COMPREHENSIVE QUALITY ASSURANCE PLAN

for

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Revision 003
October 2018

LABORATORY'S APPROVED SIGNATORIES

Michelle L. Briscoe – President/CEO (effective upon this sign-off)	10/22/18 Date
Hakan Gürleyük – Senior Scientist (Technical Director)	10/22/18 Date
Annie Carter – VP of Operations (Deputy Technical Director)	10/22/18 Date
Frank McFarland – Quality Assurance Manager	10/22/18 Date

1.0 CONTENTS

TABLE OF CONTENTS

TABLES

FIGURES

2.0 Statement of Policy

The Brooks Applied Labs (BAL) is committed to sound and useful quality assurance/quality control (QA/QC) management practices resulting in the production of accurate analytical data. The principal focus of the analytical laboratory is to provide specialized analytical services for trace metals analysis with an emphasis on ultra-low detection limits, metals speciation, and unusual or non-routine matrices.

Obtaining accurate data is dependent upon an effective and consistent quality assurance program. To meet this need, ISO/IEC 17025:2005 (ISO 17025), The NELAC Institute (TNI), and the Department of Defense/Department of Energy Consolidated Quality Systems Manual for Environmental Laboratories (DoD/DOE QSM) standards have been incorporated into BAL's quality assurance program. Internal audits are conducted by BAL, and external audits of BAL's facilities are conducted by ANSI-ASQ National Accreditation Board/ANAB every two years to ensure that BAL meets the requirements of the TNI standards (for TNI accreditation through the Florida Department of Health), the DoD QSM, and ISO 17025 accreditation requirements. BAL is also audited annually under the Department of Energy Consolidated Audit Program (DOECAP) and a document review audit is conducted by ANAB. Reciprocal TNI accreditation is granted for several other states. Refer to the BAL website for a complete list of current accreditations. Additionally, periodic external audits initiated by clients serve to ensure that Brooks Applied Labs continually meets the specific requirements of our clientele.

Brooks Applied Labs' management is committed to compliance with ISO 17025, TNI, and DoD/DOE QSM standards. Additionally, BAL is committed to meeting the requirements of the Current Good Manufacturing Practices (CGMP) regulations and the Clinical Laboratory Improvement Amendments (CLIA) for clinical laboratory testing performed on humans in order to meet the needs of our pharmaceutical and clinical laboratory clients. As such, BAL management is committed to continually improving the quality assurance program. The BAL quality assurance program is implemented through a team effort across the entire laboratory. All personnel concerned with laboratory testing activities within BAL must familiarize themselves with the quality system documentation [this Comprehensive Quality Assurance Plan (CQAP) and all relevant standard operating procedures (SOP)] and implement the documented policies and procedures in their work. A listing of the general considerations and objectives of the overall program are as follows:

- *Maintenance of sample integrity.* Integrity is maintained by following documented and accepted sample handling procedures for the preservation, custody, storage, labeling, and record keeping associated with samples received by the laboratory.

- *Use of approved analytical methods.* Analytical methods and related procedures approved by the EPA are readily available. When available, the approved analytical methods form the basis of BAL SOPs. All deviations from the method are noted and these are read and followed by all analysts. In addition, BAL is on the cutting edge of method development for the analysis of trace metals and speciation. All BAL developed methods undergo rigorous testing and validation before they are approved by BAL scientists for use in the analysis of customer samples.

- *Regular evaluation of analytical results.* The results from quality control tests and from sample analyses are continually evaluated to identify method weaknesses and/or to detect a need for further analyst training.

- *Instrumentation performance and maintenance.* Determination of instrument performance level by frequent calibration and the analyses of performance evaluation samples, and through scheduled preventive maintenance, is documented on a real-time basis. Instrument calibration is performed as part of each analytical procedure.

- *Data reduction and report formatting.* Various levels of data review from acquisition to the final report are incorporated to minimize any potential errors in the final data. The report format is variable from a standard format to a customized data package, with or without electronic data deliverables (EDD).

- *Method performance (precision and bias) documentation.* Data from analyses are monitored using control charts to assess performance and to detect trends.

- *Regular evaluation of the quality system.* Brooks Applied Labs management is committed to continually improving the quality system. Reviews of all standard operating procedures and the CQAP annually, as well as routine audits of the laboratory and management reviews are some of the procedures used to find and correct deficiencies in the quality system.

Brooks Applied Labs' management is committed to professional laboratory practices and to the quality of our analytical testing in providing services to our clients including maintaining their confidential information (including national security concerns) and proprietary rights. The above considerations are documented to ensure the quality of the data generated by BAL. Subsequent sections of this manual detail the various elements of the QA program developed and practiced by the laboratory.

The QA program is structured such that the CQAP is the primary reference for quality policies and procedures. As long as the CQAP contains all of the necessary requirements of the quality system, then no additional SOP is necessary. If there is a discrepancy between the CQAP and any SOP, the policy or procedure contained in the CQAP takes precedence and the discrepancy must be resolved as quickly as feasible. Any written directions that disagree with the CQAP and SOPs is considered a departure from the approved QA plan and may not be followed unless it has been approved by the Technical Director, QA Manager, VP of Operations, or President/CEO of BAL. Refer to Section 8.4 for details on how any exceptional departure from BAL procedure is handled.

The Statement of Policy is issued under the authority of the President/CEO of Brooks Applied Labs.

Michelle L. Briscoe
President/CEO

10/22/18
Date

3.0 Organization and Responsibility

3.1 Duties and Responsibilities of Personnel

The laboratory staff is organized in such a way that all analytical personnel are trained in a variety of laboratory duties. Individuals are specialized in their area of primary responsibility, but training overlaps so that there are always secondary personnel trained to perform the primary functions of staff that may be absent. Specific responsibilities have different minimum qualification requirements. The descriptions of responsibilities and their minimum qualifications are listed in Table 3.1.

TABLE 3.1 RESPONSIBILITIES AND MINIMUM QUALIFICATIONS

Title	Responsibilities	Deputies	Minimum Qualifications
President/CEO	Oversees operations of the laboratory, including: management of personnel and analytical services, contracting, client services, sales & marketing, QA/QC, R&D, budgeting, and financial controls	VP of Operations	Bachelor's degree in physical sciences (advanced degree or equivalent experience preferred) and 15 years experience in the analytical lab business, with 10 years in management positions
VP of Operations Lab Manager	Oversees operations of the laboratory, including: management of all lab personnel and all analytical services, R&D, method development, facilities improvements, and scheduling	President/CEO of Brooks Applied Labs	Bachelor's degree in physical sciences and 6 years experience in the analytical lab business, with 5 years in management positions
Technical Director	Maintains technical oversight of the laboratory, including: maintaining instrumentation and equipment, method development, validation, and approval, R&D, and identification of method limitations which may result in degradation of representativeness of results	President/CEO of Brooks Applied Labs and the VP of Operations both serve as backup Technical Director. If backup exceeds 35 consecutive calendar days, the primary accrediting authority must be notified in writing.	Bachelor's degree in physical sciences (advanced degree or equivalent experience preferred) with 24 hours of college chemistry credits, and 3 years experience in the trace metals analytical lab business, with 1 year in a supervisory position
IT Manager	Maintains servers, backups and cyber security. Ensures laboratory personnel are supplied with secure computers and software necessary to perform their jobs.	President/CEO of Brooks Applied Labs or Web Administrator	Bachelor's degree in physical or computer sciences and 3 years working as an IT professional

Title	Responsibilities	Deputies	Minimum Qualifications
Client Services Manager	Oversees the project management group and manages client projects, including: internal communication of client requirements and reporting to client. Performs report level review of the data prior to issuing reports to clients.	President/CEO of Brooks Applied Labs	Bachelor's degree in physical sciences or equivalent and 3 years experience in the analytical lab business; including at least 1 year in Project Management positions
Project Manager	Manages client projects including: internal communication of client requirements, reporting to client. Performs report level review of the data prior to issuing reports to clients.	Another Project Manager or Project Coordinator	Bachelor's degree in physical sciences or equivalent and 1 year experience in the analytical lab business
Project Coordinator	Assist Project Manager.	Another Project Manager or Project Coordinator	Bachelor's degree in physical sciences or equivalent
LIMS Administrator	Maintains test codes, creates custom EDDs, creates and maintains all Crystal Reports, support the PM group	QA Manager, IT Manager, or Project Manager	Bachelor's degree in physical sciences or equivalent of 3 years of experience in the analytical lab business
QA Manager	Oversees QA group; has the authority and is responsible for implementing, maintaining, and improving the QA program, ensuring that all personnel understand their contribution to the QA program, ensure that communication takes place at all levels regarding the effectiveness of the QA program, evaluate the effectiveness of training, using all available tools to monitor trends and continually improve the QA program. Responsible for ensuring all data undergoes final data review prior to release to clients.	President/CEO of Brooks Applied Labs and Sr QA Associate both serve as backup QA Managers. If backup exceeds 35 consecutive calendar days, the primary accrediting authority must be notified in writing.	Bachelor's degree in physical sciences (advanced degree preferred) and 3 years lab experience with 1 year of applied QA principles. Must have documented training and/or experience in QA/QC procedures and have general knowledge of analytical methods for which data review is performed.
Senior QA Associate	Be able to mirror QA Manager in QA related duties. Cover for QA Manager in their absence. Authorized to perform final review of laboratory data.	A QA Associate or QA Manager	Bachelor's degree in physical sciences and 1 year experience in the analytical lab business or with applied QA principles
Regulatory Compliance Manager - Environmental	Ensure laboratory compliance with all Environmental accrediting authority standards. Scheduling and responding to external environmental compliance audits. Maintaining state and federal environmental accreditations.	Sr. QA Associate or QA Manager	Bachelor's degree in physical sciences and 1 year experience in the analytical lab business or with applied QA principles

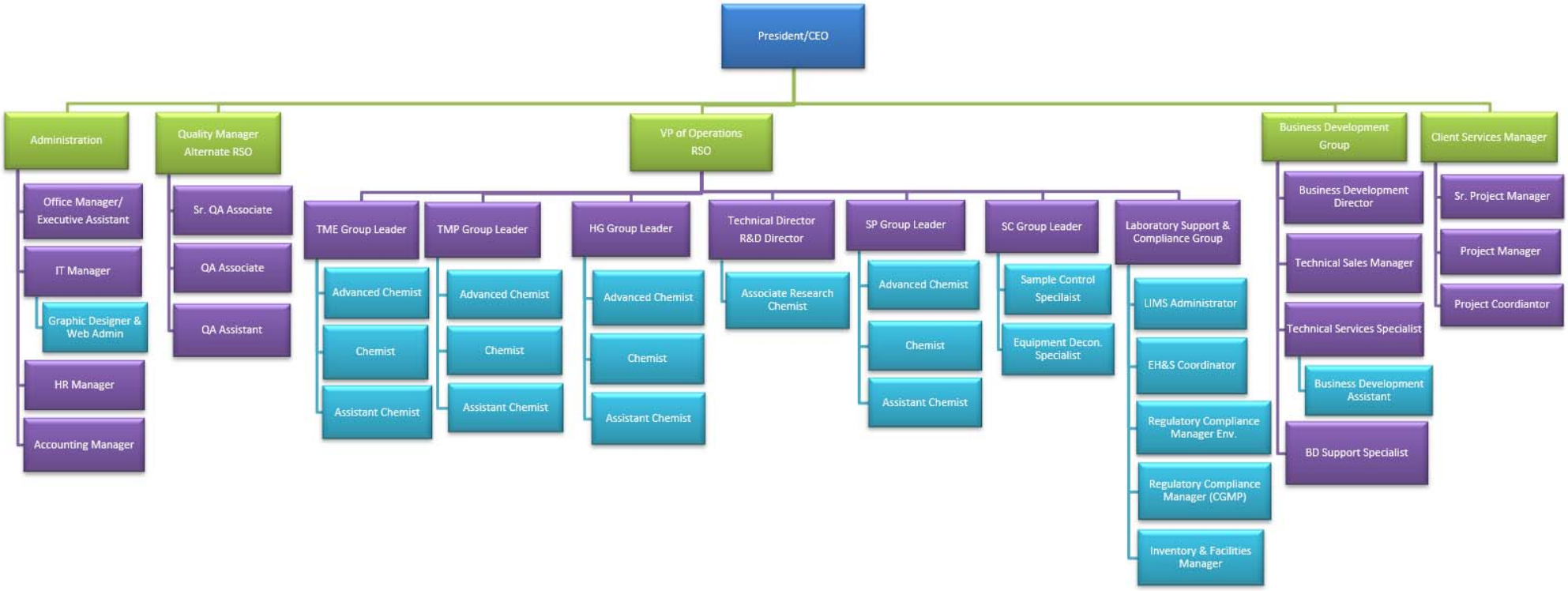
Title	Responsibilities	Deputies	Minimum Qualifications
QA Associate	Assist QA Manager in duties. Authorized to perform final review of laboratory data.	Another QA Associate, Sr. QA Associate, or QA Manager	Bachelor's degree in physical sciences and 6 months experience in the analytical lab business or with applied QA principles
QA Assistant	Assist QA Manager in duties. Performs final review of laboratory data.	Another QA Assistant or QA Associate	Bachelor's degree in physical sciences
HG Group Leader	Oversees the analytical group responsible for Hg, MeHg, and As speciation by Hydride Generation, and related methods including: training records, sample preparation, analysis, detection limit studies, troubleshooting, scheduling within the group, and primary data review.	VP of Operations	Bachelor's Degree in physical sciences and 1 year of analytical lab experience as a metals chemist
TME Group Leader	Oversees the analytical group responsible for trace metals in environmental samples by ICP-MS including: training records, sample preparation, analysis, detection limit studies, troubleshooting, scheduling within the group, and primary data review	VP of Operations	Bachelor's Degree in physical sciences and 1 year of analytical lab experience as a metals chemist
TMP Group Leader	Oversees the analytical group responsible for trace metals in pharmaceutical or other samples requiring cGMP compliance by ICP-MS including: training records, sample preparation, analysis, detection limit studies, troubleshooting, scheduling within the group, and primary data review	VP of Operations	Bachelor's Degree in physical sciences and 1 year of analytical lab experience as a metals chemist
SP Group Leader	Oversees the analytical group responsible for IC-ICP-MS, CN and related methods including: training records, sample preparation, analysis, detection limit studies, troubleshooting, scheduling within the group, and primary data review.	VP of Operations	Bachelor's Degree in physical sciences and 1 year of analytical lab experience as a metals chemist
Advanced Chemist and Special Project Chemist	Perform and document sample preparations and analyses following SOPs, instrument calibration and reagent/standard preparation. Perform primary review of data for methods trained to perform. Train new personnel.	Another Chemist or Group Leader	Bachelor's Degree in physical sciences.
Chemist	Perform and document sample preparations and analyses following SOPs, instrument calibration and reagent/standard preparation. Perform primary review of data for methods trained to perform.	Another Chemist or Advanced Chemist	Bachelor's Degree in physical sciences.
Assistant Chemist	Perform and document sample preparations following SOPs. Perform primary review of data for methods trained to perform.	Another Assistant Chemist or Chemist	A.S. Degree or equivalent schooling in physical sciences

Title	Responsibilities	Deputies	Minimum Qualifications
Sample Control Group Leader	Oversees sample control group including: training, custody of samples, and scheduling within the group. Oversees classical chemistry method for total suspended solids (TSS) performed by the sample control group and sample homogenization. Responsible for ensuring all data produced by the SC Group undergoes primary review prior to turning data in to QA.	VP of Operations	Bachelor's Degree in physical sciences and 1 year of analytical lab experience
Sample Control Specialist	Oversees custody of sample, sample receipt, sample log-in, sample homogenization, TSS and primary review.	Another Sample Control Specialist or Group Leader	High School Diploma (Bachelor's Degree in physical sciences preferred)
Equipment Decontamination Specialist	Cleaning and decontamination of laboratory equipment; sample disposal	Group Leader	High School Diploma (Bachelor's Degree in physical sciences preferred)
Radiation Safety Officer (RSO) and Alternate RSO	Conduct training, identify potential radiation safety issues, review all radiation level surveys, ensures safety of laboratory personnel from radiation risks, ensure compliance with all licenses and regulations.	Another Radiation Safety Officer or the Alternate Radiation Safety Officer	Bachelor's Degree in physical sciences. Current certificate of RSO training.
EH&S Coordinator	Maintain the Chemical Hygiene Plan (CHP), design and implement safety program including training, safety meetings, preventative measures, and responses to accidents/injuries, ensure monthly safety inspections are performed and any deficiencies are corrected.	BAL President/CEO and VP of Operations	Bachelor's Degree in physical sciences.

