Quality Assurance Plan
Hg Analytical Laboratory

National Atmospheric Deposition Program

Eurofins Frontier Global Sciences
11720 North Creek Pkwy N
Bothell, WA 98019
P: 425-686-1996 § F: 425-686-3096
http://eurofinsus.com/environment-testing/laboratories/eurofins-frontier-global-sciences

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Prepared by:
Dave Wunderlich

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Mark Rhodes
David Gay

General Mission of the MDN:

The National Atmospheric Deposition Program – Mercury Deposition Network (NADP/MDN) is a long-term monitoring program in support of research on the effects of atmospheric Mercury deposition. Our mission is to monitor our chemical climate for total mercury and methyl mercury in rain and snow precipitation through a North America network of standardized monitoring stations. This enables the measurement of temporal and geographic trends.

Quality Assurance Plan Approval Form

The Quality Assurance Plan has been reviewed and approved by the following authorized National Atmospheric Deposition Program and Eurofins Frontier Global Sciences signatories for quality assurance documents.

Signature _____ Date _____
Michael Flournoy, MDN HAL Business Unit Manager,
Eurofins Frontier Global Sciences

Signature _____ Date _____
Robert Brunette, MDN HAL Director,
Eurofins Frontier Global Sciences

Signature _____ Date _____
Patrick Garcia-Strickland, MDN Laboratory Manager/Science Advisor,
Eurofins Frontier Global Sciences

Signature _____ Date _____
Douglas Disney, MDN Site Liaison/Project Manager
Eurofins Frontier Global Sciences

Signature _____ Date _____
Ryan Nelson, MDN Laboratory Group Leader,
Eurofins Frontier Global Sciences

Signature _____ Date _____
Dave Wunderlich, MDN Quality Assurance Officer,
Eurofins Frontier Global Sciences

Signature _____ Date _____
David Gay, NADP Program Coordinator,
National Atmospheric Deposition Program

Signature _____ Date _____
Mark Rhodes, NADP Quality Assurance Manager,
National Atmospheric Deposition Program

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Appendixes

Figures and Tables

CHANGES FROM PREVIOUS REVISION

- Added contact and control information to cover page
- Added titles of MDN Business Unit Manager, Laboratory Manager/Science Advisor and MDN Lab Group Leader to the Approval Form and combined MDN Project Manager and Site Liaison into a single approval line.
- Added this revision log
- Updated list of abbreviations and acronyms
- Declared ownership change and updated number of sites in section A1
- Updated organization chart in section A2
- Added responsibilities for Business Unit Manager, Science Advisor and MDN Lab Group Leader; updated responsibilities for the HAL Director, Laboratory Manager, Project Manager, Site Liaison and QA Officer in Section A2
- Updated training program components in sections A3.1 and A3.2
- Updated Quality Assurance Policy Statement in Section A4.1
- Updated shipping services and website address in section A4.3
- Updated facility security measures in section A4.3.1
- Updated capital equipment in Section A5
- Added frequency of air monitoring in section A6.1
- Weekly monitoring of reagent water was changed to monthly in section A6.2
- Changed title of section B1.4 to Calibration and Verification of Support Equipment
- Added Lumex analyzer and in-line conductivity meter to support equipment in Table 1.
- Corrected concentration of the ongoing calibration standard for methyl mercury in section B2.4.1
- Adjusted aliquot volumes for the Tekran 2600 in Section B2.7.1
- Added QC summary table following section B2.9.2
- Specified the inception of total low level mercury PT testing and added more guidance on the handling of PT samples in section B5
- Updated server backup strategy and added backup testing in section C1.1
- Moved section C4 (Network Field Supplies) from Data Management section to Lab Operation and section B7.
- Updated MDN-specific SOP list in Table 3
- Updated EFGS supplemental SOP list in Table 4

Definitions of Abbreviations and Acronyms

APDC	Ammonium PyrrolidineDithioCarbamate
AS/ASD	Analytical Spike/ Analytical Spike Duplicate
ASTM	American Society for Testing and Materials
BrCl	Bromine Monochloride
BS/BSD	Blank Spike/ Blank Spike Duplicate
BU	Business Unit
CCB	Continued Calibration Blank
CCV	Continued Calibration Verification
CFR	Code of Federal Regulations
CHP	Chemical Hygiene Plan
CRM	Certified Reference Material
DQO	Data Quality Objectives
EPA	Environmental Protection Agency
EFGS	Eurofins Frontier Global Sciences
HAL	Mercury (Hg) Analytical Laboratory
IAEA	International Atomic Energy Agency
ICB	Initial Calibration Blank
ICV	Initial Calibration Verification
IDOC	Initial Demonstration of Competency
IR	Incident Report
LCS/LCSD	Laboratory Control Sample/Laboratory Control Sample Duplicate
LIMS	Laboratory Information Management System
LOD	Limit of Detection
LOQ	Limit of Quantitation
MD	Matrix Duplicate
MDL	Method Detection Limit
MDN	Mercury Deposition Network
MMHg	Methyl Mercury
MOF	Mercury Observer Form
MRL	Method Reporting Limit
MS/MSD	Matrix Spike/ Matrix Spike Duplicate
NADP	National Atmospheric Deposition Program
NELAC	National Environmental Laboratory Accreditation Conference
NELAP	National Environmental Laboratory Accreditation Program
NIST	National Institute of Standards and Technology
NRCC	National Research Council Canada
PO	Program Office
PQL	Practical Quantitation Limit
PT	Proficiency Test
QA	Quality Assurance
QC	Quality Control
QAP	Quality Assurance Plan
RO	Reverse Osmosis
RPD	Relative Percent Difference
SOP	Standard Operating Procedure

Definitions of Abbreviations and Acronyms Continued

THg	Total Mercury (Hg)
TNI	The NELAC Institute
USGS	United States Geological Survey

Section A:
Program Overview

A1 Introduction

Since January 1996, Eurofins Frontier Global Sciences Inc. (EFGS), previously named Frontier Global Sciences Inc. and Frontier GeoSciences Inc., (FGS) has served as the Mercury Analytical Laboratory (HAL) and the Network Operations Center, for the Mercury Deposition Network (MDN). The MDN, coordinated through the National Atmospheric Deposition Program (NADP), was designed with the primary objective of quantifying wet deposition of mercury in North America to determine long-term geographic and temporal distributions. The Mercury Deposition Network has grown to incorporate and consistently operate over 100 sites in North America by the end of 2015. For the current site map, go to http://nadp.isws.illinois.edu/maps.

EFGS, serving as the HAL, provides site technical support, glassware and field supply cleaning, sample processing, sample analysis, and data validation services for precipitation samples collected at the NADP/MDN monitoring sites. All these processes must follow documented quality assurance and quality control procedures. The Quality Assurance Plan (QAP) describes these procedures and indicates how they are to be monitored and quantified. The QAP is reviewed annually and updated as necessary.

