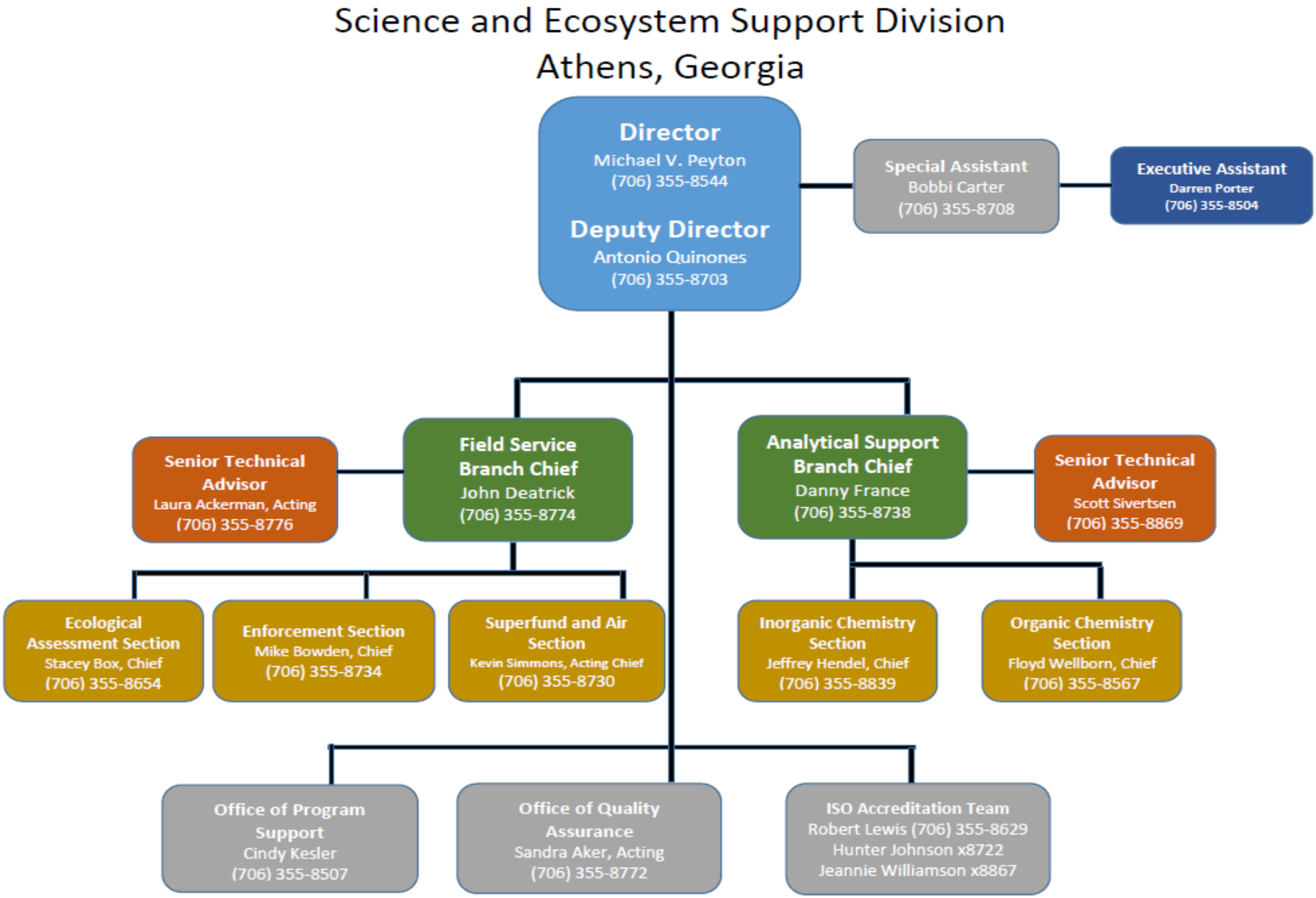


Figure 2-2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CHAPTER 3

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 chemical analysis received by ASB are controlled with documented custody procedures. Custody and handling procedures for field operations are detailed in the Field Branches Quality System and Technical Procedures.

3.2 Sample Collection

3.2.1 Procedures Samples are collected using standard field sampling techniques consistent with the parameter being determined. Sampling procedures are followed that minimize the possibility of sample adulteration by either the sample collector or sampling device. Field sample collection procedures are detailed in the Field Branches Quality System and Technical Procedures. ASB staff does not routinely perform field sampling activities.

3.2.2 Containers and Holding Times Selection of sample container types and preservation techniques are guided by the method 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 analysis, sample matrices, preservatives, and recommended holding times.

3.3 Sample Scheduling

3.3.1 Initial Scheduling ASB uses Element®, a commercially available laboratory information management system (LIMS) for the management and reporting of analytical data. However, because of the versatile and powerful project scheduling features of the existing in-house Region 4 LIMS (R4LIMS), it is being actively maintained for project scheduling. Projects requiring chemical analyses are routinely scheduled into R4LIMS directly by project leaders; occasionally, the scheduling of the samples may be made directly with the Regional Sample Control Coordinator (RSCC) or ASB management. ASB management or Laboratory Quality Manager (LQM) will use R4LIMS to schedule QA projects for in-house analysis. Each time samples are scheduled, a unique project number is assigned automatically by R4LIMSThe R4LIMS project number is 'passed through' to Element® and is used in conjunction with project specific Element® work order number(s) for tracking of the analyses. An additional feature of R4LIMS is a function called "Project Notes" which allows project leaders and lab management to communicate on the project's analytical needs. The laboratory will not accept samples that arrive without first being

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.

3.3.2 Sample Acceptance

3.3.2.1 Review of Requested Analyses The ASB Section Chiefs routinely review scheduled projects to determine if there is internal lab capacity to accept a project, or if the project should be contracted outside the laboratory through the national contracts such as the Superfund Contract Laboratory Program (CLP). Normally all samples in support of criminal projects will be analyzed within the ASB laboratories or sent to the National Enforcement Investigations Center (NEIC) for analysis.

3.3.2.2 Sample Acceptance Responsibility and Considerations The acceptance of samples into the ASB laboratories 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 lab staff have the necessary skills and expertise to perform the environmental tests requested, a demonstration of capability is on file, and the laboratory has accreditation for a specific method/analyte 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.

3.3.2.3 Special Project Needs The requirement for management to review laboratory capability, capacity, and accreditation status in no way limits management's ability to develop new testing methods or accept projects requiring unique or unusual analytes. 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.

3.3.2.4 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.

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

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

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

3.3.2.5 NPDES Analyses 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; however, they do not require the laboratory to be accredited for those methods. When a project is requested in support of NPDES regulations, the requestor will include this information in R4LIMS Project Notes.

3.3.2.6 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.

3.3.2.7 Documenting Communication in R4Lims and Element®'s Workorder Notes Documentation of communication between the project leader and ASB personnel should be documented. This includes documenting special method requests, clarification to requests, and deviations from the Standard Operating Procedure (SOPs) are noted in R4LIMS and Element®.

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

3.3.2.7.2 After the samples arrive in-house, the chain-of-custody reflects the official request and **will be the final authority used for the analytical work performed**. When changes are requested in parameters after sample receipt at the laboratory, they must be approved by the appropriate ASB manager (or designee) and documented by the ASB manager submitting the change in writing along with the reason to the SCC. The SCC will obtain a revised COC from the originator of the original COC and include it in the project file. Refer to SOP ASB 105G for more procedures related to sample receipt.

3.3.2.8 Quick Turn-Around analyses R4LIMS shows the scheduled projects in blue in the projects report 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. TATs other than the standard of 35 calendar days

(or 45 calendar days for projects with TCLPs) must also be included in discussions with ASB mangers for acceptance. While it is important that the RSCC/designee communicate all issues that may arise concerning the samples for a specific project, it is especially critical that quick turn-around samples be monitored for receipt and communicated to managers and analysts so that analyses may begin as soon as possible in order to accommodate the TAT request.

3.3.3 Canceled Projects The RSCC notes in R4LIMS projects that were scheduled and subsequently canceled. The electronic record also tracks these changes.

3.4 Sample Receipt

3.4.1 Sample Acceptance Policy Samples requested for analysis within ASB are typically from internal Agency sampling organizations 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., preservatives, 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. Most often 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 Organics with Short Holding Time Requirements Some organic samples such as waters requiring analysis for semivolatiles, pesticides/PCBs, and unpreserved volatiles require quick shipping to SESD due to the short holding time of 7 days from sample collection to extraction. Also, there is a 48-hour holding time requirement from collection to preservation for VOA soil samples collected in coring devices or 40-mL containers. Therefore, the following sample shipping guidelines should be observed by field sampling organizations.

3.4.3.1 Semivolatiles, Pesticides/PCBs, unpreserved VOA waters: Water samples collected during the week must be shipped to the lab within 48 hours of collection and cannot be driven back to the lab on a Friday if collected on Monday or Tuesday. Samples collected on Monday should not be held for shipping later in the week, especially before a Monday holiday. Samples with 7-day holding times can be "walked in" by samplers to the lab if they arrive by Thursday; Wednesday if the following Monday is a holiday.

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.

3.4.4 Acceptance of Samples Known or Suspected to Contain Dioxin

3.4.4.1 Environmental samples (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 to the best of their knowledge 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 to the best of their ability whether samples are contaminated with the listed RCRA wastes. The references cited in references 1 and 2 may be consulted for additional information on the management of RCRA wastes.

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

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

3.4.4.2.2 Sample Disposal ASB will depend upon 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 the EPA 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-L 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, samples are secured within the custody room walk in coolers or other specifically designated sample storage locations.

3.5.2.1 The temperature of the custody room coolers and freezer are monitored and recorded using a certified wireless temperature sensor which interfaces with a receiver and software installed on a custody room computer. The software records and sends e-mail and 'pop-up' alerts if the cooler temperature exceeds the specified range. These sensor thermometers serve as the 'official' thermometers of the coolers and freezer. Charted data from the wireless cooler and freezer temperature sensors are used to assess the extent and duration of any temperature excursions and assist in determining if any samples may have been affected by the temperature excursion. The acceptable temperature range for the coolers is 0-6°C. The freezer should maintain ≤ -10°C. A temperature excursion outside acceptance criteria may result in initiating a corrective action to determine if the excursion was a systematic issue. The coolers and freezer are additionally monitored by security personnel outside regular business hours.

3.6 Custody

Custody records on all samples received by ASB are maintained within Element®. Reports can be generated on each work order which detail the custody transactions for each sample bottle.

3.6.1 Custody Room Access Key card entry controls access to the main custody room area. Authorized 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.

3.6.2 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 does not routinely enter the custody room. The custody room is cleaned only by special coordination and scheduling through the facility representative.

3.6.3 Documentation of Custody Documentation of sample custody is accomplished by the use of COC seals that are secured by field sampling personnel, a COC form initiated at the time of sample collection, field log books, individual analysis logs, the LIMS 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 EPA/SESD personnel responsible for sample collection. It is the project leader's responsibility to check the computer print-out against the chain-of-custody record for accuracy as it relates to analyses requested for the project and the sampling station identification information.

3.6.4 Assuming Custody for Analysis ASB utilizes the LIMS to perform a check-out, check-in system 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).

3.6.5 Tracking of Sample Extracts and Digests

3.6.5.1 Each batch of samples prepared for analysis, or taken for direct analysis, is assigned a unique Batch ID number for tracking purposes. Batch IDs are assigned automatically by Element® and are in the format 'YYMMnnnn' where:

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

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

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

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

3.6.5.3 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 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 information will be performed by the LQM or designee(s). These reviews typically are performed along with the annual technical system review of custody procedures and will include an examination of custody documentation of randomly selected samples for traceability, completeness, and accuracy.