3.2 BAL Organization

There is a defined chain of responsibility along which the laboratory staff is organized. The organization of the laboratory personnel is shown in Figure 3.2. This organizational chart is current as of the finalization of the current version of the CQAP and is subject to change prior to the next revision of the CQAP.

FIGURE 3.2 – ORGANIZATIONAL CHART



3.3 Managerial Responsibilities for the Detection of Improper, Unethical, and Illegal Actions

Brooks Applied Labs holds management responsible for ensuring that all client data is properly and accurately reported. To this end, management works diligently to ensure that all employees of BAL are free from undue commercial, financial, and any other pressures that may adversely affect the quality of their work. In addition, the management of BAL works proactively to detect any improper, unethical, or illegal actions that might arise due to such pressures before such actions can adversely affect client data.

All information relevant to client results, from sample receipt to the analysis and reporting of data, goes through several levels of review. Management keeps track of all work that appears suspect or contains mistakes and all changes made to the Laboratory Information Management System (LIMS) are electronically tracked and stamped with the date/time of the change and who instigated it. All employees routinely meet with senior management at which time work-related problems are discussed so that any undue pressures can be brought out into the open. Immediate supervisors speak daily with all employees and strive to keep aware of the activities and general attitudes of those employees for which they are responsible. Employee work is additionally reviewed during monthly audits.

All employees receive training to ensure that each is fully aware as to what constitutes improper, unethical, and illegal behavior and what the consequences are for such behavior (refer to BAL SOP BAL-0001) and must sign the "Brooks Applied Labs Ethical and Legal Responsibilities Agreement" form stating that they agree to adhere to all aspects of the ethics program at BAL prior to working with client samples. Any employee who becomes aware of unethical behavior is encouraged to report the behavior to BAL management. Any employee should feel free to report such behavior to any manager, including the President/CEO of BAL. BAL management assures that any reporting of improper, unethical, or illegal behavior will remain strictly confidential. If such behavior is detected, the responsible employee is immediately brought before senior management. If the behavior is unintentional and appears to be caused by undue pressures, every effort is made by management to eradicate the pressures. If the behavior is deemed willful, then senior management is responsible for determining the best course of action for BAL and its clients. Under no circumstances is any behavior that might adversely affect the quality of the data produced by BAL tolerated.

All investigations that result in finding inappropriate activity shall be documented and shall include any disciplinary action that was taken, corrective actions taken, and all appropriate notifications of affected clients. All instances of inappropriate and prohibited practices must be reported to the Accrediting Bodies (AB) within 15 business days of discovery and records of corrections taken or planned corrective actions reported to the AB within 30 business days of discovery. It is the responsibility of the AB to notify the Environmental Data Quality Workgroup (EDQW) of any issues that might affect DoD projects. All documentation of the investigation's actions shall be maintained for a minimum of 7 years. Documentation may be stored either electronically or by hardcopy but must be readily available.

4.0 Training

4.1 Technical Training

Brooks Applied Labs personnel are trained prior to the analysis of client samples. An experienced chemist who has previously demonstrated their capability to perform the procedures for which a new employee is being trained directly supervises all training.

Brooks Applied Labs is also on the cutting edge in the development of new techniques for the analysis of trace metals. In such situations where a scientist is developing a new technique, they must train themselves in the new procedures. The lead scientist is expected to develop the training protocol by which future technicians will be trained in the method.

Regardless of how training takes place, an initial demonstration of capability (IDOC) for all accredited analytes/methods must be successfully completed as per ISO 17025, TNI, and DoD/DOE QSM standards prior to any preparation or analysis of client samples. The IDOC serves as an indicator of the successful completion of training. From then on, the consistent meeting of quality control criteria serves as the ongoing DOC. The QA Manager reviews the ongoing DOC annually to ensure the continued proficiency of each technician. The conclusions of these reviews are recorded in each employee's training records.

4.2 Safety Training

All BAL employees receive training in laboratory safety that they are required to review on an annual basis. Safety training includes "Right-to-Know" training as to the potential chemical and physical hazards of working in an analytical laboratory and how best to reduce these hazards. Training also includes what procedures to take in the case of a laboratory accident and the locations of all safety and first aid equipment.

4.3 Training in Legal and Ethical Rights and Responsibilities

All Brooks Applied Labs employees receive initial and then annual training in their legal and ethical rights and responsibilities. This training includes the following topics: Freedom from undue-pressures; data manipulation, and workplace ethics. This training also specifies the potential punishments and penalties for improper, unethical, or illegal actions performed by BAL employees. The BAL data integrity plan (SOP BAL-0001) discusses in detail the specific legal and ethical rights and responsibilities of employees at BAL. Training on this SOP is required for all personnel.

In conjunction with this training, each employee must attest that they are free from any commercial, financial, and other undue pressures, which might adversely affect the quality of their work prior to working with client samples.

4.4 Documentation of Training

Each employee is responsible for documenting all training in their personal training records and each Group Lead is responsible for the completeness of the training records for all employees within their group. At a minimum, the QA Manager or their designee audits the training records quarterly and reports any deficiencies to the Group Leads and the VP of Operations so that they can be corrected. Training records are updated at least annually to ensure that all personnel continue to be proficient in their assigned tasks. Records for each type of training (i.e. technical, safety, and legal and ethical) are maintained and stored for a minimum of 7 years from when employment at BAL ends.

4.5 Additional Training

The QA Manager or their designee reviews each employee's training records quarterly. Any deficiencies found are recorded in the monthly QA audits. In addition, the technical abilities of each employee are constantly monitored through the analysis of quality control samples. If quality control criteria are not consistently met, additional training is required under the supervision of the VP of Operations. Any additional training is fully documented and a new DOC must be successfully completed before the technician may restart analyzing client samples.

5.0 Capabilities and Quality Assurance Objectives

5.1 Acceptance of New Work

The President/CEO (along with any necessary assistance from the Client Services Manager, project managers, VP of Operations, and the QA Manager, as designated) carefully reviews the specific requirements of every contract before any new work is accepted by BAL. BAL will accept a project only after the President/CEO or their designee has ensured that BAL possesses the appropriate facilities and resources to carry out the work as specified by the client. This is further addressed in Section 6.6 of this document.

5.2 Capabilities of Organization

Brooks Applied Labs is an analytical laboratory primarily focused on providing analytical services for the determination of low-level trace metals and metals speciation in environmental samples, food, biologicals, and pharmaceuticals. BAL's specialties are fourfold: 1) providing the lowest detection limits commercially available, 2) speciation of oxidation state and organometallic forms, 3) analysis of non-routine matrices using specialty/proprietary methods, and 4) providing our clients with expert consulting to ensure their data objectives are met.

Current scopes of accreditation and licenses can be found on the BAL website at http://brooksapplied.com/resources/certificates-permits/.

Each sample preparation method followed at BAL is dependent upon the analyte of interest and the type of matrix being analyzed. Refer to the specific SOP for a description of each particular preparation method utilized at BAL. Other sample preparation methods may be used upon request.

5.3 Quality Assurance Objectives

Brooks Applied Labs is dedicated to providing high quality services to its clients. To meet this objective, every position at BAL is staffed with trained personnel and competent managers who possess the authority and resources to produce meaningful metals data that meet the needs of our clients.

The primary purpose of the Quality Assurance Program at BAL is to ensure that all data reported to our clients are accurate and reproducible. To this end, BAL implements procedures to ensure that all staff are qualified and fully trained to perform their specific laboratory duties, that laboratory instrumentation is properly maintained and calibrated, and that materials are adequately stocked and tested prior to use in the laboratory. All data reported by the laboratory undergoes several levels of review before being approved for release by the Quality Assurance Group.

5.4 Subcontracted Work

Occasionally, a client may wish to work directly through Brooks Applied Labs even for analyses that BAL does not currently perform. Under these conditions, BAL may subcontract work to other laboratories with the client's approval in writing (typically an email or signed laboratory service agreement referencing the quotation). The subcontracted laboratories must meet all project specific requirements, including required accreditations, before samples may be delivered. BAL is responsible to our clients for the subcontract laboratory's work, except in cases where the client specifies which subcontract laboratory is to be used.

Subcontract laboratories are selected by BAL based on a culmination of many factors. These include, but are not limited to, laboratory capabilities, past work experience, data quality (including data presentation), accreditation / certification, price, turnaround time, customer service, and electronic data deliverable (EDD) capabilities. Example reports can be requested from a potential subcontract laboratory.

Once subcontracted work has been contracted between BAL and the client, a purchase order is issued by BAL to the subcontract laboratory.

All samples are logged into the BAL LIMS and a subcontract order is created. When samples are submitted to the subcontract laboratory, they are accompanied with a copy of the subcontract order.

The subcontract laboratory will email the appropriate BAL Project Manager with the final report and EDD (if requested). The Project Manager reviews level II reports. Level IV reports need to be forwarded to the QA Manager. Level IV report data is reviewed by the QA Group and notes are written regarding any issues with the subcontracted data set. If there are outstanding issues, the Project Manager will contact the subcontract laboratory and work to resolve the issue/gain clarification. Depending on the reporting level, BAL may supply a cover letter to the client regarding the data set provided by the subcontract laboratory. Final reports (and EDDs) will be emailed by the Project Manager to the client.

6.0 Sampling Procedures and Requirements

6.1 Sampling Capabilities

Brooks Applied Labs conducts field sampling on a very infrequent basis. Therefore, the following sampling procedure topics are only briefly addressed:

Sampling Equipment - All sampling equipment is decontaminated and/or tested prior to and following every sampling event and stored in a secure designated area. Any equipment requiring calibration or maintenance, as specified by the manufacturer's instructions, is placed on a routine calibration/maintenance schedule.

Field Sample Documentation - During site visits, minimal notes regarding specific field parameter measurements, general observations, hydrologic conditions, and overall suitability are documented, if applicable. These notes are entered as Work Order Memo in BAL's LIMS for the related project(s).

Sample Dispatch - Field samples are relinquished by the sample collection team to BAL's Sample Control Group following a strict chain-of-custody process. Time, date, samplers' and receipt signatures, and any relevant environmental conditions are documented.

Field Reagent and Waste Disposal - All field reagents and wastes generated or used during field sampling activities should be collected and disposed of in accordance with all state and federal regulations.

6.2 List of Equipment Provided by BAL for Sampling

TABLE 6.2 - CONTENTS OF BAL SAMPLING KITS

Equipment	Construction	Use	Parameter Groups
Sample Container(s)	Teflon® – FEP, PFA Fluorinated – FLPE Glass – I-Chem 200 series HDPE (for solids only) zip-type bags (some biota)	Sampling/Storage	Mercury and monomethyl mercury in water/soil/biota
	HDPE bottles LDPE, HDPE, or PP jars zip-type bags	Sampling/Storage	Trace Metals and metals species, except Hg and MeHg, in water/soil/biota
	Iodated carbon (IC) Traps or Gold Coated Media in zip-type bags	Sampling/Storage	Mercury in air
Sampling Equipment	Teflon® tubing, inline filter units	Sampling	Trace metals and mercury in water

Shipping Containers	Plastic cooler or cardboard box	Sample Transport	All parameter groups
Gloves, Clean Room	Vinyl, non-powdered	Sampling	All parameter groups
Trip Blanks	FEP, PFA, FLPE, HDPE bottle (dependent on analysis) filled with reagent water and sealed with custody seal.	Test for potential contamination during transit	Mercury, monomethyl mercury, trace metals and metals species.
Reagent Water	FEP, PFA, FLPE, HDPE bottle (dependent on analysis) filled with reagent water	Rinsing of equipment in the field, preparation of field blanks	Mercury, monomethyl mercury, trace metals and metals species.

6.3 Decontamination Procedures

Client Equipment

Brooks Applied Labs supplies sampling equipment and sample containers for all analyses. Clients may provide their own sample containers at their discretion, but BAL cannot guarantee the cleanliness of containers that have not been cleaned and/or tested by BAL.

Cooler/shipping containers

All coolers are cleaned prior to use for shipping samples or sample containers. Appropriate sample containers are placed in the coolers or boxes to make a sampling kit. Sampling kits are sent via freight carrier (UPS or FedEx) to the requested location.

Sample Containers

Due to the possible occurrence of false positive results due to trace metals contamination, it is extremely important that all water samples are collected in rigorously acid-cleaned or pretested containers that are double-bagged in poly bags and suitable to the analyses to be performed.

6.4 Sampling Protocol

Brooks Applied Labs recommends that all samples to be analyzed for trace metals are collected following the guidelines laid out in EPA Method 1669 (7/96): Sampling Ambient Water for Trace Metals at EPA Water Quality Criteria Levels, unless alternative procedures are more appropriate (e.g., for speciation analyses by IC-ICP-MS).

6.5 Sample Preservation, Holding Times, Container Types, and Required Volumes

All preservation reagents used by Brooks Applied Labs are reagent grade or better. For total metals analyses, samples not being filtered in the lab are typically sent to BAL at ambient temperature via ground shipment. For most speciation parameters, containers are sent to the field pre-preserved or the field sampling crew can add the appropriate preservative. Samples can also be sent to BAL on ice via overnight shipping to be preserved at BAL. All samples for “dissolved” analyses must be filtered before preservation. Refer to SOP BAL-2000 for the Container Details and Preservation Requirements table.

6.6 BAL's Policy on Accepting Samples

Brooks Applied Labs will only accept samples for analysis from parties with whom a written contract or agreement has been jointly signed or otherwise agreed to in writing, or from long-time clients in good standing with BAL's accounts receivable department. If a signed contract is not in place, samples are still received and placed on "hold." Any time-sensitive work, such as preservation or filtering, is still performed and the samples are stored appropriately. Once the Project Manager establishes the contracting paperwork, the samples are taken off hold status and the agreed to TAT begins. The terms under which BAL would enter into a contract are explained in section 5.1 of this document.

Once a legal contract is in place, BAL will accept client samples even if the samples have not been preserved or handled properly, but all evidence of improper preservation and/or handling will be fully documented by BAL at the time of receipt. Examples of improperly handled samples include those with holding time exceedances, temperature exceedances, pH exceedances, improper container, evidence of improper sampling technique, improper handling (i.e., broken custody seal), improper documentation of samples on the chain-of-custody (COC) form, etc.

In the case of samples that have been collected or preserved improperly, the Project Manager immediately contacts the client to determine whether the client desires to continue with the analysis of samples. At the request of the client, BAL will perform analyses of samples even if they have non-conformance issues, but all such samples shall have their results qualified to indicate the non-conformance.

In the case of improperly documented samples, the Project Manager will contact the client to clarify any questions concerning the COC before samples are batched.

At all times, BAL reserves the right to refuse to accept samples at their sole discretion. Typically this would be reserved for samples that are deemed to be a threat to the health or safety of BAL personnel beyond what might reasonably be expected while working within an analytical laboratory.

Refer to the sample receiving SOP (BAL-2000) for specific information pertaining to BAL's sample acceptance policy.

7.0 Sample Custody

7.1 Field Custody

Formal custody requirements begin at BAL with the shipment of sampling equipment and containers to the field. Every shipment must be documented by BAL. With the exception of HDPE bottles sent for the collection of samples for arsenic speciation by hydride, Se speciation, and all solid samples, a minimum of 10% of the bottles from 10% of the cases for each manufacturer lot number (or 10% of the bottles from each acid-cleaning lot number) are tested for all applicable analytes prior to shipment. A certificate of analysis may be generated and supplied with all equipment at the request of the client. BAL provides COC forms and two custody seals with each container shipment. Sample collection dates and times should be provided on the COC by the organization conducting the field sampling. If provided, the COC is used as documentation for sample collection dates and times. In addition, all sample container shipments are documented to track container information such as bottle cleaning batch numbers, preservation lot numbers, quantity of containers shipped, and their date of shipment.

