COMPREHENSIVE QUALITY ASSURANCE PLAN

for

BROOKS RAND LABS

3958 6th Avenue NW
Seattle, Washington 98107 U.S.A.
206.632.6206
206.632.6017 (fax)
www.brooksrand.com
brl@brooksrand.com

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LABORATORY'S APPROVED SIGNATORIES

Michelle Briscoe – BRL President (Technical Director)
(effective upon sign-off Lead technical Director)
Date: 3/27/14

Annie Carter – BRL VP of Operations
(Deputy Technical Director)
Date: 3/27/14

Frank McFarland – BRL VP of Quality
Date: 3/27/14

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2.0 Statement of Policy

The Brooks Rand Labs (BRL) 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, National Environmental Laboratory Accreditation Conference (NELAC), Department of Defense Quality Systems Manual (DoD QSM), and U.S. Department of Energy Quality Systems for Analytical Services Manual (DOE QSAS) standards have been incorporated into BRL's quality assurance program. Internal audits are conducted by BRL, and external audits of BRL's facilities are conducted by ANSI-ASQ National Accreditation Board/ACLASS every two years to ensure that BRL meets the requirements of NELAC standards (for NELAC accreditation through the Florida Department of Health), the DoD QSM, and ISO 17025 accreditation requirements. Reciprocal NELAC accreditation is granted by the Oregon Environmental Laboratory Accreditation Program, the New York State DOH, the New Jersey Department of Environmental Protection, the State of Maine Department of Health and Human Services, and the State of Louisiana Department of Environmental Quality. BRL is also accredited by the Washington State Department of Ecology and reciprocal accreditation is granted by the California State Department of Health Services. The Washington State Department of Ecology routinely conducts audits of BRL's facilities every 3 years to ensure that BRL's quality assurance program meets the agency's specific requirements. Additionally, periodic external audits initiated by clients serve to ensure that Brooks Rand Labs continually meets the specific requirements of our clientele.

Brooks Rand Labs management is committed to compliance with NELAC, DoD QSM, and DOE QSAS standards. As such, BRL management is committed to continually improving the quality assurance program. The BRL quality assurance program is implemented through a team effort across the entire laboratory. All personnel concerned with environmental testing activities within BRL 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. These are read and followed by all analysts. In addition, BRL is on the cutting edge of method development for the analyses of trace metals. All BRL-developed methods undergo rigorous testing and validation before they are approved by BRL 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).

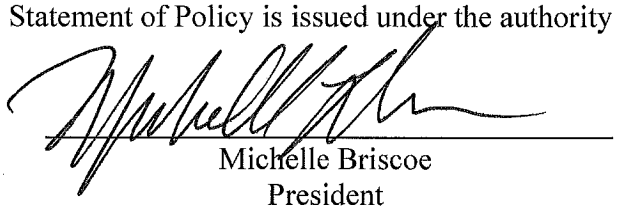
- *Method performance (precision and bias) documentation.* Data from analyses are monitored using control charts to assess performance and to detect trends.

- *Regular evaluation of the quality system.* Brooks Rand Labs management is committed to continually improving the quality system. Annual reviews of all standard operating procedures and the CQAP, 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 Rand Labs management is committed to professional laboratory practices and to the quality of our environmental testing in providing services to our clients. The above considerations are documented to ensure the quality of the data generated by BRL. Subsequent sections of this manual will 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 VP of Quality and the President of BRL. Refer to Section 8.5 for details on how any exceptional departure from BRL procedure is handled.

The Statement of Policy is issued under the authority of the President of Brooks Rand Labs.



Michelle Briscoe
President

3/27/14
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	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 and VP of Quality	Bachelors degree in physical sciences (advanced degree or equivalent experience preferred) and 15 years experience in the environmental 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 of Brooks Rand Labs	Bachelors degree in physical sciences and 6 years experience in the environmental lab business, with 5 years in management positions
Technical Directors	Oversees operations of the laboratory, including: maintaining instrumentation and equipment, technical oversight, method development, validation, and approval, R&D, responsible for ensuring laboratory compliance with the NELAC, DoD, and all other accrediting authority standards	President of Brooks Rand Labs and the VP of Operations both serve as Technical Directors	Bachelors degree in physical sciences (advanced degree or equivalent experience preferred) with 24 hours of college chemistry credits, and 3 years experience in the environmental trace metals analytical lab business, with 1 year in a supervisory position
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 of Brook Rand Labs	Bachelors degree in physical sciences or equivalent and 3 years experience in the environmental lab business; including at least 1 year in Project Management positions

TABLE 3.1 RESPONSIBILITIES AND MINIMUM QUALIFICATIONS (CONTINUED)

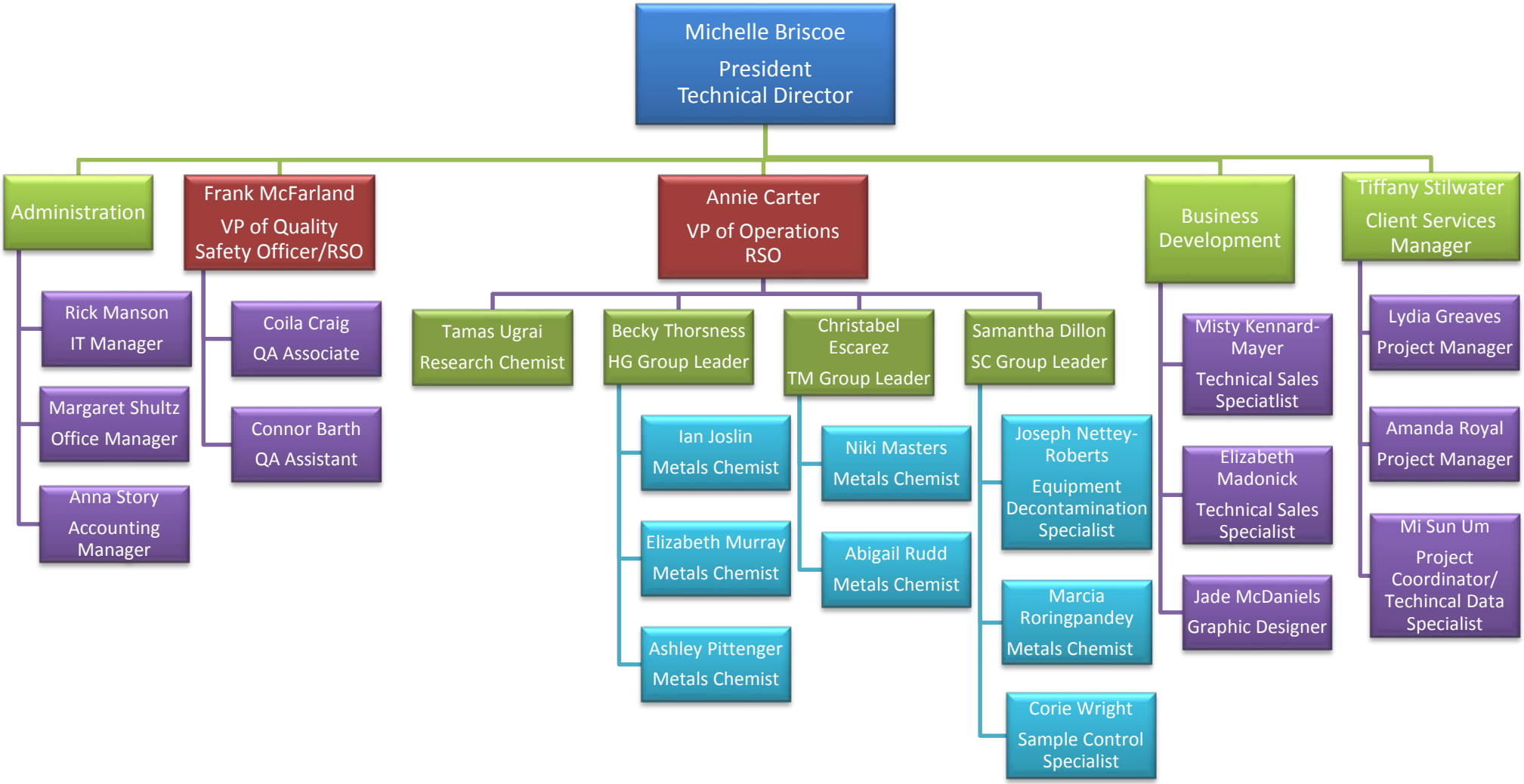
Title	Responsibilities	Deputies	Minimum Qualifications
Project Manager	Manages client projects including: internal communication of client requirements, reporting to client. Performs report level review of the data prior to issuing reports to clients.	Another Project Manager or Project Coordinator	Bachelors degree in physical sciences or equivalent and 1 year experience in the environmental lab business
VP of Quality/ 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, evaluating the effectiveness of training, using all available tools to monitor trends and continually improve the QA program, and ensuring laboratory compliance with the TNI, DoD, and all other accrediting authority standards. Performs final review of data. Responsible for ensuring all data undergoes final data review prior to release to clients.	President of Brook Rand Labs	Bachelors degree in physical sciences (advanced degree preferred) and 3 years lab experience with 1 year of applied QA principles
QA Associate	Be able to mirror VP of Quality in QA related duties. Cover for VP of Quality in his absence. Authorized to perform final review of laboratory data.	VP of Quality	Bachelors degree in physical sciences and 6 months experience in the environmental lab business or Associates degree and 1 year of analytical lab experience with 6 months of applied QA principles.
QA Assistant (as necessary)	Assist VP of Quality in duties. Performs final review of laboratory data.	QA Associate	Bachelors degree in physical sciences or Associates degree and 6 months experience in the environmental lab business
HG Group Leader	Oversees the mercury analytical group including: training records, sample preparation, analysis, and scheduling within the group. Oversees classical chemistry methods for percent total solids and total volatile solids performed by the mercury group. Responsible for ensuring all data produced by the	VP of Operations	Bachelors Degree in physical sciences and 1 year of analytical lab experience as an analyst, or 5 years experience in the environmental lab business

	HG Group undergoes primary review prior to turning data in to QA.		
TM Group Leader	Oversees the trace metals analytical group including: training records, sample preparation, analysis, and scheduling within the group. Oversees classical chemistry methods for percent total solids and total volatile solids performed by the trace metals group. Responsible for ensuring all data produced by the TM Group undergoes primary review prior to turning data in to QA.	VP of Operations	Bachelors Degree in physical sciences and 1 year of analytical lab experience as an analyst, or 5 years experience in the environmental lab business
Metals Chemist	Perform and document sample preparations and analyses following SOPs, instrument calibration and reagent/standard preparation. May perform primary review of data for methods trained to perform.	Group Leader	Bachelors Degree in physical sciences or equivalent or Associates degree with 2 years analytical lab experience.
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 performed by the sample control group. Responsible for ensuring all data produced by the SC Group undergoes primary review prior to turning data in to QA.	VP of Operations	Associates Degree and 1 year of analytical lab experience
Sample Control Specialist	Oversees custody of sample, sample receipt, and sample log-in.	Group Leader	Bachelors Degree in physical sciences or equivalent or Associates degree with 1 year analytical lab experience.
Equipment Decontamination Specialist	Cleaning and decontamination of laboratory equipment; sample disposal	Group Leader	Bachelors Degree in physical sciences or equivalent or Associates degree with 1 year analytical lab experience.

3.2 BRL 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 (next page).

FIGURE 3.2 - BRL ORGANIZATIONAL CHART



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

Brooks Rand 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 BRL are free from undue commercial, financial, and any other pressures that may adversely affect the quality of their work. In addition, the management of BRL 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 sloppy or contains mistakes and all changes made to the 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 BRL SOP BR-1101) and must sign the "Brooks Rand Labs Ethical and Legal Responsibilities Agreement" form stating that they agree to adhere to all aspects of the ethics program at BRL prior to working with client samples. Any employee who becomes aware of unethical behavior is encouraged to report the behavior to BRL management. Any employee should feel free to report such behavior to any manager, on up to the President of BRL. Brooks Rand Labs 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 BRL and its clients. Under no circumstances is any behavior that might adversely affect the quality of the data produced by BRL 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 documentation of the investigation's actions shall be maintained for a minimum of five years. Documentation may be stored either electronically or by hardcopy, but must be readily available.

4.0 Training

4.1 Technical Training

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

Brooks Rand 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 each method must be successfully completed as per NELAC, DoD QSM, and DOE QSAS standards prior to any 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 VP of Quality 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 BRL 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 environmental 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 BRL employees receive annual training in their legal and ethical rights and responsibilities. This training includes the following topics: Non-discriminatory workplace; drug free workplace; data manipulation; and workplace ethics. This training also specifies the potential punishments and penalties for improper, unethical, or illegal actions performed by BRL employees. The Brooks Rand Labs data integrity plan (SOP BR-1101) discusses in detail the specific legal and ethical rights and responsibilities of employees at BRL. 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 must review the training records for all employees within their group to ensure completeness. At a minimum, the VP of Quality 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 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 five years, regardless of the employment status of the individual.