A2 Organization and Responsibilities

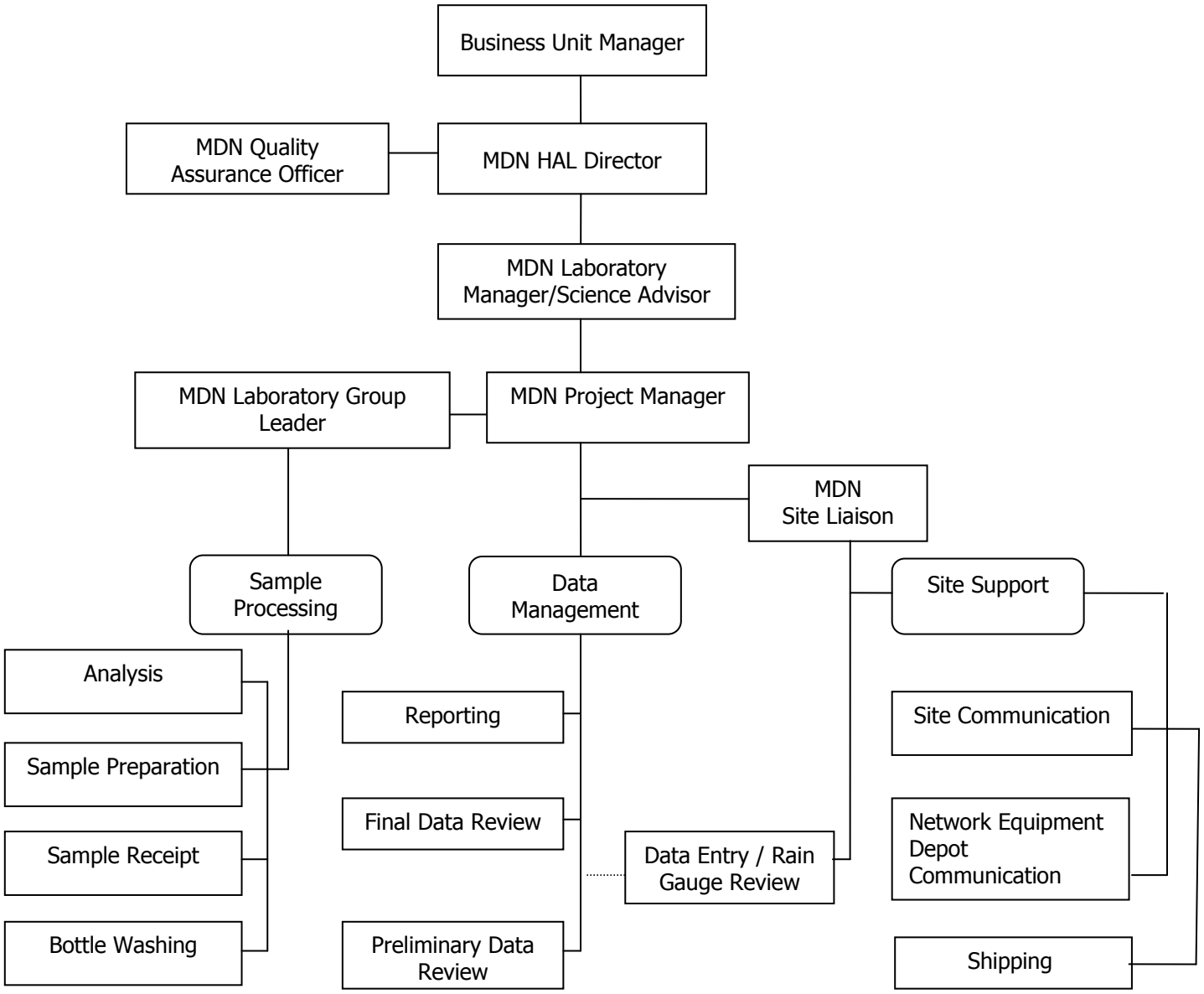


Figure 1: Mercury Analytical Laboratory Organizational Chart

A2.1 Business Unit (BU) Manager

The BU Manager oversees all business and operational strategies and is responsible for the laboratory's performance.

- Improves efficiency of laboratory operations and logistics. Streamlines the operational processes and makes good use of IT so as to achieve high efficiency. Supports the development, implementation and optimal use of the Company's LIMS and ensures its efficient usage at all levels of the laboratory to improve productivity
- Hires, motivates, develops and retains excellent Managers and employees
- Improves the technical and scientific aspects of the business by ensuring the appropriate development and application of new techniques and business processes
- Ensures that HAL/EFGS has appropriate systems, work ethics and policies in place to guarantee high quality of service to clients
- Maintains Customer satisfaction, including that of internal customers
- Guarantees and confirms that all relevant legal and Health & Safety requirements are being abided to, that the accreditations are being maintained and regularly expanded and that all employees act according to Eurofins values
- Leads according to Eurofins Leadership Charter and act as a role model for leadership and staff
- May assist project manager or their backup with reviewing and reporting MDN analytical and field data

A2.2 MDN HAL Director

The HAL Director oversees HAL's involvement in the NADP. The Director serves as the HAL contact for the multiple agencies currently sponsoring the MDN. The Director provides oversight, guidance and direction to all HAL Management staff to help ensure that HAL is meeting the NADP Program Requirement needs. The MDN HAL Director activities include the following:

- To help if necessary with site selection and equipment installation of MDN Sites
- If necessary, acts as a back up to the MDN HAL Site Liaison for equipment troubleshooting
- Presents HAL reports to committees at the semiannual NADP meetings
- Helps provide outreach to promote MDN site growth through presentations and support others using MDN data for research
- Helps support new initiatives related to the MDN program either through the NADP or with MDN sponsors

A2.3 MDN Laboratory Manager

The MDN Laboratory Manager oversees HAL's day-to-day operations as follows:

- The Laboratory Manager's goal is to produce data that meets the Data Quality Objectives (DQO), while maintaining required supplies and data turn-around-times, cost effectiveness, sustainable laboratory practices, employee job satisfaction, and supportive customer relations.
- The Laboratory Manager has ultimate responsibility for the quality of all analytical laboratory data, reports, practices, and safety.
- The Laboratory Manager ensures that data meet all quality control requirements, and takes appropriate and documented corrective action if it does not.
- It is the Laboratory Manager's responsibility to ensure that all staff members understand and adhere to this QAP and relevant Standard Operating Procedures (SOP).
- The Laboratory Manager maintains proficiency in laboratory training, analysis and report writing and ensures all MDN staff are trained and proficient at their duties.
- Serves as a backup to the MDN Project Manager
- Participates in the NADP committees at the spring and fall meetings

A2.4 MDN Science Advisor

- Guides/oversees research initiatives for new mercury/trace metals monitoring efforts
- Provides science oversight to the MDN program
- Looks to develop new science initiatives to support the NADP MDN program and end user needs

A2.5 MDN Project Manager

The MDN Project Manager supports the HAL by reviewing and transmitting data and providing backup assistance with glass cleaning and preparation, shipping and receiving and sample preservation and preparation.

- Reviews and reports MDN analytical and field data to site operators and to the NADP Program Office
- Reviews data when necessary
- Ensures that the MDN area is maintained and kept clean
- Demonstrates proactive commitment and adherence to industry and company safety regulations and procedures through scientific literature, user groups and seminars
- With the MDN Lab Group Leader, reviews and revises the SOPs used by the group according to the schedule
- Prepares and updates operation manuals and QA documents
- Conducts data analysis and review from sites for completeness and accuracy, and communicates with site operators and data validation staff to ensure high quality data records are obtained

- With the MDN Site Liaison, plans and performs training sessions for MDN Site Operators
- May attend the semiannual NADP meetings and present reports

A2.6 MDN Site Liaison

The Site Liaison is the primary contact with the monitoring sites and supports tracking shipments, troubleshooting equipment, reading rain gauge charts, assisting clients with requests, and providing backup assistance with glass cleaning and preparation, shipping and receiving, sample preparation, digestion and preservation.

- Proactively tracks and manages all shipments to MDN sites, ensuring that they arrive promptly and to the correct location
- Assists clients with troubleshooting MDN equipment when issues arise
- Logs all MDN communications and documents all conversations with clients
- Prepares and updates communications related to site operations including support materials for operational protocols, and equipment troubleshooting and repair
- Troubleshoots precipitation collection and rain gauge equipment
- Reads, interprets and monitors rain gauge charts and electronic records
- With the MDN Project Manager, plans and performs training sessions for MDN Site Operators

A2.7 MDN Laboratory Group Leader

The MDN Lab Group Leader supports the HAL by scheduling and supervising the laboratory staff that are responsible for glass cleaning and preparation, shipping and receiving, sample preservation, preparation and analysis and data review.

- Together with Quality Assurance Officer, ensures training protocols are up-to-date and properly documented
- With the MDN Project Manager, reviews and revises the SOPs used by the group according to the schedule
- Trains or organizes the technical training of MDN Lab employees
- Ensures that all group members have up-to-date documentation of Demonstration of Capability
- Organizes the day to day activities of the MDN Lab
- Establishes sample preparation and analysis priorities
- Reviews data generated by the lab against method and program requirements
- Schedules, compiles and reviews method detection limit studies
- Conducts monthly monitoring for mercury contamination in laboratory areas using a direct measurement, real- time, low level atmospheric Hg detector
- Ensures that the HAL meets data reporting deadlines
- Serves as a backup to the MDN Site Liaison
- May attend the semiannual NADP meetings and present reports

A2.8 MDN Quality Assurance Officer

The Quality Assurance (QA) Officer's goal is to improve the laboratory's quality assurance/quality control (QA/QC) processes in a manner consistent with the MDN mission statement. This requires support from the Management and laboratory staff. The QA Officer ensures that all laboratory decisions are considered from a QA standpoint. Specifically, the QA Officer has the following responsibilities relating to laboratory QA systems:

- Coordinates and documents non-technical training procedures for laboratory staff, including QA orientations and ethics training
- Notifies the lab when problems occur from facilities testing programs (e.g., reagent water, bottles, equipment, and air)
- Investigates rejected datasets
- Investigates corrective actions and ensures their proper implementation
- Manages proficiency tests and inter-laboratory comparison studies
- Maintains, reviews, and updates controlled documents including the QAP and SOPs used by HAL
- Provides staff members with QA information as needed
- Performs other relevant tasks associated with EFGS's QA requirements
- Generates the annual Quality Assurance Report for MDN
- Reviews and revises the MDN Quality Assurance Plan according to schedule
- Member of the NADP Quality Assurance Advisory Group
- Performs annual internal audit of the HAL

The QA Officer works closely with the HAL Laboratory Manager, MDN Project Manager/Site Liaison and the MDN Lab Group Leader to ensure that all staff members adhere to this QAP and relevant SOPs, and that scientific excellence remains EFGS's top priority.