Table 3-1
Recommended Preservation & Holding Times

Analytical Group	Soil/Sediment [1]		Water [1, 2] and Waste Water		Waste		Tissue	
	Pres[3] Amt[4] Container Type[5]	**Hold[6]**	**Pres[3] Amt[4] Container Type[5]**	**Hold[6]**	**Pres[3] Amt[4] Container Type[5]**	**Hold[6]**	**Pres[3] Amt[4] Container Type[5]**	**Hold[6]**
Inorganics								
Acidity	NA	NA	Ice-4ºC 500 mL P, FP Fill completely and cap tightly	14 days	NA	NA	NA	NA
Alkalinity	NA	NA	Ice-4ºC 500 mL P, FP Fill completely and cap tightly	14 days	NA	NA	NA	NA
BOD5	NA	NA	Ice-4ºC 2 L P, FP	48 hrs	NA	NA	NA	NA
BOD - Long Term	NA	NA	Ice-4ºC 1 gal(x2)[15] P, FP	48 hrs	NA	NA	NA	NA
Bromide	NA	NA	Ice-4ºC 500 mL P, FP	28 days	NA	NA	NA	NA
Chloride	None 8 oz G	Not specified	None 500 mL P, FP	28 days	NA	NA	NA	NA
Chlorine - Residual	NA	NA	None 500 mL P	Immed	NA	NA	NA	NA
Conductivity	NA	NA	Ice-4ºC 500 mL P, FP	28 days	NA	NA	NA	NA
Chromium VI (hexavalent)	Ice-4ºC 8 oz G	Extract - 1 month Analysis - 4 days[7]	Filter immed.[9] Ice-4ºC 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-4ºC 8 oz G	14 days	NaOH to pH>12, ascorbic acid[8], Ice-4ºC 500 mL P, FP	14 days	None 8oz G	Not specified	NA	NA
Dissolved P, total	NA	NA	Filter immed.[9] Ice-4ºC H_2SO_4 to pH<2 500 mL P, FP	28 days	NA	NA	NA	NA
Fluoride	None 8 oz G	Not specified	None 500 mL P	28 days	NA	NA	NA	NA
Grain Size	None 8oz G	Not Specified	NA	NA	NA	NA	NA	NA
Hardness	NA	NA	HNO_3 to pH<2 500 mL P, FP	180 days 6 months	NA	NA	NA	NA
Mercury	Ice-4ºC 8 oz G	28 days	HNO_3 to pH<2 1L P, FP	28 days	None 8oz G	180 days	Freeze, 5 g 8 oz G, Al foil or plastic	Not specified
Mercury - TCLP	None 8 oz G	28 days 56 days[10]	None 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 multiphase (>0.5% and <10% solids)	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	
	Pres[3] Amt[4] Container Type[5]	**Hold[6]**	**Pres[3] Amt[4] Container Type[5]**	**Hold[6]**	**Pres[3] Amt[4] Container Type[5]**	**Hold[6]**	**Pres[3] Amt[4] Container Type[5]**	**Hold[6]**
Mercury - UTL	Ice-4ºC 8 oz G	90 days	HCl to pH<2 1L FP	90 days	None 8oz G	Not specified	Freeze, 5 g 8 oz G, Al foil or plastic	Not specified
Metals, except mercury	Ice-4ºC 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 specified
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 2nd preserved)	Ice-4ºC 8 oz G	Not specified	Ice-4ºC 500 mL P, FP AND 2nd container Ice-4ºC, H_2SO_4 to pH<2 500 mL	48 hrs	NA	NA	NA	NA
Nitrite	Ice-4ºC 8 oz G	Not specified	Ice-4ºC 500 mL P, FP	48 hrs	NA	NA	NA	NA
Nutrients (ammonia, TKN, NO_3+NO_2 -N, total phosphorus)	Ice-4ºC 8 oz G	Not specified	Ice-4ºC, H_2SO_4 to pH<2 500 mL/paramtr or 1L total P,FP	28 days	NA	NA	NA	NA
Ortho-P	NA	NA	Ice-4ºC 500 mL P, FP	48 hrs	NA	NA	NA	NA
Ortho-P (when equating dissolved with Ortho-P)	NA	NA	Filter immed[9], Ice-4ºC 500 mL P, FP	48 hrs	NA	NA	NA	NA
pH	None 8 oz G	Not specified	None 500 mL P, FP	Immed except 24 hrs for RCRA[12]	None 8 oz G	24 hrs for aqueous, otherwise not specified	NA	NA
Settleable Solids	NA	NA	Ice-4ºC 1 L P, FP	48 hrs	NA	NA	NA	NA
Solids Series (TS, TSS, TDS, TVSS, etc.)	NA	NA	Ice-4ºC 1L P, FP	7 days	NA	NA	NA	NA
Sulfates	Ice-4ºC 8 oz G	Not specified	Ice-4ºC 500 mL P, FP	28 days	NA	NA	NA	NA
TOC	Ice-4°C 8 oz G	Not Specified	Ice-4ºC, H_2SO_4 to pH<2 500mL P, FP	28 days	NA	NA	NA	NA
Dissolved TOC	NA	NA	Filter immed[9] , Ice 4ºC, H_2SO_4 to pH<2 500mL, P, FP	28 days	NA	NA	NA	NA
Turbidity	NA	NA	Ice-4ºC 500 mL P, FP	48 hrs	NA	NA	NA	NA

Table 3-1
Recommended Preservation & Holding Times

Analytical Group	Soil/Sediment [1]		Water [1, 2] and Waste Water		Waste		Tissue	
	Pres[3]/ Amt[4]/ Container Type[5]	**Hold[6]**	**Pres[3]/ Amt[4]/ Container Type[5]**	**Hold[6]**	**Pres[3]/ Amt[4]/ Container Type[5]**	**Hold[6]**	**Pres[3]/ Amt[4]/ Container Type[5]**	**Hold[6]**
Organics								
Alcohol - Percent	NA	NA	Ice 1gal.G/	Not Specified	None 8 oz G	Not Specified	NA	NA
TCLP Extractables (Pesticides, Herbicides, Semivolatiles)	Ice-4ºC 8 oz G	61 days[13]	Ice-4ºC 1 L (x2 per fraction)[15] G/A	61 days[13]	None 8 oz G	61 days[13]	NA	NA
Extractables (Pesticides/PCBs, Herbicides, Semivolatiles)	Ice-4ºC 8 oz G	54 days[14]	Ice-4ºC 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	3 ml of 10 % sodium thiosulfate per gallon 1 L (x2 per fraction)[15] G/A	44 days[17]	NA	NA	NA	NA
Flashpoint	N A	NA	NA	NA	None 8 oz G	Not specified	NA	NA
Methane/Ethane/ Ethene	NA	NA	HCL (pH<2), Ice-4ºC 40 mL(x3)[15] G/S	14 days	NA	NA	NA	NA
Org Halide (TOX)	Ice-4ºC 8 oz G	28 days	Ice-4ºC H_2SO_4 to pH<2 1 L G/A	28 days	NA	NA	NA	NA
Perfluorocarbons	NA	NA	Ice-4-15ºC lab ambient+ 10-40 % acetonitrile 125-mL Polypropylene (x2)	194 days[27]	NA	NA	NA	NA
Carbamates	NA	NA	Ice-4ºC 60-mL G/A (x2)	14 days[28]				
Phenols (analyzed as semivolatile compounds)	Ice 4ºC 8 oz G	54 days[14]	Ice-4ºC 1 L(x2)[15] G/A	47 days[16]	None 8 oz G	54 days[14]	Ice & Freeze 30 g Al Foil	Not specified
Volatile Organics								
Volatile Organics Method 5035A	Ice-4ºC 5 g (x3)[18] E or equivalent[19]	48 hours iced/14 days frozen[20]	NA	NA	Ice-4ºC 8-oz G[26]	14 days[21]	NA	NA
Volatile Organics Method 5035A	Ice-4ºC 5 g (x3)[18] into tared 40-mL VOA vials[19]	48 hours iced/14 days frozen[20]	NA	NA	NA	NA	NA	NA

Table 3-1
Recommended Preservation & Holding Times

Analytical Group	Soil/Sediment [1]		Water [1, 2] and Waste Water		Waste		Tissue	
	Pres[3]/ Amt[4]/ Container Type[5]	**Hold[6]**	**Pres[3]/ Amt[4]/ Container Type[5]**	**Hold[6]**	**Pres[3]/ Amt[4]/ Container Type[5]**	**Hold[6]**	**Pres[3]/ Amt[4]/ Container Type[5]**	**Hold[6]**
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 $Na_2S_20_3$, 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 - Hydrochloric Acid used as a preservative must be present at concentrations ≤ 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.

H_2SO_4 - Sulfuric Acid used as a preservative must be present at concentrations ≤ 0.35% by weight (pH about 1.15 or greater), as specified in 40 CFR 136.3, Table II, footnote 3. Approximately 5 ml. of the laboratory-prepared preservative is added to the sample.

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

HNO_3 - Nitric Acid used as a preservative must be present at concentrations ≤ 0.15% by weight (pH about 1.62 or greater), as specified in 40 CFR 136.3, Table II, footnote 3. Approximately 5 ml. of the laboratory prepared preservative is added to the sample.

Chromium VI buffer – A concentrated buffer is used to extend the holding time for hexavalent chromium samples from 24 hours to 28 days and uses constituents as allowed by EPA guidance found at: http://water.epa.gov/scitech/methods/cwa/questions-cr6.cfm. The sample preservation buffer is prepared by carefully dissolving 330 g ammonium sulfate and 50 g sodium hydroxide in about 500 mL of deionized water. The solution is allowed to cool and 260 mL of 29% ammonium hydroxide is added and solution is diluted with deionized water to a final volume of 1L. In house studies revealed that the equivalent of 1% of buffer volume is needed to preserve samples to attain the pH range (pH 9.3 to 9.7, 10 mL buffer for a 1 L sample) as specified in 40 CFR 136.3 Table II. Adding preservative to sample bottles prior to shipment to the fieldis 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 $4^{\pm}2^{\circ}C$ until analyzed (SW-846, Table 3-1).

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

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

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

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

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

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

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

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

CHAPTER 4

General Laboratory Practices

4.1 Good Lab 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 hard-copy 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 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 standard operating procedures (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 SOP shall be coordinated with the appropriate supervisor and Laboratory Quality Manager (LQM) and the rationale for the deviation shall be clearly documented and included in the project file. In those instances where 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 manager.

4.1.3.1 Current revisions of SOPs are located on the LAN at K:\ASB\Current Documents\SOPs. SOPs no longer in use are moved to an 'Archived' folder with the archived date indicated by a watermark on each page of the document. The effective date (which is in the SOP header and incorporated into the unique name of the SOP) and the archived date serve to document the date range during which the SOP was effective.

4.1.4 Manual Peak Integration Electronic data reduction is used for several of the more complex instrumental analysis systems. Analysts are required to review the electronic data

processing for accuracy and consistency with appropriate data reduction techniques. Some electronic reduction can result in incorrect actions by the system software and for these instances manual override and correction of the electronic processing is appropriate. Examples of this may be such items as integration of an incorrect peak, errors in calculations, or misplacement of baseline in peak integration. Guidance related to manual integrations is documented in individual workgroup Data Review Guidelines. When manual override of the electronic process is deemed appropriate, analysts shall document on the hard-copy instrument printouts, which shall be of both before and after the correction, the action taken and why. The action should be concurred by the Secondary Review Analyst. Concurrence is documented by a secondary reviewer dating and initialing 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.

4.1.5 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 check list. Checklists are designed for the procedure(s) being performed. The individual data review check lists for organic and inorganic analyses are maintained on the Region 4 SESD's local network drive (LAN).

4.2 Document Control/File Management

4.2.1 Policy 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 not be necessarily limited to): equipment, 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 and/or the LQM.

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 Archived documents shall be marked as such.

4.2.1.7 ASB's procedures for document control are detailed in SOP ASB 107G, SOP for Document Control.

4.2.2 Internal Chain-of-Custody (COC) ASB analysts check samples in/out of the Custody Room through the LIMS (see SOP ASB 105G).

4.2.3 COC Receipt Form The sample custodian/designee receives a COC record with every shipment of samples (see SOP ASB 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 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 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.

4.2.4.2 Preparation logs shall document all information to reconstruct the preparation such as weights, volumes, lot number of digestion tubes, balance used, reagents 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 Analysis logs: 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.

4.2.4.4 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.

4.2.4.5 Spreadsheets or other calculating software used as logbooks or used in support of data generation in ASB analyses will be controlled. All cells, except information 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 K: drive. Prior to posting and use, all calculations in spreadsheets will be hand-validated by the responsible party and submitted through the technical director 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 K: drive 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:

4.2.6.1 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.

4.2.6.2 The annual review and versions of less comprehensive reviews, as described in section 4.2.6.3 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 4.2.6.1 above.

4.2.6.3 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 (SOPs)/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 ASB 106G (SOP for the Preparation of Standard Operating Procedures). Analytical SOPs for the various methods in use in each laboratory may be placed within the lab 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 may 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 If corrections are deemed necessary to the original after the project file has been completed, the primary analyst or group leader will ensure that a copy of the corrected page(s) are placed in the file and the incorrect copy removed. If the final data reports, either in part or in total, must be corrected or clarified and reported again, a new memo 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.

4.2.7.3 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. At a minimum, the following hard-copy information is retained in the project file.