4.5 Additional Training

The VP of Quality 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 (along with any necessary assistance from the Client Services Manager, project managers, VP of Operations, and the VP of Quality, as designated) carefully reviews the specific requirements of every contract before any new work is accepted by BRL. BRL will accept a project only after the President or her designee has ensured that BRL possesses the appropriate facilities and resources to carry out the work as specified by the client.

5.2 Capabilities of Organization

BRL is an analytical laboratory primarily focused on providing analytical services for the determination of low-level trace metals and metals speciation in environmental and food samples. Brooks Rand Labs' specialties are threefold: 1) providing the lowest detection limits commercially available, 2) speciation of oxidation state and organometallic forms; and 3) analysis of non-routine matrices.

Each sample preparation method followed at Brooks Rand Labs is dependent upon the analyte of interest and the type of matrix being analyzed. Refer to the specific analytical method or standard operating procedure (SOP) for a description of each particular preparation method utilized at BRL. Other sample preparation methods may be used upon request for specific enforcement or compliance-based contracts.

5.3 Quality Assurance Objectives

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

The primary purpose of the Quality Assurance Program at BRL is to ensure that all data reported to our clients are accurate and reproducible. To this end, BRL 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 Department.

5.4 Subcontracted Work

Occasionally, a client may wish to work directly through Brooks Rand Labs even for analyses that BRL does not currently perform. Under these conditions, BRL will subcontract work to other laboratories with the client's approval. The subcontracted laboratories must meet all project specific requirements before samples may be delivered.

Subcontract laboratories are selected by Brooks Rand Labs 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 Brooks Rand Labs and the client, a purchase order agreement is issued by Brooks Rand Labs to the subcontract laboratory.

All samples are logged into Brooks Rand Labs' Laboratory Information Management System 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 Brooks Rand Labs Project Manager with the final report and EDD (if requested). The Project Manager will then forward the report to the VP of Quality. Data is reviewed and notes will be written regarding the 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, Brooks Rand Labs 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

BRL 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 Comments in BRL's LIMS for the related project(s).

Sample Dispatch - Field samples are relinquished by the sample collection team to BRL's Sample Control Group following a strict chain-of-custody process. Time, date, and samplers signatures 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 BRL for Sampling

TABLE 6.2 - CONTENTS OF BRL SAMPLING KITS

Equipment	Construction	Use	Parameter Groups
Sample Container(s)	Teflon® – FEP, PFA Fluorinated – FLPE Glass – I-Chem 200 series HDPE (for solids only) Ziploc® bags (some biota)	Sampling/Storage	Mercury and monomethyl mercury in water/soil/biota
	HDPE bottles LDPE, HDPE, or PP jars Ziploc® bags	Sampling/Storage	Trace Metals, except Hg, in water/soil/biota
	Iodated carbon (IC) Traps or Gold Coated Media in Ziploc® bags	Sampling/Storage	Mercury in air
Shipping Containers	Plastic cooler or cardboard box	Sample Transport	All parameter groups
Gloves, Clean Room	Vinyl, non-powdered	Sampling	All parameter groups

6.3 Decontamination Procedures

Client Equipment

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

Cooler/shipping containers

All coolers are cleaned prior to use for shipping samples or sample containers. Appropriate sample containers are placed in the coolers 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

BRL 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.

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

All preservation reagents used by BRL are analytical grade or better. For total metals analyses, samples not being filtered in the lab may be sent to BRL 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 (See Table 6.5). Samples can also be sent to BRL on ice via overnight shipping to be preserved at BRL. All samples for “dissolved” analyses must be filtered before preservation.

TABLE 6.5 - SUMMARY OF SAMPLE CONTAINERS & PRESERVATIVES

Parameter	Method	Minimum Volume	Container	Means of Preservation	Holding Time
A. Water[1]					
As and Se (for HGAAS analysis ONLY)	BR-0020	500 mL	HDPE[2]	0.4% (v/v) 12 M HCl; pH < 2 within 28 days of collection	6 months
Arsenic (As) Speciation	EPA 1632	125 mL		0.4% (v/v) 6 M HCl at time of collection; Store in dark at 0-4 °C[3]	28 days
Selenium (Se) Speciation Natural Waters	BR-0061	2 x 125 mL		Field-filtration recommended, especially for samples with high levels of solids; unpreserved; zero headspace; keep dark; maintain collection temperature as best possible	14 days (target analysis within 2 days of receipt)
Selenium (Se) Speciation Industrial Wastewaters		2 x 125 mL		Field-filtration recommended, especially for samples with high levels of solids; HCl to pH < 2 (pre-preserved containers with 0.1% HCl); zero headspace; keep dark; maintain collection temperature as best possible (keep cool)	14 days (target analysis within 2 days of receipt)
MeHg (Freshwater)	EPA 1630	250 mL	Fluoropolymer, FLPE, or Glass with Fluoropolymer lined lids	0-4 ºC and dark immediately; 0.4% (v/v) 12 M HCl within 48 hours of collection	6 months
MeHg (Salt Water ≥ 10 ppth salinity)	EPA 1630	250 mL		0-4 ºC and dark immediately; 0.2% (v/v) 18 M H_2SO_4 within 48 hours of collection	6 months
EtHg	In-House	250 mL		0-4 °C and dark immediately; HCl to 0.4% in lab within 48 hrs of collection	6 months
Total Hg	EPA 1631E	125 mL		Preserve to pH < 2 with BrCl in original sample container within 28 days	90 days
Acid-labile Hg	BR-0003	250 mL		0.8% 12 M HCl within 24 hrs of collection; Store in Dark at 0-4 °C	Analyze 21 days (± 3 days) after preserv. and < 90 days
Total Volatile Hg	BR-0005	1-L	Glass with Fluoropolymer lined lids	No head space; Store in Dark at 0-4 ºC	24 hours
Reactive Hg	In-House	n/a	Glass septa-top bottles; gold-coated sand trap (requires special sampling equipment)	Field collect reactive Hg onto gold traps and mail overnight; dark	Analyze gold traps within 7 days of collection
Fe (II)	SM3500B	40 mL	Glass with Teflon-lined lid w/preservation	0-4 ºC, dark, 1% (v/v) 12 M HCl at collection (pre-preserved vial)	48 hours
ICP-MS Metals (Freshwater)	EPA 1638, Mod.	125 mL	HDPE	1% (v/v) HNO_3; pH < 2 in lab, should be acidified within 14 days of collection[4]	6 months

[1] Samples to be analyzed for dissolved analytes must be filtered prior to preservation. This should preferably be done on site in the field. If this is not possible, the sample must be chilled (0-4 ºC) and filtered within 48 hours of collection (24 hours for mercury samples).
[2] High Density Polyethylene
[3] All temperature ranges of 0-4 ºC assume an acceptable measured temperature of ± 2 ºC from either extreme as long as the samples do not freeze.
[4] While Brooks Rand Labs suggests that samples be acidified within 14 days of collection, there is no good scientific evidence to indicate that not doing so will impact data quality as long as samples are acidified in the original collection container for a minimum of 24 hours prior to analysis.

ICP-MS Metals (Brackish or Seawater)	EPA 1640, Mod.	1 L	HDPE	0.1% (v/v) HNO_3 (APDC Prep) or 0.2% (v/v) HNO_3 (RP prep) or 1% (v/v) HNO_3 (chelating column procedure) in lab, should be acidified within 14 days of collection	6 months
Cr (VI)	SW 7196A	125 mL	HDPE	Store at 0-4 °C	24 hours
	SW 7196A, Mod.			16 mL/L of 25% NaOH (pre-preserved bottle); store at 0-4 ºC	30 days
Cr(III) and Cr(VI), dissolved drinking waters, ground waters, and fresh surface waters	BRL SOP #BR-0061	2 x 40 mL	40-mL glass vials or 60/125-mL HDPE (not acid cleaned)	Field-filtration required (optional for drinking water samples); unpreserved; 0-4 °C and dark immediately; zero headspace; keep dark	ASAP, but not to exceed 14 days
Cr(VI), dissolved drinking waters, ground waters, fresh surface waters, and most relatively clean wastewaters	BRL SOP #BR-0061/ EPA 218.7 (modified)	40 mL	40-mL glass vial or 60/125-mL HDPE (not acid cleaned)	Field-filtration required (optional for drinking water samples); container preserved with $NH_4OH/(NH_4)_2SO_4$ buffer to pH > 8; zero headspace; keep dark; keep at ≤ 25 °C during shipment; store refrigerated at lab	14 days
Au	EPA 1638, Modified	40 mL	Glass with fluoropolymer-lined lid	HCl + HNO_3 (3% + 1%) in lab upon receipt (closed-vessel digestion in the original sample vial)	ASAP
Os	EPA 1638, Modified	125 mL	HDPE	1% HCl	6 months
TSS	EPA 160.2	1 L	HDPE	Store at 0-4 °C	7 days
Lab pH, hydrogen ion	EPA 150.1	25 mL	Glass or HDPE	0-4 °C	ASAP (to be determined the day of receipt)

TABLE 6.5 - SUMMARY OF SAMPLE CONTAINERS & PRESERVATIVES (CONTINUED)

Parameter	Method	Minimum Volume	Container	Means of Preservation	Holding Time
B. Wet Sediments and Soils					
Total Metals	Various	Fill 4 oz jar	FLPE, Glass or HDPE	0-4 ºC during shipping, ≤ 4 °C and in dark in lab	1 year[5]
Metal Species (other than As species and Reactive Hg)	Various	Fill 4 oz jar	FLPE, Glass or HDPE	If possible, place on dry ice or freeze immediately following collection. Otherwise, maintain a temperature of 0-4 °C following collection and during shipping, and ship ASAP (within 48 hours) to the lab; store at -15 °C.	7 days to freeze; 1 year to analyze
As Species	EPA 1632	Fill 4 oz jar	FLPE, Glass or HDPE	0-4 ºC during shipping, < -18 °C in lab	1 year
Reactive Hg	In-House	Fill 4 oz jar	FLPE or HDPE	Flash frozen or frozen immediately (overnight) in the field, ship frozen on dry ice, ≤ -18 °C in lab	14 days to prep; 28 days to analysis
Cr (VI)	SW 3060a/ 7196a	Fill 4 oz jar	FLPE, Glass or HDPE	0-4 ºC during shipment; ≤ 4 ºC in lab	280 days
% Solids	EPA 160.3	100 mL	FLPE, Glass or HDPE	0-4 °C	7 Days[6]
C. Dry Sediments and Soils					
Total Metals	Various	Fill 4 oz jar	FLPE, Glass or HDPE	N/A (Room Temperature is OK)	1 year
Metal Species	Various	Fill 4 oz jar	FLPE, Glass or HDPE	Room Temp OK (recommended 0-4 ºC during shipment; ≤ -15 ºC in lab)	1 year
Cr (VI)	SW 3060a/ 7196a	Fill 4 oz jar	FLPE, Glass or HDPE	0-4 ºC during shipment; ≤ 4 ºC in lab	28 days
D. Wet Tissues					
Total Metals or Species (other than As)	Various	Fill 4 oz jar	FLPE, Glass, HDPE or Ziploc bags	0-4 ºC during shipping, -15 to -30 °C, dark in lab	1 year
As Species	Various	Fill 4 oz jar	FLPE, Glass, HDPE, or Ziploc bags	0-4 ºC during shipping, < -18 °C in lab	2 years
E. Tissues (freeze dried)					
Total Metals	Various	Fill 4 oz jar	FLPE, Glass or HDPE	N/A (Room Temperature is OK)	1 year
Metal Species (other than As Species)	Various	Fill 4 oz jar	FLPE, Glass, HDPE, or Ziploc bags	Room Temp OK (recommended 0-4 ºC during shipment; ≤ -18 ºC in lab)	1 year
As Species	EPA 1632	Fill 4 oz jar	FLPE, Glass, HDPE, or Ziploc bags	N/A (Room Temperature is OK)	2 years
F. Air					
Total Hg	EPA 324 / IO-5 / D6350	NA	Iodated Carbon Trap or Gold Coated media	mbient temp OK, keep dark; ends plugged, store in Ziploc® Bag	28 days
G. Biomonitoring					

[5] There are no established holding time limitations for solid samples.

[6] Although the standard temperature / holding time requirements for % solids are 0-4 ºC for 7 days, many solid samples are frozen and held for much longer periods prior to analysis for other parameters. Ideally, sample aliquots for each parameter should be removed prior to freezing. If this has not been done and % solids analysis is required for the samples, then the aliquots for % solids should be removed at the same time as the aliquots for the other parameters to ensure similar sample characteristics between aliquots.