A3 Training Program

All personnel are trained appropriately in their assigned tasks before they contribute to functions that can affect data quality. Staff members are trained in new skills or methods through a mentorship process. It is management's responsibility to ensure personnel are trained. Training records are used to document management's approval of personnel competency.

A3.1 Training of New Staff

New staff members are given the following training:

- Initial Quality Assurance training within the first two weeks of employment
- Ethics training. The employee must view an ethics slide presentation and sign an ethics agreement. This shall be done within the first two weeks of employment.
- All new full time employees shall read the general Eurofins Frontier Global Sciences Quality Manual, within the first week of employment.
- Employees providing support related to the MDN Hg Analytical Laboratory shall also read this document, the Quality Assurance Plan for the Mercury Analytical Laboratory, within the first month of employment.
- New staff members receive a list of the SOPs they need to read. The list contains

general SOPs that all employees need to read and also SOPs specific to their work area. The list includes a statement that says the employee has been given an opportunity to ask questions about the procedures, that all of the responses to their questions have been complete, that they agree to follow the procedures and that they agree to comply with all future changes in these procedures. The employee signs the list and returns it to the QA office.

- The Chemical Hygiene Plan (CHP). The employee must read and correctly answer the questions in the CHP before doing any laboratory work. Incorrect answers will be discussed with the new employee.
- The employee shall be given an initial safety tour on the first day of employment.
- An overview of the Employee Handbook will be given by the Human Resource Manager.
- Review of Eurofins Quality Policy Statement, followed by signing the Statement
- Review of Eurofins Confidentiality Agreement, followed by signing the Agreement.

All initial training must be documented and signed. Signed records are maintained in the employee training file.

New employees or staff members, who are learning a new skill or method, are assigned to their immediate supervisor or a senior coworker. The new method is taught according to the following steps:

- Reading the SOP
- Observing performance of the method
- Closer reading of the SOP, associated literature, and other notes
- Supervised practice of the method on non-critical work, until the supervisor is satisfied that the employee is competent. Competency is demonstrated when an acceptable Initial Demonstration of Capability (IDOC) has been submitted and approved by the Quality Assurance Officer. This shall be done both for methyl mercury distillation process, total mercury analysis, and methyl mercury analysis.

A3.2 Ongoing Training

All technical staff shall be given the following annual training:

- Annual Ethics and Data Integrity training. All employees are required to attend ethics and data integrity training annually and sign the Ethics and Data Integrity Policy.
- All technical staff members shall undergo annual general laboratory safety training.
- All employees shall attest that they have read, understood, and agreed to follow the current version of the QAP and any SOPs that are related to their job duties.
- The analysts shall show ongoing demonstration of capability in applicable areas on an annual basis.

A4 Quality Assurance

A4.1 Quality Assurance Policy Statement

HAL recognizes quality as a key element of the laboratory's standard of service. HAL is committed to quality through the strict adherence to the Quality Policy Statement. The Quality Policy Statement gives employees clear requirements for the production of analytical data. Employees are trained on the components of the Quality Policy Statement during their first day of orientation. Each employee signs the statement as agreement to implement the policy in all aspects of their work. The statement is as follows:

- Describing clearly and accurately all activities performed; documenting "real time" as the task is carried out; understanding that it is never acceptable to "back date" entries and should additional information be required at a later date, the actual date and by whom the notation is made must be documented.
- Providing accountability and traceability for each sample analyzed through proper sample handling, labeling, preparation, instrument calibration/qualification, analysis, and reporting; establishing an audit trail that identifies date, time, analyst, instrument used, instrument conditions, quality control samples (where appropriate and/or required by the method), and associated standard material.
- Emphasizing a total quality management process and commitment to continuous improvement which provides accuracy, and strict compliance with agency regulations and client requirements, giving the highest degree of confidence; understanding that meeting the requirements of the next employee in the work flow process is just as important as meeting the needs of the external client.
- Providing thorough documentation and explanation to qualify reported data that may not meet all requirements and specifications, but is still of use to the client; understanding this occurs only after discussion with the client on the data limitations and acceptability of this approach.
- Responding immediately to indications of questionable data, out-of-specification occurrences, equipment malfunctions, and other types of laboratory problems, with investigation and applicable corrective action; documenting these activities completely, including the reasons for the decisions made.
- Providing a work environment that ensures accessibility to all levels of management and encourages questions and expression of concern on quality issues to management.

We each take personal responsibility to provide this quality product while meeting the company's high standards of integrity and ethics, understanding that improprieties, such as failure to conduct the required test, manipulation of test procedures or data, or inaccurate documentation will not be tolerated. Intentional misrepresentation of the activities performed is considered fraud and is grounds for termination.

HAL is committed to Quality Assurance, viewing it as both a program and a philosophy. Quality control begins at the bench level, and management continuously works to improve processes with a focus on prevention of analytical problems. HAL's laboratory staff is trained to troubleshoot and initiate corrective actions. Process improvements and problem solving are solicited from the technicians and analysts, and management implements the solutions. This helps keep management informed while at the same time promoting the professional growth of HAL's staff.

HAL is dedicated to providing high-quality data that meets the needs of the MDN. Accurate and precise data depends on these basic principles:

- Sample integrity must be preserved. All documented sample handling procedures for preservation, custody, storage, labeling, and recordkeeping are followed.
- Trace metal-free ("ultra-clean") sample handling must be employed. Samples that are analyzed for low-level mercury or methyl mercury concentrations are handled according to established protocols. This includes the use of Class-100 clean hoods, clean gloves, and pre-tested and approved reagents, water, and equipment. High-level

(contaminated) samples are kept segregated from ultra-clean samples during storage and sample preparation.

- Approved analytical methods must be followed. The analyst's fundamental understanding of analytical methods is paramount for effective quality control. Emphasis on scientific understanding and adherence to procedure is part of every analyst's training. QC results from each method are evaluated to identify and correct method weaknesses, and to detect any need for further training.
- Analytical instrumentation must be in proper working order. Optimum instrument performance is ensured by analyzing daily calibration and performance evaluation samples. Preventative maintenance is performed on a regular basis and is documented in the instrument logbooks.
- Raw data must be reduced properly and accurately transcribed into the correct reporting format.
- Data review, from acquisition to the final report, is performed throughout to minimize error.

A4.2 Quality Assurance Objectives

HAL data quality is assessed against EFGS's DQO to ensure production of high-quality coherent data. The DQO consist of five elements: precision, accuracy (bias), representativeness, comparability, and completeness. These elements are evaluated annually and the results are presented in Annual QA Reports.

- *Precision* is a measure of our ability to use our methods to analyze a sample repeatedly and get the same results each time. To demonstrate precision of a method, sample duplicates are analyzed and the results compared. The acceptance criterion for Matrix Duplicates is ≤ 25% Relative Percent Difference (RPD).
- *Accuracy* or bias is a measure of how close the result is to the true or expected value of the target analyte in the sample. Accuracy may be determined by the analysis of reference materials, blank spikes, or matrix spikes where the results can be compared with a true or expected value. The acceptance criteria for Reference Materials and Matrix Spikes are 75-125% recovery.
- *Representativeness* describes how well a single sample can characterize the conditions of the entire sample population. Appropriate sampling techniques and artifact-free procedures, combined with sample homogenization, help achieve representative data.
- *Comparability* is a particularly important QA criterion for long-term projects. Individual data sets are evaluated with respect to other data from the same project to ensure the validity of trends in the data.
- *Completeness* is a measure of how many collected data points are usable. HAL strives for at least 95% analytical data completeness for the MDN project.