4.2.7.3.1 COC record(s)

4.2.7.3.2 Final production data reports complete with signed transmittal memos

4.2.7.3.3 All raw data used in the decision making process of obtaining reported results including, but not limited to the items below;

4.2.7.3.3.1 Laboratory sample ID,

4.2.7.3.3.2 Date of analysis,

4.2.7.3.3.3 Instrument ID and operating conditions,

4.2.7.3.3.4 Analysis type,

4.2.7.3.3.5 Documentation of any manual calculations, including manual integrations,

4.2.7.3.3.6 A record of who performed the analysis indicated by the analyst's initials or signature,

4.2.7.3.3.7 Standard traceability and reagent preparation (or references which would include enough information to locate the traceability records),

4.2.7.3.3.8 QA/QC information,

4.2.7.3.3.9 Secondary review check lists,

4.2.7.3.3.10 Instrument output,

4.2.7.3.3.11 Special notes concerning the project,

4.2.7.3.3.12 Copies of logbook pages,

4.2.7.3.3.13 Correspondence (memos or electronic mails or other documents relevant to the project).

4.2.8.4 The analytical information is maintained by analysts while the project is in progress. When completed, the completed data packet is transferred by the group leader or designee to 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. The hard copy project file is the official record and the e-data is not required to be maintained. Ultimate retention and disposal will be according to 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.

4.2.8.4.1 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 this period of time, even if the software/instrumentation has been removed from routine service.

4.2.8 Confidentiality of Data

4.2.9.1 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.

4.2.9.2 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 and 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 supervisor and shall have the supervisor 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 filed according to the ASB Divisional File Plan and with the LQM for general correspondence.

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.

4.2.11 QA/QC Records Maintained within SESD either in the project file or by the LQM.

4.2.12.1 The following raw QA/QC data are typically associated with a project and are found in the project file.

4.2.12.1.1 DOCs, IDLs, MDLs, etc.,

4.2.12.1.2 Records of all spikes, replicates, and surrogates and updates of acceptance limits for each,

4.2.12.1.3 Data related to validation of new methods/techniques,

4.2.12.2 The following QA/QC documents shall be maintained by the LQM,

4.2.12.2.1 General quality assurance records such as performance testing, copies of acceptance limits and updates of acceptance limits,

4.2.12.2.2 Certification of temperature devices, balances and weights,

4.2.12.2.3 Internal and external audit records and responses,

4.2.12.2.4 Copies of method validation studies,

4.2.12.2.5 Special issue investigations,

4.2.12.2.6 Summaries of QC data,

4.2.12.2.7 Summaries of DOCs; IDLs; MDLs,

4.2.12.2.8 Summaries of acceptance limits for QC parameters,

4.2.12.2.9 Managerial reports and memos.

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. All forms will be maintained by the LQM in the appropriate subdirectory at K:\ASB\Current Documents\Forms\. These forms shall be reviewed and revised as necessary. Changes to these 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 K drive. The LQM shall then post the changed copy to the K: drive and notify all appropriate staff of the change completion. Forms will be assigned a version number by the LQM and the most current version will available for access by all staff. It is the responsibility of each staff member to ensure the current version as listed on the K: drive is being used. If there is ever any question, confirmation with the LQM is required. Specific document control procedures are detailed in SOP ASB 107G.

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. 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. 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.

4.3.2 Incubators If an automatic temperature recorder is not used for incubators, place a verified thermometer (preferably within a bottle of water) on a central shelf and record

temperature at least once each working day (more frequently if required by methods/SOPs) when the incubator 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 did 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. 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 then daily checks must resume. Check temperature variations when incubators are loaded to capacity and document this check in the 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.

4.3.3 Water Baths Monitor and record temperature in the analysis log at least once each working day while in use or as may be specified by the method/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 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 2hours is allowed for units equipped with automatic temperature recording devices. Exceedances lasting longer than 2hours 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 analytical methods.

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.

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 SOP ASB 101G: Standard Operating Procedure (SOP) for the Certification of Laboratory Weights.

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 ASB's analytical specifications as stated in SOP ASB101G. 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.

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. This is required on an annual basis and re-certification is coordinated by the LQM as detailed in SOP ASB 100G.

4.3.8 Mechanical Dispensing Devices Mechanical volumetric dispensing devices (except Class A glassware) shall be checked for accuracy on at least a quarterly basis. Glass micro-liter 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 SOP ASB 102G: Standard Operating Procedure (SOP) for the Certification of Laboratory Pipets, Syringes, or Automated Diluters.

4.3.8.1 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 analytical syringes 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 references.

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, date of receipt, any recommended storage conditions, expiration date and a cross reference to the Element® ID assigned to the standard. Certificates of

analysis for purchased standards are maintained in individual laboratories for a minimum of five years after the date of last use.

4.3.10 Major Instrumentation

4.3.10.1 These instruments include but are not limited to the Inductively Coupled Plasma; ICP/Mass Spectrometer (ICP/MS); Gas Chromatograph/Mass Spectrometer (GC/MS); Gas Chromatograph (GCs); Liquid Chromatograph/Mass Spectrometer/Mass Spectrometer (LC/MS/MS); 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 individual SOPs.

4.3.10.3 A list of all major instrumentation, including unique IDs, is maintained on the LAN:ASB directory.

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 lab.

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 Group Leader 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 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 for accuracy; accreditations dates updated, first time use updated, unacceptable supplies noted and suppliers taken off the list, etc.

4.4.2 Glassware

4.4.2.1 Glassware used in general laboratory operations must be high quality borosilicate glass, e.g., "Pyrex" or "Kimax". Volumetric glassware must be Class "A" quality.

4.4.2.2 Clean glassware in accordance with individual SOPs.

4.4.2.3 If a new washing compound or cleaning application is used, tests shall be performed to ensure that the glassware is free of interferences before routine analyses are begun.

4.4.3 Chemicals, Reagents, Solvents, Standards, Gases

4.4.3.1 The quality of chemicals, reagents, solvents and standard gases is determined by the sensitivity and specificity of the analytical techniques being used. Reagents of lesser purity than specified by a method will not be used. When not specified by the method, analytical reagent grade materials should be used.

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 corrective action to determine whether the contamination is the result of unsuitable reagents, 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 for five years after the expiration or consumption of standards.

4.4.3.1.3 If any consumables, supplies or services evaluated through the above procedures prove to be unsuitable for use, the person 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 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.

4.4.3.3 Date all reagents, chemicals, solvents, and standard reference materials when received and when opened or prepared, and discard when outdated, or when evidence of deterioration is detected.

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 is not required.

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 as either: '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 person making these intermediate preparations must have possession of the material and must label it if he or she leaves the material unattended.

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 compounds, 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.

4.4.3.3.5.1 Reagents which are not deemed critical to the success of the analysis, or those that are used in negligible quantities 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 Verification of an expired material will be performed by comparison with the same material from a second source that is within the 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®.

4.4.3.3.5.1 Acceptance Criteria for Verifying Expired Calibration Standard The stability of the expired calibration standard is considered to be verified if:

4.4.3.3.5.1.1 The ICAL prepared using the expired standard meets method acceptance criteria and

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

4.4.3.3.5.1.3 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 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 SOP ASB 112G for chemical purchasing procedures.

4.4.4.5 Receipt of Chemicals The CHO will be listed on the purchase 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 followed in individual labs 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 as recommended by the manufacturer or as indicated by water quality. Date modules when changed.

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 method SOPs.

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

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

4.5.1 Procedures for Satellite Hazardous Waste Accumulation Many laboratory operations necessitate the generation of hazardous wastes (e.g., solvents, acids, etc.) which are required to be near the point of generation. The in-laboratory "satellite" accumulation of such waste should be carefully controlled by the laboratory analyst(s) working with the SHEM so as to avoid creating an unsafe situation and also comply with RCRA temporary storage requirements. Laboratory managers or designees shall conduct periodic walk-through inspections to ensure the proper application of temporary waste accumulation procedures. The quarterly safety inspection by the branch Safety Officer serves this purpose.

4.5.1.1 The RCRA regulations (40 CFR 262.34(c)(1)) permit temporary accumulation of hazardous waste or acutely hazardous wastes at or near the point of generation. Waste accumulated in this manner is considered to be in "satellite accumulation". See SOP ASB 104G for procedures that apply to satellite accumulation of hazardous waste in ASB.

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 **b**e accounted for separately from non-acute wastes. See SOP ASB 104G for procedures that apply to satellite accumulation of acutely hazardous waste in ASB.

4.5.2.1 P-Listed Chemicals When any unused chemical and/or the empty container(s) for a P-Listed Chemical are ready for disposal, the analyst will notify the CHO and coordinate transfer of the items to the CHO. **[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.]**

4.5.3 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. The SHEM should be consulted to verify appropriate procedures.

4.5.4 Non-P-Listed Chemicals Follow all Standard Procedures for disposal as specified in the "SHEMP, Procedures and Policy Manual" and the SOP ASB 112G, Maintaining a Chemical Inventory System. Any questions about disposal of unused chemicals should be referred to the appropriate supervisor or the SHEM.

4.5.5 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, mixed paper, Styrofoam and plastics. This accounts for a large amount of the total waste stream generated by the Branch and Division.

4.5.5.1 Branch management is responsible for ensuring that staff adhere to all Region 4 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.

4.5.5.2 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 operational and cost effectiveness of our overall hazardous waste management program.

4.6 Laboratory Cleanliness

Each analyst is responsible for keeping the laboratories 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 to final data reporting, has associated with it degrees of error. This laboratory does not attempt to quantify total error, since it includes both sampling and analytical error. The purpose of a laboratory quality assurance program is to determine when the analytical measurement error has exceeded acceptance limits for precision and bias. 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. All performance quality control data (Organic and Inorganic Sections) are transferred from the log books and forms to the appropriate quality control logs, data entry forms, or directly into ASB's Laboratory Information Management System (Element®).

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

5.2.7 Batch: environmental samples that are prepared and/or analyzed together with the same process and personnel, using the same lot(s) of reagents. A preparation batch is composed of one to 20 environmental samples of the same matrix, meeting the above mentioned criteria and with a maximum time between the start of processing of the first and last sample in the batch to be 24 hours. An analytical batch is composed of prepared environmental samples (extracts, digestates or concentrates) which are analyzed together as a group. An analytical batch can include prepared samples originating from various environmental matrices and can exceed 20 samples.

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

5.2.9 Blank: sample that has not been exposed to the analyzed sample stream in order to monitor contamination during sampling, transport, storage or analysis. The blank is subjected to the usual analytical and measurement process to establish a zero baseline or background value.

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

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

5.2.9.3 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 pure de-ionized water and appropriate preservative, if any, for the specific sampling activity being undertaken.

5.2.9.4 Instrument Blank: clean sample (e.g., distilled water) processed through the instrumental steps of the measurement process; used to determine instrument contamination.

5.2.9.5 Method Blank: sample of a matrix similar to the batch of associated samples (when available) that is free from the analytes of interest, which is processed simultaneously with and under the same conditions as samples through all steps of the analytical procedures, and in which no target analytes or interferences are present at concentrations that impact the analytical results for sample analyses.

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

5.2.10 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.

5.2.11 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.

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

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

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

5.2.15 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.

5.2.16 Chain of Custody: 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.

5.2.17 Check Standard: reference standard used to verify the concentration of the calibration standard and which is obtained from a source that is independent of the calibration standard.

5.2.18 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.

5.2.19 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.

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

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

5.2.21.1 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.

5.2.21.2 Technical Corrective Action: routine action taken in the laboratory, or elsewhere, to correct expected occasional deviations from method acceptance criteria, instrument malfunctions, or other causes. Trends or frequent re-occurrences of technical corrective actions will initiate a formal corrective action.

5.2.22 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).

5.2.23 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.

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

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

5.2.26 Demonstration of Capability (DOC): procedure to establish the ability of the analyst to generate acceptable accuracy.

5.2.27 Detection Limit: lowest concentration or amount of the target analyte that can be identified, measured, and reported with confidence that the analyte concentration is not a false positive value.

5.2.28 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.

5.2.29 Estimated Detection Limit: based on 40CFR Part 136 Appendix B, an estimate of the detection limit using one of the following:

5.2.29.1 Concentration value that corresponds to an instrumental signal/noise ratio in the range of 2.5 to 5;

5.2.29.2 Concentration equivalent of three times the standard deviation of replicate instrumental measurements of the analyte in reagent water (or matrix of interest);

5.2.29.3 Region of the standard curve where there is a significant change in sensitivity, i.e., a break in the slope of the standard curve; and

5.2.29.4 Instrumental limitations.

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

5.2.31 Field of Accreditation: NELAC's approach to accrediting laboratories by matrix, technology/method and analyte/analyte group.

5.2.32 Holding Time: period of time (usually in hours or days) from sample collection until sample preparation or analysis. The initial time is when a grab sample is collected or the time the last aliquot of a composite is collected. The 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. Samples may be analyzed after the holding time has expired; however, results should be flagged with an appropriate remark. 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.

5.2.33 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.

5.2.34 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.

5.2.35 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.