While BAL does not typically provide field sample collection services, we do recommend that certain precautions be taken when collecting samples. Special consideration should be given to the procurement, transportation, preservation, and storage of samples to be analyzed. These procedures are intended to ensure that any analyte originally present in the sample matrix has not degraded and that contamination has not been introduced. See SOP BAL-2000 for container, preservation, and holding time requirements for other analyses.

The courier is responsible for documenting the custody of the samples while the samples are in transit from the field to BAL.

7.2 Laboratory Custody

7.2.1 BAL Definition of Laboratory Custody

A sample is considered to be "in custody" in the laboratory if it meets any one of the following criteria:

- It is in the possession of a BAL employee.
- It was in the possession of a BAL employee, and then locked or sealed to prevent tampering.
- It is in a secure area (i.e., storage).

7.2.2 Sample Receipt

All samples delivered to BAL are received by a sample control specialist or designated alternate in the laboratory receiving area. Upon delivery of samples, the sample control specialist signs and dates the COC form (refer to SOP BAL-2000 for a detailed description of sample receipt and SOP BAL-2001 for example COC).

Immediately after opening the cooler or other container, the sample control specialist confirms the presence of ice, measures sample temperatures (if required), and documents the condition

of the samples (intact, broken, leaking, etc.). The sample control specialist also verifies that each container is properly labeled and sealed and compares the sample ID or field ID against the COC form. The temperature of the samples at the time of receipt is determined with a calibrated IR as outlined in SOP BAL-2000.

For all methods with temperature requirements, the thermal preservation of the samples must be maintained during the sample receiving to storage procedures. Therefore, samples with temperature requirements should be prioritized for login and placed in cold storage (refrigeration or freezer) if there is any doubt that temperatures might go out of criteria during the receiving procedure. The laboratory must document if any required thermal preservation is not maintained during sample receipt and login. If thermal preservation is not maintained, the Project Manager must be notified immediately and the client shall be notified in writing (typically by email).

The sample control specialist is also responsible for ensuring that all samples are properly preserved. If any filtration or analysis of volatile mercury species is required, this should be performed before the preservation of samples. All samples must be preserved in accordance with the preservation instructions in each appropriate analytical methodology. If water samples that do require preservation are preserved in the field, the sample control specialist checks the pH and documents that it meets the acceptance criteria. If it does not, additional preservation reagent is added to the sample(s) and the amount of preservative required to adjust the pH to the appropriate level is documented on the Sample Receiving Log.

If the sample ID listed on the bottle label and the COC form do not match, the custody seals on any of the containers are broken, the temperature of the samples is above the method specified storage limit, or the samples are not properly preserved, the sample control specialist notes the problem directly in the LIMS “Work Order Comments” for the affected work order and notifies the project manager. The project manager then notifies the client of any concerns via email or telephone within 1 business day.

If sample containers arrive with too little sample for analysis and this is noticed at receipt, then the sample control specialist logs in the sample for all analyses requested on the COC form, sets the status for the analyses on the affected sample to “cancelled”, and writes “insufficient sample for analysis” in the container comments field. The Project Manager is also notified and will communicate the issue with the client.

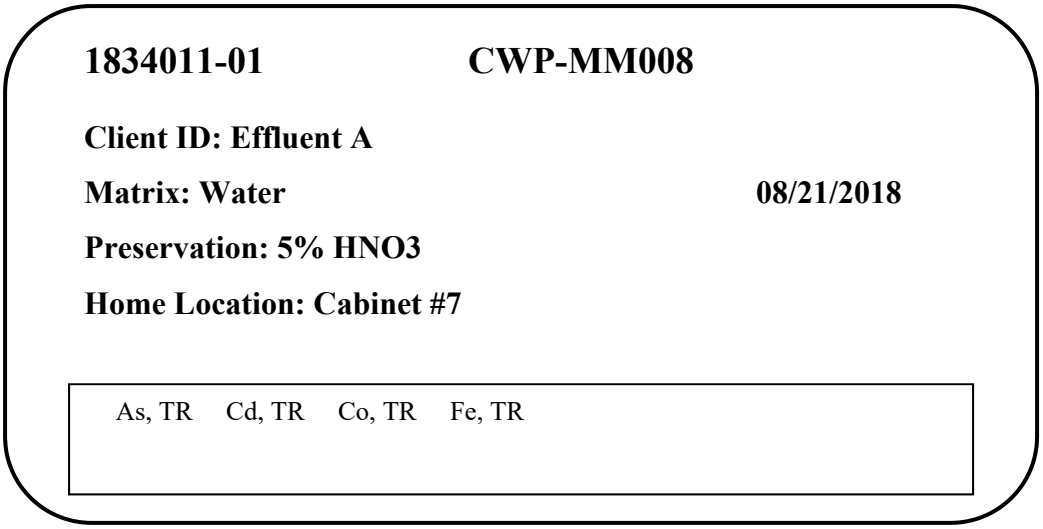
7.2.3 Sample Log In

When logging in samples, the sample control specialist notifies the PM if any parameters are requested by the client that are not in the LIMS Project. The Project Manager will confirm the contracted analyses and update either the LIMS Project or the original COC form prior to sample log-in.

All samples are given a unique sample identification number at the time of sample log in. This number consists of a work order number that is unique to each sample shipment received and a sample number for each sample within that particular shipment. Work order numbers consist of a 7-digit code (yyww###) where the first two numbers are associated with the year, the

next two are associated with the week of the year, and the final three are associated with the number of shipments received in that week (e.g., the eleventh sample shipment received in the 8th week of 2017 is given the work order number 1708011). The samples within a shipment are then each identified by sequential numbering. For example, if three samples were received in the 1708011 shipment they would be given the sample numbers 1708011-01, 1708011-02, and 1708011-03. LIMS automatically generates a unique sample number for each client sample and the client's sample name is recorded. The maximum number of samples per work order number is 99; therefore, if more than 99 samples are received in a sample delivery group, then the shipment must be broken up into more than one work order. The unique BAL work order and sample numbers are referenced during all laboratory preparations and analyses.

When a sample container is removed from the zip-type bag(s) in which it was sent, the container should be rinsed with clean DIW, if necessary (e.g. visibly dirty), and/or wiped with a clean paper towel. Containers are then placed in the clean hood or on the clean bench with a clean, new benchliner. Bottles are then labeled with the BAL sample number, BAL project number, client sample ID, matrix, date of sample receipt, receipt preservation, storage location, and list of analytes to be analyzed for. An example of one type of BAL sample label is shown here:



Vials and other small containers may not be large enough to fit the type of label shown above. The information included on labels used for vials placed in the cryo-freezer or mini labels for small vials may be truncated. At a minimum, all sample labels will include the samples unique LIMS ID and the client assigned sample ID. All the other information will be still be entered and stored in LIMS.
After all of the log-in information is recorded in the LIMS, the BAL Sample Receipt Log (refer to SOP BAL-2000) is generated from the LIMS and is signed and dated by the sample control specialist.

Custody of the original samples is tracked in the LIMS by updating the storage location of the samples every time that they are moved.

7.2.4 Sample Storage

All samples are stored in a secure area. A secure area is defined as an area within the premises of BAL with restricted access. To satisfy these custody provisions, the laboratory implements the following procedures:

- Access doors to the laboratory facility are kept locked
- Visitors must sign in and are escorted while in the laboratory
- Samples remain in the secure area until they are removed for sample preparation or analysis

After the samples are logged in, the sample control specialist stores them, according to their specific holding requirements, in either the refrigerator, freezer, or ambient sample storage area.

Samples requiring refrigeration are stored in a walk in fridge in the center of the BAL facility, or rarely a stand-alone fridge is used in certain instances. Samples requiring freezing are stored in a walk in freezer located within Prep Lab 2 or a stand-alone freezer located within Prep Lab 1. Samples requiring cryogenic freezing are placed in the cryogenic freezer located in Prep Lab 1. Samples not requiring refrigeration are stored on shelves in the sample storage cabinets, which helps to protect samples from UV radiation. All standards and other chemicals used at BAL are stored separately from samples.

After the samples are stored, all sample information is placed in a folder. This information includes the original COC form(s), a copy of the BAL Sample Receipt Log, the shipping way bill (or a copy of it), and any other documents included with the shipment. The folder is labeled with the work order number, BAL project number, received date, and due date. The folder is given to the Project Manager/Project Coordinator who then reviews the information, signs and dates the BAL Sample Receipt Log, updates the status in LIMS from “received” to “available” and files the folder in the “Active Customer” file located in the Project Manager’s office.

Samples for all projects are assigned a due date. Each analytical Group Leader is responsible for ensuring that all due dates and sample turn-around times are met. This is done by checking the LIMS to see what work orders are approaching the due dates and by checking the collection dates of samples with short holding times (less than 60 days). In addition, all Sample Processing Forms (SPF) have due dates recorded on them for each project.

The minimal duration of original sample storage at BAL is set at 60 days following the submittal of the final report, unless contractual requirements indicate a longer period of storage.

7.2.5 Sample Distribution and Tracking

The system for tracking samples through preparation and analysis consists of the LIMS, laboratory benchsheets, laboratory notebooks, instrument operation logbooks, instrument printouts (raw data), and final analytical reports.

7.2.5.1 Sample Batching - After samples are set to "available" by the project manager, the samples are then batched by the analytical Group Leaders or their designee. There may be instances where the samples are batched when they are at "received" status; when this happens, the project manager must be notified. Batches are sequentially numbered starting with the letter B, then the last two digits of the year, followed by a four digit sequential number (e.g. the 805th batch in 2018 is numbered B180805). Samples are assigned to each batch in the LIMS. Sample custody is tracked electronically in the LIMS.

Original samples are batched according to the method by which they are to be prepared and/or analyzed. At the time of batching, the SPF is printed from the LIMS. Before the samples are set to available, the project manager is responsible for adding any special QA requirements and/or pertinent information provided by the client concerning the sample preparation/analysis to the project comments in the LIMS so that it will automatically appear on any SPF including samples for that project. If the project comments field is not large enough, additional notes can be made in the project notes field, with instructions in the project comments field to "see project notes." Once batched, the status of the samples is automatically changed from "available" to "batched" in the LIMS.

7.2.5.2 Sample Preparation - The SPF is given to the chemist responsible for sample preparation. All sample preparation details must be documented on the SPF or sample preparation benchsheet. Copies of all preparation documentation, once complete, must accompany the SPF. In order to track both original samples and sample preparations, the chemist documents the removal of original samples from their primary storage location to the preparation location and back to their storage location in real time in the LIMS. For samples that aren't prepared in their original container, after the original samples are logged in as being returned to storage, the preparation technician changes the samples in the prep bench sheet to "extracts." These extracts can now be tracked separately from the original samples. The location of the extracts is entered as the preparation location. When sample preparation is finished, and the extracts are moved to the lab or other storage location, this information is entered into the LIMS bench sheet. From then on, every time the extracts are moved, up to and including disposal, this information is updated in real time.

During sample preparation, any comments on unusual observances or deviations from the analytical method or SOP must be documented. (Note: Senior management must approve any deviations from the analytical method or SOP prior to the preparation of the samples.) Following sample preparation, the prepared samples are stored in a secure laboratory area. Once prepared, the status of the samples is changed from "batched" to "prepared" and the date/time of prep and chemist's initials are updated in the LIMS benchsheet.

Note: If the sample has insufficient mass for sample preparation or is exhausted following preparation, then the technician should make a note on the

benchsheet and send an email to the Group Leader and the affected Project Manager. The PM then adds a note to the container comments field for all affected samples. Should the sample be inadvertently rebatched, this note will show up on any subsequent SPFs making it clear that the sample is already exhausted and cannot be reanalyzed. The Project manager should alert the other Group Leaders if any other analyses are affected and set the remaining analyses to "Cancelled".

7.2.5.3 Sample Analysis – When analyzing the samples, the chemist builds a sequence that contains the calibration and other sequence specific QC (ICVs, CCVs, CCBs) as well as all samples, including batch QC, from all batches analyzed as part of the sequence. Any comments on unusual observances or deviations from the analytical SOP must be documented and must be referenced on the SPF or the analytical benchsheet. The analytical benchsheet is also used to record any dilutions prepared at the instrument. As previously mentioned, movement of the batch throughout the lab is documented in the LIMS bench sheet.

7.2.5.4 Primary Data Review - The chemist uploads the data into the LIMS and then must review the data. Primary data review includes checking all LIMS entries in the prep bench sheet and the data upload for accuracy, determining whether criteria is met, and preparing analyst notes about the quality of the data. The chemist should also rebatch samples that need to be reprepared or reanalyzed and alert the Group Leader. The chemist then puts together the data package, which includes the analytical bench sheet, all SPFs, all sample preparation documentation, the data review checklist, and any chemist notes. Instrument raw data is saved electronically. Once the data has been primary reviewed, the status of the samples is changed from "prepared" to "reviewed – primary" in the LIMS and the SPF is signed and dated. Refer to SOP BAL-1501 for a complete discussion of primary data review.

7.2.5.5 Final Review - A member of the QA Group reviews the final data. After the final review, either the QA Manager or his designee (QA Associate, QA Assistant) must sign and date the SPF and include comments on any unusual observations and/or deviations from the analytical method or SOP. The batch status in LIMS is updated from "reviewed – primary" to "reviewed – final." If any samples require rebatching, this is performed by a member of the QA Group prior to changing the status of the samples in the analyzed batch to "reviewed – final." Refer to SOP BAL-1502 for a complete discussion of final data review. Refer to section 12 of this document for additional discussion of the data reduction, validation, storage, and reporting process.

7.2.5.6 Deviation Traceability - All documents are used to track any deviations from generally accepted handling of the samples. The main forms for tracking deviation are the SPF, preparation and analytical bench sheets, and analyst notes. These forms must contain any mention of unusual events or occurrences or deviations from SOPs and should list where this information can be found if

relevant. Examples of possible entries include, but are not limited to the following:

- Samples not cold when removed from refrigeration.
- Samples over distilled.
- Sample over diluted.
- Out-of-Control calibration curve.

Each person is responsible for filling-out the appropriate information for the task performed. The next responsible person will not accept the data and SPF unless the data package is complete for what has been performed so far. In this way all necessary information concerning samples and all sample handling steps can be traced and noted in the report to the customer.

7.2.5.7 Subcontracted Samples - Subcontracting samples is only done only with the prior consent of the client and the subcontractor laboratory must have an established and documented laboratory quality system that complies with all of the requirements of the original contract. For example, if a project has been contracted under DoD/DOE QSM requirements, then the subcontracted laboratory would have to meet the same requirements and also have DoD accreditation for all of the subcontracted analyses prior to analyzing any of the samples. A COC always accompanies samples that are transferred from BAL to a subcontracted laboratory. When transferring samples, the documentation includes collection date and time (if available from the field samplers), the BAL Lab ID #, the date of preparation (if extracts are transferred), the requested analyses, the signature of the person relinquishing the samples, and date samples relinquished by BAL. Any transfer of samples that have been logged in the BAL LIMS must be tracked under sample location.

7.2.6 Sample Disposal

7.2.6.1 Sample Preparations – Unless otherwise specified in the contract, sample preparations may be disposed of once the preparations have been analyzed and the data has been reviewed and reported to the client. Additionally, sample preparations may be disposed of if the holding time has been exceeded. The disposal of each batch must be documented in the LIMS.

7.2.6.2 Original Samples – It is BAL policy to maintain all samples (aqueous and solid) for no less than 60 days after reporting results unless previous arrangements have been made with the client. Samples that require longer storage should be separated from those that may be disposed after the two-month period. The location in the LIMS must be updated to indicate that the samples have been disposed.

7.2.6.2.1 Original aqueous sample extra volume for Se speciation may be disposed of after 60 days from receipt as the necessary aliquots have already been removed and stored.