A4.3 Facilities and Equipment

EFGS's 18,000 ft^2 analytical laboratory is located in Bothell, Washington. The location is close to both UPS and FedEx shipping centers and has easy access to interstate 405. The space contains large laboratory areas for sample analysis, sample preparation, and bottle washing and is equipped with several Class-100 clean air stations. The facility has specially designed areas for sample storage, shipping and receiving, and hazardous waste disposal. HAL has a sample shipping and receiving area for the Mercury Deposition Network (MDN) that was designed especially for MDN. EFGS's entire space is secured and monitored.

The laboratories are served by a custom-designed HVAC system, providing an atmosphere that is clean and well isolated from outside dust and dirt. Disposal of all other toxic materials is carried out under contract with a certified disposal company. The entire EFGS space is inspected periodically for compliance with all city and state code requirements for fire and emissions.

The offices are equipped with document production equipment including laser printers, document and image-processing software, high volume photographic-quality color printer, large-capacity collating copiers, and a binding machine. A LAN connects staff computers and printers for local access, as well as providing external email, fax, and Internet access. EFGS also maintains a web site at http://www.eurofinsus.com/environment-testing/laboratories/eurofins-frontier-global-sciences/services/metals-and-metals-speciation and has both UPS and FedEx shipping capabilities with access to pick-up as late as 5:00 PM Pacific time. The laboratory has staff available Monday through Friday from 7:00 AM until 6:00 PM Pacific time to receive sample shipments.

A4.3.1 Building Security

Access to EFGS offices and laboratories is regulated and limited to authorized personnel. All outside doors are kept locked at all times. The facility is monitored 24/7 by a security system. Visitors must first identify themselves before being admitted by an EFGS employee. Visitors are required to check in and sign the logbook (LOG- HS-001 Visitor Log) on arrival, and to sign out on departure. All visitors must wear a "Visitor" batch while on-site. Regular visitors may be allowed unescorted access to general areas of the building, provided that their names and other details are recorded at the front desk. All other visitors in laboratory areas must be accompanied by an EFGS employee.

A5 HAL Capital Equipment for Analytical Use

Quantity	Instrumentation
3	Flow Injection Cold Vapor Atomic Fluorescence Spectrophotometers for Mercury Analysis
1	Flow Injection Cold Vapor Atomic Fluorescence Spectrophotometer for Methyl Mercury Analysis
3	Manual Isothermal GC/Bubbler Systems for Hg Speciation
4	Class-100 Clean Air Hood
6	Methyl Hg Distillation Units
1	Gold Sputter Coater
1	RO Reagent Water System

A6 Ultra Clean Facilities Monitoring

A6.1 Air Monitoring

HAL's mercury analyses require ultra low levels of mercury in laboratory air. All laboratories at HAL are monitored monthly for mercury contamination by direct measurements with a real time low level atmospheric Hg detector. The action limit for laboratory air is 25 ng/m^3. If a laboratory's air exceeds the action limit, air flow is increased and the location is monitored until

levels agree with previously collected background data from this location. Records of each test are maintained by the MDN Laboratory Group Leader and are available upon request.

A6.2 Reagent Water Monitoring

Ensuring that reagent water is free of contamination sources for sampling and analysis of ambient water is critical to HAL's laboratory facilities. The reagent water monitoring program gives additional quantitative evidence that the process is contaminant-free. Records of each test performed under the reagent water monitoring program are maintained in the LIMS.

The mercury warning limit for the monthly waters is set at <0.25 ng/L. If the concentration of mercury is greater than 0.5 ng/L, a new sample shall be collected and tested. If the source comes out high two consecutive times, the Laboratory Manager, the MDN Project Manager/Site Liaison, and the Quality Assurance Officer shall be notified in an email message and the water shall not be used. A maintenance visit may need to be scheduled to resolve the problem.

Section B:
Laboratory Operations

B1 Laboratory Procedures

B1.1 Documentation

HAL's goal is to be able to trace all laboratory measurements to their original source. The HAL uses traceable reagents, standards, and reference materials in all procedures. Instrument calibration, reagent and bottle testing, and equipment maintenance are all thoroughly documented. All calibrations are traceable to certified standards or manufacturer lot numbers. For analytical instruments, high purity calibration standards are obtained from chemical suppliers. Certificates attesting to the concentration ranges of the covered analytes are retained by the QA Officer.

B1.2 Reagents and Standards

Standards and reagents are documented in LIMS upon receipt or creation. A LIMS generated label is affixed to each standard and reagent, with the name of the solution, the person who prepared or received it, the date it was prepared or received, and the expiration date. All standards and reagents are logged into LIMS individually (one LIMS ID per sample container).
The LIMS documentation must include the following:

- A description of the standard;
- Department,
- Expiration date of the standard (not to exceed the expiration of the parent standard),
- The name of the person that prepared the standard or reagent,
- The date it was prepared (or received),
- Final volume,
- A reference date (date entered into LIMS),
- Concentration units (µg/mL),
- The vendor and the vendor lot (the solvent lot is not applicable),
- The correct parent standard must be documented, as well as the aliquot used; and
- Analytes are entered individually.

Where possible, the HAL uses reference materials that are certified and traceable to national or international standards of measurement. These do not require testing, provided there is a Certificate of Analysis on file with the QA Officer.

The mercury analyst shall notify the QA Officer each time a new standard is prepared, the LIMS ID of the new standard, and requests a work order to document standard testing. The QA Officer creates a work order and enters the work order into the comments section of the standard. Working reagents are prepared by the analyst and logged into LIMS and assigned a unique identifier. All reagents used during analysis and prep should be added to the bench sheets. Additionally, reagents undergo continuous monitoring through analysis of method blanks. A method blank is a sample of reagent water and analytical reagents that undergoes the same analytical process as the corresponding samples. A minimum of three method blank samples are prepared with each analytical batch. For MDN, a typical analytical batch consists of 30 samples.

B1.3 Expiration Date and Opening Dates on Standards and Reagents:

The expiration date is set to no longer than three years if no expiration date is given by the manufacturer. Expiration dates can be extended if the reference standard's or material's integrity is verified. The extended date may not be beyond the expiration date of the referenced standards used to re-verify. All standards and reagents shall be labeled with the date they were opened and the initials of the analyst who opened the container.

B1.4 Calibration and Verification of Support Equipment

Support equipment used at HAL is calibrated an external ISO certified calibration company or calibrated internally. The equipment used includes:

Equipment	Frequency	Source	Comments
Balances	bi-yearly	externally	used to weigh out sample aliquot size
Balances	daily before usage	internally	used to weigh out sample aliquot size
ASTM Class 1 and 3 Weights	yearly	externally	used for internal balance calibration
Pipettes	daily	internally	used for BrCl preservation
Pipettes	weekly	internally	all, except the pipettes used for BrCl preservation
Repipettors	quarterly	internally	used for preparing 1% HCl charge solution
Digital Thermometers	Quarterly	internally	used for temperature monitoring
Liquid-Filled Thermometers	yearly	internally	used for temperature monitoring
Refrigerators	daily	internally	temperature verified daily
Lumex Hg analyzer	yearly	externally	used for air monitoring
RO system in-line conductivity meter	yearly	externally	used for reagent water monitoring

Table 1: Calibration Frequency of Support Equipment used at HAL

Equipment that has been subject to overloading, mishandling, given suspect results, or shown to be defective or outside specifications is taken out of service. The equipment shall be labeled clearly as being out of service until it has been shown to function properly and the QA department has related paperwork on file for documentation. If it is shown that previous tests are affected, then procedures for nonconforming work in EFQA-Q-QD-SOP2812 are followed and results are documented.

When support equipment is checked or calibrated, measurements are recorded in laboratory logbooks or in the calibration file in the QA Office. The calibration tolerances of the analytical support equipment are listed in their respective SOPs (EFQA-R-MT-SOP2710 "Balance Verification, Calibration and Maintenance," EFQA-R-EQ-SOP2711 "Pipette and Dispenser Operation, Calibration and Maintenance," and EFSR-P-SH-SOP2712 "Refrigerator and Freezer Temp Monitoring and Maintenance").