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

5.2.37 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. For example, in addition to a laboratory duplicate (performed as a matrix duplicate), four replicate analyses are performed for a Demonstration of Capability and seven replicates are performed for an MDL study.

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

5.2.39 Limit of Detection (LOD): estimate of the minimum amount of a substance that an analytical process can reliably detect. An LOD is analyte-and matrix-specific and may be laboratory-dependent.

5.2.40 Limit of Quantitation (LOQ): see **Minimum Reporting Limit.**

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.

5.2.43 Matrix: substrate of a test sample.

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

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

5.2.43.1.2 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.

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

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

5.2.43.1.5 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.

5.2.44 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, for example, 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 Blank: sample of a matrix similar to the batch of associated samples (when available) that is free from the analytes of interest, which is processed simultaneously with and under the same conditions as samples through all steps of the analytical procedures, and in which no target analytes or interferences are present at concentrations that impact the analytical results for sample analyses.

5.2.50 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.

5.2.51 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.

5.2.52 Must: denotes required action.

5.2.53 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 <u>not</u> 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 as tentative or provisional. The amounts reported are calculated relative to known concentrations of other reference materials and as reported are considered to be estimated. These analytes are also often referred to as tentatively identified compounds (TICs).

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

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

5.2.56 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.

5.2.57 Preservation: refrigeration and/or reagents added at the time of sample collection (or later) to maintain the chemical and/or biological integrity of the sample.

5.2.58 Preventive Action: pro-active 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 and is provided to test whether the analyst/laboratory can produce analytical results within specified acceptance criteria.

5.2.60 Pure Reagent Water: shall be 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. Quality control samples may be Certified Reference Materials, Standardized Reference Materials or second-source materials.

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 the 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 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 do so.

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 Analytical Support Branch data reporting.

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 containers depending on the analytical and/or preservation requirements.

5.2.71 Second-Source Material: term typically applied to a quality control 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.

5.2.72 Selectivity: (Analytical chemistry) the capability of a test method or instrument to respond to a target substance or constituent in the presence of non-target substances.

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

5.2.74 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.

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

5.2.76 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.

5.2.77 Standardized 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.

5.2.78 Target Analyte: individual analyte specifically targeted for analysis by using a method designed and validated for the analyte. The technique will include calibration

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

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

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

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

5.2.82 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.

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

5.2.84 Work Cell: defined as a group of analysts (more than one individual) that share responsibility for a specified analysis.

5.3 Essential Quality Control Requirements

5.3.1 Demonstration of Capability (DOC) Performed initially (prior to the independent analysis of any samples) and with a significant change in instrument type, personnel, matrix, or test method where applicable. Each analyst will have a DOC on file for the portion of the analysis that they work on. Procedures for performing a DOC are detailed in SOP ASB 110G: Standard Operating Procedure for Initial Test Method Evaluations, Demonstrations of Capability and Continuing Demonstrations of Proficiency. When performing an analyst DOC, method requirements take precedence. If analyst DOC requirements are not specified in the method, then SOP requirements are followed. If analyst DOC requirements are not specified in the SOP then the requirements of SOP ASB 110G are followed. If employees have not performed a DOC/CDOP within a three year time frame, a training form and DOC must be conducted again. In special cases, the Technical Director and LQM may approve an alternative to a DOC, but it will be evaluated on a case by case basis.

5.3.1.1 DOC and the Work Cell ASB employs the work cell concept in the organic extraction laboratory. Because the nature of the workflow is such that multiple extractions may be in process at any time, a single analyst usually does not perform the entire extraction start-to-finish, but rather in a shared manner. Each analyst must have a DOC on file for each extraction, and they must have participated in the extraction. When a new analyst joins the extraction lab work cell, either a new DOC must be performed which includes participation of the new analyst, or a DOC must be compiled from four consecutive LCSs.

5.3.1.2 Continuing Demonstrations of Proficiency (CDOP) Procedures for documenting analyst CDOPs (or ongoing DOC) are documented in SOP ASB 110G. CDOPs are documented by technology. For example, an analyst CDOP using Method 8081 (GC-ECD technology) has demonstrated continuing proficiency for all GC-ECD methods for which the analyst has performed an initial DOC.

5.3.2 Limit of Detection (LOD) Performed initially (prior to the analysis of any samples) and with a significant change in instrument type, personnel or test method where applicable. The LOD shall be considered current as long as it has been performed at a frequency required by the method or program. In those instances where an LOD is highly dependent on analyst technique (as opposed to instrumental sensitivity), or is required by method or regulation, Branch Management or the LQM may require LODs to be performed by individual analysts on a case-by-case basis. Procedures for performing an LOD are detailed in SOP ASB 110G. ASB performs two types of LOD studies: 1) MDL studies as specified at 40CFR Part 136 Appendix B and 2) modified MDL studies, hereafter referred to as LOD studies.

5.3.2.1 MDL Studies Performed by ASB when specifically mandated by a test method. In general, this only applies to Initial Demonstrations of Capability (however named) for drinking water methods.

5.3.2.2 LOD Studies ASB only reports non-detects at the LOD by special request. As such, the primary function of LOD studies is in setting Minimum Reporting Levels (MRLs) at a level greater than the LOD. LOD studies in ASB consist of a simple modification to the MDL study in that strict adherence to the LOD to spike concentration ratio of 1:10 is not required (with concurrence of the Section Chief and LQM as detailed in SOP ASB 110G). (When performing an MDL study as specified at 40CFR Part 136 Appendix B for multi-analyte methods, it is highly unlikely that all analytes will meet the requirement that the calculated MDL must be greater than or equal to 0.1 times the concentration chosen for spiking. This typically results in an extensive use of resources performing multiple iterations of the study at varying concentration levels.)

5.3.3 Instrument Calibration

5.3.3.1 Initial Calibration Curve (ICAL) Standard curve with concentrations bracketing the range of interest must be performed prior to sample analysis. Unless otherwise specified in the method, the following items are essential elements of initial instrument calibration:

5.3.3.1.1 Details of the ICAL procedure shall be documented in the method SOP. Refer to SOP ASB 106G for details.

5.3.3.1.2 Raw data records shall be retained to permit reconstruction of the ICAL. Records shall include: calibration date, method, unique instrument ID, analysis date, each analyte name, analyst's initials or signature, concentration and response,

calibration curve or response factor; or unique equation or coefficient used to reduce instrument responses to concentration.

5.3.3.1.3 Sample results must be calculated from the ICAL unless otherwise required by regulation, method or program.

5.3.3.1.4 All ICALs shall be verified with a second source standard, where available. Traceability shall be to a national standard, when commercially available.

5.3.3.1.5 Acceptance criteria for the ICAL shall be established which are appropriate to the calibration technique being used.

5.3.3.1.6 The lowest (non-zero) calibration standard shall be at or below the MRL. Any data reported below the MRL shall be considered to have an increased quantitative uncertainty and shall be reported with a qualifier.

5.3.3.1.7 The highest calibration standard shall be at or above the highest concentration for which quantitative data is reported. Any data above the calibration range shall be considered to have an increased quantitative uncertainty and shall be reported with a qualifier.

5.3.3.1.8 For instrument technology (such as ICP-OES or ICP-MS) with a validated technique by the manufacturer or method using standardization with a zero point and a single point calibration the following criteria will apply:

5.3.3.1.8.1 The Linear Dynamic Range of the instrument shall be established as described in the method if quantitative data is to be reported that is at a concentration higher than the calibration standard. The range may also be verified in an analytical sequence by analyzing a standard equal to or higher than the highest concentration in a sample to be reported.

5.3.3.1.8.2 A standard corresponding to the MRL shall be analyzed with each analytical sequence.

5.3.3.1.9 If ICAL results are outside of established acceptance criteria, corrective action shall be performed and all associated samples shall be reanalyzed. If re-calibration and/or reanalysis is not possible, data associated with the unacceptable ICAL shall be appropriately qualified. If analyses continue using an ICAL that does not meet criteria, there must be complete documentation of the process that includes clear justification for the action.

5.3.3.1.10 If the method does not specify the number of calibration standards, at least three points shall be used for the calibration.

5.3.3.2 Continuing Calibration Verification (CCV) An analytical reference standard at a concentration near the mid-point of the initial curve, or as specified by the method, is to

be analyzed at the beginning of each analytical batch and on a frequency determined by the analytical method utilized.

5.3.3.3 Calibration Standard Verification A check standard obtained from a source that is independent of the calibration standard is used to verify the concentration of the calibration standard on a frequency determined by method requirements or by a specified frequency to be established in SOPs. The calibration check standard must be analyzed each time an ICAL is generated and prepared at a minimum of one concentration in the calibration range covered by the ICAL standards.

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. A minimum of 20 results will be required for developing acceptance limits. After the initial limits are determined, they should be updated again as needed or as soon as practical. The time period over which limits are calculated may vary depending on the number of data points collected. Limits are calculated from data stored in Element®. Acceptance limits for recovery (bias) are set at ± three standard deviations from the mean recovery. Precision limits are calculated as relative percent difference (RPD) and are set at zero to three standard deviations of the data set above the mean bias.

5.3.4.1 Recovery limits will not be set tighter than ± 10% around the mean recovery.

5.3.4.2 Precision limits will not be set tighter than an RPD of 10.

5.3.4.3 Limits supplied with SRMs and purchased LCS/QC materials will be used as follows.

5.3.4.3.1 Values supplied with SRMs (such as those purchased from NIST) are supplied with a true value, and an uncertainty range around that true value, which are not meant to be acceptance criteria. In-house acceptance criteria shall be determined for these materials as described above.

5.3.4.3.2 Matrix-specific QC materials are sometimes purchased for use as an LCS (such as nutrients in soil). These materials are typically received with vendor recommended acceptance criteria. Because limits may be dependent on the actual material received, these QC samples will be designated as 'Reference' materials in Element® and vendor-supplied limits will be used for the life of the material rather than attempting to generate specific in-house limits. (The Section Chief and/or LQM may set acceptance criteria tighter than those supplied by the vendor based on experience with the specific analytical method.) Designating these as 'Reference' materials will facilitate segregating results of these samples from LCSs which have been prepared in reagent water or other analyte-free matrix for control charting purposes. While it is desirable to use matrix-specific LCS materials when possible, it is actually quite difficult to locate materials that contain all the analytes of interest at

concentrations amenable to use as an LCS. For this reason, results for these materials are kept separate from the routine LCS database.

5.3.4.4 When 20 data points are not available, interim limits must be established using one of the following guidance procedures.

5.3.4.4.1 Setting Interim Bias (Recovery) Limits If at least seven spiked samples are available, interim limits may be calculated against these seven data points. Statistics indicate when at least seven data points are available, the likelihood of producing representative acceptance limits increases. Recovery limits should be recalculated once 20 data points have been collected. If seven data points are not available, one of the following options should be used.

5.3.4.4.1.1 If guidance on recovery control limits is provided in the referenced method, these control limits should be utilized until at least seven spike values are generated.

5.3.4.4.1.2 If the referenced method does not provide recovery acceptance limits, the acceptance limits from an alternate source may be used. These may include: the acceptance limits from the most recent PT sample for the matrix being analyzed, or limits from a method/analyte expected to perform similarly (e.g., Method 8270C limits could be used for Method 8270D until enough data points have been generated.)

5.3.4.4.1.3 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. In most instances, limits for inorganic analyses should be set at 85-115% and limits for organic analyses at 70-130%.

5.3.4.4.2 Setting Interim Precision Limits Set at an RPD of 20 for most analyses until enough data points are available to generate acceptance criteria. At the discretion of the LQM or Section Chief, interim RPD limits of 30 may be set for historically difficult analyses such as tissue or waste.

5.3.5 Negative Controls

5.3.5.1 Method Blanks are samples of a matrix (when available) similar to the batch of associated samples that is processed simultaneously with and under the same conditions as samples through all steps of the analytical procedures. Method blanks are performed at a frequency of at least one per batch of samples per matrix type per sample preparation method. Results of the method blank analysis are used to assess potential contamination of the associated sample batch.

5.3.6 Positive Controls

5.3.6.1 Bias Refers to the difference between an estimate based on the data and the true value of the parameter being estimated. Bias is expressed as percent recovery (%R) and calculated by formulas below for both LCS and matrix spikes.

5.3.6.1.1 Formulas

Spike Samples

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

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.

5.3.6.1.2 Laboratory Control Samples (LCS) Performed at a frequency of one per batch of samples per matrix type per sample preparation method. The LCS is to be carried through the entire analytical process. Control limits should be established as specified in the section on Acceptance Criteria in this chapter. The LCS 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. ASB uses the LCS to serve as a "best case" indicator of the overall performance of the analytical system.

5.3.6.1.2.1 LCS spikes may be prepared using reference materials (including performance evaluation or proficiency testing samples) or internally-prepared spiking mixtures. Clean matrices such as reagent grade water and sand are used to provide consistency for determining system performance. When clean matrices are not available, an alternate clean matrix such as reagent water shall be used as a substitute.