Total Metals (including Hg) for Urine	Various	90 mL	Polyethylene Urine Specimen Container (transfer to FPE for Hg analysis)	0-4 ºC during shipment; 0-4 ºC in lab (or ≤ -15 ºC to -30 ºC in lab)	28 days (1 year if frozen – being investigated)
Total Metals and Metals Speciation in Whole Blood	Various	6 mL	6-mL royal blue top K2 EDTA tube	0-4 ºC during shipment; ≤ -15 ºC to -30 ºC in lab	1 year
Total Metals and Metals Speciation in Plasma/Serum	Various	6 mL	6-mL royal blue top Vacutainer with clot activator	0-4 °C during shipment; ≤ -15 °C in lab	1 year
Total Metals and Metals Speciation in Hair	Various	100 mg	Zip-type plastic bag	Dark, room temperature	1 year

6.6 BRL's Policy on Accepting Samples

Brooks Rand 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 BRL'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 samples may be batched for further analysis. The terms under which BRL would enter into a contract are explained in section 5.1 of this document.

Once a legal contract is in place, BRL 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 BRL 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, BRL 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 Field Sampler to clarify any questions concerning the COC before samples are batched.

At all times, BRL reserves the right to not accept samples that are deemed to be a threat to the health or safety of BRL personnel beyond what might reasonably be expected while working within an environmental laboratory.

Refer to the sample receiving SOP (BR-0300) for specific information pertaining to BRL's sample acceptance policy.

7.0 Sample Custody

7.1 Field Custody

Formal custody requirements begin at BRL with the shipment of sample containers to the field. Every shipment must be documented by BRL. With the exception of HDPE bottles sent for the collection of hydride, Se speciation, and all solid samples, a minimum of 10% of the bottles from 10% of the cases for each manufacturer lot # are tested for all applicable analytes prior to shipment. BRL provides Chain-of-Custody (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, quantity of containers shipped and their date of shipment.

While BRL does not typically provide field 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. For example, for mercury work, only rigorously acid-cleaned FEP, or pre-tested FLPE and glass containers with fluoropolymer lined lids may be used for water samples. See section 6.5 of this document for container, preservation, and holding time requirements for other analyses. Tissues and other solid matrices may be stored in FLPE containers, HDPE containers, glass containers with fluoropolymer lined lids, or plastic bags/wrap. Solid samples are preserved by shipping on ice, followed by storage in a freezer at -15 to -30 °C (solids to be analyzed for arsenic speciation must be stored ≤ -18 ºC).

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

7.2 Laboratory Custody

7.2.1 BRL Definition of Laboratory Custody

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

- It is in the possession of a sample control specialist, a laboratory chemist, or a group leader.
- It was in the possession of a sample control specialist, a laboratory chemist, or a group leader, 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 BRL 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 BR-0301 for example of form).

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 number against the COC form. The temperature of the samples at the time of receipt is determined by aiming a calibrated IR thermometer directly into a sample.

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 acidification are preserved in the field, the sample control specialist checks the pH and documents that it is less than 2. If it is not less than 2, additional preservation reagent is added to the sample(s) and the amount of acid required to adjust the pH to < 2 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 immediately notifies the client of any concerns.

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.

Refer to SOP BR-0300 for a detailed description of BRL's sample acceptance policy.

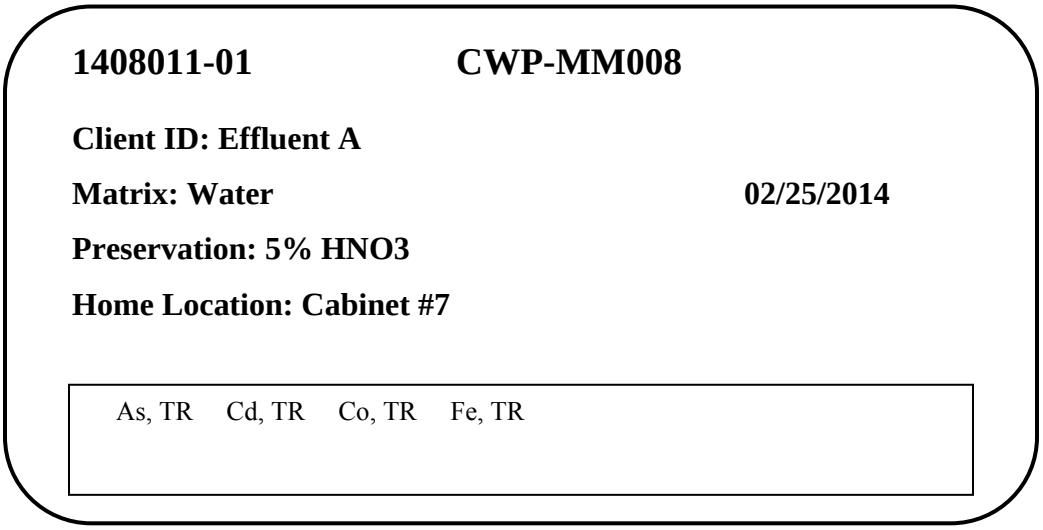
7.2.3 Sample Log In

When logging in samples, the sample control specialist must check the LIMS Project Information against the COC form received to ensure that the work has been authorized. If any discrepancies exist, the Project Manager is immediately notified. The Project Manager will confirm the contracted analyses and update either the LIMS 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 2014 is given the work order number 1408011). The samples within a shipment are then each identified by sequential numbering. For example, if three samples were received in the 1245011 shipment they would be given the sample numbers 1408011-01, 1408011-02, and 1408011-03. 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 may be broken

up into more than work order. The BRL work order and sample numbers are referenced during all laboratory preparations and analyses.

When a bottle is removed from the Ziploc® bags in which it was sent, the bottle should be rinsed with clean DIW (for low-level samples) and/or wiped with a clean cloth. Bottles are then labeled with the BRL sample number, BRL project number, client sample ID, matrix, date of sample receipt, preservation, storage location, and list of analytes to be analyzed for. An example of a BRL sample label is as follows:



After all of the log-in information is recorded in the LIMS, the BRL Sample Receipt Log (refer to SOP BR-0300) 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 a locked area within the premises of BRL with restricted access. To satisfy these custody provisions, the laboratory implements the following procedures:

- Access doors to the laboratory 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 on shelf space in the secure sample storage cabinets

Samples requiring refrigeration or freezing are stored in facilities dedicated to secure sample storage in a sample storage room located in the northwest corner of the facilities. The samples are removed from the shipping cooler and stored in their original containers, unless damaged. Samples not requiring refrigeration are stored on shelves in the secure sample storage cabinets,

which helps to protect samples from UV radiation. All standards and other chemicals used at BRL 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 BRL 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, BRL project number, received date, and due date. The folder is given to the Project Manager who then reviews the information, signs and dates the BRL Sample Receipt Log, and files the folder in the "Active Customer" file located in the Project Manager's office. The "Active Customer" files are sorted alphabetically by the BRL project number.

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 deliverables 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, which correlates to the date that the sample preparation, analysis, data review and report need to be completed. This date is generally four days prior to the final report due date for a standard 20 business day turnaround time, in order to allow sufficient time for data validation, final report generation, review, and submittal. If samples are on a rush turn-around schedule, then the due date will be adjusted to give less time between the date on the SPF and the date that the final report must be sent to the client.

The duration of original sample storage at BRL 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 worksheets, laboratory notebooks, instrument operation logbooks, instrument printouts (raw data), and final analytical reports.

7.2.5.1 Sample Batching - After samples are received and logged-in, the samples are then batched by the analytical Group Leaders. 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 2014 is numbered B140805. 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 analyzed. At the time of batching the SPF is printed from the LIMS. Any special QA requirements and/or pertinent information provided by the client concerning the sample preparation/analysis should be noted on the SPF. This information should be added 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 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. From the time the chemist removes samples from the storage area the SPF must remain with the sample batch. All sample preparation details must be documented on the SPF or in a logbook. 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 prep 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, along with the SPF and all preparation documentation, are stored in a secure laboratory area. Once prepared, the status of the samples is changed from "batched" to "prepared" in the LIMS.

Note: If during sample preparation the technician notices that there is insufficient sample for preparation, the technician should send an email informing their Group Lead and the appropriate Project Manager of this information and write the information on both the SPF and the benchsheet. The Group Leader should set the analysis for the affected sample to "cancelled", write "insufficient sample for analysis" in the container comments field, and send an email to the affected Project Manager and the other Group Leaders if any other analyses are affected. It is up to the Group Leaders to coordinate who will set any other affected test code's analytical status to "cancelled."

If the sample is exhausted following preparation, then the technician should make a note on the benchsheet and send an email to the Group Leader, affected Project Manager, and the QA Group. 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.

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 of the batches analyzed as part of the sequence. When a batch is analyzed, the chemist must sign and date the SPF and update the LIMS. Any comments on unusual observances or deviations from the analytical SOP must be documented and must be referenced on the SPF. As previously mentioned, movement of the batch throughout the lab is documented in the LIMS at the prep bench sheet. The chemist then uploads the data into the LIMS and signs and dates the SPF with when this action was

performed. During the upload of the data, the status of all samples is changed from "prepared" to "analyzed" in the LIMS. The chemist then performs the primary review of the data.

7.2.5.4 Primary Data Review - When the data is reviewed, the reviewer must sign and date the SPF, update LIMS, and comment on any unusual observances or deviations from appropriate. 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 then puts together the data package, which includes a printout of the Analysis Sequence form, any sequence lab bench sheets, all SPFs, all sample preparation documentation, and any chemist notes. Instrument raw data is saved electronically as a PDF file. Once the data has been primary reviewed, the status of the samples is changed from "analyzed" to "reviewed – primary" in the LIMS and the SPF is signed and dated.

7.2.5.5 Final Review - A member of the QA Group reviews the final data. After the final review, either the VP of Quality 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 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 form for tracking deviation is the SPF. This form should 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 on the SPF include, but are not limited to the following:

- Samples not cold when removed from refrigeration-see instrument log book.
- Samples over distilled-see distillation prep sheet.
- Samples prepared differently from SOP-see preparation notes.
- Out-of-Control calibration curve - see chemist's notes.

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 Subcontracting - Only in extremely rare occasions will BRL subcontract samples for analysis. This is 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 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. In such a case, the

documentation to transfer samples includes collection date and time (if available from the field samplers), the Field ID#, the BRL Lab ID #, the date of preparation (if extracts are transferred), and the requested analyses. Refer to section 5.4 for further discussion.

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 VP of Operations has determined that there is no further value in analyzing the preparations, such as water preparations for methyl mercury that are only stable for a maximum of two days. The disposal of each batch must be documented in the LIMS.

7.2.6.2 Original Samples – It is BRL policy to maintain all samples (aqueous and solid) for a minimum of 60 days after reporting results unless previous arrangements have been made with the client. At least once a year, all refrigerators and freezers should be cleared of any samples for which results have been reported more than two months ago. Samples that require longer storage should be separated from those that may be disposed after the two month period. The Sample Receiving Log in the LIMS must be updated to indicate that the samples have been disposed.

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 BRL SOP BR-0303 for disposal limits for specific elements). The method of disposal (routine verses high-level disposal) must be indicated on the appropriate form.

7.2.6.3.1 Routine Disposal

7.2.6.3.1.1 Water Samples and Acid Digestions - All water samples (including preparations) and acid digested solid samples that are not designated as being hazardous may be disposed down the drain. All acidic samples must be neutralized with soda ash prior to disposal.

7.2.6.3.1.2 Native Solid Samples and Dry Weights - All native solid samples (not sample preparations) that are not designated as hazardous may be discarded directly into the garbage.

7.2.6.3.2 High Level Disposal - All High Level metal sample waste (as well as other waste that is considered to be hazardous) must be recorded in the Sample Receiving Log (original samples) or on the SPF (sample preparations). The total volume added to the High Level Waste containers must be logged in the "Waste Disposal Log."

7.2.6.3.2.1 Water Samples and Acid Digestions - All water samples (including preparations) and acid digested solid samples that are designated as hazardous are placed directly into the high level metals/corrosives waste storage container.

7.2.6.3.2.2 Native Solid Samples and Dry Weights - All native solid samples (not sample preparations) that are designated as hazardous are disposed of directly into the hazardous waste container.

7.2.6.3.2.3 All Solvent Extracts - All solvent extracts must be treated as hazardous waste. Solvent extracts may be consolidated in clearly marked containers near the hazardous waste fume hood, and then disposed of as hazardous waste.