B1.5 Calibration of Analytical Instruments

Every instrument used to analyze samples at the HAL must pass the calibration criteria in the relevant SOP. Initial calibration criteria for instrument reproducibility and sensitivity must be met before samples may be analyzed. Continuing calibration verification (CCV) checks establish whether ongoing instrument calibration is acceptable. Before a new mercury instrument is used a PQL/MDL study must be performed on the instrument. Instrument approval must be documented and kept on file with the Quality Assurance Officer. New support equipment, such as pipettes, must be received with a record of calibration, which is turned in to the QA department for archiving.

The calibration protocols and the control criteria for total mercury and methyl mercury analysis are outlined in SOPs EFMDN-T-MDN-SOP5510 "MDN Total Mercury Sample Analysis" and EFAFS-T-AFS-SOP2808 "Determination of Methyl Mercury in Various Matrices.

The lowest calibration standard is the lowest concentration for which quantitative results can be reported without qualification. The lowest calibration standard is at the Limit of Quantitation (LOQ or PQL), or lower, and is greater than the Limit of Detection (MDL or LOD). Results that are less than the lower calibration standard are considered to have increased uncertainty.

The highest calibration standard is the highest concentration for which quantitative results can be reported. The sample shall be diluted and reanalyzed within the calibration curve. If the volume has been exhausted and the analytical result was above the highest calibration point, a standard greater than the sample result shall be analyzed to verify the linearity of the calibration curve at the level of the sample. The sample shall be reported with a qualifier.

B1.6 Calibration Verification for Analytical Instruments

An Initial Calibration Verification (ICV) is analyzed following each calibration curve to verify the accuracy of the primary standard solution. The ICV is a solution made from a second source standard, traceable to a national standard when commercially available, and independent of that used in the primary standard solution.

CCVs verify that the analytical system is in control, or to demonstrate analytical drift. The CCV is a standard solution that is made from a traceable stock standard (usually the same source as the primary calibration stock). CCVs are analyzed at a frequency of every ten samples or less and at the end of each analytical sequence. All ICV/CCVs reference a unique identification number and are traceable through LIMS. All MDN raw data references a unique laboratory ID number and includes a unique identifier for each standard used in the analysis. These identification numbers are traceable through LIMS.

B2 Sample Analysis

B2.1 Laboratory Quality Control Samples

A laboratory control sample (LCS) may be a certified reference material or a Blank Spike (BS). A BS is a sample of reagent water or analytical reagents that has predetermined quantities of analyte added. It undergoes the same preparation and analytical processes as the corresponding samples. Blank spikes are used to evaluate the daily performance of a method, but are not subject to matrix effects that may occur in matrix spikes (MS). They are used primarily when no appropriate reference material is available for a particular matrix. The quality control criteria are discussed in the annual QA report, which also contain an example of an Analytical Run Sequence, see Table 5.

B2.2 Laboratory Bottle Blanks

B2.2.1 Description

Following cleaning, MDN sample bottles are charged with 20 mL of 1% hydrochloric acid. One sample bottle is selected randomly from each cleaning event and is analyzed for total mercury. On average, 2-3 laboratory bottle blanks are analyzed each week for total mercury. At least one bottle blank should be collected per month and analyzed for methyl mercury. The quality control criteria are discussed in the annual QA report. For each bottle blank, the vat in which the bottle was cleaned is recorded on the bottle blank form in Appendix A, EFGS EFMDN-T-MDN-SOP5697. The acid vats in the bottle washing rooms are tested on an as needed basis. The cleanliness of the bottles demonstrates the effectiveness of the vats used for bottle cleaning. Each vat should include what acid it contains, the acid's LIMS ID, the concentration of the acid, when the vat was started, and by whom. This information should be included when additional acid is added.

B2.2.2 Purpose

Even in an ultra-clean laboratory, mercury exposure is inherent to the handling of MDN sample bottles. Because such contamination is inevitable, it must be analyzed and quantified so that it can be subtracted objectively from final sample results. The final sample results for mercury are corrected by the average bottle blank results from the previous quarter. The final sample results for methyl mercury are not bottle blank corrected. The result of the methyl mercury bottle blanks are used to monitor for lab-introduced contamination.

B2.3 Preparation Blanks

B2.3.1 Description

Preparation blanks for total mercury consist of bromine monochloride (1 % (v/v) BrCl), hydroxylamine hydrochloride (0.200mL), and stannous chloride (0.300mL) in 100mL of reagent water. The quality control criteria are discussed in the annual QA report.

Preparation blanks for methyl mercury consist of 45 mL reagent water, hydrochloric acid (0.4%), ammonium pyrrolidine dithiocarbamate (0.200mL of APDC) solution, ethylating agent (38.5µL), acetate buffer (0.300mL), and reagent water. The quality control criteria are discussed in the annual QA report.

B2.3.2 Purpose

Mercury contamination is inherent in sample preparation and in analytical reagents, in any laboratory setting. Preparation blanks are a measure of how much of each sample result can be attributed from these necessary reagents. Preparation blanks also help when investigating possible sources of contamination.

B2.4 Initial and Continuing Calibration Verification Standards

B2.4.1 Description

The Initial Calibration Verification (ICV) is a solution made from a second source standard, independent of what is used in the primary standard solution. For the MDN total mercury project, NIST 1641d is the secondary source analyzed after the calibration curve and also after the second set of matrix spikes, and is discussed under the Certified Reference Material (CRM) section.

A minimum of one Continuing Calibration Verification (CCV) standard is analyzed for every 10 samples during the course of the run, and at the end of each analytical run. The CCV is a standard solution that is made from a traceable stock standard (usually the same source as the primary calibration stock). A 10ng/L standard for total mercury and a 0.5ng/L standard for methyl mercury are analyzed as an ongoing calibration standard. The MDN control limits for ICVs for total and methyl mercury are set to 80-120%; the CCV control limits for total mercury are 77-123%, while the CCV limits for methyl mercury are 67-133%.

B2.4.2 Purpose

An ICV is analyzed following each calibration curve to verify the accuracy of the primary standard solution and to validate the calibration curve. CCVs verify that the analytical system is in control, or demonstrate analytical drift. All ICV/CCVs reference a unique identification number and are traceable through LIMS. All raw data references a unique laboratory ID number and includes a unique identifier for each standard used in the analysis.

B2.5 Continuing Calibration Blanks

B2.5.1 Description

Continuing Calibration Blanks (CCBs) are analyzed during the course of sample analysis directly after the ICV and the CCVs, with a minimum frequency of one per 10 samples, and at the end of each analytical run. The run must end with a CCB. Individually, the Initial and Continuing Calibration Blanks (ICB/CCBs) shall be less than 0.25ng/L to be within control limits for total mercury. For MMHg the mean of the ICB/ CCB shall be less than 0.025 ng/L.

B2.5.2 Purpose

Instrument blanks are used to demonstrate freedom from system contamination, carryover, and to monitor baseline drift.

B2.6 Matrix Duplicates

B2.6.1 Description

Matrix Duplicates (MD) are created when an existing sample is split into two portions and then are compared analytically. The MDN control limit for the Matrix Duplicates is set at 25% RPD for total mercury. US EPA methods 1630 and 1631 do not require a MD. One MD is performed for every ten analyzed samples and during a standard MDN THg analytical run three MDs are analyzed. The source samples are selected depending on the available volume. For total mercury analysis, 100 mL is ideal, though when samples with sufficient volume are not available, HAL uses 50 mL or 75 mL aliquot volumes for the source sample, the MD and the MS, and for potential reanalysis of these QC samples. The quality control criteria are discussed in the annual QA report.

B2.6.2 Purpose

Replicate samples provide information about analytical precision. MDs are part of the same sample. As such, their Relative Percent Difference (RPD) is expected to be less than 25%. Out of control results are indications of a heterogeneous sample matrix and/or poor analytical precision.

B2.7 Matrix Spikes

B2.7.1 Description

A Matrix Spike (MS) for total mercury is created when a sample with known mercury content is split in two fractions and one fraction is supplemented with an additional 1.00ng of mercury standard.

For both EPA method 1631 and 1630, there must be 1 MS and 1 MSD sample for every 10 samples (a frequency of 10%) and the spiking level shall be at 1–5 times the background concentration or at 1-5 times the MRL (0.5ng/L for THg and 0.06ng/L for MMHg), whichever is greater.