7.2.6.3 Disposal Guidelines – The concentration of all elements of interest in each sample preparation and each original sample is calculated to determine the proper disposal method for each sample (Refer to BAL SOP BAL-2003 for disposal limits for specific elements). The method of disposal (routine verses high-level disposal) must be indicated on the appropriate form. Refer to SOP BAL-2003 for routine disposal guidelines and high-level metals waste transport and ultimate disposal.

7.2.6.4 Non-Routine Disposal - Samples that are designated by the client to be high level in an analyte not performed by BAL shall be considered hazardous and treated as hazardous waste upon release for disposal. In certain cases, BAL may contract with a client to analyze samples that are known to be hazardous beyond the scope of our analysis (such as samples containing a high level of organic contaminants or dioxins), these samples will be flagged as requiring special disposal (as per the VP of Operations) and disposed of through a licensed hazardous waste acceptance facility. BAL may also arrange with the client to return the remaining samples after analysis.

7.2.6.4.1 Low-level Radioactive Waste - All samples required to be disposed as low-level radioactive waste need to be in accordance with all local, state, and federal regulations regardless of the concentrations of other constituents.

7.2.6.5 Documentation – The LIMS is updated when necessary to both document disposal of samples (and sample preparations) and to initiate disposal or transfer of samples.

7.2.7 Catastrophic Failure of Storage Equipment

Refer to the Chemical Hygiene Plan in the event of a catastrophe (fire, loss of power, equipment failure) for the procedures to follow. If any samples are affected (go outside of holding temperature requirements or are otherwise damaged), it is the responsibility of the Project Manager to make sure that all affected samples are identified and documented in the associated Incident Report. The Project Manager is also responsible for informing the associated clients of all affected samples that have not yet been prepared. Clients must be notified as soon as it is practical to do so.

8.0 Analytical Procedures

8.1 Methods

All methods applied by Brooks Applied Labs are designed and approved to provide the most representative data available for any trace metal application. Only validated methods are applied to client samples unless research and method development is necessitated by the nature of the project. Validated methods are identified by either of the following criteria: the method was a promulgated or draft method issued by the EPA or other recognized regulatory agency, or the method of validation conforms to BAL SOP BAL-7000. BAL prides itself on application of cutting edge science to generate real world solutions by investigating new methodologies on a continual basis. BAL offers clients the ability to custom tailor digestion, extraction, and analytical techniques to fit their needs.

All methods generated by BAL which are promulgated into an official SOP must identify if the method does not conform to US EPA standards. All reports which are generated using such methods must clarify that the results are not generated using a US EPA approved method. A modification to a method performed is typically accomplished by stating "mod" after the method number.

All BAL methods, standard operating procedures, inventions, ideas, processes, improvements, designs and techniques included or referred to therein, must be considered and treated as Proprietary Information, protected by the Washington State Trade Secret Act, RCW 19.108 *et. seq,* and other laws. All Proprietary Information, written or implied, will not be distributed, copied, or altered in any fashion without prior written consent from BAL. All Proprietary Information (including originals, copies, summaries or other reproductions thereof) shall remain the property of BAL at all times and must be returned upon demand.

TABLE 8.1.1 – LIST OF ROUTINE METHODS AT BAL

Analyte	**Method Description**	**Reference**
Trace Metals	ICP-MS	EPA Methods 200.8 mod, 6020B mod, and 1638 mod
Mercury	CVAFS	EPA 1631E (Appendix for solids)
Methyl Mercury	CVAFS	EPA 1630 (mod for non-aqueous matrices)
As Speciation Analysis	HG-CT-AAS	EPA 1632A (mod for soils)
As Speciation Analysis	IC-ICP-MS	in-house
Se Speciation Analysis	IC-ICP-MS	in-house
Cr Speciation Analysis	IC-ICP-MS	in-house
Total Cyanide	IC/PAD	in-house

Refer to Appendix A for list of common abbreviations.

A complete list of BAL methods is found below.

TABLE 8.1.2 – LIST OF BAL STANDARD OPERATING PROCEDURES

SOP #	Title
BAL-0001	Prevention and Detection of Improper, Unethical, and Illegal Actions – Brooks Applied Labs' Data Integrity Plan
BAL-0020	Preventative Maintenance of Support Equipment
BAL-0021	Maintaining Instrument and Equipment Records and Logbooks
BAL-0102	Preventing Trace Metals Contamination of Samples
BAL-0103	Element for Analysts
BAL-0104	Documentation of Reagents and Standards
BAL-0105	Security of Laboratory and Samples
BAL-0106	BAL Training Program
BAL-0301	Metals "Free" Filtration
BAL-0302	Filtration for the Collection of Plankton from Water Samples
BAL-0303	Total Suspended Solids in Water
BAL-0304	Sample Homogenization
BAL-0308	Radiation Protection Program: Sample Handling, Storage, and Disposal under Brooks Applied Labs' Radioactive Materials License
BAL-0501	Dry Weight Determination
BAL-0600	Maintenance of Analytical Instrumentation
BAL-0700	Security of Electronic Data
BAL-0800	Purchase, Receipt, Lot Testing, Storage of Consumable Materials, and Vetting Service Providers
BAL-1000	Writing, Reviewing and Revising Standard Operating Procedures
BAL-1001	Document Control for Standard Operating Procedures (SOPs) and Comprehensive Quality Assurance Plan (CQAP)
BAL-1002	Records of Client Sample Results
BAL-1003	Records of QC Results
BAL-1004	Internal Laboratory Audits
BAL-1005	Evaluating Precision and Accuracy and Estimating Uncertainty in Results
BAL-1006	Identification and Generation of Method Variability
BAL-1007	Identifying Systematic Errors using Control Charts
BAL-1008	Corrective / Preventative Action and Resolution
BAL-1009	Method Detection Limit Studies

BAL-1010	Demonstration of Capability
BAL-1500	Data Flow and Handling
BAL-1501	Primary Data Review
BAL-1502	Final Data Review
BAL-1504	Acceptable Deviation from Documented Protocols
BAL-1700	Test Code Creation and Maintenance
BAL-2000	Receipt of Samples
BAL-2001	Sample Custody Maintenance and Tracking
BAL-2003	Sample Storage and Disposal
BAL-2300	Decontamination of Containers and Sample Preparation Equipment
BAL-2301	Decontamination of Silicon and Teflon® FEP Tubing and Filter Units for Sample Collection
BAL-2302	Metals Decontamination of Glassware
BAL-3000	Manual Integration of Chromatograms and Integrity of Electronic Data in GURU®
BAL-3100	Procedure for EPA Method 1631, Revision E: Mercury in Water by Oxidation, Purge and Trap, and Cold Vapor Atomic Fluorescence Spectrometry
BAL-3101	BAL Procedure for EPA Method 1631, Appendix to (1/01): Total Mercury in Tissue, Sludge, Sediment, and Soil by Acid Digestion and BrCl Oxidation by Cold Vapor Atomic Fluorescence Spectrometry (CVAFS)
BAL-3200	Determination of Methyl Mercury by Aqueous Phase Ethylation, Trap Pre-Collection, Isothermal GC Separation, and CVAFS Detection: BAL Procedures for EPA Method 1630 (Aqueous Samples) and EPA Method 1630, Modified (Solid Samples)
BAL-3300	BAL Procedure for the Analysis of Water, Sediment, and Tissue by EPA Method 1632, Revision A (1/01): Chemical Speciation of Arsenic in Water and Tissue by Hydride Generation Quartz Furnace Atomic Absorption Spectrometry
BAL-3900	Five-Step Selective Sequential Extraction (SSE) Procedure to Quantify Mercury Fractions in Sediments, Soils, and Other Solids
BAL-4000	Manual Integration of Chromatograms and Integrity of Electronic Data
BAL-4100	As Speciation for Waters and Tissues (Basic Anion Exchange) by Ion Chromatography – Inductively Coupled Plasma Mass Spectrometry (IC-ICP-MS)
BAL-4101	Arsenic Speciation Analysis of Aqueous Samples and Trifluoroacetic Acid Extracts of Tissue Samples Using Ion Chromatography – Inductively Coupled Plasma Mass Spectrometry IC-ICP-MS)
BAL-4115	Extraction of Tissue for Arsenic Species
BAL-4116	Organic Acid Extraction for As Speciation from Rice
BAL-4191	Cationic As Speciation for Tissues (Cation Exchange) by Ion Chromatography – Inductively Coupled Plasma Mass Spectrometry (IC-ICP-MS)

BAL-4200	Selenium Speciation by Ion Chromatography – Inductively Coupled Plasma Mass Spectrometry (IC-ICP-MS)
BAL-4201	Selenium Speciation by Ion Chromatography – Inductively Coupled Plasma Mass Spectrometry (IC-ICP-MS)
BAL-4300	Determination of Hexavalent Chromium by Ion Chromatography – Inductively Coupled Plasma Mass Spectrometry (IC-ICP-MS)
BAL-4310	Extraction of Hexavalent Chromium from Soils and Sediments
BAL-4500	Determination of Fe(II) and Fe(III) by Colorimetry
BAL-4701	Mercury Speciation in Waters by Ion Pair Chromatography – Inductively Coupled Plasma Mass Spectrometry (IPC-CV-ICP-MS)
BAL-4800	Determination of Magnesium and Chloride in Aqueous Samples using Ion Chromatography – Inductively Coupled Plasma Mass Spectrometry (IC-ICP-MS)
BAL-5000	Determination of Trace Elements by Inductively Coupled Plasma – Mass Spectrometry using an Agilent 8800/8900 QQQ
BAL-5001	Determination of Trace Elements by Inductively Coupled Plasma – Mass Spectrometry using a Perkin-Elmer Elan DRC II BAL Procedure for the Analysis of Samples following EPA Methods 1638 (Jan. 1996), 1640 (Jan. 1996), 200.8 (Rev 5.5), 6020A (Rev. 1), and AOAC 2015.01
BAL-5002	Determination of Hardness in Water by Calculation
BAL-5010	Aqueous Sample Digestion by Hotblock Heating of Total Recoverable Metals
BAL-5011	Aqueous Sample Digestion by In-Bottle Oven Heating for Total and Total Recoverable Metals
BAL-5012	Aqueous Sample Digestion by Oven Heating of Digestion Bombs for Total and Total Recoverable Metals
BAL-5020	Reductive Precipitation of Total Recoverable and Dissolved Metals from Brackish and Seawater Samples
BAL-5021	Determination of Trace Elements in Seawaters and Low Level Waters by Online Column Chelation Preconcentration – Inductively Coupled Plasma – Mass Spectrometry with Dynamic Reaction Cell Technology
BAL-5030	Acid Digestion for Environmentally Available Metals Digestion in Sediments and Biological Samples (EPA 3050B modified)
BAL-5040	Microwave Digestion for Total Recoverable Metals Digestion in Food Matrices (AOAC Method 2051.01 modified)
BAL-5913	Sequential Extraction of As in Soils and Sediment
BAL-6000	Reporting
BAL-6001	Review of Request for Work & Contracts
BAL-6002	Handling of Customer Complaints
BAL-7000	Method Development and Validation
BAL-8000	Procedure for Modified EPA Method 1669: Collection and Preservation of Samples for Trace Level Analysis
BAL-9001	Out of Specification (OOS) Investigations (GMP)
BAL-9002	Qualification of Legacy Equipment Used for the Generation of cGMP Data (GMP)

BAL-9003	Change Control Management (GMP)
BAL-9004	Document Control (GMP)
BAL-9005	Event and Deviation Management (GMP)
BAL-9006	Corrective and Preventative Actions (GMP)
BAL-9007	Quality Risk Management (GMP)
BAL-9008	Good Documentation Practices (GMP)
BAL-9050	Acid Digestion for Environmentally Available Metals in Chemically Defined Media Powders and Solutions (GMP)
BAL-9100	Trace Metal Analysis by Triple Quadrupole Inductively Coupled Plasma – Mass Spectrometry (ICP-MS) using Current Good Manufacturing Practices (CGMP)
BAL-9110	Maintenance for Agilent 8900 Inductively Coupled Plasma – Mass Spectrometry (ICP-MS) Instrumentation (GMP)
BAL-9201	Preventative Maintenance of Walk-In Cold Storage (GMP)
BAL-9202	Preventative Maintenance of Balances (GMP)
BAL-9203	Preventative Maintenance of Pipettes (GMP)
BAL-9240	Materials Management (GMP)
BAL-9250	Vendor Qualification (GMP)
BAL-9600	Reporting for CGMP
BAL-9700	Security of Electronic Data (GMP)
ASC-2001	Inorganic Arsenic Analysis of Melarsamine Dihydrochloride and Associated Final Drug Product by Solid Phase Extraction (SPE) and Ion Chromatography – Inductively Coupled Plasma Mass Spectrometry (IC-ICP-MS)

8.2 Method Modifications

Brooks Applied Labs is on the forefront of method development and improvement for trace metals and speciation. BAL has identified several modifications that improve the performance of standard EPA methods. All modifications are clearly stated in BAL standard operating procedures.

8.3 Laboratory Operations

8.3.1 Laboratory Containers Used for Preparation and Analysis

All containers for ultra-trace metals preparation and analysis must be rigorously cleaned and/or tested to minimize possible contamination. Refer to SOPs BAL-2300 and BAL-2301 for description of cleaning procedures for laboratory preparatory equipment.

8.3.2 Reagent Storage

All supplies (i.e., glassware, chemicals, and reagents) are of the highest necessary quality to ensure quality assurance and to avoid contamination. Reagents purchased from commercial vendors are assigned a unique LIMS identification number and are labeled with the date received, the date opened, and the expiration date. Reagents used for stock and working standards are prepared from analytical reagent grade chemicals or higher purity grades, unless such purity is not available. Reagent water is prepared by deionization of city water using reverse osmosis. Each prepared reagent is clearly labeled with the composition, concentration, date prepared, initials of preparer, expiration date, BAL lot # and special storage requirements, if any.

Reagent solutions are stored in appropriate glass or plastic containers under conditions designed to maintain their integrity (refrigerated, dark, etc.). Expiration date is listed on the label, and the reagent is removed from general lab use after it has expired. They may however still be used for research. Expired standards must be adequately segregated from working standards to prevent their accidental misuse. Reagent solutions are checked for contamination by testing reagent blanks. The expiration date of a standard or reagent may only be extended upon the approval of the Technical Director, Quality Manager, or VP of Operations. Such approval must be indicated in the notes section of the LIMS entry along with justification for extending the expiration date.

Refer to BAL's Chemical Inventory list stored on the BAL server for a list of all chemicals used at BAL. The Chemical Hygiene Plan describes where and how these chemicals are stored at BAL. Safety Data Sheets (SDS) are stored on the server as PDF files and should be consulted for detail concerning potential hazards associated with specific chemicals. SDSs are organized on the server by both chemical name and by CAS number. SOP BAL-0104 describes the documentation of standards and reagents in the LIMS and the testing of standards and reagents.

8.3.3 Waste Disposal

Handling, storage, and disposal of laboratory-related hazardous wastes are subject to the regulations contained in the Resource Conservation and Recovery Act (RCRA). BAL shall store, package, label, ship, and dispose of hazardous wastes in a manner which ensures compliance with all federal, state, and local laws. Potentially hazardous wastes include all standards, reagent solutions, process wastes, solvents, native samples, sample extracts, and digestates.

A waste is considered hazardous if:

1. The waste material is listed as hazardous in 40 CFR Part 261.30-261.33.
2. The material exhibits any of the characteristics of hazardous waste: ignitability, corrosiveness, reactivity, or extraction procedure toxicity.
3. The waste is listed in 1 or 2 above and is not excluded by any provisions under the Resource Conservation and Recovery Act.

A waste is considered an acute hazardous waste if it is identified in 40 CFR Part 261.31, 261.32, 261.33 (e) as an acute hazardous waste.

BAL is categorized as a Medium Quantity Generator. This category is defined as: A generator who generates 220 - 2200 kilograms of hazardous waste or < 1 kg of acute hazardous waste in a calendar month and stores all generated waste for no more than 180 days (40 CFR Part 261.5).

BAL shall ensure delivery of hazardous waste to a treatment, storage, or disposal facility, which is:

1. Permitted under 40 CFR Part 270
2. In the interim status under 40 CFR Parts 270 and 265
3. Authorized to manage hazardous waste by a state with a hazardous waste management program approved under Part 271; or
4. Permitted, licensed, or registered by a state to manage municipal or industrial solid waste (subject to local regulations).