7.2.6.3.3 Non-Routine Disposal - Samples that are designated by the client to be high level in an analyte not performed by BRL shall be considered hazardous and treated as hazardous waste upon release for disposal. In certain cases, BRL 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 Laboratory Director's instruction) and disposed of through a licensed hazardous waste acceptance facility. BRL may also arrange with the client to return the leftover samples after analysis.

7.2.6.3.4 High-Level Metals Waste Transport and Ultimate Disposal - Once a sufficient volume of waste is generated, warranting proper disposal, a waste disposal company should be contacted, and the waste scheduled for pick-up. It shall be the responsibility of the waste handling company to transport and dispose of the high level metal waste in a manner consistent with local and federal environmental laws and regulations.

7.2.6.3.5 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.

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

8.0 Analytical Procedures

8.1 Method References

Refer to the tables in section 6.0 for method number references and to Appendix A for a list of BRL analytical Standard Operating Procedures (SOP).

8.2 Field Methods

Not Applicable. BRL is not currently involved in the analysis of samples directly in the field.

8.3 Analytical Method Modifications

Methods BR-0006 and BR-0002 (Modifications of EPA Method 1631, Revision E and EPA Method 1631, appendix to) - EPA Method 1631.E describes the determination of mercury in ambient water while the Appendix to EPA Method 1631 describes the determination of mercury in solids. Any modifications that Brooks Rand Labs has made to these methods are fully documented in the appropriate SOP. All solid (biological, sediments and soils) matrices are prepared in accordance with SOP BR-0002, *BRL 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 Spectrophotometry (CVAFS)*. All aqueous matrices are prepared in accordance with SOP BR-0006, *BRL Procedure for EPA Method 1631, Revision E (8/02): Mercury in Water by Oxidation, Purge and Trap, and Cold Vapor Atomic Fluorescence Spectrometry (CVAFS)*. Please refer to the method validation package for SOPs BR-0002 and BR-0006 in Appendix C (available upon request).

Method BR-0011 (Modification to EPA Draft Method 1630) – BRL has developed a method for the determination of methyl mercury at the ultra-trace level in various matrices. The SOP for this method, BR-0011: *Determination of Methyl Mercury by Aqueous Phase Ethylation, Trapping Pre-Collection, Isothermal GC Separation and CVAFS Detection*, is included in Appendix B (available upon request). By this method waters are distilled to remove methyl mercury from the matrix. Alternatively, water samples may be extracted to remove methyl mercury from the matrix or analyzed by direct ethylation. Biological samples (i.e. animal and plant tissue) are digested in a KOH/Methanol solution. Sediment samples are extracted using dichloromethane. After sample preparation all samples are ethylated with tetraethyl borate which forms ethyl derivatives of the mercury species. The ethylated mercury species are collected on a Tenax® trap that is then introduced into the GC oven, pyrolytic column, and AFS detector. Under the flow of an inert carrier gas, the Tenax® trap is gently heated to release the mercury species, which are then separated chromatographically prior to being decomposed to elemental mercury (Hg^0), in the pyrolytic column. The AFS detector then detects Hg^0. Please refer to the method validation package for SOP BR-0011 in Appendix C (available upon request).

Method BR-0021 (Modification to EPA Draft Method 1632, Revision A) – BRL has developed a method for the determination of inorganic arsenic, trivalent arsenic,

monomethyl arsinic acid, and dimethyl arsinic acid at the trace level in various matrices. The SOP for this method, BR-0021: *BRL Procedure for EPA Method 1632, Revision A (1/01): Chemical Speciation of Arsenic in Water and Tissue by Hydride Generation Quartz Furnace Atomic Absorption Spectrometry*, is included in Appendix C (available upon request).

Method BR-0060 (Modification to EPA Methods 200.8, 1638, and 6020) –The SOP for this method, BR-0060: *Determination of Trace Elements by Inductively Coupled Plasma – Mass Spectrometry (ICP-MS) using a Perkin-Elmer ELAN DRC II*, is included in Appendix C (available upon request). Any modifications that Brooks Rand Labs has made to this method are fully documented in the SOP.

Methods BR-0063, BR-0066, and BR-0069 (Modification to EPA Method 1640) – The SOPs for these methods, BR-0063: *Determination of Trace Elements in Seawaters and Low Level Waters by Online Column Chelation Preconcentration – Inductively Coupled Plasma – Mass Spectrometry using a Perkin-Elmer ELAN DRC II*, BR-0066: *Reductive Precipitation of Total recoverable and Dissolved Metals from Brackish and Seawater Samples* and BR-0069: *Extraction Using Co-APDC for Nickel, Copper, Silver, Cadmium, and Lead in Water*, are included in Appendix C (available upon request). Any modifications that Brooks Rand Labs has made to this method are fully documented in the SOPs. Modifications to the analysis of samples are documented in BR-0060: *Determination of Trace Elements by Inductively Coupled Plasma – Mass Spectrometry (ICP-MS) using a Perkin-Elmer ELAN DRC II*.

Method BR-0080 (Modification to SM 3500 – Fe B) – The SOP for this method, BR-0080: *Determination of Iron Speciation in Water Samples by Colorimetric Detection*, is included in Appendix C (available upon request).

Method BR-0085 (Modification to EPA Methods SW 3060A and SW 7196A) – The SOP for this method, BR-0085: *Determination of Hexavalent Chromium (Cr^{6+}) in Sediment and Aqueous Samples* is included in Appendix C (available upon request).

8.4 Laboratory Operations

8.4.1 Laboratory Glassware

All glassware for ultra-trace metals analysis must be rigorously cleaned, in order to minimize possible contamination. Glassware is initially cleaned using Alconox® and DIW and scrubbing with a brush. After thorough rinsing with DIW, the glassware is immersed into either a large rectangular vat containing 50% HCl or one containing 50% HNO_3, depending on how great the concentration of analyte in the glassware was prior to cleaning. Each of these acid vats is equipped with an immersion heater and a calibrated voltage regulator, or Variac). After filling the vat with glassware, the Variac and heater are turned on to heat the vat to 75°C and the samples are soaked for a period of 48 hours. Glassware to be used for ICP-MS analyses is stored in the cold 30% nitric acid vats.

Glassware for certain parameters other than ultra-trace metals need only be cleaned with Alconox and DIW followed by copious rinsing with DIW. A detailed account of the decontamination procedures for laboratory glassware is described in SOP BR-0402.

8.4.2 Reagent Storage

All supplies (i.e., glassware, chemicals, and reagents) are of the highest possible quality to ensure quality assurance and to avoid contamination. Reagents purchased from commercial vendors 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, BRL lot # and special storage requirements, if any.

Reagent water used in the laboratory is produced by reverse osmosis. The level of mercury in the reagent water is less than 0.2 ng/L and is checked during mercury analyses. Reagent water is tested monthly for ICP-MS metals at each sink used to clean bottles, prepare samples/reagents, or analyze samples. The reagent water is at metal concentrations below the method reporting limits (MRL) for each metal. If reagent water fails to meet the criteria for any metal, then the water may not be used for analysis of the metal until reagent water blanks have met the criteria.

Reagent solutions are stored in appropriate glass or plastic containers under conditions designed to maintain their integrity (refrigerated, dark, etc.). Shelf life is listed on the label, and the reagent is discarded after it has expired. Acids used are either glass-distilled or analytical reagent grade for trace metal analysis. Reagent solutions are checked for contamination by testing reagent blanks before use.

Refer to BRL's Chemical Hygiene Plan for a list of all chemicals used at BRL. The aforementioned document also describes where and how these chemicals are stored at BRL. Material Safety and Data Sheets (MSDS) are stored on the server as PDF files and should be consulted for detail concerning potential hazards associated with specific chemicals. MSDSs are organized on the server by both chemical name and by CAS number. BRL SOP BR-0500 describes the documentation of standards and reagents in the LIMS and the testing of standards and reagents.

8.4.3 Waste Disposal

Handling, storage, and disposal of laboratory-related hazardous wastes are subject to the regulations contained in the Resource Conservation and Recovery Act. BRL 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 EP 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.

BRL 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).

BRL 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 BRL 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 a decision is made to dispose of the laboratory samples, 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 Resource Conservation and Recovery Act (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.

Native samples and sample preparations must be disposed of in accordance with local, state, and federal regulations. Residual native samples must be separated by matrix (water, sediment/soil, and biota) and placed in the appropriate containers for disposal. Remaining sample preparation solutions must be separated by digestion type (acid, base, solvent extract) and placed in the appropriately labeled disposal containers.

8.4.4 Laboratory Procedures

BRL currently has one clean room for ultra-trace mercury analysis and a semi-clean room for other trace metal analyses.

The mercury laboratory, prep laboratory, receiving laboratory, bottle washing and sample storage 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 for mercury each day prior to beginning analysis by testing bubbler blanks. The average bubbler blank must contain less than 20 pg Hg. Clean room sticky mats are located at the entrance to minimize tracking in particles.

The ICP-MS lab is where other metals analyses are conducted. This lab contains two laminar flow hoods with HEPA filters. Incoming air is pre-filtered. The laminar flow hood is at one end of the room with the air intake at the other to facilitate efficient circulation through the HEPA-filter. In addition, the entrance to this lab has an enclosed gowning area to reduce direct flow from outside air. Reagent water is tested by ICP-MS at least once a month. Clean room sticky mats are located at the entrance to minimize tracking in particles.

8.5 Exceptional Departures from Standard Operating Procedures (SOPs)

The VP of Quality, VP of Operations, or President must approve of any departure from BRL standard operating procedures. Under no circumstances is the Sample Preparation Technician or the Analyst authorized to alter any standard procedure without managerial approval. Such approval and the actual departure from BRL procedure must be fully documented and conveyed to the affected client. All quality control criteria must still be met for all reported data.

9.0 Calibration Procedures

9.1 Instrumentation

Refer to the current revision of SOP BR-1205 for an up-to-date list of the equipment maintained at Brooks Rand Labs.

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. Original certifications for all stock standards are maintained in a designated binder located in the main office area.

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, BRL lot #, and the expiration date. Documentation for working standards must include: identification of the stock and intermediate standards used, the preparation date, method of preparation (specifically dilution information), the preparer's name, the concentration prepared, BRL 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 Table 9.3 and/or the analytical method or SOP for the specific procedures followed for the preparation 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%. Standards that are made daily are not tested against the old standard prior to use. Instead, they are verified against the second source standard.

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

TABLE 9.3 - STANDARD SOURCES AND PREPARATION

Instrument Group	Standard Source(s)	How Received	Storage	Preparation from Source	Lab Storage	Preparation Frequency
Atomic Fluorescence	Total Hg – High Purity Standards (calibration)	Solutions of 1000 µg/mL	Room temp. 2% HNO_3	Intermediate standards are prepared from Stock standard	10000 ng/mL, 2% BrCl at 4 °C	annually
				Intermediate standards are prepared from Stock standard	1000 ng/mL, 2% BrCl at 4 °C	annually
				Working standards are prepared from intermediate	10ng/mL & 1 ng/mL, 1% BrCl at room temperature	monthly
	NIST 1641d (independent check)	CRM Solution of 1601 µg/mL	4 ºC; 2% HNO_3	Verification standards are prepared from a source other than the calibration standards	1.601 ng/mL, 1% BrCl at room temperature	monthly
Atomic Fluorescence	Methyl Hg – Strem Chemicals, Alfa Aesar, or Environmental Resource Associates	Solutions of ~ 1000 ppm MeHg	Fridge 0-4 ºC	Intermediate standards are prepared from stock standard	1000 ng/mL, 0.2% HCl 0.5% HOAc in dark at 4 ºC	annually
				Working standards are prepared from intermediate	1 ng/mL & 10 ng/mL, 0.2% HCl 0.5% HOAc in dark	monthly
	DOLT-4 (Dogfish Liver)	Solid CRM 1330 ng/g	Room temp. in desiccator	~50 mg of CRM into 1 mL of KOH/methanol and diluted to 2.5 mL with methanol; final concentration of ~25 ng/mL	Room temp. in the dark	monthly

TABLE 9.3 - STANDARD SOURCES AND PREPARATION (CONTINUED)

Instrument Group	Standard Source(s)	How Received	Storage	Preparation from Source	Lab Storage	Preparation Frequency
ICP-MS	Single-Element High Purity Standards	Solutions of 1000 or 10000 ppm	Room temp. in the dark	Intermediate standards are prepared from Stock standards	Plastic cabinet under TM clean hood, 2% nitric	semi-annually
				Working standards are prepared from intermediate	Plastic cabinet under TM clean hood, 2% nitric	daily/ as needed
	Multi-Element CPI International (independent check)	Various conc. Depending on element	Room temp. in the dark	400x dilution working standard	Clean hood of ICP-MS room, 2% nitric	semi-annually / as needed
				10x dilution prepared from intermediate	Clean hood of ICP-MS room, 2% nitric	monthly / as needed
	NIST 1643e National Institute of Standards and Technology	Various conc. Depending on element	Room temp. in the dark	5x dilution at the instrument	Not stored	made daily

TABLE 9.3 - STANDARD SOURCES AND PREPARATION (CONTINUED)

Instrument Group	Standard Source(s)	How Received	Storage	Preparation from Source	Lab Storage	Preparation Frequency
HPLC-DRC-ICP-MS	Se(IV) Spex Certi Prep	Solutions of 1000 ppm	In TM drawer	Intermediate standards are prepared from Stock standards	Not stored	made daily
	Se(VI) High Purity Standards	Solutions of 1000 ppm	In TM drawer	Intermediate standards are prepared from Stock standards	Not stored	made daily
	Se (species) Inorganic Ventures (independent check)	Solutions of 1000 ppm	In TM drawer	Intermediate standards are prepared from Stock standards	Not stored	made daily
Atomic Absorption	As (species) High Purity Standards	Solutions of 1000 ppm	In TM drawer	Working Standard are prepared from stock standards	In TM drawer	monthly
	As (species) Lab Chem (independent check)	Solutions of 1000 ppm	In TM drawer	Working Standard are prepared from stock standards	In TM drawer	monthly
pH Meters	Scientific Products	pH 4, 7, 10	Room temp.	NA	NA	NA
Conductivity Meters	Fisher Scientific	Solutions of 9.77 µmhos 96 µmhos and 974 µmhos	Room temp.	NA	NA	NA

9.4 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 in the sample data. In addition, each instrument has a log book in which summarized information is documented. This summarized documentation includes date, analyst, batch #, calibration coefficient (or response factor), correlation coefficient (r), average blank level, and instrument noise level or other relevant instrument information. In addition, any instrument maintenance is documented in the instrument log books.