5.3.6.1.2.2 Components to be spiked shall be as specified by the mandated test method. Any permit specified analytes or client requested analytes shall also be included. If there are no specified components, the laboratory shall spike per the following instructions.

5.3.6.1.2.2.1 For components that interfere with an accurate assessment such as spiking simultaneously with technical chlordane, toxaphene and PCBs, the spike should be chosen that represents the chemistries and elution patterns of the components to be reported.

5.3.6.1.2.2.2 For test methods that have extremely long lists of analytes, a representative number may be chosen. Analytes selected should be

representative of all analytes reported. The following criteria shall be used for determining the minimum number of analytes to be spiked.

5.3.6.1.2.2.2.1 For methods that include 1-10 target analytes, spike all analytes.

5.3.6.1.2.2.2.2 For methods that include 11-20 target analytes, spike at least 10 or 80%, whichever is greater.

5.3.6.1.2.2.2.3 For methods with more than 20 target analytes, spike at least 16 analytes.

5.3.6.1.2.2.3 Over a period of 2 years, all routine target analytes must have been included in the LCS spike.

5.3.6.1.2.2.4 All analyte concentrations shall be within the calibration range of the methods.

5.3.6.1.2.3 The LCS is to be carried through the entire analytical process. Control limits should be established as specified in the section on Acceptance Criteria in this chapter.

5.3.6.1.2.4 Due to some constraints of Element®, the LCSs for some methods are designated as Standard Reference Materials (SRM or REF) in order to correctly evaluate recoveries. For example, pH buffers are designated as SRMs because they are evaluated at ± 0.1 standard pH unit around the true value, which is not a true %R calculation

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

5.3.6.1.2.5.1 Samples which can be analyzed by direct analysis (e.g., metals and nutrients by autoanalyzer) do not require a separate LCS or matrix spike. The ICV for the analytical run serves as both an LCS and matrix spike (reagent water spiked with analytes of interest.). Acceptance criteria for ICVs are always equal to or tighter than LCS and matrix spike acceptance criteria.

5.3.6.1.2.5.2 Samples requiring some type of preparation step prior to analysis (e.g., extraction of equipment rinse blanks or solvent reduction of bottle rinsate blanks) require a LCS. However, the LCS will also serve as the matrix spike because the matrix for the LCS is the same as for the sample (reagent water or solvent). Acceptance criteria for an LCS are always equal to or tighter than matrix spike requirements.

5.3.6.1.2.5.3 Matrix spike duplicates are not required for reagent water matrices because long-term precision in reagent water can be viewed in the control charting function of the LIMS for LCSs.

5.3.6.1.3 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 mandated test method. Unless otherwise allowed by the analytical SOP, a minimum of at least one MS should be prepared per batch of samples for all methods amenable to performing a MS. MS recoveries may be used only to assess the sample matrix which was spiked and not to evaluate matrix effects of non-spiked samples in the associated sample batch. ASB does not qualify any batch results based on the MS analysis. Only the sample spiked is flagged if QC results are outside of the MS limits for that sample.

5.3.6.1.3.1 The analytes to be spiked shall be as specified by the mandated test method. Any permit-specified analytes or client-requested analytes shall also be included. If there are no specified analytes, the laboratory shall spike per the following.

5.3.6.1.3.1.1 For analytes that interfere with an accurate assessment such as spiking simultaneously with technical chlordane, toxaphene and PCBs, the spike should be chosen that represents the chemistries and elution patterns of the analytes to be reported.

5.3.6.1.3.1.2 For test methods that have extremely long lists of analytes, a representative number may be chosen. The analytes selected should be representative of all analytes reported. The following criteria shall be used for determining the minimum number of analytes to be spiked.

5.3.6.1.3.1.2.1 For methods that include 1-10 targets analytes, spike all analytes.

5.3.6.1.3.1.2.2 For methods that include 11-20 target analytes, spike at least 10 analytes or 80%, whichever is greater.

5.3.6.1.3.1.2.3 For methods with more than 20 target analytes, spike at least 16 analytes.

5.3.6.1.3.2 All analyte concentrations shall be within the calibration range of the methods. Over a period of 2 years, all routine target analytes must have been included in the MS.

5.3.6.1.4 Surrogate Spike 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.

5.3.6.1.4.1 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. Sample results with surrogate limits that fall outside acceptance criteria are qualified appropriately.

5.3.6.1.4.2 Acceptance limits are calculated at the same time that control limits for the LCS and MS/MSD are calculated. Surrogate recovery acceptance limits are calculated by Element® in the control chart function of the application.

5.3.6.1.4.2.1 Only results from sample analyses are used to calculate surrogate acceptance criteria. (Note: Blanks and LCS results are not to be used in the calculation of limits; however, surrogate recoveries for these batch QC samples can be viewed in the Element® database and evaluated for trends for use in preventive actions or corrective actions.)

5.3.6.1.4.2.2 Obvious outliers can be flagged for exclusion from the Element® database by using an appropriate 'X-series' qualifier. Any data flagged with an 'X' qualifier will be not be used in determining surrogate control limits if after review, the LQM determines there is a legitimate reason for excluding the data point.

5.3.6.1.4.2.3 Limits are established in the Control Chart function of Element® and are set at ± three standard deviations of the data around the mean recovery of the surrogate.

5.3.6.1.5 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. When analyzing a PT sample, this laboratory shall employ the same calibration, laboratory quality control and acceptance criteria, sequence of analytical steps, number of replicates and other procedures as used when analyzing routine samples. Neither management nor staff shall communicate with another laboratory concerning an open PT study or attempt to obtain the assigned value of a PT sample prior to the close of the study.

5.3.6.1.5.1 Special Handling Procedures Due to the unique nature of PT samples, the following special handling procedures are used.

5.3.6.1.5.1.1 Holding times Most PT samples are received as concentrates in sealed ampules from which whole volume samples are prepared for analysis. Holding times are evaluated against the preparation date of the whole volume, since an actual collection date is not available or practical. An explanatory data qualifier will be added to the data explaining any holding time excursions.

5.3.6.1.5.1.2 Percent Solids Many analyses for soil samples require the use of a wet sample, with conversion of results to a dry-weight basis based on the % solids content of the sample. Most soil PT samples do not contain enough soil for a % solids determination. Experience has shown that these samples are close enough to 100% solids to allow for that assumption to be made. For those analyses which require a % solids number to be entered into Element® for calculation of final results (e.g. cyanide, volatiles, hexavalent chromium), the PT sample numbers will be batched along with a set of samples being prepared for % solids determination, and a result of 100% solids will be entered into Element® even though an actual determination has not been performed.

Note: An exception to this policy occurs in the case of PT soil metals samples. Since metals analyses consume a much smaller amount of sample in the digestion process, and drying of the sample prior to digestion is the normal procedure for other samples, metals' PT samples are dried prior to digestion and an actual % solids value is entered into Element®.

5.3.6.1.5.1.3 Preservation Aliquots for calcium, magnesium, sodium and potassium are received unpreserved in 'Hardness' and 'Mineral' samples for WP PTs. These samples are not preserved prior to preparation and analysis as the preservation aliquot could impact other analytes contained in these samples.

5.3.6.1.5.1.4 Instructions Where PT instructions differ from ASB's normal protocols, the PT instructions will be followed. Examples include sample preparation and significant figures.

5.3.6.1.5.1.5 Additional documentation requirements Many PT samples are received that require preparation steps in addition to the steps required by normal environmental samples. These additional steps are allowed as follows.

5.3.6.1.5.1.5.1 If the PT sample is received as concentrates from which whole volume samples are prepared, the analyst shall document that either the whole volume was prepared according to PT instructions or state exactly how the whole volume was prepared. For those samples received as concentrates, custody is maintained on the concentrate only.

5.3.6.1.5.1.5.2 Air PTs are received in high pressure lecture bottles and must be stepped down to a pressure into a canister that is able to be analyzed. Due to the orders of magnitude difference in pressure from the lecture bottle to the analysis can, multiple attempts at a usable canister pressure are sometimes required. The analyst shall document in the sample preparation/dilution log all attempts including necessary adjustment steps until the final canister for analysis is prepared. Canisters

outside of normal initial pressure ranges may be analyzed only if necessary and only if all preparation steps are properly documented in the log.

5.3.6.1.5.1.6 Ancillary Test Hexavalent chromium analysis in soil requires additional tests to confirm the presence of the analyte. Typically, there is not enough material to perform all of these tests. pH and ORP analyses may be dropped if there is not enough sample to perform these tests.

5.3.6.1.5.1.7 Record Retention Requirements In addition to record retention requirements listed elsewhere in this manual, a copy of all reporting forms and a summary of on-line data entry shall be maintained in the project file.

5.3.6.1.5.1.8 Reporting Requirements For some analyses, the PT Reporting Limit (PTRL) may be less than the routine MRL. If analyte is present at greater than the PTRL, but less than the MRL, report the data as less than the MRL as would be done for routine samples.

5.3.6.1.5.1.9 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.

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

5.3.6.1.7 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.

5.3.6.2 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 as published in the mandated test method. Components of precision are described below.

5.3.6.2.1 Laboratory Replicate Analyses The frequency of analysis of matrix replicates may be determined as part of a systematic planning process (e.g., DQOs) or as specified by the mandated test method. Replicates analyses are usually part of LOD/DOC studies, recovery studies, or in method development studies.

5.3.6.2.1.1 Replicates are performed on replicate aliquots of actual samples, matrix spikes, or an LCS. Precision of replicates is expressed as % relative standard deviation and is calculated by the formula:

$$\% \, RSD = \frac{s}{\overline{X}} \times 100$$

Where: s = Standard Deviation

$\overline{X}$ = Mean

For replicate analysis (any number >2):

$$s = \sqrt{\frac{\sum (X - \overline{X})^2}{n-1}}$$

Where: X = individual observations

$\overline{X}$ = mean

n = number of observations.

Do not use this formula for n = 2. (See Section 5.3.6.2.2.1 for precision calculation when n = 2.)

5.3.6.2.2 Matrix Duplicate Analyses Frequency of the analysis of matrix duplicates may be determined as part of a systematic planning process (e.g. Data Quality Objectives) or as specified by the mandated test method. At a minimum, either a matrix duplicate or MS duplicate (see below) shall be prepared with each batch of samples where the method is amenable to performing one.

5.3.6.2.2.1 The results from matrix duplicates are primarily 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. Precision of duplicates is expressed as relative percent difference (RPD) and is calculated by the formula:

$$RPD = \frac{D}{\overline{X}} \times 100$$

Where: D = Difference between measurements

$\overline{X}$ = Mean

5.3.6.2.3 Laboratory Control Sample Duplicate (LCSD) A LCS may be prepared in duplicate at a frequency of one per batch of samples per matrix type per sample preparation method. An LCSD is optional unless mandated by the method or project specific DQOs.

5.3.6.2.3.1 The results of the LCSDs are primarily designed to assess the precision of analytical results for a specific batch. Results are compared to established limits for that specific matrix if available. If precision results from an

LCS 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" and an appropriate explanatory qualifier.

5.3.6.2.3.2 Precision of the LCSD is expressed as relative percent difference (RPD). Acceptance criteria are calculated as specified above in the section on Acceptance Criteria.

5.3.6.2.4 Matrix Spike Duplicates (MSD) The frequency of analysis of MSDs may be determined as part of a systematic planning process (e.g., Data Quality Objectives) or as specified by the mandated test method. At a minimum, either a matrix duplicate (see above) or MSD shall be prepared with each batch of samples where the method is amenable to performing one and adequate sample volume is received to perform the analysis.

5.3.6.2.4.1 The results from MSDs are primarily designed to assess the precision of analytical results in a given matrix. Results are compared to established limits for that specific matrix if available.

5.3.6.2.4.2 Precision of MSDs is expressed as RPD.

5.3.6.2.4.3 MSD acceptance limits are calculated as specified above in the section on Acceptance Criteria.

5.3.6.2.4.4 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.

5.3.6.3 Internal Standards Added to each organic sample or sample extract analyzed by GC-MS as appropriate as well as samples analyzed for metals by ICP and ICP-MS. Internal standards are also added to all calibration standards and QC samples (method blank, MS/MSD, LCS/LCSD, MRL verification).

5.4 Data Handling

5.4.1 Holding Time Results are considered to be within holding times if the preparation and/or analysis are performed within the recommended period of time. Holding time starts at the end of the composite period for composite samples. Holding times are evaluated in the same units as the Maximum Holding Time Requirements (e.g., Holding times specified in terms of hours will be evaluated based on the hour of collection. Holding times specified in terms of days will be evaluated based on the day of collection.) If analyses are performed outside defined recommended maximum holding times, results will be "J"-qualified and an appropriate explanatory qualifier 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), then for purposes of this evaluation, consider the holding time for that sample to have been exceeded.