Hazardous waste solvents as identified in the 40 CFR Part 261 may not be evaporated off in a fume hood. Solvents evaporated off during the extraction/testing process are exempt. Acidic and basic wastes may be neutralized and disposed of via the sanitary sewer if they are not hazardous due to the presence of other constituents (as subject to local regulations). Heavy metals may be precipitated from the liquid portion and disposed via the sanitary sewer (subject to local regulations).

Hazardous waste storage is limited to quantity and/or accumulation time and must comply with RCRA regulations as specified in the 40 CFR. These wastes should be packaged and separated according to the compatible groups (e.g. solvents, acids etc.).

Samples submitted to BAL for analysis are excluded from regulation as hazardous waste under 40 CFR Part 261.4(d) provided the samples are being transported to or from the laboratory, are being analyzed, are being held for analysis or are being maintained in custody for legal reasons. However, once the laboratory samples or extracts are removed from their original container and placed in a waste container, the exclusion provisions of 40 CFR Part 261.4(d) no longer apply. Samples that have been identified as hazardous may be either: 1) returned to the generator; or, 2) disposed of according to applicable RCRA regulations. Samples, which are determined to be non-hazardous, may be subject to local environmental regulations. A sample collector shipping sample to a laboratory and a laboratory returning samples to a sample collector must comply with U.S. Department of Transportation (DOT), U.S. Postal Service (USPS), or any other applicable shipping requirements.

8.3.4 Facility Description

BAL's research and analytical laboratory facility is located at 18804 North Creek Parkway, Suite 100, Bothell, Washington 98011. This is also the mailing address for the company, including all packages and samples. BAL's administrative offices are located nearby at 18912 North Creek Parkway, Suite 105, Bothell, Washington 98011, These locations are close to Sea-Tac Airport, numerous colleges and universities, and state regulatory offices. The entire laboratory section of the facility is identified as a clean area, which is continually monitored for contamination through routine testing of the DIW system, air, and preparatory blanks.

BAL has two designated sample preparation laboratories, a designated Inductively Coupled Plasma – Mass Spectrometry (ICP-MS) laboratory, and a Cold Vapor Atomic Fluorescence Spectrophotometry (CVAFS) laboratory which shares space with Hydride Generation Atomic Absorption (HGAAS) isolated from the rest of the facility to maximize cleanliness. The facility also includes office space, as well as areas for consumables storage, bottle washing, sample receipt, sample storage, and waste neutralization/disposal. Clean room sticky mats are located at the entrance to each lab to minimize tracking in particles.

The CVAFS laboratory, sample preparation laboratories, receiving laboratory, bottle washing, shipping and sample processing areas are monitored monthly for atmospheric mercury levels to ensure that levels are suitably low for ultra-trace level mercury analysis. A warning level has been established at 15 ng/m^3 with a shutdown control level at 25 ng/m^3. Reagent water is tested prior to beginning analysis by testing blanks.

The ICP-MS lab is where other metals analyses are conducted. DIW from all prep lab, sample receipt lab and ICP-MS lab sinks is tested by ICP-MS monthly.

All acid neutralized waste disposals are performed on a batch basis to eliminate the possibility of high level waste release. Disposal of all other toxic materials is carried out under contract with a licensed disposal company.

8.4 Exceptional Departures from Standard Operating Procedures (SOPs)

The Technical Director, QA Manager, VP of Operations, or President/CEO must approve of any departure from BAL standard operating procedures. This approval must be documented in some way (Note on SPF, email, etc.). Documentation must include the reason for the departure. Under no circumstances is the Chemist or Group Leader authorized to alter any standard procedure without managerial approval. Such approval and the actual departure from BAL procedure must be fully documented and conveyed to the affected client before issuing the final report. All quality control criteria must still be met for all reported data.

9.0 Calibration Procedures

9.1 Instrumentation

Refer to the Equipment List stored on the Brooks Applied Labs server for an up-to-date list of the equipment maintained at BAL.

9.2 Standard Receipt and Traceability

All stock standard solutions are received by the analytical laboratory and are documented in the LIMS. Information documented in the LIMS includes source, type of standard, date of receipt, lot number (if applicable), and expiration date. PDF copies of stock standard certificates are attached to the LIMS standard page for the standard.

All standard solutions are stored in a manner that is consistent with the manufacturers' recommendations.

Standards traceability is achieved by documenting all standard solution information in the LIMS. In addition to the previously mentioned documentation for stock standards, documentation for intermediate standard solutions must include: identification of primary (stock) standard used, the preparation date, method of preparation (specifically dilution information), the preparer's name, the concentration prepared, BAL lot #, and the expiration date. Documentation for working standards must include: identification of the stock and/or intermediate standards used, the preparation date, method of preparation (specifically dilution information), the preparer's name, the concentration prepared, BAL lot #, and the expiration date.

9.3 Standard Sources, Preparation, and Testing

All working standards are documented for traceability as discussed in section 9.2. All intermediate and working standards are made in accordance with the protocols of the specific procedure for which the standards shall be used. Refer to the analytical method SOP for the specific procedures followed for the preparation and holding time of any intermediate or working standard.

Any new standard must be tested prior to use. The acceptance criterion is that the average recovery of the new standard is within ± 5% of the average recovery of the previous standard. A minimum of three replicates of the old and the new standard must be analyzed for comparison. The RSD of the measurements of each standard may not exceed 5.0%. Only the Technical Director or QA Manager can authorize the use of any untested standard or standard that does not meet the testing criteria. Standards that are made daily are not tested against the old standard prior to use. Instead, they are verified against the second source standard as per method specific criteria.

Quality control reference materials are typically acquired from the National Resource Council of Canada (NRCC), the National Institute of Standards and Technology (NIST), the International Atomic Energy Agency (IAEA), RT Corp (RTC), or Community Bureau of Reference (BCR).

9.4 Analytical Instrument Calibration

The analytical methods or the SOP for the specific method specifies all calibration protocols, frequency and acceptance criteria. Full documentation for calibration is included with the sample data package. Each instrument used to analyze samples must pass the calibration criteria established in the appropriate method or operating procedure.

All standards used to prepare the calibration standard solution are obtained from accredited chemical suppliers and are tested for purity prior to use. Working standards are made from the stock standards at appropriate concentrations to cover the linear range of the calibration curve as outlined in the individual procedures. All laboratory analysis is documented by the analyst on the analytical bench sheets. All information concerning the calibration must be stored such that the calibration can be recreated if need be.

9.4.1 Instrument Calibration for CVAFS and HGAAS Methods

The instrument calibration consists of analyzing a minimum of three initial calibration blanks (used to correct all results, including those for the calibration standards, at the instrument) and a minimum of five standards. These standards should span the linear range of the instrument. Calibration coefficients are calculated for each concentration of the calibration and the average calibration coefficient is used to calculate results. For CVAFS the RSD of the calibration coefficients must be ≤ 15% and for HGAAS the RSD of the calibration coefficients must be ≤ 20%. Only calibration standards that meet recovery criteria as stated in the analytical methods are used in the calibration. Allowances for dropping calibration points are discussed in section 9.4.5.

9.4.2 Instrument Calibration for ICP-MS Methods

The instrument calibration consists of analyzing a minimum of one initial calibration blank (used to blank offset the calibration) and a minimum of five standards (8 to 9 standards are routinely analyzed for ICP-MS analyses). These standards should span the linear range of the instrument. The calibration routinely used for ICP-MS analysis is linear, not forced through zero. The correlation coefficient (r) of the initial calibration for ICP-MS must be ≥ 0.995, but in general should be ≥ 0.998. If a weighted linear calibration forced through zero is used for ICP-MS analyses, then the r value must still be ≥ 0.995. All calibration standards > the MRL must recover between 75 – 125%. All reportable calibration points ≤ the MRL must recover between 70 – 130%. Since unweighted linear calibrations are heavily influenced by the standards at the high end of the calibration, calibration points near the bottom of the calibration will not significantly affect the linear regression and need not be removed even when failing to meet recovery criteria. However, the lowest calibration standard used to set the quantitation level of the analysis must recover within 70 – 130% and all subsequent calibration standards must recover within the 75 – 125% acceptance criteria.

More than one calibration may be run during a single sequence. This is done for large analyte lists due to potential internal standard failures and instrument drift. In such cases, the best calibration may be used on an analyte-specific basis.

9.4.3 Instrument Calibration for Ion Chromatography – Inductively Coupled Plasma – Mass Spectrometry (IC-ICP-MS) and Ultraviolet Visible Spectroscopy (UV/Vis)

The instrument calibration consists of analyzing a minimum of one initial calibration blank (used to blank offset the calibration) and a minimum of five standards. These standards should span the linear range of the instrument. Additionally, four replicates of the low calibration standard are typically analyzed. The average response is used for the low calibration standard. Additionally, the standard deviation of the low level standards may be used to estimate the batch specific MDL for procedures where the method blank results at the instrument are typically 0 µg/L. The calibration routinely used for metals speciation analysis is linear, forced through zero. The correlation coefficient (r) of the initial calibration for these methods must be ≥ 0.995. Additionally, if multiple species are being calibrated in an analysis, the RSD of the various slopes should be ≤ 10. All calibration standards > the MRL must recover between 75 – 125%. All reportable calibration points ≤ the MRL must recover between 70 – 130%. Since unweighted linear calibrations are heavily influenced by the standards at the high end of the calibration, calibration points near the bottom of the calibration will not significantly affect the linear regression and need not be removed even when failing to meet recovery criteria. However, the lowest calibration standard used to set the quantitation level of the analysis must recover within 70 -130% and all subsequent calibration standards must recover within the 75 – 125% acceptance criteria.

9.4.4 Instrument Calibration for Ion Chromatography – Pulsed Amperometric Detection (IC-PAD) Methods

The instrument calibration consists of analyzing a minimum of one initial calibration blanks (used to blank offset the calibration) and a minimum of five standards. These standards should span the linear range of the instrument. Additionally, four replicates of the low calibration standard are typically analyzed. The average response is used for the low calibration standard. Additionally, the standard deviation of the low level standards may be used to estimate the batch specific MDL for procedures where the method blank results at the instrument are typically 0 µg/L. The calibration routinely used for cyanide analysis is a polynomial curve to the second order, forced through zero. The correlation coefficient (r) of the initial calibration for these methods must be ≥ 0.999. All calibration standards > the MRL must recover between 75 – 125%. All reportable calibration points ≤ the MRL must recover between 70 – 130%.

9.4.5 Allowances for Dropping Standards from the Calibration

When calibrating the instrument, the low calibration standard must be equal to or less than the method reporting limit (MRL). Standards may be removed from the bottom end or top end of the calibration when they do not meet acceptance criteria as long as at least three consecutive standards remain, however, this will result in a reduced range of quantitation. It is not permissible to drop a mid-level point from the calibration without also dropping either all of the points below or all of the points above it as well. The only exceptions for dropping a mid-point standard from a calibration is for a known misinjection or obvious spiking error. Even then, it is not allowable to drop two consecutive mid-point standards without the documented approval of the Technical Director or QA Manager.

9.4.6 Results Outside of the Calibration

Results should not be reported if the instrument result for the sample is above the result for the high calibration standard. It is standard practice to dilute the high-level sample and reanalyze it such that the result at the instrument falls within the calibration. A result that is outside of the calibration range would not be reported without appropriate qualification or narration. An additional linearity check standard (high calibration check or HCV) that is above the calibration may be analyzed later in the run. The recovery of the HCV must be 90 – 110% for it to be used. If the HCV is acceptable, then a result that was above the calibration, but below the level of the HCV may be reported without qualification or narration. If this is done, results must be carefully evaluated to determine if there is any risk for potential carry over from the high sample or the HCV. Samples analyzed at dilutions beyond the default dilution for the method yielding results below the MRL should be reanalyzed at less dilution to be within the calibration curve.

9.4.7 Reverification of a calibration

If an instrument stops for any unplanned reason or for more than 4 hours, the calibration must be reverified by analyzing passing ICV, CCV, and CCB prior to analyzing any client samples.

9.4.8 Reanalysis of Samples and Calibration Procedures for All Automated Methods

When sample reanalysis is necessary, it must be performed on the most recent instrument calibration. If more than 48 hours have passed since the original calibration was last verified or if the instrument has been recalibrated since original sample analysis, then the original calibration must not be used and a new calibration must be performed. The new calibration must include all analytes of interest, as well as the instrument modes used in the original calibration (e.g., HEHe, H_2, O_2, etc.).

If a new calibration is used, the samples would be analyzed in a new sequence but would remain in the same batch. If the original calibration is to be re-used within 48 hours, the calibration must be re-verified by the analysis of instrument blanks and an ICV before sample reanalysis, and the samples must be bracketed with the appropriate number of CCV/CCB sets. In this case, the samples remain in the same batch and sequence.

It is not necessary to re-analyze the entire batch or even all of the associated batch QC with the samples. When samples are reanalyzed, a 10% frequency of the associated MS or MS/MSD sets (including native samples) must be reanalyzed as well to verify that elements have not precipitated out of the digestate and to verify that the analytical precision between the initial analysis and the reanalysis meets criteria for all samples that have a valid initial result. If no matrix spikes are prepared with the samples, then a BS or SRM may be analyzed in place of the matrix spikes. The acceptance criteria for analytical precision is equivalent to the analytical duplicate precision criteria listed in each method. If the reanalysis of the samples with valid initial results (including QC samples) do not meet precision criteria, the QA Manager and/or the Technical Director must be consulted to prescribe what corrective action is appropriate. Results from the reanalysis of the QC (that meet precision criteria) are not required to be reported to the client, but should be kept as validation data for the reanalyzed samples.

Other batch QC samples (DUPs, Blanks, SRMs, Blank Spikes, etc.) are not required to be reanalyzed unless there were specific issues with the initial analysis of these samples. If samples that do not have pre-prepared QC samples are reanalyzed (such as waters prepared with the closed-vessel original-container oven digestion), then the MS/MSD sets must be performed on the reanalyzed samples at a 10% frequency (not necessarily the samples that were initially spiked).

9.5 Periodic Calibration Procedures for Other Laboratory Equipment

Periodic calibration checks are performed for associated equipment such as balances, thermometers, pipettes, ovens, and refrigerators that are required in support of the preparation and analytical methods, but that are not routinely calibrated as part of the analytical procedure. All the calibration measurements are recorded in a laboratory log book as outlined in SOP BAL-0021.

BALANCES

Balances are calibrated annually by a contracted, certified professional. Balances are also checked with Class ASTM1 weights on a daily, as-used basis. At the beginning of each day that the balance is used, the analyst is required to perform at least one calibration check in the range of the material to be weighed. All calibration checks are documented in a laboratory log book. All weights used to calibrate balances are themselves calibrated at a minimum every 5 years against NIST traceable weights.

TABLE 9.5 - CRITERIA FOR BALANCE CALIBRATION CHECKS

Balance Type	**Criteria**
Top-loading (2 and 3 point) balances	± 2% or ± 0.02g, whichever is greater
Analytical (4 point) balances	± 0.1% or ± 0.5mg, whichever is greater

PIPETTES

All pipettes are calibrated weekly and are checked the day of use if a DOD/DOE sample is being processed. Pipette performance is monitored by gravimetrically measuring the volume of DIW dispensed by each pipette over the range of its use with the assumption that the density of the water in the laboratory is 1.000 g/mL (± 0.003 g/mL). If the pipette does not meet criteria, it must be adjusted or removed from service. After adjustment, the pipette check procedure must be re-performed. Both the pre-adjustment and post-adjustment measurements are maintained in a laboratory log book. Bias and precision are measured for all new pipettes prior to being put into service and then quarterly thereafter. Bias and precision are based on 10 measurements. The criteria are that the average measurement must be within ± 2% of the measured volume and the RSD of the measurements must be ≤ 1%.