9.4.1 CVAFS, HGAAS, and ICP-MS Instrument and Method Calibration

Instrument abbreviations are as follows: Cold Vapor Atomic Fluorescence Spectrophotometry (CVAFS), Hydride Generation Atomic Absorption (HGAAS), and Inductively Coupled Plasma – Mass Spectrometry (ICP-MS). Each instrument used to analyze samples must pass the calibration criteria established in the appropriate method or operating procedure. The instrument calibration consists of analyzing a minimum of three standards (HGAAS and ICP-MS) covering at least one order of magnitude (one at the low end, one in the middle and one high standard below the upper limits of linearity within the calibration curve) and a calibration blank (Note: A minimum of five standards must be used in the calibration for all CVAFS work). These standards should span the linear range of the instrument. The correlation coefficient (r) of the initial calibration for ICP-MS must be ≥ 0.995. If the squared correlation coefficient (r^2) is calculated for the ICP-MS calibration, then r^2 must be ≥ 0.990. If a weighted linear calibration is used for ICP-MS analyses, then the r must be ≥ 0.990. For CVAFS the RSD of the calibration coefficients must be $\leq 15\%$ and for HGAAS the RSD of the calibration coefficients must be $\leq 20\%$. The initial calibration check, consisting of one standard at the mid-point of the calibration curve and one calibration blank, is performed immediately following this calibration. Continuing calibration verification standards are analyzed at the end of each batch or sequence and, depending on the analytical method, at a frequency of 10% during the course of the analytical sequence. Initial and continuing calibration checks are used to establish whether ongoing instrument calibration is acceptable. The calibration is verified with a standard prepared from a source independent of the calibration standards.

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 never 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. 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 explanation.

It is standard procedure at Brooks Rand Labs to calibrate analytical instruments daily prior to analyzing any client samples. Under certain circumstances further analysis of samples may be

reinitiated without recalibrating the instrument. If this is to be done for any reason, the following procedures must take place prior to the analysis of any blanks or client samples:

1) Less than 24 hours must have passed since the calibration was last verified
2) The VP of Operations, the VP of Quality, or specific Group Leader must approve of the deviation from standard BRL procedure and document the approval on the SPF
3) Both an ICV and a CCV must be run to verify the calibration
4) Either the VP of Quality or the Group Leader must approve of the calibration verification results prior to beginning to analyze samples

All standards used to prepare the calibration standard solution are obtained from chemical suppliers and are of high purity and concentration. The standards are routinely checked by the laboratory for traceability to National Research Council of Canada (NRCC) or National Institute of Standards and Technology (NIST) Standard Reference materials. These commercial standards are used as stock standards. 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. Preparation of all standards is recorded in the LIMS (as described in SOP BR-0500). All solutions are labeled as follows: name of solution, concentration of solution, date prepared, LIMS ID number, expiration date, and analyst's initials. All laboratory analysis (including instrument calibration) 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.

9.4.2 Periodic Calibration Procedures for other Laboratory Equipment

Periodic calibration checks are performed for associated equipment such as balances, thermometers, ovens, and refrigerators that are required in 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 BR-1200.

BALANCES

Balances are calibrated annually by a contracted, certified professional. Balances are also checked with Class S weights on a daily or 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 on a daily basis are themselves calibrated at a minimum every 5 years against NIST traceable weights.

TABLE 9.4 - CRITERIA FOR BALANCE CALIBRATION CHECKS
4-POINT BALANCE

Class S / 1 Weight (g)	Warning Level (g)	Control Level (g)
0.0100	0.0099 - 0.0101	0.0098 - 0.0102
0.1000	0.0997 - 0.1003	0.0995 - 0.1005
1.0000	0.9995 - 1.0005	0.9990 - 1.0010
10.0000	9.9950 - 10.0050	9.9900 - 10.0100
100.0000	99.9500 - 100.0500	99.9000 - 100.1000

3-POINT BALANCE

Class S / 1 Weight (g)	Warning Level (g)	Control Level (g)
0.100	0.099 - 0.101	0.098 - 0.102
1.000	0.995 - 1.005	0.990 - 1.010
10.000	9.950 - 10.050	9.900 - 10.100
100.000	99.750 - 100.250	99.500 - 100.500

TOP LOADING BALANCES

Class S / 1 Weight (g)	Warning Level (g)	Control Level (g)
1.00	0.99 - 1.01	0.98 - 1.02
10.00	9.95 - 10.05	9.90 - 10.10
100.00	99.5 - 100.50	99.00 - 101.00
200.00	199.00 - 201.00	198.00 - 202.00

PIPETTES

All pipettes are calibrated weekly. Pipettes performance is monitored by gravimetrically measuring the volume of DIW dispensed by each pipette over the range of its use prior to calibration with the assumption that the density of the water in the laboratory is 1.000 g/mL (± 0.003 g/mL). The volume dispensed by each pipette is then re-measured after calibration and both 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 monthly 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, SAND BATHS, WATER BATHS, 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, sand baths, and water baths are checked at least once during each use. Refrigerator and freezer temperatures are recorded in laboratory logs that are maintained by the Sample Control Group Lead. Oven, hotplate, sand bath, and water bath temperature are recorded in the sample preparation logs.

THERMOMETERS

The performance of each thermometer is compared annually to a certified NIST-grade thermometer and correction factors are posted for each thermometer in the area where it is used.

10.0 Preventative Maintenance

10.1 Routine Maintenance Measures

Refer to SOP BR-1205 (Preventative Maintenance) for greater detail of specific procedures.

TABLE 10.1 - PREVENTATIVE MAINTENANCE

Instrument	Activity	Frequency
ICP-MS	Inspect and/or change all tubing	Daily
	Clean sampler and skimmer cones	Daily
	Inspect torch and injector	Daily
	Check gas and coolant levels	Daily
	Change roughing pump oil	Monthly*
	Update mass calibration (tuning)	Monthly
	Replace quartz torch and injector	Semi-Annually*
	Replace sampler and skimmer cones	Semi-Annually*
	Replace RF coil	Semi-Annually*
	Replace chiller air filter	Annually*
	Replace ion lens	Annually*
	Schedule annual maintenance w/ Perkin-Elmer	Annually
HPLC (for IEC)	Change eluents	Daily
	Inspect lines for leaks and obstructions	Daily
	Monitor suppressor flow	Daily
	Clean and regenerate suppressor	Monthly*
	Clean and test analytical column	Monthly*
CVAFS	Prepare new soda lime pre-traps	Daily
(Manual Total Hg)	Condition bubblers and blank traps	Daily
	Check all fittings	Daily
	Soak bubblers for 15 min. in 1% KOH and then over the weekend with 10% HCl	Weekly*
	Inspect and/or change all tubing	Monthly*
	Make, test, and change out traps	Quarterly*
	Change lamp	Semi-Annually*
	Blank traps on incoming gas lines	Semi-Annually*
	Clean/change quartz cell	Annually*
	Check Electronics	Annually

* or as needed

TABLE 10.1 - PREVENTATIVE MAINTENANCE (CONTINUED)

Instrument	Activity	Frequency
CVAFS	Prepare new soda lime pre-traps	Daily
(MERX-T)	Check all tubing and clear any liquid in tubing	Daily
	Inspect and/or change all tubing	Monthly*
	Change Traps	Semi-Annually*
	Change lamp	Semi-Annually*
	Change traps on incoming gas lines	Semi-Annually*
	Clean/change quartz cell	Annually*
CVAFS	Inspect and/or change all tubing	Monthly*
(MERX-M MeHg)	Condition GC column at 80 ºC overnight	Quarterly*
	Change lamp	Semi-Annually*
	Check and change out traps	Semi-Annually*
	Blank traps on incoming gas lines	Semi-Annually*
	Clean/change quartz cell and replace GC column	Annually*
AA - Flame	Check tubing, pump and lamps	Daily
(As Speciation)	Rinse water removal trap with DIW	Daily*
	Clean spectrophotometer windows	Weekly
	Soak bubblers over weekend in 30% nitric acid	Monthly*
	Inspect and/or change all tubing	Monthly*
	Clean nebulizer	Semi-Annually
	Fine tune the instrument wavelength	Annually*
	Check instrument optics	Annually*
Colorimetric /	Clean sample compartment	Daily
Spectrophotometer	Windows cleaned	Monthly
	Check Electronics and lamp alignment	Annually
pH Meter	Clean; 2 pt. Calibration	After each use
Balances	Clean pans and compartment	Before/after every use
	3 to 4 pt. Calibration check	Before every use
	Certified Calibration	Annually
Pipettes	Check at mid-point volume	Daily
	Check bias and precision	Quarterly*
Conductivity Meter	Check batteries and probe cables	Weekly*
Refrigerator /	Check temperature	Daily
Freezers	Clean interior	Monthly*
	Check temp. against NIST cert. thermometer	Annually

* or as needed

10.1.1 Air Testing

The mercury lab, the sample preparation lab, the receiving laboratory, and bottle washing are monitored monthly for atmospheric mercury levels to ensure that these levels are sufficiently low for ultra-trace level mercury analysis. Air from each lab is pumped through a soda lime pre-trap and onto either a gold wire or gold-coated sand trap at a flow rate of 1 L/min until at least 20 L of air have been collected per trap. A warning level has been established at 15 ng Hg/m^3 with a shutdown control level at 25 ng Hg/m^3. Results from the monthly air tests are electronically on the server.

10.1.2 Water Testing

Reagent water is monitored for Hg on a daily basis when calibration blanks are analyzed. A minimum of four 100 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 average results must be < 5 pg Hg with a standard deviation < 2.5 pg Hg. A high level of mercury detected in the reagent water analysis may also be attributed to the bubbler itself, the $SnCl_2$, 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. 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 BRL acceptance criteria.

10.2 Documentation

Instrument logbooks are maintained for all equipment. These logbooks contain a complete history of past performance and maintenance. For each CVAFS and AA instrument, a logbook is kept to 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. BRL's sister company, Brooks Rand Instruments, is an instrument manufacturer specializing in ultra-trace level mercury analyzers; 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 the ICP-MS, BRL has three Perkin Elmer ICP-MS instruments. Perkin Elmer's service record is excellent, and none of our ICP-MS instruments has ever been out of service for more than 1 week.

BRL 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 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 VP of Quality 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 VP of Quality 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 certified reference materials (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 Rand Labs is rarely involved in field sampling. The client is typically responsible for field sampling activities and therefore mandates the requirements for field QC checks. However, BRL 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 level 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 level 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 client must be notified and every effort should be made to determine the source of the contamination and to eliminate it if possible.

Many methods require that field blanks be collected and analyzed if results are to be reported for regulatory purposes. While Brooks Rand Labs does not require that clients provide field blanks for analysis, BRL 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

11.1.2.1 Method Blanks

A method blank is a sample of 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 in equivalent amounts, digested in the same type container 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 minimum frequency and acceptance criteria are method specific (Refer to specific SOP). Also 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 11.2.2 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. If no spot contamination is evident in the batch (e.g. elevated trip or field blanks, random dissolved results significantly greater than total results), then the rejected method blank is not considered when evaluating the rest of the data. However, if there is evidence of additional spot contamination, then the data is evaluated against the method blank considered to be a

Grubb's Outlier such that any data point not ≥ 10 times the rejected method blank would require reanalysis or appropriate qualification.