5.4.1.1 Element® measures holding times in either hours or days as specified in individual methods in the following manner.

5.4.1.1.1 Sampled to Prepared Holding time is measured from the date (and time) of collection as listed on the Work Order/Sample page to the beginning of sample preparation as recorded on the bench sheet.

5.4.1.1.2 Prepared to Analyzed Holding time is measured from the preparation time listed on the bench sheet to the date (and time) of analysis as either entered manually in the data entry and review screen or imported electronically through DataTool®.

5.4.1.1.3 Sampled to Analyzed Holding time is measured from the date (and time) of collection as listed on the Work Order/Sample page to the date (and time) of analysis as either entered manually in the data entry and review screen or imported electronically through DataTool®.

5.4.2 Minimum Reporting Limit (MRL) 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. An alternative acceptable procedure, when not prohibited by the method, is to analyze a low level check standard at the MRL (or reporting limit) as a sample after the instrument has been calibrated to demonstrate that the instrument is quantitating within acceptance criteria. (This approach is used, for example, in ICP analysis when the instrument is calibrated with a blank and a high standard.) The MRL is established at a level that:

5.4.2.1 Is greater than the LOD;

5.4.2.2 Will meet or exceed the majority of DQO requests for routine projects;

5.4.2.3 Will accommodate convenient preparation of standards and/or check samples (e.g., 5, 10, 20); and

5.4.2.4 Will promote efficient laboratory operations.

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

5.4.3.1 Organic GC/MS data Because GC/MS analysis uses both retention time and a spectral match, there is qualitative evidence for the presence of these analyses at concentrations between the LOD and MRL from the target analyte mass spectrum produced during the analysis.

5.4.3.1.1 Non-detects are reported as less than the MRL.

5.4.3.1.2 Detects between the LOD and the MRL are reported with a 'J'-qualifier.

5.4.3.1.3 Any requests for non-detects to be reported as less than the LOD must be approved by the Section Chief or LQM who will verify that a current LOD study or LOD verification 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 as less than the MRL.

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

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

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

5.4.4 Units

5.4.4.1 Sediment/Soil Percent solids must be determined on all samples unless otherwise specified by the sample requestor. All soil/sediment samples shall be reported on a dry-weight basis.

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

5.4.4.2.1 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.

5.4.4.3 Tissue samples Reported on a wet-weight basis unless otherwise specified by the sample requestor.

5.4.5 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 for a result reported at "75.6" mg/L the analyst would be certain of the "75" , 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; therefore 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, 18th edition) Because the accuracy and/or uncertainty of every procedure is not always precisely known, it is the general practice of ASB to report most analytical results to two significant figures.

5.4.6 Rounding Rules

5.4.6.1 Manual Rounding Round numbers by dropping digits that are not significant. If the digit 6, 7, 8, or 9 is dropped, increase preceding digit by one unit; if the digit 0, l, 2, 3, or 4 is dropped, do not alter preceding digit. If the digit 5 is dropped, round off preceding digit to the nearest even number: thus 2.25 becomes 2.2 and 2.35 becomes 2.4. Use only the digit beyond the last significant figure for rounding. Rounding should be performed only after arriving at the final result in the calculation. (Based on Standard Methods (SM) for the Examination of Water and Wastewater, 18th edition)

5.4.6.2 Rounding in the 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 when rounded up to the MRL are reported as detects. That is, if the MRL is 0.5 and the 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

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, LOD, 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 Calculate the sample mean ($\overline{X}$) and the standard deviation (s) of the data including the suspect extreme value.

5.4.7.3.2 Calculate the ratio

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

5.4.7.3.3 Apply the following decision rule.

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

5.4.7.3.3.2 Critical values of *t* as a function of sample size, n, at the 95% level of confidence (level of significance, α =0.05) are given in Table 5-1.

5.4.7.3.4 Example

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

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

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

The critical value of *t* for $\alpha = 0.05$ and for n=7 is 2.02. The calculated value of *t* 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:

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

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

5.4.7.4.2 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*:

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

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

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 ($Q_{critical}$) 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 (µg/L)
1	40.3
2	41.0
3	40.1
4	38.0
5	40.7
6	41.3
7	41.1

The data sorted in ascending order are:

MDL Rep	Lead (µg/L)
4	38.0
3	40.1
1	40.3
5	40.7

MDL Rep	Lead (µg/L)
2	41.0
7	41.1
6	41.3

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

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

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

5.4.8 Uncertainty Differs from error in that it takes the form of a range of values as opposed to error which is the difference from a true value and is represented by a single value. In most cases ASB uses 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. For most purposes, customers can use the quality control results reported with each final report as an estimate of the uncertainty of the results of their dataset. If ASB is 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.

5.4.8.1 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:

$$100(c / \overline{R})(1 \pm L / \overline{R})$$

Where: c = measured concentration of the analyte

$\overline{R}$ = mean historical LCS recovery

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

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

5.4.8.2 Standard Methods 1030B Measurement Uncertainty

5.5 Data Reporting

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

5.5.1 Procedures for entering and verifying data in Element®

5.5.1.1 All samples must be batched in Element® in order for analyses to proceed. All QC data associated with a batch of samples will be identified by the batch ID assigned by Element®. Data are entered into Element® either manually or electronically through the DataTool® interface. Element® will then calculate a final result for the sample/analyte based on the preparation weights/volumes entered into the batch bench sheet and other calculation parameters established in the analysis. Calculation algorithms are documented in the Element® software, as well as on ASB's K drive.

5.5.1.2 The primary analyst generating the results and a secondary review analyst are responsible for entering, proofing, and verifying the results in Element®.

5.5.1.3 The primary or secondary review analyst is responsible for producing a Draft Report of the results for the Section Chief (or designee). This serves as notification to the Section Chief (or designee) that the project has been completed and is ready for final review and reporting.

5.5.1.4 The Section Chief or designee reviews data for completeness and accuracy, setting the status of data to 'Final Review' and then producing a Final Report.

5.5.1.5 Once the data is released, an e-mail notification, with the data attached in a non-alterable file format (.pdf), is sent to the project leader and/or requestor of the analyses. The "file" hard copy of the final data is sent to SESD's Central Records for inclusion in the appropriate project file and is the official record.

5.5.1.5.1 A hyperlink to a Customer Survey Form on the Region 4 Intranet is included on each email notification of released data. Responses to the survey are delivered to the LQM who forwards them to the appropriate managers. The LQM summarizes the responses for quarterly reports and the management review.

5.5.1.6 If a correction or change needs to be made to reported and transmitted data, the Section Chief is responsible for insuring corrected data is produced and re-transmitted to the customer. A new memo should be created transmitting the corrected data. The report narrative is used to describe the changes to the customer.

5.5.2 Analytical Data Qualifiers Added to data in an effort to best describe the quality of the data to the end-user. These flags are applied during data reduction by primary analysts based on appropriate QC criteria. There are two levels of qualifiers: (1) a descriptive qualifier which denotes a broad qualifier, such as 'J-the result is an estimated value'; and an explanatory qualifier, which as the name implies, explains why the general descriptive qualifier was applied. Explanatory qualifiers are applied to many, but not all of the general

descriptive qualifiers. Refer to the Qualifier Guidance Table on the K drive for a complete list of descriptive and explanatory qualifiers used for ASB data qualifications.

5.5.3 Report Narrative Additional explanatory remarks about the data can be added by **t**he Section Chief (or designee) in the Case 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. 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. ASB routinely reports quality control data with each final report (both specific batch QC results and acceptance criteria) which the customer may use to estimate uncertainty. If a more rigorous treatment is requested by the customer, ASB will use the approach detailed in this manual.

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

5.5.4 Chemical Abstract Service (CAS) Registry Numbers and EPA Identifiers (EPA ID) Each analyte reported from Element® is also reported with the analytes corresponding CAS number. Where a CAS number is not appropriate, an EPA ID number is reported with the analyte. EPA's Substance Registry System (SRS) is the source of CAS numbers and EPA IDs reported with all data. The SRS data base 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.

5.5.5 Opinions and Interpretations ASB rarely, if ever, offers opinions and interpretations of the reported data. However, if included with a laboratory report, both 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.

5.6 Data Management and Data Security

5.6.1 Data is managed using both the Region 4 Laboratory Information Management System (R4LIMS) and a commercially available software package from Promium called 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), 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 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 login to the system using an R4LIMS or Element® application USERID and PASSWORD. An R4LIMS PUBLIC account and the Coldfusion web server, both with limited access as described later are the only exceptions to this requirement. Otherwise, access is controlled by USERID, with varying rights assigned to each user.

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 Waste 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 the LQM after the project has been entered.

5.6.2.1.3.3 Sample logging in Element® is restricted to 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 or 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 and any significant observations 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 LODs/MDLs have been updated.

5.8 QC Study Plans

5.8.1 A QC Study Plan is developed when planning a non-routine LOD/DOC study, method development studies, 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 EPA Region 4 LAN. It should be noted that 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 group leaders 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 review 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 to the complaint/inquiry, which shall be maintained by the LQM.

5.10 Corrective Actions

A corrective action is any action or procedure to rectify non-conformity. The corrective action procedure is detailed in SOP ASB 116G.

5.11 Preventive Actions and Improvements

Preventive Actions consist of pro-active processes to prevent problems or complaints and are used as opportunities for improvement. The preventive action procedure is detailed in SOP ASB 116G.

5.12 Control of Nonconforming Testing Work Nonconforming work is 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 SOP ASB 116G.

5.13 Annual Management Review

During the first quarter of each fiscal year, branch management (Branch Chief and Section Chiefs) and the LQM conduct an annual review of the quality system and environmental testing activities to verify their continued effectiveness and plan for any needed improvements to the system. The review will be documented and maintained by the LQM and will cover the branch'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 ($a = 0.05$, two-sided test).[1]	
Sample size, n	**Critical value ($t_{critical}$)**
3	1.15
4	1.48
5	1.71
6	1.89
7	2.02
8	2.13
9	2.21
10	2.29
11	2.36
12	2.41
13	2.46
14	2.51
15	2.55
16	2.59
17	2.62
18	2.65
19	2.68
20	2.71
21	2.73
22	2.76
23	2.78
24	2.80
25	2.82
[1] Pearson, E. S.; Hartley, H.O., Eds, *Biometrika Tables for Statisticians*, Vol. I, 3rd ed., Cambridge University Press, London, 1966.	

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

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

CHAPTER 6

Methodology

6.1 General

Methods used by the Analytical Support Branch are guided by data quality objectives of specific projects and by program requirements. Occasionally, matrices and samples present analytical challenges or are not amenable to standardized methodology. Deviations from SOPs will be 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. Details on method applications and limitations are found within the SOPs. (Any reference to an analytical method refers to the current version of ASB's SOP for that 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 minimum reporting limit (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 Appendices 1 through 7 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 appendices 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 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 Land Disposal Restrictions (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 level of concerns for LDR regulations are presented in Figure 6-1.

6.4.2 When placing requests for LDR, sufficient lead time will be needed. LDR analyses require special reporting conventions that are not routine for ASB's LIMS. The laboratory will need 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 6-2 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 ASB LQM when analyzing samples for LDR purposes.