OVENS, HOTPLATES, REFRIGERATORS, AND FREEZERS

Temperatures are checked with calibrated thermometers and necessary adjustments to the temperature settings are made as required. Refrigerators and freezers are checked on a daily basis and all ovens, hotplates, are checked at least once during each use. Refrigerator and freezer temperatures are recorded on temperature chart recorders or in laboratory logs that are maintained by the Sample Control Group Lead. Oven and hotplate temperatures are recorded in the sample preparation logs.

THERMOMETERS

The performance of each thermometer is checked prior to being put into service and then annually by comparison to a certified NIST-grade thermometer and correction factors and the calibration due date are posted on each thermometer.

10.0 Preventative Maintenance

10.1 Routine Maintenance Measures

Refer to SOPs BAL-0020 and BAL-0600 (Preventative Maintenance of Support Equipment and Analytical Instruments) for greater detail of all required preventative maintenance.

10.1.1 Air Testing

Volatile mercury air measurements are taken monthly as described in section 8.3.4 of this procedure. Air from each lab is pumped through a soda lime pre-trap and onto gold-coated substrate trap at a flow rate of 1 L/min until at least 20 L of air have been collected per trap. Results from the monthly air tests are saved electronically on the server.

10.1.2 Water Testing

Reagent water is monitored for Hg each day the instrument is run when calibration blanks are analyzed. A minimum of four 25 mL aliquot of fresh reagent water, each with 0.2 mL $NH_2OH{\cdot}HCl$ and 0.2 mL of $SnCl_2$, are analyzed at the beginning of the run sequence. The results must be < 10 pg Hg. A high level of mercury detected in the reagent water analysis may also be attributed to the MERX system itself, the reagents, or the soda lime pre-traps. Regardless of the source, all analysis is stopped until the source of contamination is determined and the problem is corrected. The results are stored with each batch.

Reagent water is tested for trace metals by ICP-MS at a minimum of once per month when instrument water blanks collected from every sink used to clean equipment, prepare reagents/samples, or analyze samples are analyzed. Specific elements are tested for with each batch. Currently, water blanks must be less than the element specific MRL or client specific requirements. Results for water testing are stored on the server in Excel© spreadsheets.

10.1.3 Equipment and Reagent Testing

All reagents (acids, standards, etc.) and equipment (bottles, vials, etc.) are tested prior to use for THg and Trace Metals. The acceptance criteria for specific reagents and equipment are specified in the individual SOPs describing the use of the reagents or the decontamination of equipment. In all cases, contract specified requirements take precedence over BAL acceptance criteria.

10.2 Documentation

Instrument logbooks are maintained for all equipment. These logbooks contain a complete history of past performance and maintenance. Analytical instrument logbooks document instrument usage, routine maintenance, and non-routine repairs.

10.3 Contingency Plans

10.3.1 Major Equipment Failure

For major equipment failure of CVAFS instruments, the laboratory has backup instrumentation. BAL has close ties to Brooks Rand Instruments, an instrument manufacturer

specializing in ultra-trace level mercury analyzers in Seattle; therefore, a stock of replacement parts and complete analyzers exist and expert service personnel are readily available.

For flame AA's, rental equipment is locally available in the case of a major equipment failure while instrumentation is being repaired.

For ICP-MS, BAL has ten instruments that can be used for total metals and speciation analysis giving the lab plenty of redundancy.

BAL currently has an excess of balances and refrigerators/freezers. If any of this equipment fails backup equipment is immediately available. Other equipment such as the conductivity meter and the pH meter are relatively inexpensive and will be purchased immediately if major equipment failure is determined.

10.3.2 Loss of Power

BAL has a backup generator to reduce the risk of loss of power to critical instrumentation and key support equipment. However, if there is a total or partial loss of power to the lab, then the procedures outlined in Appendix D, Section 6.0 of the Chemical Hygiene Plan must be followed.

10.3.3 Invalidation of work

Results for all sample analyses affected by equipment failure may be ruled invalid depending upon the circumstances. When QC criteria are not met during analysis, all instrumentation is thoroughly checked and appropriate maintenance action is taken. Subsequent reanalysis of the affected samples is then initiated after the instrumentation is proven to be functioning properly. The Technical Director, BAL President/CEO, QA Manager, and the VP of Operations have the authority to stop work whenever there is evidence of non-conforming work. Once work is stopped, corrective action must take place and be documented. Permission to restart work must be granted by the Technical Director, BAL President/CEO, QA Manager, or the VP of Operations.

11.0 Quality Control Checks and Routines to Assess Precision and Accuracy and the Calculation of Method Detection Limits

The laboratory uses quality control samples to assist in assessing the validity of the analytical results of field samples. The use of quality control samples helps to assess analytical accuracy and precision in the laboratory. Quality control samples are analyzed in the same manner as field samples at a frequency described either in the individual procedures or in the contract with the client. If the quality control sample results fall within acceptable criteria (also detailed in the method), then the field sample data are considered to be valid or acceptable as is. However, it is important to keep in mind that errors made during sample collection can seriously affect the analytical results of field samples. In other words, the quality or validity of the field sample data is only partially supported by the laboratory quality control sample results. Field quality control samples are the other necessary component for the validity of field sample results.

Laboratory quality control (QC) samples include method blanks, calibration checks, replicates, spiked samples, and standard or certified reference materials (SRM/CRM). The specific frequency and type of QC samples analyzed are described in the individual analytical method, SOP, or client-specific Statement of Work (SOW). In some cases, contracts may specify additional or more stringent QC requirements beyond what the method requires. In these cases, the contract specific QC requirements are followed. In addition to these routine QC samples, performance evaluation samples required for certification are analyzed semi-annually.

11.1 Quality Control Checks

11.1.1 Field QC Checks

Brooks Applied Labs is rarely involved in field sampling. The client is responsible for field sampling activities and therefore mandates the requirements for field QC checks. However, BAL suggests that the following field QC be collected.

11.1.1.1 Trip Blanks

Trip blanks are used to demonstrate that sampling equipment and collected samples have not been contaminated during transit. Trip blanks consist of laboratory reagent water collected into a sampling container at the laboratory. The trip blank is then double bagged (as per sampling containers for use in the field) and affixed with a custody seal to indicate if it has been tampered with. The trip blank is then shipped with the sampling kit to and from the field. The trip blank must not be opened again until it has returned to the laboratory.

When collected and analyzed, the concentration of the analyte of interest in the trip blank should be less than the reporting limits or less than 10% of any affected sample results. If criteria are not met, then the client must be notified and every effort should be made to determine the source of the contamination and to eliminate it if possible.

11.1.1.2 Field Blanks

Field blanks are used to demonstrate that the samples were not contaminated during the collection procedure or while in transit (Note: The analysis of trip blanks in conjunction with field blanks can better pinpoint the source of contamination). Field blanks are collected in the field, typically using lab-supplied reagent water, and simulating the collection of actual samples as well as can be done. Once collected, the field blank is treated in every way as an actual sample.

When collected and analyzed, the concentration of the analyte of interest in the field blank should be less than the reporting limits or less than 10% of any affected sample results. If criteria are not met, then the source of the contamination should be determined and eliminated if possible.

Many methods require that field blanks be collected and analyzed if results are to be reported for regulatory purposes. While BAL does not require that clients provide field blanks for analysis, BAL does inform clients of this regulatory requirement in the quote signed by the client prior to any work performed as well as in any case narrative that includes relevant results.

11.1.1.3 Field Duplicates

Field duplicates are used to assess precision in the collection procedures. When collected, the field duplicate relative percent difference (RPD) should be no greater than that allowed for method duplicates by the specific analytical method, the SOP, or the SOW. If the RPD is greater than the acceptance criterion, then the sampling team should be notified. When analyzed in conjunction with method duplicates (Section 11.1.2.7), field duplicates will aid in determining the source of any imprecision.

11.1.2 Lab QC Checks

Laboratory QC samples are analyzed with every batch and sequence to validate method performance. The number of QC checks analyzed will depend on batch size and the number of analyses associated with the sequence. Batches at BAL may contain more than 20 client samples as long as QC frequency requirements are met. For instance, most EPA methods only require one method blank per batch of 20 samples. BAL routinely prepares four method blanks; therefore, a batch could contain up to 80 samples as long as SRM, BS, DUP, and MS/MSD frequency requirements were also met.

11.1.2.1 Method Blanks (BLK)

A method blank is a sample of reagents or reagent water treated as a sample such that it is prepared in conjunction with and undergoes the same analytical processes (i.e. same reagents added at time of sample preparation, digested in the same type container, if available, at the same temperatures/times, etc.) as the corresponding field samples. Method blanks are used to monitor laboratory performance and contamination introduced during sample preparation and analysis. The method blank acceptance criteria are method specific (Refer to the specific analytical method, the SOP, or the contractual requirements).

In cases where a sufficient number of method blanks (minimum of four) have been prepared and analyzed with the batch to characterize the nature of the blanks and the potential for any reagent or spot contamination, one blank may be rejected as a Grubb's Outlier if it meets the criteria for doing so at the 5% or less risk of false rejection level (refer to Section 11.2.3 for further discussion on how the Grubb's Test for Outliers is applied to data). If a method blank is rejected as a Grubb's Outlier, then its value is not used to calculate the mean or the standard deviation of the method blanks used to blank-correct the batch data or calculate any batch specific reporting limits. However, the data should still be evaluated against any Grubb's Outlier that doesn't meet method blank acceptance criteria.

Typically if a method blank is determined to be a Grubb's Outlier, it is uploaded into LIMS and left as reportable with narration. There are some circumstances in which a method blank may not be uploaded such as when it can be shown that the removed method blank does not impact the data quality. Examples include a misinjected method blank or a method blank that is elevated due to carry over from a previously analyzed high-level sample. For blank corrected data, the Grubb's Outlier method blank must be removed from the LIMS upload in order for the sample results to be appropriately corrected.

The discarding of any data point as a Grubb's Outlier and the potential effect on overall data quality must be narrated to the client. Current LIMS limitations do not allow method blanks rejected as Grubb's Outliers to appear on the "Method Blanks & Reporting Limits" page of the report. Therefore, the value of any rejected method blank must be reported in the case narrative section of the data report. Grubb's Outliers may never be discarded for non-method blank corrected data except for reasons noted above. Refer to Section 12.6 (Data Reporting) for specific instructions on how method blanks are evaluated and reported for uncorrected results.

11.1.2.2 Matrix Spikes (MS)

Matrix spikes are routinely included in the analytical batch as they are required for most methods utilized at BAL. Method-specific or client-specific frequency and recovery requirements are variable and available in the method, the SOP, or the SOW, whichever is applicable. Matrix spikes are typically analyzed at a frequency of one per every ten client samples. Although not a requirement, if a batch contains samples of different submatrices, matrix spikes should be prepared and analyzed for each submatrix type to ensure that there is no matrix-specific interference. It is up to the client to request additional matrix spikes on their samples if they suspect matrix issues. Unless specified by the client, matrix spikes should not be performed on any type of blank sample.

The native sample should always be run at the same dilution as the spikes to check for possible signal suppression or enhancement but may be reported from a different dilution if necessary to obtain results that are within the calibration. At any time, a post-spike sample may be prepared and analyzed at the instrument. The recovery criteria for post-spikes is typically equivalent to the recovery criteria for the associated CCVs.

11.1.2.3 Blank Spikes (BS)

If reference materials are unavailable, it is BAL policy to prepare a Blank Spike (BS) at a frequency of at least 5% per batch of samples. The BS is typically spiked at approximately 10 – 20 times the MRL. Acceptance criteria is defined in the method or the client contract.

11.1.2.4 Performance Evaluation (PE) Samples

Performance Evaluation samples are analyzed as blind samples and are analyzed at a minimum of semi-annually. BAL purchases PE samples from Environmental Resource Associates (ERA). All PE studies utilize samples that are blind not only to the analyst but also the entire laboratory staff until after the results have been submitted to the appropriate agency and the final report for the study is issued. ERA PE results are forwarded directly to BAL and all appropriate accrediting bodies.

Additionally, BAL routinely participates in optional laboratory intercomparison studies offered by such institutes as MAPEP (U.S. Department of Energy's Mixed Analyte Performance Evaluation Program), the International Atomic Energy Agency (IAEA), the United States Geological Survey (USGS), etc. Laboratory intercomparison studies such as these allow BAL the opportunity to evaluate our performance on more non-traditional matrices not typically available from PE providers.

11.1.2.5 Calibration Verification

Independent Calibration Verification (ICV) standards are standards that are from a different source than the working standards. The ICV is analyzed once immediately following the calibration. Verification standards made directly from the working standards are also used throughout the analysis to check the continuing accuracy of the calibration. They are often referred to as Continuing Calibration Verification (CCV) standards. For most methods utilized at BAL, the CCV samples must be analyzed at the beginning and the end of an analytical batch and after every 10 injections throughout the analysis. For most analyses, Continuing Calibration Blanks (CCB) are analyzed after each CCV sample to ensure that there is no carry-over of analyte to the field sample analysis. Additional requirements may be specified in the specific analytical method, SOP, or contractual requirements.

11.1.2.6 Quality Control Samples

Quality control (QC) samples are additional QC checks for evaluating the accuracy of the analysis. These samples may be prepared by BAL (as with Blank Spikes) or purchased from an outside source (as with SRMs) depending upon their availability. Frequency and recovery criteria for QC samples are method specific. Refer to the specific analytical method, the SOP, or the SOW for specific frequency and recovery requirements.

11.1.2.7 Duplicates (Method Duplicates (DUP) or Matrix Spike Duplicates (MSD))

Duplicate samples and/or matrix spike duplicates are typically analyzed at a minimum frequency of 10% per analytical batch and should be performed on each matrix in the batch for all analytical methods employed at BAL. Refer to the specific analytical method, the SOP, or the SOW for specific frequency and precision requirements.

11.1.2.8 Reagents and Standards Purity Checks

All reagents used in the preservation, preparation or analysis of samples by accredited methods must be checked for the appropriate parameters prior to use.

Likewise, all standards (except those made daily or weekly) are tested against previously tested, non-expired standards prior to use to ensure that they are acceptable for use as calibration, calibration verification, or spiking standards.

11.2 Routine Methods Used to Assess Precision and Accuracy

11.2.1 Accuracy and Precision

11.2.1.1 Precision

Precision from two replicates is expressed as Relative Percent Difference (RPD). Precision from more than two replicates is expressed as % Relative Standard Deviation (% RSD) and shall be calculated from the following formulae:

$$RPD = \left(\frac{|a - b|}{\bar{x}} \right) \times 100$$

Where: a = result a from native sample, or for matrix spike samples, result from the matrix spike (native + spike concentration) sample
b = result b from native sample duplicate, or for matrix spike samples, result from the matrix spike duplicate (native + spike duplicate concentration) sample
$\bar{x}$ = Mean (average) of the two results

$$\%RSD = \left(\frac{s}{\bar{x}} \right) \times 100$$

Where: $\bar{x}$ = Mean (average) of the data points
s = Standard deviation calculated as:

$$s = \sqrt{\frac{\sum_{i=1}^{n} \left(x_i - \bar{x} \right)^2}{n - 1}}$$

Where: x_i = the individual data point for each n
n = the total number of data points

11.2.1.2 Accuracy from Spiked Samples

The accuracy of a measurement shall be determined by the recovery of a known amount of analyte in a real sample as:

$$\%\,R = \left(\frac{Cs - Cu}{S} \right) \times 100$$

Where: Cs = concentration of spiked sample
Cu = concentration in non-spiked sample (can be 0 for results < MDL)
S = expected concentration (spiking level)
%R = percent recovery

11.2.1.3 Accuracy from Known Concentrations
The accuracy of a measurement based on known concentrations shall be calculated as:

$$\%\,R = \left(\frac{\text{Sample concentration}}{\text{Reported True Value}} \right) \times 100$$

11.2.1.4 Upper and Lower Warning and Control Limits for Acceptance Criteria
When not method based, Upper and Lower Warning Limits (WL) and Control Limits (CL) for determining acceptance criteria shall be calculated as follows:

$$CL = P_{av} \pm 3s$$

where: CL = Control Limit (upper and/or lower)
P_{av} = Mean of P (percent recovery or RPD)
s = standard deviation of the mean of P

and

$$WL = P_{av} \pm 2s$$

where: WL = Warning Limit (upper and/or lower)

11.2.2 Quality Control Charts

Quality Control charts are used to determine acceptance criteria for in-house developed methods and review the relevance of QA criteria parameters used in each analytical method. Separate quality control charts should be established for each analytical method, for each parameter or analyte, and for each matrix type, both for precision and for accuracy. Control charts are automatically updated for all test codes in the LIMS as data is uploaded.