Note: Some projects do not allow for the rejection of any method blank, such as those under the DoD QSM. For such projects the Grubb's Outlier method blank is still not used to correct the results, but all results are judged against the Grubb's Outlier as described above regardless of whether there is any further evidence of spot contamination in the batch or not.

The discarding of any data point as a Grubb's Outlier and the potential affect 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. Refer to Section 12.6 (Data Reporting) for specific instructions on how method blanks are evaluated and reported for non-method blank corrected results.

11.1.2.2 Matrix Spikes

Matrix spikes are routinely included in the analytical batch as they are required for most methods utilized at BRL. 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.

The target spiking level of the matrix spikes is 2-5 times the native sample concentration or 5 times the MRL, whichever is greater. However, it is not always possible to know the concentration of the native sample before preparing the matrix spikes. Historical data should always be consulted prior to spiking in-house samples if this data is available. If historical data is available, no further action is required as long as the spiking level is still within 1-20 times the native concentration. If the native result agrees with the historical data and the sample is spiked incorrectly, then the native and its associated matrix spikes should be reprepared and reanalyzed. Lacking historical data for the samples, most methods have default spiking levels. If these levels end up being less than the native concentration, then a post spike should be analyzed and the MS/MSD do not require analysis. If it is spiked at more than 20x the native concentration, then no post spike is required. The native sample should always be run at the same dilution as the MS/MSD to check for suppression, but may be reported from a different dilution if needed to obtain results that are above the MRL.

11.1.2.3 Blank Spikes

When spike blanks are employed at the request of a client as an additional QC check to monitor the efficiency of the method, a minimum frequency of one per sample batch must be prepared and analyzed.

The policy at BRL is to analyze one BS at approximately 10 – 20 times the MRL with default acceptance criteria of 75-125% if an appropriate CRM is not available.

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. BRL purchases PE samples from Environmental Resource Associates (ERA) semi-annually. 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 BRL, the Washington State Dept. of Ecology, Sate of Florida Dept. of Health, Oregon State Environmental Laboratory Accreditation Program, State of Louisiana Dept. of Environmental Quality, State of New Jersey Dept. of Environmental Protection, New York State Dept. of Health, California Dept. of Health Services, and the Maine Dept. of Health and Human Services for accreditation purposes.

Additionally, BRL routinely participates in laboratory intercomparison studies offered by such institutes as the International Atomic Energy Agency (IAEA), the United States Geological Survey (USGS), Florida Department of Environmental Protection, etc. Laboratory intercomparison studies such as these allow BRL 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 or at the beginning of an analytical batch. 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 or Ongoing Precision Recovery (OPR) samples. For most methods utilized at BRL, the CCV/OPR samples must be analyzed at the beginning and the end of an analytical batch and at a frequency of at least 10% throughout the analysis. For most analyses, Continuing Check Blanks (CCB) are analyzed after each CCV/OPR 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 BRL (as with Laboratory Fortified Blanks) or purchased from an outside source (as with CRMs) depending upon their availability. QC samples range from Laboratory Certified Standards (LCS) to matrix specific CRMs. 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 or Matrix Spike Duplicates)

Duplicate samples and/or matrix spike duplicates must be analyzed at a minimum frequency of 10% per analytical batch for all analytical methods employed at BRL. If a batch contains samples with different matrices, then duplicates should be analyzed for each matrix. 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 must be checked for the appropriate parameters prior to use. All reagent testing results are stored electronically on the server and the reagents are then labeled with the BRL assigned lot number, the date of testing and the measured concentrations of the analytes of interest.

Likewise, all standards 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 or more replicates is expressed as % Relative Percent Difference (% RPD) or % Relative Standard Deviation (or % 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

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 are constructed and used to monitor laboratory certified standards and SRMs/CRMs performances, 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.

It takes a minimum of 10 data points from at least 3 non-consecutive calendar days of analysis to "define" a control chart. The use of "real time" control charts is instrumental in indicating when an analysis is out-of-control. Out-of-control events may be indicated by the following occurrences.

1) QC sample result that exceeds the control limit even after reanalysis
2) Three out of four consecutive QC sample results that exceed the warning limit
3) Five out of six consecutive QC sample results exceed ± one standard deviation from the mean
4) Five consecutive QC sample results in decreasing or increasing order in the same calibration
5) Seven consecutive QC sample results are on the same side of the mean (Only applies to QC samples used to measure accuracy and not those used to measure precision)

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. 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 12.1). Outliers at BRL 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. This procedure can be reiterated using the next extreme value until no outliers remain.

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

The method detection limit (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 are determined by replicate analysis of a sample that is one to five times the estimated detection limit for the analyte of concern or up to 10 times the level for multi-element tests. The sample aliquots to be used may be from a native sample or a representative matrix that has sufficient analyte (present or spiked) to make the concentration one to five times the estimated MDL. If a sample low enough in the analyte of interest is not available, then method blanks may be spiked at the appropriate level for the MDL study. A minimum of seven sample aliquots (BRL routinely prepares and analyzes eight MDL samples) must be analyzed for the determination of the MDL. As long as seven sample aliquots remain, one MDL sample may be discarded, but only if there is a defensible reason for doing so (e.g. the auto sampler did not sample the cup, sample was not spiked, etc.). The reason for the abnormal result must be known and it must be shown that no other sample results could have been affected. The MDL is then calculated as the standard deviation of the replicate analysis multiplied by the "student's t" value for the number of replicates analyzed (3.143 for seven replicates; 2.998 for eight replicates). When evaluating a MDL, the sample aliquots must be carried through the entire method as per client samples. If, for a particular method of analysis, the concentration in the sample aliquots is below the MDL, then they cannot be used to calculate the MDL. In such a case, the MDL study is repeated with appropriately spiked MDL samples. More specific information on the MDL procedure and calculation are found in the EPA in "Definition and Procedure for the Determination of the Method Detection Limit - Revision 1.11", 40 CFR 136, Appendix B.

MDLs are determined for each method used at BRL prior to the analysis of client samples by that particular method. For research methods or methods that BRL is not accredited for use, MDLs may be estimated from the standard deviation of the method blanks. New MDL studies are performed

annually for aqueous and for solids methods. More frequent MDL studies may be required by accrediting bodies or at the discretion of BRL management. Additionally, the MDL must be verified any time major changes in a procedure are made. Major changes include changes in personnel, instrumentation, procedural changes to either the preparation or the analysis of samples, etc. The MDL study is intended to represent the capabilities of BRL and; therefore, should only be performed by experienced analysts.

As part of the MDL study, a Limit of Detection (LOD) validation check must be performed either during or immediately following the study (within one week and prior to analyzing any samples using the new MDL) on all instruments that might be used for the analysis. The LOD validation check consists of an appropriate sample prepared at 2 – 3 times the determined MDL for single analyte tests and 1 – 4 times the MDL for multi-analyte tests. The response produced during the analysis of the LOD validation check sample must be greater than 3 times above the instrument's noise level to be acceptable. If the LOD validation sample analysis fails to meet the acceptance criterion, then additional LOD validation checks must be performed at a higher level to set a higher MDL or the MDL study must be re-conducted. For some accreditations, such as DOD, the MDL verification check sample must be analyzed quarterly on each instrument used to analyze samples for each analyte for which we maintain accreditation. As a rule, the MDL verification sample is prepared and analyzed at levels of 1, 2, and 3 times the MDL when first establishing the MDL/LOD and then at 2 to 3 times the MDL quarterly. It is BRL policy to always set the LOD at a level of at least 2 times the MDL and no more than 3 times the MDL.

Brooks Rand Labs currently defines the different units for the MDL validation test as such.

- **Background Noise**: The average response of the method blanks during a single analytical run (sequence).
- **Instrument Noise**: The variation of the individual method blanks around the background noise during the sequence. To be as conservative as possible, this is measured for any single sequence as the absolute difference between the response of the lowest and the highest method blanks.
- **MDL Verification Sample Response**: This is measured as the instrument response for the analysis of the MDL verification sample corrected for the background noise.

For example, instrument response for total mercury analysis is measured in units of peak area (PA). If four method blanks were analyzed and gave responses of 59 PA, 98 PA, 81 PA, and 69 PA, then the background noise would be the average (76.75 PA or 77 PA) and the instrument noise would be 98 PA – 59 PA = 39 PA. For this example, the instrument response for the MDL validation sample would need to be greater than 77 PA + (3 x 39 PA) or greater than 194 PA in order for the MDL validation criteria to be met.

NOTE: The requirement of the MDL verification check may be met through the analysis of client samples prepared with a batch that happen to be approximately 2 times the determined MDL. That is, a specific MDL verification sample need not be prepared if another sample prepared with the batch meets the requirements of the MDL verification sample. However, the initial MDL verification immediately following a new MDL study must be performed prior to any sample analysis.

Some additional clarification is required between how NELAC and the DoD QSM use the MDL verification sample to establish the Limit of Detection (LOD). NELAC treats the LOD as

BRL treats the MDL; namely, the LOD is the detection limit (DL) for the method of interest and is verified by analyzing an LOD validation check sample. The DoD QSM defines the DL for the method in the same way that BRL defines the MDL. However, by the DoD QSM, the LOD is set by the level of the LOD validation check sample and it will always be greater than the DL. What this means is that for work covered by the NELAC standard and any work not requiring accreditation, BRL will report down to the MDL unless otherwise specified in the contract. Whereas, for work covered by the DoD QSM, BRL will report down to the level of the lowest LOD validation sample that meets the acceptance criteria, but not below the MDL.

All MDL studies are documented and the Quality Assurance Manager keeps the documentation on file. Documentation includes the date of the study, the name of the analyst conducting the study, the analytical method(s), the analyte of interest, preparation notes, and all raw data from analysis.

The method reporting limit (MRL) is based on the level of the low standard used in the instrument calibration and the volumes/weights used in the analysis of samples. The MRL cannot be less than the MDL and is typically 3 to 10 times the MDL. The Limit of Quantitation (LOQ) is the concentration of an analyte of interest where the relative confidence in the measured value is ± 30% at the 95% confidence level. LOQs are estimated as 9 to 12 times the standard deviation from the MDL determination. Whenever possible, BRL sets the MRL to be equivalent to the LOQ by adjusting the level of the low standard to be equal to the LOQ.

The validity of the LOQ/MRL is confirmed quarterly for each instrument used with the analysis of four laboratory fortified blank samples spiked at 1 – 2 times the level of the LOQ/MRL. Recovery of the LOQ/MRL LFB is judged against the established method acceptance criteria or client data quality objectives, whichever are more stringent, for precision and accuracy.

The instrument detection limit (IDL) is determined at initial set-up of the instrument and after any significant change (such as change in equipment or reagents used, carrier gases, gas pressures, etc.). Additionally, an IDL study may be used as part of an analyst's demonstration of capability as long as their ability to prepare samples is not being evaluated. A minimum of seven analytical spikes prepared at the level of the low calibration standard are analyzed and the IDL is then calculated as the standard deviation of the replicate analysis multiplied by the Student t value ($t_{.99}$) for the number of replicates analyzed (3.143 for seven replicates; 2.998 for eight replicates). The calculated IDL must be less than the MDL for all methods analyzed on the instrument. Additionally, a minimum of four analytical spikes prepared at approximately 10 times the MRL are analyzed. The recovery and standard deviation of these QCS samples must meet sample specific requirements of the method being performed.

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 analyzing or preparing samples. An MDL study is typically part of an analyst's IDOC, but is not required since it will also be affected by the preparation of the samples and isn't fully indicative of the analyst's ability to properly analyze sample preparations. If an MDL is not part of the IDOC, then an IDL study is performed where a minimum of seven analytical spikes prepared at the level of the low

calibration standard by the analyst at the instrument must be analyzed. These samples are analyzed as per an MDL study and the resulting detection limit must be less than the MDL for the analytical method. In addition, a minimum of four QCS samples are prepared as per the specific analytical method requirements (typically at a concentration that is 10 times the level of the MRL) and analyzed. The average recovery and the RSD yielded by the analysis of the QCS samples must meet the specific requirements of the analytical method being performed. A new IDOC must be performed anytime there is a change in instrument type, personnel, or test method (including changes to either the preparation or analysis of samples). Specific requirements for a passing IDOC are outlined in BRL SOP BR-1206 (MDL Studies, Validation, and Demonstration of Capability).