Figure 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)	Nonwastewater		
Antimony	6µg/L		1.9 mg/L	1.15 mg/L TCLP	11.5 mg/L TCLP	* See publication at www.epa.gov/ost/pc/revcom.pdf
Arsenic	10 µg/L * proposed in 2002	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	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		1.2 mg/L	590 mg/kg (by 9010 or 9012)	5900 mg/kg (by 9010 or 9012, inferred)	
Cyanides (Amenable)			0.86 mg/L	30 mg/kg (by 9010 or 9012)	300 mg/kg (by 9010 or 9012, inferred)	
Fluoride	4.0 mg/L		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 wstwtr/retort)			NA	0.20 mg/L TCLP	2.0 mg/L TCLP	
Mercury	2 µg/L	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	10 mg/L					
Nitrite	1 mg/L					
Nitrate + Nitrite	10 mg/L					
pH		$\leq$ 2 and $\geq$ 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	

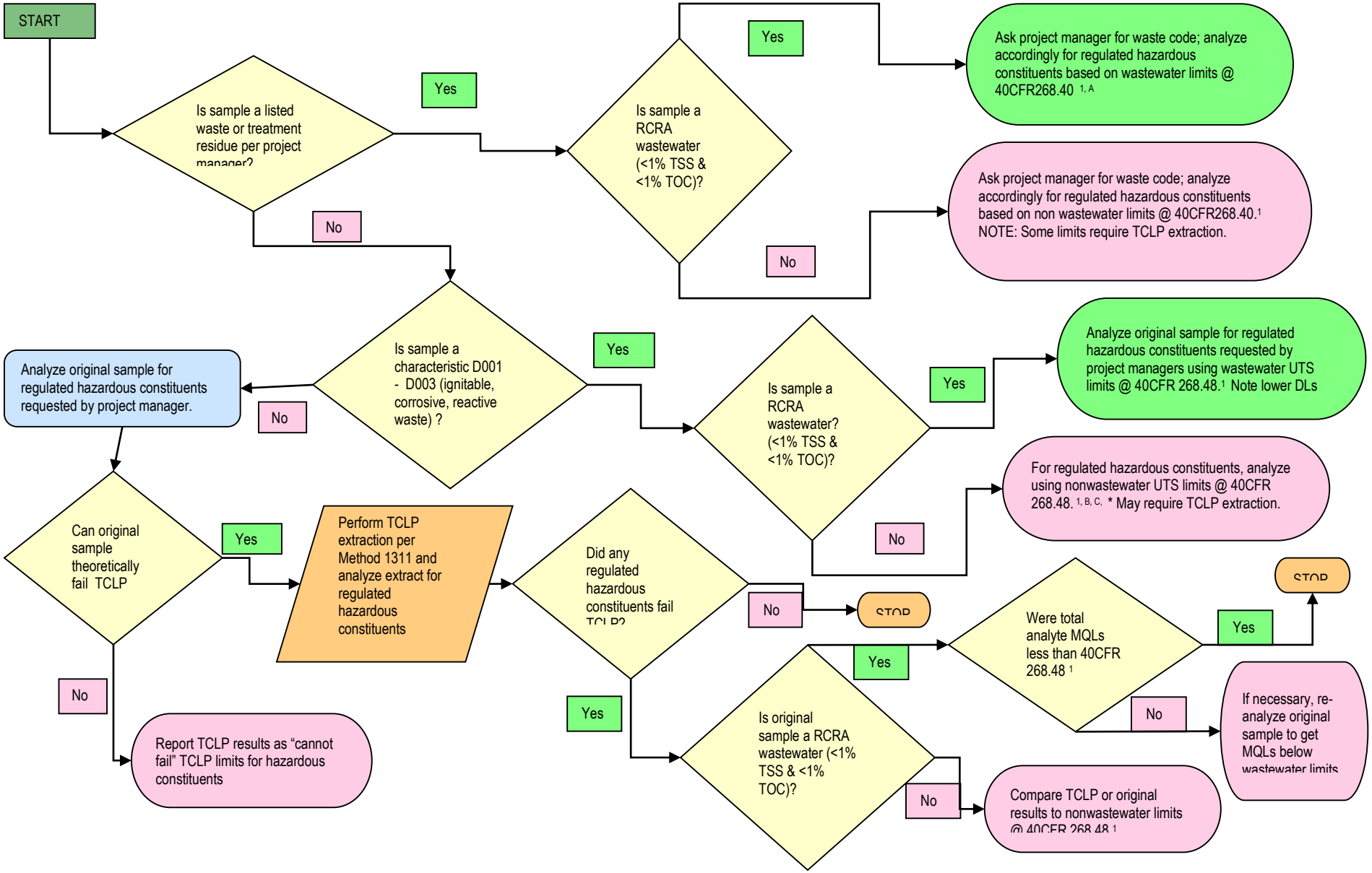

Figure 6-2: Decision Tree for Analysis of Samples for Land Disposal Restriction

Footnotes for Figure 6-2

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

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

[B] 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.

[C] 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 6-1

Capability for Potable Waters-Inorganics					
SDWA Analyte	SDWA MCL (mg/L)	SDWA Method	ASB SDWA MRL (mg/L)	ASB Routine Method	ASB MRL for routine low level request (mg/L)
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	1.3	200.7 or 200.8	0.01	200.7 or 200.8	0.01
Chromium	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
Mercury	0.002	245.1	0.0001	245.1	0.0001
Selenium	0.05	200.8	0.001	200.8	0.001
Thallium	0.002	200.8	0.0005	200.8	0.0005
Asbestos[2]	7MF/L>10u	NA	NA	NA	NA
Bromate[2]	0.010	NA	NA	NA	NA
Chlorite[2]	1.0	NA	NA	NA	NA
Residual Disinfectant[2]	detectable	NA	NA	NA	NA
Cyanide	0.2	335.4	0.015	335.4	0.015
Fluoride	4.0	300.0	0.05	300.0	0.05
Nitrate	10	353.2	0.05	300.0 or 353.2	0.05
Nitrite	1	353.2	0.05	300.0 or 353.2	0.05
Nitrate/Nitrite (as N)	10	353.2	0.05	353.2	0.05
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 units and not mg/L.

Table 6-2

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]	8151A	0.000025
Alachlor	0.002	NA[1]	NA[1]	8141A	0.00005
Atrazine	0.003	NA[1]	NA[1]	8141A	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]	8151A[3]	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]	8151A[3]	0.0000125
Diquat	0.02	NA[2]	NA[2]	NA[2]	NA[2]

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)
Endothall	0.1	NA[2]	NA[2]	NA[2]	NA[2]
Endrin	0.002	525.2	0.002	8081B	0.00005
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]	8151A[3]	0.0000125
Simazine	0.004	NA[2]	NA[2]	NA[2]	NA[2]
2,4,5-TP (Silvex)	0.05	NA[1]	NA[1]	8151A	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.

ASB LOQAM Chapter 6 Appendix 1 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	0.005
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 245/7473[5]	0.10	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*
Titanium	EPA 6010C	5.0	0.5	0.5	0.1
Thallium	EPA 200.8	0.5	0.05	0.05	0.01

ASB LOQAM Chapter 6 Appendix 1
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]
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; Soil, Waste, and Tissue: 7473

* 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.

ASB LOQAM Chapter 6 Appendix 2 Nutrients and Classicals Analyte List Minimum Reporting Limits by Matrices					
ANALYTE	**Analytical Method [5]**	**Water mg/L (ppm)[1]**	**Soil/Sed mg/kg (ppm)**	**Waste mg/kg (ppm)**	**Tissue mg/kg (ppm)**
Acidity	SM 2310	10	NA	NA	NA
Alkalinity	SM 2320B	1.0	NA	NA	NA
Ammonia	EPA 350.1	0.05	2.5[2]	2.5[2]	NA
BOD	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
UBOD	SM 5210C	2.0	NA	NA	NA

ASB LOQAM Chapter 6 Appendix 2
Nutrients and Classicals Analyte List
Minimum Reporting Limits by Matrices

ANALYTE	Analytical Method [5]	Water mg/L (ppm)[1]	Soil/Sed mg/kg (ppm)	Waste mg/kg (ppm)	Tissue mg/kg (ppm)

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

[1] Units as specified unless otherwise noted.

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

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

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

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

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

[7] The 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.

ASB LOQAM Chapter 6 Appendix 3 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/m³)**
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	0.20
Acrylonitrile	EPA 8260C	NA	NA	NA	0.20
Benzene	EPA 8260C EPA 8260C SIM	0.50 0.05	1.0	0.20	0.20
Benzyl Chloride	EPA 8260C	NA	NA	NA	0.20
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	0.20
Bromoform	EPA 8260C EPA 8260C SIM	1.0-4.0 0.10	2.0-10	0.40-1.6	0.20
Bromomethane	EPA 8260C	2.0-5.0	2.0	0.80-2.0	0.20
1,3-Butadiene	EPA 8260C	NA	NA	NA	0.20
n-Butylbenzene	EPA 8260C 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	0.20
Carbon Disulfide	EPA 8260C	2.0	2.0	0.80	0.20
Chlorobenzene	EPA 8260C EPA 8260C SIM	0.50 0.05	1.0	0.20	0.20
Chloroethane	EPA 8260C EPA 8260C SIM	2.0-5.0 0.05	2.0	0.80-2.0	0.20
Chloroform	EPA 8260C EPA 8260C SIM	0.50 0.015	1.0	0.20	0.20
Chloromethane	EPA 8260C	0.50	1.0	0.20	0.20
3-Chloroprene(2-Chloro-1,3-butadiene)	EPA 8260C	NA	NA	NA	0.20
o-Chlorotoluene	EPA 8260C EPA 8260C SIM	0.50 0.05	1.0	0.20	NA

ASB LOQAM Chapter 6 Appendix 3 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/m³)**
ANALYTE	**Analytical Method**	**Routine Level**	**Routine Level (Encore®/ Tared Vial)**	**Routine Level**	**Routine Level EPA TO-15[6]**
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	0.20
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	0.20
1,3-Dichlorobenzene	EPA 8260C EPA 8260C SIM	0.50 0.05	1.0	0.20	0.20
1,4-Dichlorobenzene	EPA 8260C EPA 8260C SIM	0.50 0.05	1.0	0.20	0.20
Dichlorodifluoromethane (R12)	EPA 8260C EPA 8260C SIM	0.50 0.015	1.0	0.20	0.20
1,1-Dichloroethene[7]	EPA 8260C EPA 8260C SIM	0.50 0.015	1.0	0.20	0.20
cis-1,2-Dichloroethene	EPA 8260C EPA 8260C SIM	0.50 0.015	1.0	0.20	0.20
trans-1,2-Dichloroethene[7]	EPA 8260C EPA 8260C SIM	0.50 0.015	1.0	0.20	0.20
1,1-Dichloroethane[7]	EPA 8260C EPA 8260C SIM	0.50 0.015	1.0	0.20	0.20
1,2-Dichloroethane[7]	EPA 8260C EPA 8260C SIM	0.50 0.015	1.0	0.20	0.20
1,2-Dichloropropane	EPA 8260C EPA 8260C SIM	0.50 0.015	1.0	0.20	0.20
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	0.20

ASB LOQAM Chapter 6 Appendix 3 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/m³)**
ANALYTE	**Analytical Method**	**Routine Level**	**Routine Level (Encore®/ Tared Vial)**	**Routine Level**	**Routine Level EPA TO-15[6]**
Dichlorotetrafluoroethane (R114)	EPA 8260C	NA	NA	NA	0.20
trans-1,3-Dichloropropene	EPA 8260C EPA 8260C SIM	0.50 0.050	1.0	0.20	0.20
1,4-Dioxane	EPA 8260C	NA	NA	NA	0.20
Ethyl acetate	EPA 8260C	NA	NA	NA	0.20
Ethyl benzene	EPA 8260C EPA 8260C SIM	0.50 0.05	1.0	0.20	0.20
4-Ethyltoluene (1-Ethyl-4-methyl benzene)	EPA 8260C	NA	NA	NA	0.20
Heptane	EPA 8260C	NA	NA	NA	0.20
Hexachlorobutadiene	EPA 8260C EPA 8260C SIM	0.50 0.05	1.0	0.20	0.20
Hexane	EPA 8260C	NA	NA	NA	0.20
Isooctane (2,2,4-Trimethylpentane)	EPA 8260C	NA	NA	NA	0.20
Isopropanol	EPA 8260C	NA	NA	NA	0.20
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	0.20
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	0.20
Methyl-t-butyl ether	EPA 8260C EPA 8260C SIM	0.50 0.015	1.0	0.20	0.20

ASB LOQAM Chapter 6 Appendix 3 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/m³)**
ANALYTE	**Analytical Method**	**Routine Level**	**Routine Level (Encore®/ Tared Vial)**	**Routine Level**	**Routine Level EPA TO-15[6]**
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
Styrene	EPA 8260C EPA 8260C SIM	0.50 0.05	1.0	0.20	0.20
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	0.20
Tetrachloroethene	EPA 8260C EPA 8260C SIM	0.50 0.05	1.0	0.20	0.20
Tetrahydrofuran	EPA 8260C	NA	NA	NA	0.20
Toluene	EPA 8260C EPA 8260C SIM	0.50 0.05	1.0	0.20	0.20
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	0.20
1,1,1-Trichloroethane[7]	EPA 8260C EPA 8260C SIM	0.50 0.05	1.0	0.20	0.20
1,1,2-Trichloroethane	EPA 8260C EPA 8260C SIM	0.50 0.015	1.0	0.20	0.20
Trichloroethene	EPA 8260C EPA 8260C SIM	0.50 0.015	1.0	0.20	0.20
Trichlorofluoromethane (R11)	EPA 8260C EPA 8260C SIM	0.50 0.015	1.0	0.20	0.20
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	0.20
1,2,4-Trimethylbenzene	EPA 8260C EPA 8260C SIM	0.50 0.05	1.0	0.20	0.20
1,3,5-Trimethylbenzene	EPA 8260C EPA 8260C SIM	0.50 0.05	1.0	0.20	0.20
Vinyl acetate	EPA 8260C	NA	NA	NA	0.20
Vinyl bromide (Bromoethene)	EPA 8260C	NA	NA	NA	0.20

ASB LOQAM Chapter 6 Appendix 3
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/m³)
ANALYTE	**Analytical Method**	**Routine Level**	**Routine Level (Encore®/ Tared Vial)**	**Routine Level**	**Routine Level EPA TO-15[6]**
Vinyl chloride[7]	EPA 8260C EPA 8260C SIM	0.50 0.015	1.0	0.20	0.20
o-Xylene	EPA 8260C EPA 8260C SIM	0.50 0.05	1.0	0.20	0.20
(m- and/or p-) Xylene	EPA 8260C EPA 8260C SIM	1.0 0.10	2.0	0.40	0.40

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

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

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

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

[4] Air - 250 cc from 6-L passivated canister - nominal values. MRLs in µg/m³ units depend on molecular weight and vary depending on the analyte and the standard lot.