Control charts for SRMs or blank spikes are reviewed quarterly. However, control charts can be constructed and used to monitor laboratory and method performance for other several other parameters such as spike recoveries, duplicate analyses, calibration verification standard recoveries, and blank analyses. Control charts use both the mean and standard deviation in order to identify out-of-control events as per Standard Methods, 21st Edition, Section 1020 B.

The LIMS automatically generates control charts, where the mean and the standard deviation, warning limits, and control limits are automatically calculated and updated. Control charts can be determined by method, who prepared the samples, who analyzed the samples, which instrument was used to analyze the samples, and over what dates the samples were analyzed. The individual data points are plotted against the mean and the ± 2 (warning limit), ± 3 (control limit), and ± 4 standard deviations.

Refer to SOP BAL-1007 for further discussion of the use of control charts for identifying systematic errors.

11.2.3 Grubb's Outlier

An outlier is an extreme value, high or low, that has questionable validity as a member of the measurement set with which it is associated. Outliers are **not** used in assembling the quality control charts for purposes of setting acceptance limits or determining reporting limits. Outliers may be rejected from the data set for the following reasons:

- A known experimental aberration occurred, such as instrument failure or inconsistency in the procedure or technique

- The T value for the data is larger than the tabulated values using the Grubb's test for outliers (Table 11.2). Outliers at BAL are determined with a 95% confidence level (or 5% risk of false rejection). The T value is calculated using the following equation:

$$T = \frac{\left|X_0 - \overline{X}\right|}{SD}$$

where: X_0 is the extreme value being measured
$\overline{X}$ is the mean of the measurement set for *n* observations including X_0
SD is the standard deviation associated with X including X_0

If a value is rejected, the mean and standard deviation are recalculated using the remaining data.

TABLE 11.2 - GRUBB'S TEST FOR OUTLIERS

Number of	Risk of False Rejection				
Data Points	0.1%	0.5%	1%	**5%**	10%
3	1.155	1.155	1.155	**1.153**	1.148
4	1.496	1.496	1.492	**1.463**	1.425
5	1.780	1.764	1.749	**1.672**	1.602
6	2.011	1.973	1.944	**1.822**	1.729
7	2.201	2.139	2.097	**1.938**	1.828
8	2.358	2.274	2.221	**2.032**	1.909
9	2.492	2.387	2.323	**2.110**	1.977
10	2.606	2.482	2.410	**2.176**	2.036
15	2.997	2.806	2.705	**2.409**	2.247
20	3.230	3.001	2.884	**2.557**	2.385

25	3.389	3.135	3.009	**2.663**	2.486
50	3.789	3.483	3.336	**2.956**	2.768
100	4.084	3.754	3.600	**3.207**	3.017

Tabulated values obtained from Quality Assurance of Chemical Measurements by John Keenan Taylor, 1987.

11.3 Method Detection Limits and Reporting Limits

11.3.1 Method Detection Limits for Routine Environmental Analyses

The MDL is the minimum concentration of an analyte of interest that can be measured and reported with 95 percent confidence that the value is above zero. MDLs for accredited methods are determined following BAL-1009.

11.3.2 MDLs and MRLs for Non-Routine Environmental Analyses, Food Testing, and R&D

For non-routine environmental analyses (anything that does not fall under TNI or DoD/DOE scopes of accreditation) that are not being used for regulatory compliance purposes, there is more flexibility in how the MDL and MRL are determined.

For methods where an MDL study does not exist or for which BAL is not accredited, the MDL can be determined on a batch specific basis. MDLs may be estimated from the standard deviation of the method blanks. If done, the MDL is estimated as 3 times the standard deviation of the method blanks for method blank corrected data and as the average of the method blanks + 3 times the standard deviation for uncorrected data. For methods where method blanks typically yield a result of 0 at the instrument, then 3 times the standard deviation of replicate analyses of the low calibration standard are used to estimate the MDL. The MRL is set at the lowest calibration standard or at a minimum of 2 times the estimated MDL, whichever is greater. Under no circumstances may the reported MDL be less than 10% the level of the MRL.

For food testing, the MDL can be determined as described in section 11.3.1, the AOAC Method 2015.01, or in a similar fashion, as long as the procedure for determining the MDL is documented.

MDLs can also be calculated using historic data for method blanks. Data for at least 20 method blanks should be available in the LIMS control charts before calculating an MDL by this procedure. For uncorrected data, the MDL is determined as the average of the method blanks plus the standard deviation of the method blanks multiplied by the Students' T value for the number of replicates at the 99% confidence interval. For method blank corrected data, the MDL would just be the standard deviation of the method blanks multiplied by the Students' T value for the number of replicates at the 99% confidence interval. As before, the MDL may not be less than 10% the level of the usable MRL for the method.

11.4 Initial and Continuing Demonstration of Capability

11.4.1 Initial Demonstration of Capability (IDOC)

Every analyst must perform an IDOC study prior to independently analyzing or preparing samples by each method. Specific requirements for a passing IDOC are outlined in SOP BAL-1010.

11.4.2 Continuing Demonstration of Capability (CDOC)

Each analyst must demonstrate that they are continuously capable of performing the analysis. Specific requirements for a passing CDOC are outlined in SOP BAL-1010. If the analyst has not demonstrated continuing capability for a method, then the VP of Operations will determine if additional training is required and the analyst must successfully perform an IDOC prior to analyzing any further client samples.

11.4.3 Documentation

All raw data, including preparation logs, analytical bench sheets, and instrument printouts, used to perform the IDOC or CDOC study are scanned and attached to the relevant sequence in the LIMS and saved for no less than seven years. All IDOC and CDOC studies must be reviewed by the QA Department and the QA Manager must annually certify that each analyst is capable of performing their respective duties by completing a Demonstration of Capability Certification Statement form for each method an analyst performs. The QA Manager's signature (or Technical Director's signature in the absence of the QA Manager) and date on the Demonstration of Capability Certification Statement form indicates authorization by management for the specified person to perform the indicated laboratory procedure. These forms are kept in the employee training records.

11.5 General QC Requirement Statement

The QC requirements previously listed are general requirements only. Specific methods or client-specific Statements of Work may have more stringent requirements that take precedence.

12.0 Data Reduction, Validation, Reporting and Storage

Prior to release of analytical results, all unknown sample and associated quality control data are subjected to the full review process briefly described below. Refer to BAL SOPs BAL-1500, BAL-1501, and BAL-1502 for a detailed description of the data review procedure.

12.1 Analytical Integration

Analytical instrumentation signal output is integrated by manufacturer specific software (i.e., Guru™, MassHunter, Perkin Elmer ELAN, or Total Chrome software). Analytical runs are stored electronically. Integration software is verified by the QA samples. Any integration software related problem that affects samples would also affect QA samples; therefore, as long as QA criteria are met, the software is assumed to be operating properly. The IT department at BAL maintains all documentation of integration software upgrades.

12.2 Data Entry

The preparation technician or analyst is responsible for entering all sample masses/volumes and preparation volumes into the bench sheet in the LIMS, as well as any batch specific QC information. The analyst is responsible for checking this information, entering all analytical specific information into the instrument software, and uploading all of the instrument results into the LIMS. The analyst ensures that all data is present. For most test codes, final results are automatically calculated in the LIMS using formulas specific to the analytical method used.

12.3 Data Reduction

The analyst or other data uploader is responsible for uploading all instrument data into the LIMS and performing primary validation of the data. Initial data reduction is performed by the instrument software to obtain initial results in units of measured pg, measured ng, measured absorbance, grams, or µg/L. This information, along with volumes/masses used in the preparation/analysis of the samples is either uploaded or hand entered into the LIMS where final results are calculated according to the method used to analyze the samples. There are some test codes that have final results calculated in a locked Excel spreadsheet and the final result is entered into LIMS (such as iron speciation and %TS).

Although rare, BAL does allow for CCV correction of data as long as the client has been consulted and agreed to allow CCV correction. In order for CCV correction to be applied to any results, the CCVs must show a consistent trend or bias through the analytical run. Any use of CCV correction must be approved by either the Technical Director, VP of Operations, or QA Manager. Approval must be documented in writing with initials and date of approval.

The following documentation must be present with every data package: preparation notes, SPFs, lab bench sheets, analysis bench sheet, and analyst's notes. All information required to reproduce the data, including volumes/masses prepared, final preparation volumes, and any dilutions performed at the instrument (volume of original preparation and final dilution volume and diluent and any intermediate dilutions) must be included with the data package. All instrument printouts

are stored electronically as PDF files and must have the analytical sequence recorded on them and the sample ID for each instrument response.

12.4 Primary Data Review

After the data has been acquired and any necessary calculations performed, the primary data review is performed by the analyst. Items to be reviewed include correct upload of the data, sample identity, instrument calibration, QC samples, detection limits, numerical computations, accuracy of transcriptions, sample preparation logs, instrument/analytical logs, and compliance with the individual method.

12.5 Final Data Review and Validation

Following the analyst's review, the raw data and calculations undergo final review by the QA Group. The QA Group also reviews comments about analytical conditions as well as any interpretations made by the analyst. Additionally, the QA Group examines the QC sample data and ensures that the analytical results meet or exceed the acceptance criteria for frequency, accuracy, and precision.

After final data review and validation is complete, the QA Group applies any necessary data qualifiers, sets data to be reported to “reportable,” and signs-off on the sample processing form. Only the Technical Director, QA Manager or delegate and, in extreme cases, the VP of Operations or BAL President/CEO have authority to change the reportability of data after final data review. All changes in the status of data (e.g. batched, prepared, reviewed – primary, reviewed – final, reportable versus non-reportable, etc.) is tracked and updated automatically in the LIMS with a time stamp and identity of the person that made the change.

12.6 Data Reporting

Prior to data reporting, the Project Manager responsible for the report reviews the data a final time for any discrepancies. The final client report is generated only when the Project Manager is satisfied that the data is valid and all project specific requirements have been met. Any Level IV report or reports where issues require atypical narration goes through secondary review by another Project Manager or Project Coordinator who then signs-off on the report as well. Only then is the report sent to the client.

Typically at BAL, results are reported down to the MDL. Results ≤ the MDL are reported at the MDL and qualified “U” as non-detectable. Results at or below the MRL but above the MDL are reported as the calculated result and qualified “J” or “B” (depending on the requirements of the client) as an estimate. Results above the MRL are reported as the calculated result without qualification. For Department of Defense (DoD) work, results are either reported only down to the LOQ or by the rules defined in the DoD/DOE QSM. All sample results are reported to three significant figures except for percent total solids results and results for QC samples, which are both reported up to four significant figures. Sediment and soil results are typically reported on a dry-weight basis by dividing the wet weight result by the percent total solids result. Biota results

are typically reported on a wet-weight basis. However, upon request, biota results may be reported on a dry-weight basis as well.

Any sample that yields a non-detectable result and shows <30% recovery of the matrix spike cannot be reported unqualified. The sample is qualified "R" to indicate that all generated results for the sample are unusable and no result for the sample is reported. Refer to section 11.3 for differences in how the limits of detection are defined between work performed under the TNI standard and work performed under the DoD/DOE QSM and how this affects how results are reported by BAL.

If contamination is suspected due to elevated method blanks, then results may require special qualification. Unless the method specific criteria are different, the criterion for acceptable method blanks is that the absolute value of each method blank must be ≤ the MRL (the DoD/DOE specific criterion is < ½ the MRL). If this criterion is not met, then the method blank with the highest absolute value must be ≤ 10% of any quantifiable (> MRL) result. Any quantifiable result not ≥ 10 times the absolute value of the highest method blank not meeting acceptance criteria is qualified "X" and narrated as being an estimate due to elevated method blanks. Results below the MRL will already be qualified as estimates and generally do not require additional qualification due to the elevated method blanks.

Generally, data are reported in a format generated by the BAL LIMS with a case narrative or a cover letter attached. With the exception of Certificate of Analysis reports, all of the data, including standard spike recoveries, control samples, duplicate analyses, and results from blank analyses, are reported along with the sample results. Data quality issues are addressed in the cover letter or case narrative, which discusses each batch and sequence associated with the work order being reported. Final reports are submitted to all required parties (project dependent). A copy of each report is stored electronically as a PDF file for BAL's internal records (see 12.7 data storage). All laboratory report forms and reporting formats shall be in compliance with the reporting requirements of the applicable project for which they are generated. A specific statement clearly identifying any results that do not meet the specific project requirements (i.e. non-NELAP accredited or non-DoD accredited work performed by BAL) is included, if applicable, in the final report.

Every reasonable effort is made to report data with acceptable associated quality assurance sample (QC) results. However, barring this, data is qualified appropriately to indicate when batch QC does not meet specific acceptance criteria. A list of all data qualifiers and their definitions is included with every data report. While BAL has its own in-house data qualifiers that are defined on the "Report Information" page of the report, the use of project or accreditation specific qualifiers always take precedence over BAL qualifiers. In such instances, the project specific qualifier definitions would over-ride the BAL qualifier definitions on the "Reporting Information" page of the final report and discrepancies would be narrated.

Electronic files may be transferred to a client via electronic data deliverable (EDD) or by email with the following statement:

Electronic signatures as pictures of actual signatures are used for signing many legal documents, including PDF copies of data reports, at BAL. The original JPEG or TIF is maintained by the user and only the user and the IT Manager have access to the original copy. Use of another person's signature file without the express permission of the signature's originator is grounds for disciplinary action.

Disclosing information about client results or contracts to any party outside of BAL without prior permission from the client and BAL and without following all reporting policies stated in the BAL Comprehensive Quality Assurance Plan and associated standard operating procedures is forbidden by all personnel. The term "reporting" refers to any electronic, written, or spoken discussion of client data or other confidential and proprietary information. To protect the client's proprietary rights, data must never be reported over the phone. Additionally, data can only be reported directly to the client with whom BAL has a legal contract to perform work, unless BAL has written permission from the client to release the data or report to a third party.

12.7 Data Storage

For all data generated by BAL, data packets and electronic summaries are kept for a minimum of 7 years, longer if contract specific requirements call for longer storage. All computer files are stored both on computer hard drive and on backup disks. Computer files of client reports are organized by work order number, batch spreadsheets are organized by batch number, and all project information is organized by project ID. All sequence specific data is also scanned and stored electronically, this includes: SPFs, analyst notes, handwritten benchsheets, and Excel® results spreadsheets. After the scan is verified the original hardcopy data packet is stored for at least 2 months in the file cabinets in the main office before being shredded.

Data records that are stored solely on electronic media are kept on media that is readily accessible and retrieved using computers on the network. Software and hardware required to view and/or open the stored data is kept onsite in working condition. When hardware or software is changed or updated, the old hardware and software is kept available until verification that all data previously generated are compatible and retrievable with the new software and hardware. If compatibility does not exist, the old hardware and software is kept onsite in working condition in adherence to the data retention policy.

The Project Manager is responsible for maintaining all client specific files. All client reports are stored electronically as a PDF. Other information provided by the client that is important to the legal COC or analysis of the samples (e.g. COC forms, analysis request forms, airbills etc.) are attached to the work order in LIMS or stored on the server.

All MDL study documentation and other QA documentation are scanned and attached to the relevant sequence in the LIMS. Hard copies of the original data are filed by the QA Department by date and stored for at least one year before being shredded. Scanned data is kept for a minimum of 7 years.

All data generated is scanned and stored electronically for a minimum of 7 years. After five years, electronic data may be removed from the server, but is backed up to two separate external hard drives that are stored at separate, secure locations. Any paper work that has not been scanned for any reason will be stored for a minimum of 7 years. All hard copies of any documents that could be traced directly to a client are destroyed by shredding prior to disposal. The IT Manager monitors the upkeep of computer files.