For MDN runs, due to limited sample volume, only one matrix spike (MS) is performed for every ten analyzed samples and during a normal analytical run three matrix spikes are analyzed. The source samples are selected depending on the available volume. 50mL aliquot volumes for the source for the source sample, the matrix duplicate and the matrix spike are ideal. No RPD data for MS/MSD is available for total mercury since only a MS is analyzed. A MS/MSD is performed for methyl mercury and the control limit for the RPD is <25%.

B2.7.2 Purpose

The purpose of analyzing a MS and MSD is to demonstrate the performance of the analytical method in a particular sample matrix, and to recognize matrix interference. To prepare a MS/MSD, predetermined quantities of the analyte are added to a sample matrix before (when possible) extraction or digestion of samples, in this case preservation with BrCl for total mercury and preservation with HCl and distillation for methyl mercury analysis. If the sample is spiked with the analyte of interest after extraction or digestion, this is considered an analytical spike and an analytical spike duplicate (AS/ASD). Low recovery of a matrix spike is a sign of matrix interference. After investigation by trap and bubbler test, the samples should be reanalyzed at a dilution. The purpose is to ascertain the largest aliquot size a sample can be analyzed at without matrix interference. The source sample shall then also be reanalyzed at the same aliquot size.

B2.8 Certified Reference Materials

B2.8.1 Description

Certified Reference Materials (CRMs) are matrix specific standards that are accompanied by a certificate of analysis for the analytes of interest. The HAL generally purchases reference materials from the National Institute of Standards and Technology (NIST), the National Research Council of Canada (NRCC), or the International Atomic Energy Agency (IAEA). The HAL maintains the position that matrix equivalent reference materials are the best measure of precision and accuracy (bias), as issues associated with matrix type and homogeneity may be assessed.

Currently, there is no available CRM matching the MDN rainwater matrix. Therefore, HAL uses National Institute of Standards and Technology (NIST) reference material 1641d "Mercury in Water." The percent recovery control limits for total mercury are currently set at 80-120% with a RPD of 24%. There is no CRM available for methyl mercury and therefore a Blank Spike and a Blank Spike Duplicate (BS/BSD) are analyzed for methyl mercury with acceptance criteria of 70-130%, with a RPD of 25%. The US EPA methods 1630 and 1631 do not require a certified reference material.

B2.8.2 Purpose

CRM are used to demonstrate HAL's ability to recover a target analyte from a specific matrix. The first CRM is analyzed right after the calibration curve to verify the validity of the analytical curve.

B2.9 Blank Spikes

B2.9.1 Description

A BS is a sample of reagent water or analytical reagents that has predetermined quantities of analyte added. It undergoes the same preparation and analytical processes as the corresponding samples.

B2.9.2 Purpose

Blank Spikes are used to evaluate the daily performance of a method, but are not subject to matrix effects that may occur in matrix spikes. They are used primarily when no appropriate reference material is available for a particular matrix.

QC Sample ID	Frequency	Acceptance Limits	Corrective Action
ICV	Immediately after the last calibration standard	80-120% for both THg and MMHg	Recalibrate the instrument and re-analyze the affected samples.
CCV	At the end of the sequence and after every 10 or fewer samples	77-123% THg, 67-133% MMHg	Recalibrate the instrument and re-analyze the affected samples.
ICB/CCB	Immediately after each CCV	< 0.25 ng/L for THg, < 0.025 ng/L for MMHg	Recalibrate the instrument and re-analyze the affected samples.
Bottle Blanks	One sample bottle selected randomly from each cleaning event for THg; one bottle blank collected each month for MMHg.	≤ MDL for both THg and MMHg	Track results and look for trends. Conduct cause analysis if trends are identified.
Prep Blanks	Three per batch of thirty samples for THg, three per twenty samples for MMHg.	< 0.25 ng/L for THg, < 0.045 ng/L for MMHg	Re-analyze to confirm. For THg, test preservation source solution. For MMHg, reprep samples. If there is insufficient sample for a reprep, enter results into database with a note.
CRMs	One pair per batch of thirty samples	80-120% THg <24% RPD THg	Recalibrate and re-analyze to confirm.

QC Sample ID	Frequency	Acceptance Limits	Corrective Action
Blank Spikes	One pair per batch of twenty samples	70-130% MMHg <25% RPD MMHg	Recalibrate and re-analyze to confirm. Reprep samples. If there is insufficient sample for a reprep, enter results into database with a note.
Matrix Duplicates	One per batch of ten samples for THg; one per twenty samples for MMHg	<25% RPD THg <25% RPD MMHg	Re-analyze to confirm. Qualify and report
Matrix Spikes	One pair per batch of ten samples	75-125% THg (No MSD for THg) 65-135% MMHg <25% RPD MMHg	Re-analyze to confirm. Re-prepare the batch using a different source sample.

Table 2: QC Sample Limits and Corrective Actions

B2.10 Method Detection Limits

Method Detections Limits (MDL) are determined according to 40 CFR Part 136, Section B. Ten replicates (t-1, 9 degrees of freedom, where t is the Student's T-value for the number of replicates) of matrix matched samples that are spiked at 1-10 times the expected MDL are analyzed. There is no recovery criterion for an MDL analysis, but the new calculated MDL value must be within 2*times of the previous established MDL. The standard deviation (σ) is taken from the resulting data and the MDL is determined as t * σ of the replicates. For ten replicates, the MDL is calculated as follows: MDL=2.821 * σ. This value should not be interpreted as the method reporting limit.

The Practical Quantitation Limit (PQL) is the reporting limit for the method and is included as the lowest calibration point (2003 NELAC regulation 5.5.5.2.2.1.h.3). The PQL is determined by running ten replicate samples with a concentration that must normally meet a recovery of 70-130% (or the same recovery criteria which is applicable for the low non-standard calibration point, depending on the method). The PQL is also referred to as the Method Reporting Limit (MRL).

All MDL and PQL studies are on file with the Quality Assurance officer and are available upon request.

B2.11 Control Charts

For MDN the QC points include: LCS% Recovery, LCS/LCSD RPD, MS% Recovery, MS/MSD RPD, Duplicate RPD, ICV% Recovery, CCV% Recovery and Blanks are presented in graphs and are included in the annual QA report. Control charts allow the QA Officer and staff to spot unfavorable analytical trends as they are developing. Corrective actions for those trends can in turn be assessed in real time. Additionally, control charts are used periodically in the calculation of efficiency factors for the methyl mercury distillation method.

B3 Performance and System Audits

B3.1 Internal Laboratory Audits

It is the responsibility of the QA Officer to coordinate and conduct annual internal audits of the laboratory according to SOP EFQA-Q-QD-SOP2989, "Internal Quality Assurance Audit Procedures" to verify that the activities continue to comply with the requirements of the quality system and 2009 NELAC Standards/TNI standards. After the audit, the QA Officer lists the findings and observations in a table that is sent to the manager for the group. A meeting will also be held where the findings and the observations are discussed, and the time frame for the completion of the corrective actions is decided. Follow-up audit activities shall verify and record the implementation and effectiveness of the corrective action taken. Findings and Observations are defined as:

- **Findings -** items that deviate from NELAC standards, the EFGS QAP, MDN QAP, and standard operating procedures.
- **Observations -** items that suggest omissions or potential improvements in the laboratory's quality systems.

B3.2 External Laboratory Audits

EFGS views third-party audits as a form of consultation and welcomes the opportunity to improve the quality of the laboratory and the quality management system. On average, EFGS is audited four times each year. External audits enable EFGS to qualify for and maintain accreditation through state governments and NELAP. Additionally, clients may audit us as part of a potential or ongoing contract. The Quality Assurance Officer maintains records of all such audits, the findings, and the corrective actions. Currently the HAL is reviewed by the NADP once every three years.

B4 Corrective Actions

Corrective action is the taken to eliminate the causes of an existing non-conformity, laboratory error, or other problematic situations and prevent its reoccurrence. Corrective action can be initiated by external or internal audits findings, employee observations, data reviews, customer feedback/complaints, or management reviews. All deficiencies are investigated and a corrective action plan is developed and implemented, if necessary. The investigation and action taken is depending of the problem and the degree of risk. Corrective actions may include filing an incident report, revising an SOP, revising the QAP, revising a safety procedure, or writing a new SOP.