[5] Naphthalene not routinely reported by VOC 8260 or TO-15analysis 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.

NA = Not Available

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

ASB LOQAM Chapter 6 Appendix 4 Semivolatile Organics Target Analyte List Minimum Reporting Limits by Matrices					
		Water[1] µg/L (ppb)	Soil/Sed[2] µg/kg (ppb)	Waste[3] mg/kg (ppm)	Tissue[4] mg/kg (ppm)
ANALYTE	**Analytical Method**	**Routine Level**	**Routine Level**	**Routine Level**	**Routine Level**
1-Methylnaphthalene	EPA 8270D	2.0	66	20	0.066
1,1'-Biphenyl	EPA 8270D	2.0	66	20	0.066
1,4-Dioxane	EPA 8270D	2.0	66	NA[6]	NA[6]
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	10 - 20	330 - 660	100 - 200	0.33 – 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
4-Chloro-3-methylphenol	EPA 8270D	10	330	100	0.33

ASB LOQAM Chapter 6 Appendix 4 Semivolatile Organics Target Analyte List Minimum Reporting Limits by Matrices					
		Water[1] µg/L (ppb)	Soil/Sed[2] µg/kg (ppb)	Waste[3] mg/kg (ppm)	Tissue[4] mg/kg (ppm)
ANALYTE	**Analytical Method**	**Routine Level**	**Routine Level**	**Routine Level**	**Routine Level**
4-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
Diesel Range Organics (C_{10}-C_{28})	EPA 8015C	20	670	200-SA[5]	0.67-SA[5]
Diethyl phthalate	EPA 8270D	10	330	100	0.33
Dimethyl phthalate	EPA 8270D	10	330	100	0.33

ASB LOQAM Chapter 6 Appendix 4
Semivolatile Organics Target Analyte List
Minimum Reporting Limits by Matrices

		Water[1] µg/L (ppb)	Soil/Sed[2] µg/kg (ppb)	Waste[3] mg/kg (ppm)	Tissue[4] mg/kg (ppm)
ANALYTE	**Analytical Method**	**Routine Level**	**Routine Level**	**Routine Level**	**Routine Level**
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.

[6] NA= Matrix not analyzed for compound noted.

ASB LOQAM Chapter 6 Appendix 4
Semivolatile Organics SIM Analyte List – Low Level
Minimum Reporting Limits by Matrices

		Water[1] µg/L (ppb)	Soil/Sed[2] µg/kg (ppb)	Waste mg/kg (ppm)	Tissue mg/kg (ppm)
ANALYTE	**Analytical Method**	**SIM Level**	**SIM Level**	**SIM Level**	**SIM Level**
1-Methylnaphthalene	EPA 8270D SIM	0.1	3.33	NA	NA
2-Methylnaphthalene	EPA 8270D SIM	0.1	3.33	NA	NA
Acenaphthene	EPA 8270D SIM	0.1	3.33	NA	NA
Acenaphthylene	EPA 8270D SIM	0.1	3.33	NA	NA
Anthracene	EPA 8270D SIM	0.1	3.33	NA	NA
Benzo[a]anthracene	EPA 8270D SIM	0.1	3.33	NA	NA
Benzo[a]pyrene	EPA 8270D SIM	0.1	3.33	NA	NA
Benzo[b]fluoranthene	EPA 8270D SIM	0.1	3.33	NA	NA
Benzo[k]fluoranthene	EPA 8270D SIM	0.1	3.33	NA	NA
Benzo[g,h,i]perylene	EPA 8270D SIM	0.1	3.33	NA	NA
Carbazole	EPA 8270D SIM	0.1	3.33	NA	NA
Chrysene	EPA 8270D SIM	0.1	3.33	NA	NA
Dibenz(a,h)anthracene	EPA 8270D SIM	0.1	3.33	NA	NA
Fluoranthene	EPA 8270D SIM	0.1	3.33	NA	NA
Fluorene	EPA 8270D SIM	0.1	3.33	NA	NA
Indeno[1,2,3-cd]pyrene	EPA 8270D SIM	0.1	3.33	NA	NA
Naphthalene	EPA 8270D SIM	0.1	3.33	NA	NA
Pentachlorophenol	EPA 8270D SIM	1.0[3]	33.3	NA	NA
Phenanthrene	EPA 8270D SIM	0.1	3.33	NA	NA
Pyrene	EPA 8270D SIM	0.1	3.33	NA	NA

MRLs may increase due to interferences necessitating smaller extraction amounts, dilutions and moisture content of soil samples. The above analytes can also be analyzed by full scan GC/MS at the stated MRLs.NA = Not available

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

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

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

ASB LOQAM Chapter 6 Appendix 5 Routine Pesticide/PCB Target Analyte List Minimum Reporting Limits (MRLs)* by Matrices					
		Water[1] µg/L (ppb)	**Soil/Sed[2] µg/kg (ppb)**	**Waste[3] mg/kg (ppm)**	**Tissue[4] mg/kg (ppm)**
ANALYTE	**Analytical Method(s)**	**Routine Level**	**Routine Level**	**Routine Level**	**Routine Level**
Aldrin	EPA 8081B	0.05	1.7	SA[5]-0.50	0.020
Heptachlor	EPA 8081B	0.05	1.7	SA[5]-0.50	0.020
Heptachlor epoxide	EPA 8081B	0.05	1.7	SA[5]-0.50	0.020
α-BHC	EPA 8081B	0.05	1.7	SA[5]-0.50	0.020
β-BHC	EPA 8081B	0.05	1.7	SA[5]-0.50	0.020
γ-BHC	EPA 8081B	0.05	1.7	SA[5]-0.50	0.020
δ-BHC	EPA 8081B	0.05	1.7	SA[5]-0.50	0.020
Endosulfan I	EPA 8081B	0.05	1.7	SA[5]-0.50	0.020
Dieldrin	EPA 8081B	0.05	1.7	SA[5]-0.50	0.020
p,p'-DDT	EPA 8081B	0.05	1.7	SA[5]-0.50	0.020
p,p'-DDE	EPA 8081B	0.05	1.7	SA[5]-0.50	0.020
p,p'-DDD	EPA 8081B	0.05	1.7	SA[5]-0.50	0.020
Endrin	EPA 8081B	0.05	1.7	SA[5]-0.50	0.020
Endosulfan II	EPA 8081B	0.05	1.7	SA[5]-0.50	0.020
Endosulfan sulfate	EPA 8081B	0.05	1.7	SA[5]-0.50	0.020
Endrin aldehyde	EPA 8081B	0.05	1.7	SA[5]-0.50	0.020
Endrin ketone	EPA 8081B	0.05	1.7	SA[5]-0.50	0.020
Methoxychlor	EPA 8081B	0.05	1.7	SA[5]-1.0	0.050
γ-Chlordane	EPA 8081B	0.05	1.7	SA[5]-0.50	0.020
α-Chlordane	EPA 8081B	0.05	1.7	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 Appendix 5
Routine Pesticide/PCB Target Analyte List
Minimum Reporting Limits (MRLs)* by Matrices

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

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

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

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

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

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

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

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

ASB LOQAM Chapter 6 Appendix 6 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**	**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	EPA 8081B	SA[5]-0.50	SA[5]-20	SA[5]-0.50	SA[5]-0.020
Chlordene	EPA 8081B	SA[5]-0.50	SA[5]-20	SA[5]-0.50	SA[5]-0.020
α-Chlordene	EPA 8081B	SA[5]-0.50	SA[5]-20	SA[5]-0.50	SA[5]-0.020
trans-Nonachlor	EPA 8081B	SA[5]-0.50	SA[5]-20	SA[5]-0.50	SA[5]-0.020
cis-Nonachlor	EPA 8081B	SA[5]-0.50	SA[5]-20	SA[5]-0.50	SA[5]-0.020
Dicofol	EPA 8081B	0.080	5.0	NA	NA
4,4'-Dichlorobenzophenone	EPA 8081B	0.080	5.0	NA	NA
Chlorobenzilate	EPA 8081B	SA[5]-0.020	SA[5]-0.67	NA	NA
2,4'-DDT	EPA 8081B	SA[5]-0.040	SA[5]-1.3	NA	SA[5]-0.0013
2,4'-DDE	EPA 8081B	SA[5]-0.020	SA[5]-0.67	NA	SA[5]-0.0067
2,4'-DDD	EPA 8081B	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 Appendix 6
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**	**Routine Level**	**Routine Level**	**Routine Level**	**Routine Level**
2,4,5-T	EPA 8151A	0.25	SA[5]-0.83	SA[5]-0.25	NA
2,4-D	EPA 8151A	0.25	SA[5]-0.83	SA[5]-0.50	NA
Silvex (2,4,5-TP)	EPA 8151A	0.25	SA[5]-0.83	SA[5]-0.25	NA
Dalapon	EPA 8151A	1.25	SA[5]-4.2	SA[5]-1.25	NA
Dicamba	EPA 8151A	0.25	SA[5]-0.83	SA[5]-0.25	NA
Pentachlorophenol	EPA 8151A	0.25	SA[5]-0.83	SA[5]-0.10	NA
Picloram	EPA 8151A	0.25	SA[5]-0.83	SA[5]-0.25	NA
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/8082/8151, final extract volume 10 mL; 8276/8141, final extract volume 1 mL; 1668a, final extract volume equals 0.02 mL.
- 35 mL extracted: 8011, final extract volume 2 mL.

[2] Soil - 30 g extracted (reported on dry-weight basis); 8081A/8082/8151, final extract volume 10 mL; 8141 final extract volume 1 mL; 1668a, final extract volume equals 0.02 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; 1668a, final extract volume equals 0.02 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 LOQAM Chapter 6 Appendix 7
Carbamate Pesticide Target Analyte List
Minimum Reporting Limits (MRLs) by Matrices

		Water[1] µg/L (ppb)	Soil/Sed µg/kg (ppb)	Waste mg/kg (ppm)	Tissue mg/kg (ppm)
ANALYTE	**Analytical Method**	**Routine Level**	**Routine Level**	**Routine Level**	**Routine Level**
Aldicarb	ASTM D7645-10	1.0	NA	NA	NA
Aldicarb sulfone	ASTM D7645-10	1.0	NA	NA	NA
Aldicarb sulfoxide	ASTM D7645-10	1.0	NA	NA	NA
Carbofuran	ASTM D7645-10	1.0	NA	NA	NA
Methomyl	ASTM D7645-10	1.0	NA	NA	NA
Oxamyl	ASTM D7645-10	1.0	NA	NA	NA
Thiofanox	ASTM D7645-10	1.0	NA	NA	NA

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

[1] Water - 2-uL direct injection of sample.

NA = Not Available

ASB LOQAM Chapter 6 Appendix 8
Perfluorinated Compound (PFC) Target Analyte List
Minimum Reporting Limits (MRLs) by Matrices

		Water[1] µg/L (ppb)	Soil/Sed µg/kg (ppb)	Waste mg/kg (ppm)	Tissue mg/kg (ppm)
ANALYTE	**Analytical Method**	**Routine Level**	**Routine Level**	**Routine Level**	**Routine Level**
Perfluoro-*n*-hexanoic acid	ASB 100S	0.050	NA	NA	NA
Perfluoro-*n*-heptanoic acid	ASB 100S	0.050	NA	NA	NA
Perfluoro-*n*-octanoic acid (PFOA)	ASB 100S	0.050	NA	NA	NA
Perfluoro-*n*-nonanoic acid	ASB 100S	0.050	NA	NA	NA
Perfluoro-*n*-decanoic acid	ASB 100S	0.050	NA	NA	NA
Perfluoro-*n*-butanesulfonate	ASB 100S	0.050	NA	NA	NA
Perfluoro-*n*-hexanesulfonate	ASB 100S	0.050	NA	NA	NA
Perfluoro-*n*-octanesulfonate (PFOS)	ASB 100S	0.050	NA	NA	NA

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

[1] Water - 10-uL direct injection of sample.

NA = Not Available