13.0 Document Control Policies

13.1 SOPs, Manuals, Handbooks, and Plans

All documents important to the internal operations of BAL go through formal procedures as to their writing, approval, implementation, retirement, and sharing. Documents other than SOPs or the CQAP referenced by lab personnel to perform their routine laboratory duties (such as equipment manuals, accreditation documents, etc.) are considered part of the management system and are subject to document control procedures as noted in SOP BAL-1001.

13.1.1 Writing and Approval of SOPs, Manuals, Handbooks, and Plans

Refer to SOP BAL-1000 for writing and approval of SOPs, manuals, handbooks, and plans.

13.1.2 Annual Review of SOPs, Manuals, Handbooks, and Plans

All BAL SOPs and Plans are reviewed annually. Refer to SOP BAL-1000 for specific procedures to follow when revising an SOP.

13.1.3 Retirement of SOPs, Manuals, Handbooks, and Plans

When a BAL document is retired, the original is clearly labeled “Retired” and the date of its retirement is also clearly indicated. All copies of the retired document are either destroyed or also clearly labeled as being outdated. The original is then archived as a historical record (either electronically as a PDF file or as a hardcopy) for no less than 7 years.

13.1.4 Proprietary Information

Many of the analytical methods used at BAL have been developed in-house and are considered proprietary information. Clients or other organizations requesting particular SOPs are required to first sign an “Agreement for Confidential Disclosure and Restricted Use of Proprietary Information”, see SOP BAL-1001. Whenever possible, “client ready” SOPs, where all proprietary information has been removed, are given to clients instead of full SOPs. The permission of the President/CEO or their delegate must be obtained prior to sending any non-redacted SOP containing proprietary information.

13.1.5 Uncontrolled Documents

Uncontrolled documents are defined as any document (CQAP, SOP, “cheat sheet”, password, etc.) or portion thereof that has not been signed and dated as being approved for use in the laboratory and is not under the direct control of the Quality Manager. No such document is allowed to be posted or used in the laboratory and must be immediately removed upon detection. When referencing the CQAP or an SOP, the current approved version should be opened directly from the server from the *SOP & other DOCs* folder or from SOP Tracker. All documents in these locations are secured PDF versions with signed and dated cover pages. If isolated pages from SOPs would be of value in the lab, then these must be issued by the QA Department per section 13.1.6.

13.1.6 Controlled Documents

Controlled documents may only be issued by the QA Department. The Quality Manager is responsible for ensuring that all controlled documents are tracked through the laboratory and updated or retired as needed. Controlled documents are tracked in the *QA Controlled Document Log* stored in a secured folder on the server. At a minimum, all controlled documents must contain the following:

- Title of controlled document
- Revision number
- Approval initials (applies to printed documents only)
- Date put into service
- Date retired (if applicable)

13.2 Client Records

All client reports, records of results, and correspondences are maintained by BAL for a period of no less than 7 years. All project information is electronically stored on the server for a minimum of 7 years. All "Active Client" specific files are maintained by the Project Manager.

In the event that BAL should go out of business, it is BAL's stated policy that every attempt will be made to notify all clients (past and present) and ask them how they would wish to have their records maintained or transferred. In the advent of a change in ownership, it is BAL's policy that all records become the property of the new owner unless specifically requested otherwise by the client. All reasonable demands of the client shall be met and no client information shall be removed from BAL premises without the client's written consent.

13.3 Employee Records

All employee records, including resumes, training, IDOC and CDOC studies, are maintained by BAL for a period of no less than 7 years following the departure of the employee.

14.0 Information Systems

Brooks Applied Labs' Information Systems infrastructure is a key component of the company-wide Data Security Plan. BAL officers and managers take measures to ensure all staff are aware of and actively participate in BAL's data security program. SOP BAL-0700, *Security of Electronic Data* and the employee presentation, *Computer Security Awareness* contain detailed policies, procedures, and responsibilities designed to facilitate a proactive approach by staff to maintain a high level of physical and data security.

The BAL Data Security Plan incorporates preventive measures, operational redundancies in case of small component failures, and recovery procedures in case of large component failures or a disaster. The following systems are in place and constantly being reassessed and maintained to provide a secure technology platform for BAL operations and business objectives.

14.1 Security

The firewall appliance consists of Sophos UTM 9 software installed on a Dell PowerEdge R610 server. The firewall protects the local area network (LAN) from the public internet while providing a gateway for internet traffic, Office 365 "cloud" email access, and secure VPN access to the LAN for users working remotely. The Sophos firewall also provides wireless access to the LAN and a second wireless network for guests that gives internet access but no access to the LAN.

A second passive firewall is maintained with the exact same configuration as the primary device. This provides redundancy in the event of device failure.

Workstation access is available to all authorized employees and is controlled via active directory domain logon. Shared data is available from servers connected to the local network. Specific directories or files may have restricted access by using group policy security settings.

All networked servers and workstations are protected with Sophos Endpoint Security and Control software. This provides anti-virus and host-based intrusion prevention for all devices exposed to the network and internet.

14.2 Hardware

BAL has two physical offices located in adjacent buildings. A single network and one point of entry from the wide area network (internet) are accomplished using high throughput (10 gbps) fiber optic cable connecting the two buildings.

Two Dell PowerEdge R730xd servers running VMware ESXi form the backbone of the server-client network. The domain active directory, shared file server data, Laboratory Information Management System (LIMS) database, and remote access server are each hosted on individual virtual servers distributed tactically on the Dell servers.

Additional hardware is deployed on the LAN to provide functional redundancy. An example is an additional virtual server with active directory installed.

All servers contain multiple hard drives, and they are configured with RAID 10 to minimize risk of data loss due to hard drive failure.

Each of the Dell servers acting as a VMware host can be used in a recovery situation if a single hardware server fails. The individual virtual servers can be recreated from a backup and restored to the production network on a working VMware host server. This action would be implemented in case of corruption of a virtual server instance, or if the VMware host itself had a failure, or if the server hardware failed.

14.3 Backup and Recovery

Backup software Veeam Backup & Replication version 9.5 provides scheduling, automation and monitoring of backup for all the virtual server data. Each virtual server, for example Active Directory, File server (shared network data), and LIMS database, can be backed up as a discreet data file. This allows the data file to be restored as a working server with all the accompanying data it contained at the time of backup. File level backup is also utilized with Veeam when necessary. This enables recovery of a single file or folder that has been accidentally deleted or corrupted.

Full server backups are performed weekly, with incremental backups occurring daily for each server. However, the LIMS server is backed up more frequently. Weekly full backups of the LIMS server are performed along with hourly incremental backups. The backup program is configured to send alerts for successes and failures of backup scripts via email to the IT Manager, who monitors the emailed alerts.

A second server with Veeam Backup & Replication version 9.5 is located in a separate building than the first server, and it is configured to make a copy of the full and incremental backups completed by the primary backup server. It accomplishes the objective of having an offsite backup for a potential restore operation, and the offsite backup data is the same age as the primary data backup.

In addition, ICP instrument computers with ICP data and selected network workstations are backed up directly to external disk and copied to a separate disk so there is a duplicate.

Records stored electronically are supported by the proper hardware and software to assure retrieval.

15.0 Corrective Action

15.1 Corrective Action

The laboratory has a corrective action system to identify any situations that may adversely affect data quality. These situations include, but are not limited to:

- Results outside of quality control criteria as outlined in individual SOPs
- Statistically out-of-control-events
- Deviations from normally expected results
- Suspect data
- Deviations from the method
- Special sample handling requirements

Corrective action may also be initiated as a result of other QA activities, such as performance or system audits.

Once a requirement for corrective action has been identified, the VP of Operations and/or the QA Manager must be notified immediately. A verbal notification may be initially made; however, written documentation of the problem is required typically using an incident report form (Refer to BAL SOP BAL-1008). The QA Manager, VP of Operations, or the Technical Director is responsible for evaluating the situation and determining the appropriate corrective action. The QA Manager, VP of Operations, Technical Director, and President/CEO of BAL have the authority to stop work whenever a nonconformance issue may threaten the quality of data produced by BAL. Corrective action steps include, but are not limited to:

- Problem identification
- Investigation to determine the root cause of the condition
- Action to eliminate the problem
- Increased monitoring to evaluate the effectiveness of the corrective action
- Verification that the problem has been eliminated

Documentation of problems requiring corrective action is important to overall laboratory management. Any lab personnel may initiate a corrective action, but it is the QA Manager who is responsible for ensuring that the action is documented. The QA Manager is also responsible for verifying that initial action has taken place and appears effective and, after an appropriate time, for checking to see if the problem has been fully resolved.

15.2 Reporting Improper Laboratory Practices

In addition to documenting laboratory incidents, determining root cause, and developing effective corrective action through the incident report and resolution procedure, BAL is required to report any incident that includes improper laboratory practices as defined in the current DoD/DOE QSM. Examples of prohibited practices that require notification of our DoD accrediting body (ANAB) include:

- Fabrication, falsification, or misrepresentation of data
- Improper clock setting (time traveling) or improper date/time recording
- Unwarranted manipulation of samples, software, or analytical conditions
- Misrepresenting or misreporting QC samples
- Improper calibration
- Concealing a known analytical or sample problem
- Failing to report the occurrence of a prohibited practice or known improper or unethical act to the appropriate laboratory, contact representative, or government official.

When an improper laboratory practice is discovered by the lab, BAL must report the incident and submit the associated corrective action directly to ANAB.

15.3 Client Communication and Complaints

Brooks Applied Labs is committed to providing the best laboratory services available in the industry. To this end it is vital that good and proper communication is always maintained with our clients. Clients' opinions of the services provided by BAL are very important to us. All client comments, whether positive or negative, are taken seriously. If a client has a complaint, it is recorded and kept on file by the Client Services Manager. Complaints may encompass any aspect of the services provided by BAL, including analytical services, technical services, or quality assurance.

Once a complaint has been recorded, the BAL Project Manager who is most responsible for the service to which the complaint is directed shall handle the matter with the client. If necessary, the BAL Project Manager will initiate a corrective action to deal with any legitimate deficiencies brought to our attention by clients. The resolution of all complaints shall be recorded along with the initial complaint.

Any events that cast doubt on the validity of any test results already reported must be conveyed to the affected client within one business day of when the events become evident to BAL management. In addition to any phone messages, the client must also be promptly notified in writing. This is typically done in the form of an email. If more formal documentation is required, then a signed letter may be provided, as well as copies of any associated corrective actions. Records of corrections taken or proposed corrective actions to resolve any nonconformance must be submitted to the client within 30 business days of discovery.

Both negative and positive feedback from clients are reviewed at the end of the year as part of the Managerial Review in an effort to constantly improve the quality system and products and services provided by BAL.

15.4 External Audits

Corrective action may also be initiated by external audits by regulatory agencies or clients. BAL considers audits as an opportunity to improve upon our services. Any deficiencies discovered during external audits are documented and corrective actions are initiated to address them. All root cause analyses and corrective actions are documented and maintained by the QA Manager

and must be approved of by the auditing body. Any changes to the approved corrective action plan (CAP) must be discussed with and approved by the auditing body.

Willful avoidance of approved corrective action implementation may result in loss of DoD ELAP accreditation or result in DOECAP Priority I findings. As a result, work may be discontinued until implementation is verified by the DoD ELAP AB or DOECAP Operations Team, as appropriate.

16.0 Performance and System Audits

16.1 System Audits

16.1.1 Internal Systems Audits

BAL conducts specific function audits on an annual basis. The laboratory is audited against the most current DoD or DOE checklist to ensure that BAL remains compliant with all accreditation requirements.

Additionally, laboratory walk-through audits are performed monthly throughout the laboratory. Each lab group is audited separately. Laboratory walk-through audits are not as thorough as the annual audit but serve to ensure that quality assurance procedures are being performed routinely before issues arise. The findings from monthly walk-through audits and any necessary corrective actions are presented in monthly QA reports.

16.1.2 External Systems Audits

BAL has occasional audits from various clients and accrediting agencies. The principal organizations that conduct audits of BAL's facilities and operations are the Washington State Department of Ecology, DOECAP as part of the Department of Energy audit program, and ANAB as part of Department of Defense, ISO 17025, and TNI (on behalf of Florida DOH) accreditations. BAL views external audits as an excellent tool for evaluating our quality and for finding areas for improvement. BAL always welcomes any client (current or potential) or government agency to conduct on-site audits.

16.2 Performance Evaluation

Performance Evaluation must be conducted at least biannually and may consist of blind samples, split samples with another laboratory (interlaboratory comparison study), QC samples (unknown to the analyst), performance test samples, and/or blind spiked samples. BAL frequently participates in assisting agencies to certify reference materials. Any chemist may analyze these performance evaluation samples as long as they have successfully completed training for the affected analysis. The Project Manager and QA Manager are responsible for overseeing BAL's participation in each study, and all associated documentation, reporting, and record keeping.

Any failing results from performance testing must be documented and corrective action developed to address the causes within 21 calendar days of receipt of the results by the lab.

External Performance Evaluations are as follows:

Agency	Study Title	Frequency
ERA	Blind PE samples*	Semi-Annually**

* As a better indication of overall laboratory performance, PE samples are treated like all other received samples in terms of receipt, preparation, quality control, and analysis.

** Participation in additional PE studies may be required as part of corrective action.

16.3 Annual Management Review of the Quality Systems

Brooks Applied Labs management conducts an annual review of the quality systems, including health and safety, radioactive hazardous waste, and radioactive management functions to ensure that they are still effective. All reports by managerial personnel, the outcome from all recent internal and external audits, the results from PE studies and interlaboratory comparisons, changes in the volume and type of work performed, feedback from clients, and corrective actions are taken into account during the managerial review. This review also looks ahead to anticipated issues for the coming year(s). The ultimate purpose of this review is to ensure the continued effectiveness and improvement of the quality systems in place at BAL. The summary of management review for the previous year is typically completed during the first quarter of the year. This summary includes a corrective action plan for any outstanding findings that come out of the management review.

APPENDIX A – Common Abbreviations

AA – Atomic Absorption

BLK – Method Blank

BAL – Brooks Applied Labs

BS – Blank Spike

CCB – Continuing Calibration Blank

CCV – Continuing Calibration Verification

CDOC – Continuing Demonstration of Capability

CGMP – Current Good Manufacturing Practices

CLIA – Clinical Laboratory Improvement Amendments

COC – Chain of Custody

CQAP – Comprehensive Quality Assurance Plan

CRM – Certified Reference Material

CVAFS – Cold Vapor Atomic Fluorescence Spectrophotometry

DoD – Department of Defense

DoD/DOE QSM – DoD/DOE Consolidated Quality Systems Manual for Environmental Laboratories

DOE – Department of Energy

DUP – Method Duplicate

EDD – Electronic Data Deliverables

EPA – Environmental Protection Agency

ERA – Environmental Resource Associates

FEP – Fluorinated Ethylene Propylene (Teflon™)

FLPE – Fluorinated High-Density Polyethylene

HDPE – High-Density Polyethylene

HEPA – High Efficiency Particulate Air

HGAAS – Hydride Generation Atomic Absorption Spectrometry

IDOC – Initial Demonstration of Capability

IC – Ion Chromatography

IC-PAD – Ion Chromatography with Pulsed Amperometric Detection

ICP-MS – Inductively Coupled Plasma – Mass Spectrometry

IC-ICP-MS – Ion Chromatography – Inductively Coupled Plasma – Mass Spectrometry

ICV – Initial Calibration Verification
IT – Information Technology
LCS – Laboratory Control Sample (typically a BS or SRM)
LIMS – Laboratory Information Management System
LOD – Limit of Detection
LOQ – Limit of Quantification
MDL – Method Detection Limit
MRL – Method Reporting Limit
MS/MSD – Matrix Spike / Matrix Spike Duplicate
NELAC – National Environmental Laboratory Accreditation Conference
NELAP – National Environmental Laboratory Accreditation Program
PM – Project Manager
QA – Quality Assurance
QC – Quality Control
QCS – Quality Control Standard
RPD – Relative Percent Difference
RSD – Relative Standard Deviation
SPF – Sample Processing Form
SOP – Standard Operating Procedure
SOW – Statement of Work
SRL – Sample Receiving Log
SRM – Standard Reference Material
TNI – The NELAC Institute
VP – Vice President