B4.1 Incident Reports

Mistakes and accidents occur in the course of analytical laboratory work. These must be reported immediately to the supervisor and documented on an Incident Report Form (SOP EFQA-Q-QD-SOP2835, "Incident and OOS Reports"). If there are safety concerns, a report is also filed with the Health & Safety Officer, or other assigned staff member. An Incident Report Form is completed when a problem arises that requires a deviation from the applicable SOP or method. The deviation may be due to a mistake or accident. It also may be due to unforeseen problems with a sample, instrument or dataset. Whatever the circumstance, it must be recorded as soon as possible. It is the responsibility of each Group Supervisor (or delegate) to complete the Incident Report Forms and submit them to the QA group for review and follow-up.

Completed Incident Report Forms are kept on file in the QA office. It is the procedure that the supervisor for the affected laboratory area starts the investigation and is writing up the incident report. The Quality Assurance Officer shall review the actions taken and the root cause analysis before the IR is completed and is submitted to all involved parties.

B5 Proficiency Testing Program

HAL started to participate in proficiency studies for total low level mercury in March of 2010. There are currently no available proficiency studies for methyl mercury. The PT sample shall be treated in the same manner as a client sample. Sequential PT studies are analyzed at least five months apart and no more than 7 months apart unless the PT is being used for corrective action to maintain or reinstate accreditation. In these cases, the dates of sequential PT samples for the same accreditation have to be at least fifteen days apart. A failing result for a proficiency study must be investigated, as per EFQA-Q-QD-SOP2835 "Incident and OOS Reports" and reported to primary accreditation authorities.

Before the closing date of a PT study, laboratory personnel shall not:

- Subcontract the PT sample to another laboratory.
- Knowingly receive and analyze a PT sample from another laboratory.
- Communicate with an individual from another laboratory regarding the analysis of the PT sample.
- Attempt to find out the assigned value of a PT from the PT Provider.
- Analyze a PT sample more frequently or with special QC than would be afforded to a field sample.
- Deviate from the PT sample preparation instructions by preparing a more concentrated sample.

The above listed situations are not ethically acceptable.

B6 Laboratory Intercomparison Studies

The HAL participates in inter-laboratory comparison studies provided by USGS on a monthly basis. Samples are submitted for mercury analysis in both spiked and ultrapure deionized water.

B7 Network Field Supplies

The HAL supplies necessary items to all network monitoring sites on a weekly basis. This may include, but is not limited to, the following:

- Clean sample train, i.e. bottle, thistle tubes, and funnels
- Clean unused dry-side bag (for sites with Aerochem (ACM) collectors only).
- Blank field forms
- Clean, laboratory gloves
- Reagent-grade water

Additional information may be found in EFGS EFMDN-T-MDN-SOP5511 "Shipping of MDN Glassware."

Section C:
Data Management Operations

C1 Preventive Maintenance/Service

C1.1 Computer Systems and Software

HAL employs a number of servers, all which will conform to the latest industry best practices. Some of the servers act as domain controllers to control and maintain security access and access policies. Separate servers are utilized to control remote access to the network, terminal services, file and print services as well as the email server. All servers are equipped with backup drives and appropriate backup software that provides scheduling, automation, and monitoring of back-ups.

The server room is located on the second floor in a locked room with a metal door. It is closed each evening according to the lab lockup procedure to protect against fire. The servers and other network hardware are installed at least three inches above the floor to protect from water damage. Each server is attached to a UPS system with monitoring software and has enough battery power to keep the server running for at least twenty minutes. If the power is out for more than ten minutes, the software will shut down the server automatically while storing all data before battery power runs out. Network devices such as routers, switches, and firewalls are also attached to a UPS device. All other computers also have some form of UPS to minimize data loss, and loss of instrument control due to a short power failure. The servers, network hardware, and all computers' AC power supplies are plugged into power strips with built-in surge/spike protection.

Antivirus software with immediate file protection services is installed on each server. Files on the server disks are scanned daily. Virus definitions are updated automatically each day via an Internet connection to the software vendor. All incoming email is filtered external to the HAL network by an external email protection service. It is scanned for virus, malware, phishing and SPAM content before reaching the HAL network. Potentially harmful attachments are deleted from all e-mails that are sent to HAL. E-mail alerts are sent to IT personnel upon detection of viruses or unauthorized attachments. If HAL gets a large number of infected attachments in a day, the Internet mail service may be suspended until the problems are rectified. Antivirus software is also installed on each employee's computer to protect against infected files brought in through the Internet, outside e-mail accounts, portable diskettes, or flash drives.

Access to computers and files is limited to domain users with passwords that grant access to job-specific files and folders using the file securities built into the file system. Data security has been divided into three categories: access, protection against corruption, and redundancy. Access to data is subject to levels of control. The data owner determines data criticality. Non-critical data is available throughout the network. Critical data is available to members of predefined groups only. Sensitive and proprietary data is restricted at the user level. Data is protected from corruption by a strategy of limited access and redundancy. Redundancy takes the form of data backups via computer and secure storage of data in hard copy. Incremental daily backups and full backups on Friday cover domain controllers, email server, database server, file and web servers, user files.

The testing of the backup system is conducted and verified by the Site IT Coordinator every month. The restored file is checked against the original file to verify that the backup completed successfully. The files restored, their paths, and the tape they were restored from are recorded in the backup testing logbook. Similar testing is conducted quarterly on a full backup created the previous year.

C2 Data Management Operations

C2.1 Description

The data management task involves collecting, entering, transferring, verifying, validating, summarizing, and reporting data. Data include descriptive and historical information about each network site, all field and laboratory data, quality assurance documentation, and summaries and reports of site and network operations.

Data records are transferred from the HAL to the NADP Program Office on a monthly basis. Data records include paper or hardcopy documents, as well as electronic documents.

Preliminary, monthly data reports are sent to the Site Operators and Site Supervisors within 60 days of the end of the month during which sampling occurred. Corrections to the data are made, and documented, within 30 days of the preliminary data report being sent. All changes to the database are documented with a minimum of: who made the change, when the change was made, and why the change was made.

Final data, and supporting documentation, are shipped to the NADP PO at the end of this review process. For total mercury, final data are shipped within 90 days of the end of the month during which sampling occurred. For methyl mercury, final data are shipped within 120 days of the end of the month during which sampling occurred. Methyl mercury reporting requires additional time as composite samples from multiple sampling periods are used.

C2.2 Data Completeness

The goal is to achieve a continuous record of valid samples for the duration of each site's operation. Data completeness can be impacted by problems (e.g., weather, staffing, or equipment) at the field site, problems with shipping, and/or sample handling at the laboratory.

C2.3 Data Entry and Validation

Field and laboratory data are entered into the database using the method of double entry. This ensures correct entry of the data records. Following double entry, data records are screened using automated scripts, and quality codes are assigned. Data records that are assigned a quality code of "C" (invalid) are investigated by the Site Liaison. This helps achieve the goals of data completeness.

When practical, laboratory instruments interface with the LIMS directly. This eliminates the possibility of transcription error, and reduces the effort needed to support the system.

C3 Recordkeeping

C3.1 Laboratory Records

Results of the analytical measurements including original paper records and quality assurance results from instrumentation that are filed by the analysts and the laboratory QA Officer are also archived at HAL. All records (except logbooks) are archived for the life of the project. The logbooks are archived for a period of ten years after the last entry to the logbook according to EFGS procedures.

Computerized data records (e.g., database, scanned field records) are maintained at the NADP PO.

Records maintained at the HAL are stored for the life of the project. Both paper and electronic records are kept under the supervision of the MDN Project Manager.

C3.2 Quality Assurance Reporting

Changes in chemical analysis, new laboratory equipment, laboratory personnel, data verification or validation procedures, and Site Liaison procedures are documented on an annual basis in the QA Report that is submitted to the NADP PO. The QA Report is written by the QA Officer with assistance from the MDN Laboratory Manager and MDN Project Manager. The QA Report summarizes laboratory performance during the year, including the results of all internal QA/QC activities (e.g., blank tests, spike tests, duplicate and split samples, reagent blanks, bottle blanks, etc.).