11.4.2 Continuing Demonstration of Capability (CDOC)

In addition to performing an IDOC prior to beginning analysis, each analyst must demonstrate that s/he is continuously capable of performing the analysis. This capability is judged annually by reviewing control charts for the methods performed by the analyst, looking at four consecutive batches performed by the analyst to ensure that all batch specific QA was met, or by having the analyst perform another IDOC study while analyzing the annual MDL study to demonstrate that the analyst is still capable of obtaining accurate and precise results. The full analyte list need not be reviewed for multi-analyte analyses to show continuing capability. At a minimum, 6 analytes will be reviewed. Specific requirements for a passing CDOC are outlined in BRL SOP BR-1206 (MDL Studies, Validation, and Demonstration of Capability). 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. Additionally, hard copies of the preparation logs and analytical benchsheets are filed by the QA Department and maintained for no less than seven years from when the analyst stops working at BRL. All IDOC and CDOC studies must be reviewed by the QA Department and the VP of Quality 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 VP of Quality's signature (or VP of Operations's signature in the absence of the VP of Quality) 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. A copy of this form is presented on the following page.

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.

Demonstration of Capability
Certification Statement

Date: Page __ of __

Laboratory Name: **Brooks Rand Labs**
Laboratory Address: **3958 6th Avenue NW**
Seattle, WA 98107

Analyst(s) Name(s):

Prep Tech Name (if applicable):

Matrix:

Method number, SOP#, Rev#, and Analyte or Class of Analytes or Measured Parameters:

Criterion Used (MDL Study, Consecutive Analysis, etc):

The undersigned CERTIFY that:

1) The analyst (and prep technician if applicable) identified above, using the cited test method(s), which is in use at this facility for the analyses of samples under the National Environmental Laboratory Accreditation Program, has met the requirements for the Demonstration of Capability.
2) The test method(s) was performed by the analyst(s)/prep tech(s) identified above.
3) A copy of the test method(s) and the laboratory-specific SOPs are available for all personnel on-site.
4) The data associated with the demonstration of capability are true, accurate, complete, and self-explanatory (1).
5) All raw data (including a copy of this certification form) necessary to reconstruct and validate these analyses have been retained at the facility, and that the associated information is well organized and available for review by authorized assessors.

_____	_____	_____
Quality Assurance Manager's Name	Signature	Date

This certification form must be completed annually or each time a demonstration of capability study is completed.

(1) True: Consistent with supporting data.
Accurate: Based on good laboratory practices consistent with sound scientific principles/practices.
Complete: Includes the results of all supporting performance testing.
Self-Explanatory: Data properly labeled and stored so that the results are clear and require no additional explanation.

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 BRL SOPs BR-1300, BR-1301, and BR-1302 for a detailed description of the data review procedure.

12.1 Analytical Integration

Analytical instrumentation signal output is integrated by BRL-developed integration software (i.e., Guru™) or by manufacturer specific software (i.e., Perkin Elmer ELAN or Chromera 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 electronics department at BRL maintains all documentation of integration software upgrades.

12.2 Data Reduction

The analyst is responsible for uploading all 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, 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. The following documentation must be present with every data package: preparation notes, SPFs, lab bench sheets, Analysis Sequence printout, and analyst's notes. All instrument printouts are stored electronically as PDF files and must have the analytical batch recorded on them and the sample ID for each instrument response.

12.3 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 first ensures that all data is present and that all previous sections on the SPF have been completed and signed-off. All final results are automatically calculated in the LIMS using formulas specific to the analytical method used.

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. Software used for data entry is verified to be working properly by the data entry verification protocols described in BRL SOPs BR-1300 and BR-1301. Any software problems or

failures are documented as correspondence between the IT Manager and the software manufacturer. The IT Manager keeps all such documentation/correspondence on file.

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.

Data validation is part of the review process whereby data are inspected and either accepted, rejected, or qualified based on a set of criteria. Evaluation parameters that can be used for validation include, but are not limited to:

- Calibration data
- Specific checks unique to each measurement
- Statistical tests

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 VP of Quality or delegate and, in extreme cases, the VP of Operations or BRL President have authority to change the reportability of data after final data review. All changes in the status of data (e.g. batched, prepared, analyzed, reviewed – primary, reviewed – final, reportable versus non-reportable, etc.) is 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 that 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 additional 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 BRL, 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 "B" 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 the DoD 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 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 NELAC standard and work performed under the DoD QSM and how this affects how results are reported by BRL.

It is BRL policy to always method blank correct results (with the exception of total solids and total suspended solids). In extreme cases where the client requires results that have not been method blank corrected, the criterion for acceptable method blanks is that the absolute value of the highest method blank concentration for any detectable (> MDL) method blank must be ≤ 10% of any detectable (> MDL) result. Any detectable result not ≥ 10 times the absolute value of the highest detectable method blank concentration is qualified "X" and narrated as being an estimate due to elevated method blanks with either high or low bias depending on whether the method blank concentration is a positive or negative value.

Another extreme case where it may not be appropriate to blank correct data is when there are highly variable blanks that do not meet the method blank criteria. In such cases, it is better to report results without blank correction as previously described. When reporting results without method blank correction, the reporting limits should not be adjusted. Instead, any results affected due to random or elevated method blanks are appropriately qualified using the "X" qualifier. Refer to Section 11.1.2.1 for further discussion on method blanks.

Data that is not method blank corrected is reviewed and qualified using the same acceptance criteria as blank corrected data with one caveat. Samples are not reprepared or reanalyzed if failing non-blank corrected QC would have passed if it had been blank corrected as per standard practice. In such cases, the data is qualified accordingly and reported unless specified otherwise in the client contract.

Generally, data are reported in a format generated by the BRL LIMS with a case narrative or a cover letter attached. 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 discuss each batch analyzed in the sequence. Final reports are submitted to all required parties (project dependent). A copy of each report stored electronically as a PDF file for BRL'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 BRL) 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 Brooks Rand Labs 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 BRL qualifiers. In such instances, the project specific

qualifier definitions would over-ride the BRL 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:

Disclosing information about client results or contracts to any party outside of Brooks Rand Labs without prior permission from the client and Brooks Rand Labs and without following all reporting policies stated in the Brooks Rand Labs 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 Brooks Rand Labs has a legal contract to perform work, unless BRL 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 Brooks Rand Labs prior to October 15, 2006, hard copies of all data and documentation will be kept on file for a minimum of five years unless contract specific requirements call for longer storage. Data and documentation to be stored include: SPFs, preparation notes, lab bench sheets, lab notebooks used in reduction, instrument printouts, and any results spreadsheets (i.e., Excel® spreadsheets used to calculate values). The VP of Quality is responsible for maintaining files of all batch specific hard data. The last 6 months worth of batch specific hard data is stored in cabinets located in the main office area. The previous year's batch specific hard data is stored in the overhead space of the conference room. Any earlier batch specific hard data is stored in a secure facility off BRL premises. The Project Manager is responsible for maintaining all client specific files. Most client files are saved electronically and not in hardcopy. 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. All data generated following October 15, 2006 are scanned and stored electronically for a minimum of ten 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 five years.

Note: The following procedure is done for clients with data prior to October 15, 2006, that require data to be maintained for more than 5 years to ensure that data is not inadvertently lost. Hardcopy data prior to October 15, 2006, is pulled from storage and then scanned to the server and stored as electronic data for as long as required by the project.

Electronic summaries of data will be kept for a minimum of 10 years. All computer files are stored both on computer hard drive and on backup disks. Computer files of client reports are organized by sample tracking number, batch spreadsheets are organized by batch number, and all project information is organized by project numbers. All client reports are scanned and stored electronically. These reports contain copies of the original SRL, as well as any information provided by the client including chain-of-custody forms, analysis request forms, airbills (full reports only), etc. Since October 15, 2006, all sequence specific data has also been scanned and stored electronically. The original hardcopy batch data is stored for at least 6 months in the file cabinets in the main office before being shredded. All hard copy data previous to October 15, 2006 will be stored for a minimum of five years from the date of reporting before being shredded. All employees at BRL are authorized to access electronic information. No levels of accessibility for employees exist. The IT Manager monitors the upkeep of computer files.

All hard copies of any documents that could be traced directly to a client are destroyed by shredding prior to disposal.

13.0 Document Control Policies

13.1 SOPs, Manuals, Handbooks, and Plans

All documents important to the internal operations of BRL go through formal procedures as to their writing, approval, implementation, retirement, and sharing.

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

Once it has been determined that a new policy or procedure is required at BRL, the most appropriate employee(s) (i.e., whoever has the most knowledge or experience in the given area) is/are delegated to write a document detailing the policy or procedure. Once the document has been written, it must pass up through a chain of approval specific to the type of document being written.

All SOPs begin with the appropriate person writing the procedure. The Group Leader (if applicable), the VP of Quality, and, finally, the VP of Operations then must approve the SOP (in that order). The CQAP must be approved in order by the VP of Quality and the VP of Operations. BRL Handbooks and Plans follow their own specific chain-of-approval processes, with final approval coming from the VP of Operations or BRL President.

If an error is discovered during any portion of the approval process, the person who found the error makes a note of it and sends the document back to the original writer. It is the writer's responsibility to address the error and then reinitiate the approval process from the very beginning.

Upon approval, each person in the chain of approval must sign and date the document. Only upon final approval is the document considered to be in force and all procedures within the document from that date forth are enforced until the document is retired (see section 13.1.3).

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

All BRL documents are reviewed annually. If no changes in the procedure are required, the reviewer signs and dates the document as being reviewed. If changes are required, the appropriate employee is designated to make the required revisions. The new revision of the document must then go through the same chain of approval it went through for its initial writing. Upon final approval, the new revision is considered in force, and the old revision is retired. Refer to SOP BR-1400 for specific procedures to follow when revising an SOP.

13.1.3 Retirement of SOPs, Manuals, Handbooks, and Plans

When a BRL document is retired, the original is clearly labeled "outdated" 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 five years.

13.1.4 Proprietary Information

Many of the analytical methods used at BRL 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." Whenever possible, "client ready" SOPs, where all proprietary information has been removed, are given to clients instead of full SOPs.

13.1.5 Uncontrolled Documents

Uncontrolled documents are defined as any document (CQAP, SOP, "cheat sheet", 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 VP of Quality. 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 following folder: Y:\SOP & other DOCs. All documents in this folder are PDF versions with signed and dated cover pages. If "cheat sheets" or isolated pages from SOPs would be of value in the lab, then these must be approved by the VP of Quality who then signs and dates the pages and combines them into a controlled logbook for the laboratory.

13.1.6 Master List of Controlled Documents

The Quality Assurance Manager is responsible for maintaining a master list for the location of all controlled documents. The list must contain the following information:

- Title of controlled document
- Revision number
- Location stored or name person in possession of
- Date printed or sent electronically

13.2 Client Records

All client reports, records of results, and correspondences are maintained by BRL for a period of no less than five years. All project information is electronically stored on the server for a minimum of 10 years. All "Active Client" specific files are maintained by the Project Manager. All client data generated following October 15, 2006 is scanned and stored electronically for a minimum of ten years.

In the event that BRL should go out of business, it is BRL'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 BRL'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 BRL premises without the client's written consent.

13.3 Employee Records

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

14.0 Information Systems

14.1 Hardware

A local area network (LAN) connects staff computers and printers for local access, as well as providing external email, faxes, and Internet access. The server computer is a Dell PowerEdge 1900 running Microsoft® Small Business Server 2003 R2. The server is configured as a primary domain controller and print server for networked printers. It is equipped with three 146 gigabyte hard disk drives and employs RAID 5. The redundant array will prevent data loss in the event of a single hard drive failure.

A dedicated server runs the LIMS application and is accessible via the LAN. The hardware is a Hewlett-Packard ProLiant DL180 G5 Server. It has six 250 GB hard disk drives. The data resides on a three disk RAID 5 array. The OS resides on a two disk RAID 1 array. The sixth HDD is a spare hot swappable disk. The OS is Microsoft® Windows Server 2008. The LIMS program runs on SQL Server 2005 application.

A terminal server is configured to provide remote access for offsite employees. The server is a Dell PowerEdge 700 running Microsoft® Small Business Server 2003 R2 and Terminal Services.

14.2 System Backup

Backup software, Retrospect version 7.0, provides scheduling, automation and monitoring of backup for both server and workstation files. The software is run on a networked spare workstation. All data files located on the server are backed up daily. These include the LIMS file (Microsoft® SQL Server), instrument data, and client-related files. Other selected files on the workstation are also backed up weekly.

Three external hard drives are used to perform the backups. The hard drive connected to the backup workstation remains so for three weeks, after which it is removed to an offsite location and the external hard drive with the oldest backed up data is then connected to the backup PC.

14.3 Security

A Netscreen-5GT router/firewall protects the LAN from the public internet. Workstation access is available to all authorized employees via domain logon. Shared data is available throughout the local network. Specific directories or files may be protected from access using Group Policy security settings if the data owner considers it necessary. Data loss is safeguarded through redundancy. Redundancy is accomplished via backups as mentioned and secure storage of data in hard copy.

All computer accounts are password protected so that unauthorized access is not allowed. Individuals are required to logoff of computers when they leave the workstation.

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 VP of Quality 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 Brooks Rand Labs SOP BR-1204). The VP of Quality is responsible for evaluating the situation and determining the appropriate corrective action. The VP of Quality has the authority to stop work whenever a nonconformance issue may threaten the quality of data produced by Brooks Rand Labs. Corrective action steps include, but are not limited to:

- Problem identification
- Investigation to determine the 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 VP of Quality who is responsible for ensuring that the action is documented. The VP of Quality 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. Examples of corrective action include, but are not limited to:

- Amending forms
- Reanalyzing samples if holding times permit
- Checking instrumentation to make sure that it is operating properly
- Recalibrating with fresh standards
- Replacing suspect reagents
- Examining calculations
- Additional training in sample preparation and analysis

- Evaluating and amending procedures
- Accepting the data and acknowledging the level of uncertainty or inaccuracy by flagging the data and providing an explanation for the qualification

15.2 Client Communication and Complaints

Brooks Rand 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 Brooks Rand Labs 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 BRL, including analytical services, technical services, or quality assurance.

Once a complaint has been recorded, the BRL manager who is most responsible for the service to which the complaint is directed shall handle the matter with the client. If necessary, the BRL 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 BRL management (for issues related to new R&D, clients are typically contacted within a month of finalizing the R&D report). 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.

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 Brooks Rand Labs.

15.3 External Audits

Corrective action may also be initiated by external audits by regulatory agencies or clients. Brooks Rand Labs 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.

16.0 Performance and System Audits

16.1 System Audits

16.1.1 Internal Systems Audits

BRL conducts specific function audits on an annual basis, with a different segment of the laboratory being thoroughly audited each quarter of the year. This audit process is used to ensure that:

- Approved procedures are in place and used
- Sample custody is properly maintained and documented
- Analytical methods are performed properly and documented
- Specific equipment is available, calibrated and in proper working order
- Analysts are properly trained and the training is documented
- Record keeping procedures are being followed and appropriate documentation is maintained

Additionally, laboratory walk-through audits are performed monthly throughout the laboratory. The laboratory is divided into four sections (Sample Control Lab, HG Lab, Trace Metals Lab, and Sample Preparation Lab). Each lab is audited separately. Laboratory walk-through audits are not as thorough the quarterly audits, 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.

An annual Managerial Review of the quality system is prepared, typically during the first quarter of the year (January – March). This report consists of a review of the monthly and quarterly audits over the past year, including the documentation, findings, reporting, corrective action, and follow-up. If any findings are still considered open from the past year, then the report should include a list of out-standing findings, a detailed explanation as to how those findings are being addressed, and a form on which to indicate when the findings have been resolved. 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 Brooks Rand Labs.

Various intercalibration exercises with other laboratories also serve as a performance audit of the laboratory analyses.

16.1.2 External Systems Audits

BRL has occasional audits from various clients and accrediting agencies. The principal organizations that conduct audits of BRL's facilities and operations are the Washington State Department of Ecology, the Florida State Department of Environmental Protection as part of the NELAP accreditation, and ACLASS as part of Department of Defense accreditation. BRL views external audits as an excellent tool for evaluating our quality and for finding areas for improvement. BRL always welcomes any client (current or potential) or government agency to conduct on-site audits.

16.2 Performance Audits

Internal Performance Audits 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 evaluation samples, and/or blind spiked samples. BRL frequently participates in assisting agencies to certify reference materials for use as blind interlaboratory samples. Any of the Analytical Technicians may analyze these performance audit samples. The Project Manager and VP of Operations are responsible for overseeing BRL's participation in each study, and all associated documentation, reporting, and record keeping.

External Performance Evaluations are as follows:

Agency	Study Title	Frequency
ERA	Blind PE samples*	Semi-Annually**
RTC	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 Rand Labs management conducts an annual review of the quality systems to ensure that they are still effective. This review typically takes place during the first quarter of the year. 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.

APPENDIX A – Common Abbreviations

AA – Atomic Absorption
ASD – Analytical Services Department
BLK – Method Blank
BRL – Brooks Rand Labs
BS – Blank Spike
CCV – Continuing Calibration Verification
CDOC – Continuing Demonstration of Capability
COC – Chain of Custody
CQAP – Comprehensive Quality Assurance Plan
CRM – Certified Reference Material
CVAFS – Cold Vapor Atomic Fluorescence Spectrophotometry
DoD QSM – Department of Defense Quality Systems Manual
DOE QSAS – Department of Energy Quality Systems for Analytical Services
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
ICP-MS – Inductively Coupled Plasma – Mass Spectrometry
ICV – Initial Calibration Verification
IMD – Instrument manufacturing Department
IT – Information Technology
LIMS – Laboratory Information Management System
LOD – Limit of Detection
LOQ – Limit of Quantification

MB – Method Blank
MD – Method Duplicate
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
PQL – Practical Quantitation Limit
PM – Project Manager
QA – Quality Assurance
QC – Quality Control
RPD – Relative Percent Difference
RSD – Relative Standard Deviation
SPF – Sample Processing Form
SOP – Standard Operating Procedure
SOW – Statement of Work
SRM – Standard Reference Material
TNI – The NELAC Institute
VP – Vice President

APPENDIX B - Standard Operating Procedures

Brooks Rand Labs - Standard Operating Procedures

SOP #	Title
	Analysis (BR-0001 through BR-0099)
BR-0002	BRL 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 Spectrophotometry (CVAFS)
BR-0003	Determination of Total and "Acid-Labile" Mercury in Aqueous Samples by Cold Vapor Atomic Fluorescence Spectrophotometry (CVAFS)
BR-0005	Total Volatile Mercury in Water by Purge and Trap, and Cold Vapor Atomic Fluorescence Spectrometry
BR-0006	Procedure for EPA Method 1631, Revision E: Mercury in Water by Oxidation, Purge and Trap, and Cold Vapor Atomic Fluorescence Spectrometry
BR-0007	BRL Procedure for Determination of Vapor Phase Total Mercury from Stationary Sources Using Dry Sorbent Trap Sampling and Analysis by Cold Vapor Atomic Fluorescence Spectrometry (CVAFS) – Modification of EPA Method 324
BR-0011	Determination of Methyl Mercury by Aqueous Phase Ethylation, Trap Pre-Collection, Isothermal GC Separation, and CVAFS Detection: BRL Procedure for EPA Method 1630 (Waters) and EPA Method 1630, Modified (Solids)
BR-0013	Five-Step Selective Sequential Extraction Procedure (SEP) to Quantify Mercury Fractions in Sediments, Soils and Mine Tailings
BR-0015	BRL Procedure for Reactive Mercury in Tissue, Sludge, Sediment and Soil by Cold Vapor Atomic Fluorescence Spectrophotometry (CVAFS) Modified from EPA 1631
BR-0020	Determination of Selenium (Se) and Arsenic (As) in Environmental Samples by Hydride Generation-Atomic Absorption with Cryogenic Trap Preconcentration
BR-0021	BRL 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

Brooks Rand Labs - Standard Operating Procedures

SOP #	Title
	Analysis (BR-0001 through BR-0099 continued)
BR-0060	Determination of Trace Elements by Inductively Coupled Plasma – Mass Spectrometry (ICP-MS) using a Perkin-Elmer ELAN DRC II
BR-0061	Trace Element Speciation by High Performance Liquid Chromatography – Inductively Coupled Plasma – Dynamic Reaction Cell – Mass Spectrometry using a Perkin-Elmer ELAN DRC II
BR-0062	Determination of Ag, Al, As, Cd, Cu, Cr, Ni, Sb, Se, V and Zn in Flue Gas Desulphurization Waste Waters by Inductively Coupled Plasma – Dynamic Reaction Cell – Mass Spectrometry using a Perkin Elmer ELAN DRC II
BR-0063	Determination of Trace Elements in Seawaters and Low Level Waters by Online Column Chelation Preconcentration – Inductively Coupled Plasma – Mass Spectrometry using a Perkin-Elmer ELAN DRC II
BR-0064	Chromium Speciation by High Performance Liquid Chromatography – Inductively Coupled Plasma – Dynamic Reaction Cell – Mass Spectrometry using a Perkin-Elmer ELAN DRC II
BR-0065	Aqueous Sample Digestion by Closed-Vessel Oven Heating for Total and Total Recoverable Metals
BR-0066	Reductive Precipitation of Total Recoverable and Dissolved Metals from Brackish and Seawater Samples
BR-0067	Reverse Aqua Regia Oven Bomb Digestion for Total Recoverable Metals in Sediments and Soils
BR-0068	"Total" Metals Digestion in Sediments, Soils, Coal, and Fly Ash
BR-0069	Extraction Using Co-APDC for Nickel, Copper, Silver, Cadmium, and Lead in Water
BR-0070	Total Metals Digestion for Biota Matrices
BR-0073	Microwave Digestion for Total Recoverable Metals Digestion in Food Matrices
BR-0080	Determination of Iron Speciation in Water Samples by Colorimetric Detection
BR-0085	Determination of Hexavalent Chromium (Cr^{6+}) in Sediment and Aqueous Samples
	Sample Preparation (BR-0100's)
BR-0104	Metals "Free" Filtration
BR-0106	Sample Homogenization
BR-0107	Filtration for Collection of Particulate from Water Samples

Brooks Rand Labs - Standard Operating Procedures

SOP #	Title
	Sample Collection (BR-0200's)
BR-0200	Brooks Rand Labs Procedure for Modified EPA Method 1669: Collection and Preservation of Samples for Trace Level Analysis
	Sample Receipt and Storage (BR-0300's)
BR-0300	Receipt of Samples
BR-0301	Sample Custody Maintenance and Tracking
BR-0302	Sample and Client Identification
BR-0303	Sample Storage and Disposal
BR-0304	Sample Processing
BR-0306	Purchase, Receipt, and Storage of Consumable Materials Used for the Technical Operations of the Laboratory
BR-0307	Review of Requests for Work and Contracts
	Decontamination (BR-0400's)
BR-0400	Decontamination of Sample Preparation Equipment
BR-0401	Decontamination of Silicon and Teflon® Tubing and Filter Units for Sample Collection
BR-0402	Metals Decontamination of Glassware
BR-0404	Preventing Trace Metals Contamination of Samples
	Reagents and Standards (BR-0500's)
BR-0500	Documentation of Reagents and Standards
	Security (BR-1000's)
BR-1000	Security of Laboratory and Samples
	Training (BR-1100's)
BR-1100	Training of Laboratory Personnel
BR-1101	Prevention and Detection of Improper, Unethical, and Illegal Actions – Brooks Rand Labs' Data Integrity Plan

Brooks Rand Labs - Standard Operating Procedures

SOP #	Title
	Quality Control (BR-1200's)
BR-1200	Maintaining Instrument and Equipment Records and Logbooks
BR-1201	Internal Laboratory Audits
BR-1202	Evaluating Precision and Accuracy and Estimating Uncertainty in Results
BR-1203	Identifying Systematic Errors
BR-1204	Incident Report and Resolution
BR-1205	Preventative Maintenance
BR-1206	MDL Studies, Validation, and Demonstration of Capability
	Data Validation and Reporting (BR-1300's)
BR-1300	Primary Data Review
BR-1301	Final Data Review
BR-1302	Data Flow and Handling
BR-1303	Reporting
BR-1304	Element for Analysts
	Documents (BR-1400's)
BR-1400	Writing, Reviewing and Revising Standard Operating Procedures (SOPs)
BR-1401	Records of Client Sample Results
BR-1402	Records of QC Results
BR-1403	Document Control for Standard Operating Procedures (SOPs) and Comprehensive Quality Assurance Plan (CQAP)
BR-1404	QA Admin: Test Code Creation and Maintenance

Brooks Rand Labs - Standard Operating Procedures

SOP #	Title
	General Determinations (BR-1500's)
BR-1500	Total Suspended Solids in Water
BR-1501	Dry Weight Determination
BR-1502	Determination of Hardness in Water by Calculation
BR-1503	Brooks Rand Labs Procedure for SM 2540G: Total, Fixed and Volatile Solids in Solid and Semisolid Samples – Total Volatile Solids (TVS) Ignited at 550 ºC
BR-1504	Measurement of the pH of Solid and Aqueous Samples by Suspension, Centrifugal Separation, and Quantification Using an Electrode-Based pH Meter
	Radioactive Materials (BR-1600's)
BR-1601	Sample handling, Storage, and Disposal under Brooks Rand Labs' Radioactive Materials License