Eurofins Frontier Global Sciences – MDN-Specific SOPs

EFMDN-T-MDN-SOP5694	MDN Sample Receipt Procedures
EFMDN-T-MDN-SOP5509	MDN Data Entry
EFMDN-T-MDN-SOP5695	Oxidation of Aqueous Samples for Total Mercury
EFMDN-T-MDN-SOP5510	Total Mercury Analysis
EFMDN-T-MDN-SOP5696	Methyl Mercury Preservation, Splits and Composites
EFMDN-T-MDN-SOP5697	Cleaning of MDN Sampling Glassware
EFMDN-T-MDN-SOP5511	Shipping of MDN Sampling Glassware
EFMDN-T-MDN-SOP5512	Verification of Rain Gauge and Event Recorder Data
EFMDN-T-MDN-SOP5513	MDN Data Review and Validation
EFMDN-T-MDN-SOP5514	Documentation of MDN Site Operator Communications
EFMDN-T-MDN-SOP5515	MDN Monthly Report Review Procedures

Table 3: MDN Specific SOP List

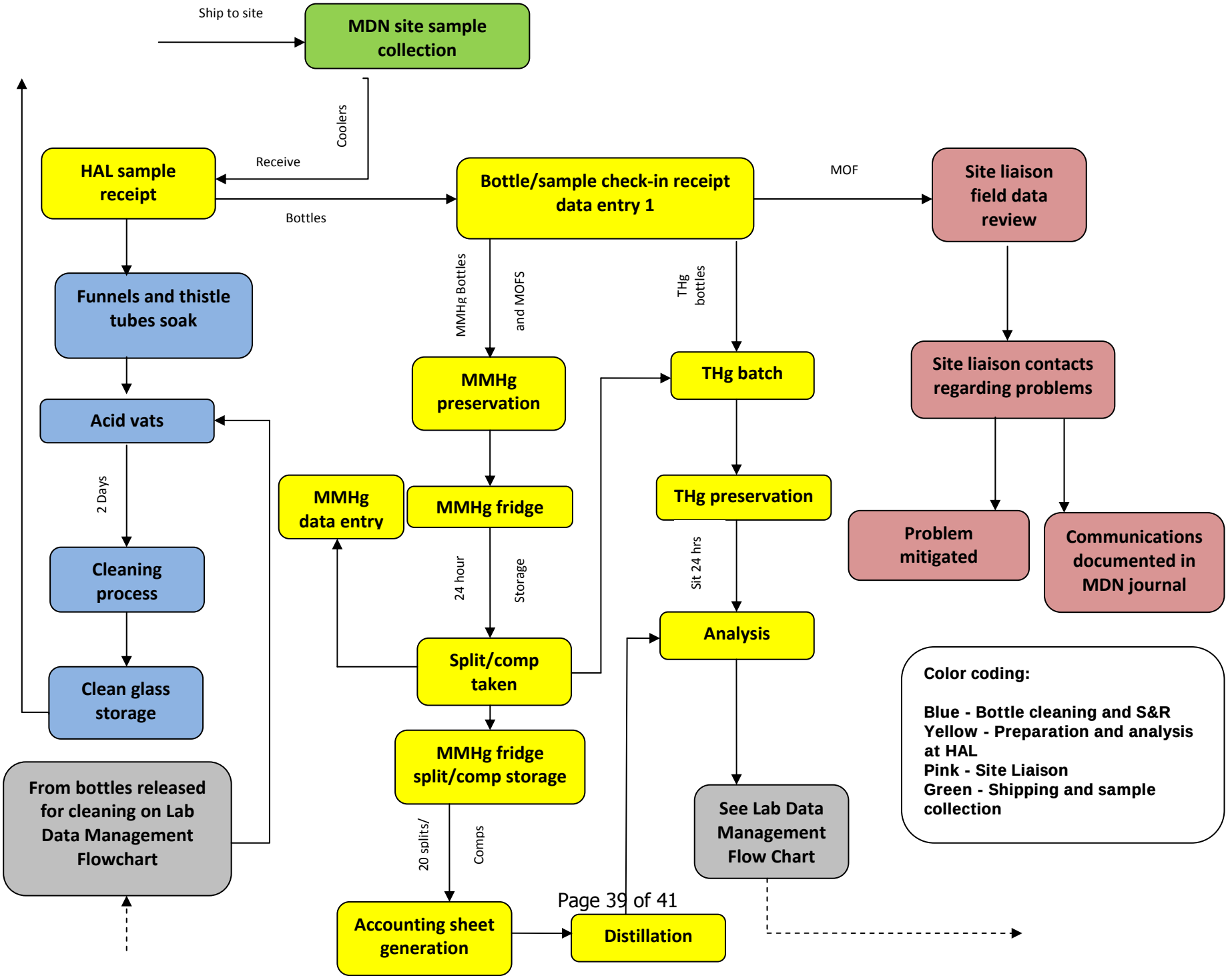
Eurofins Frontier Global Sciences – Supplemental SOPs

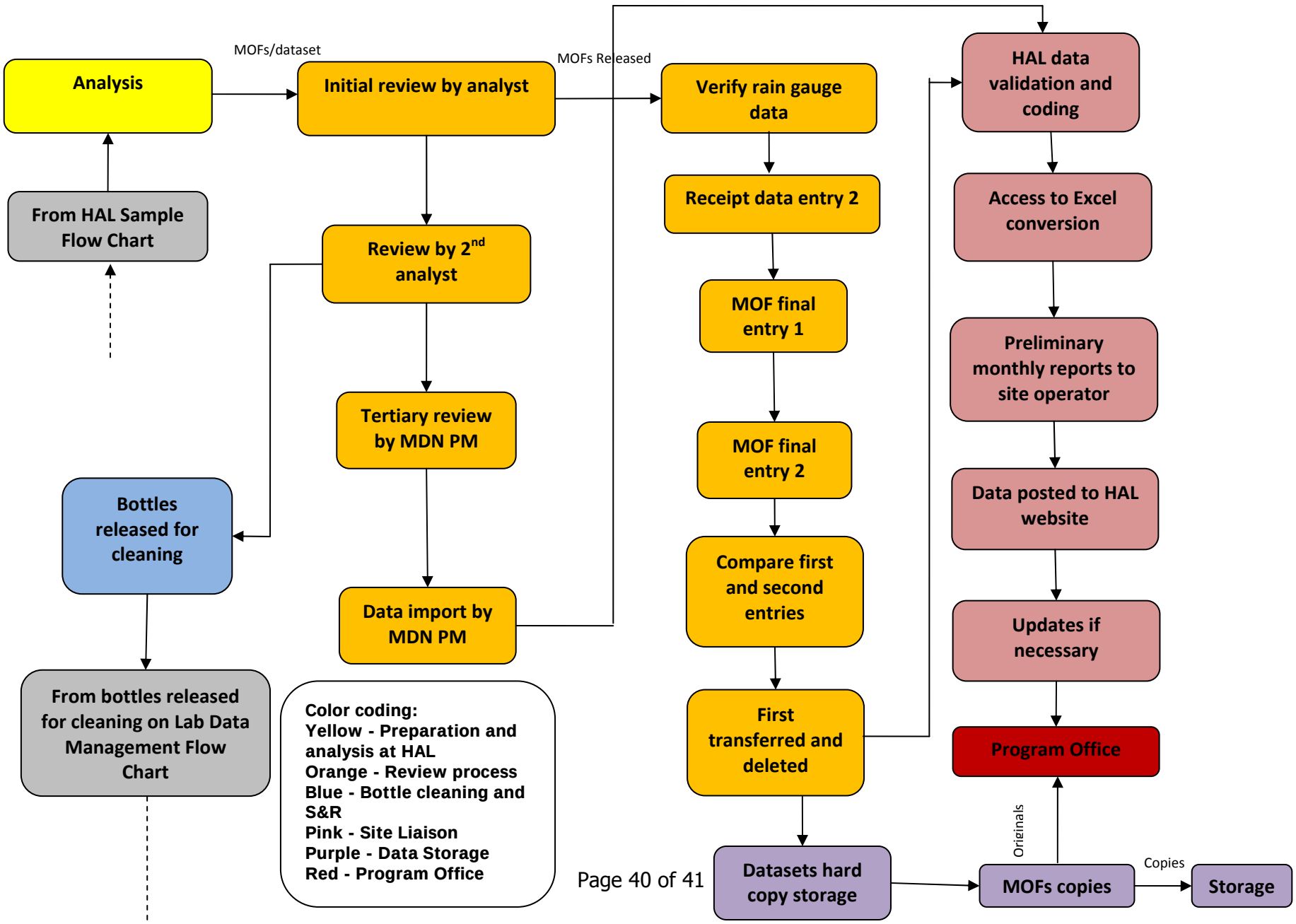
EFQA-R-MT-SOP2710 Balance Verification, Calibration and Maintenance
EFQA-R-EQ-SOP2711 Pipette and Dispenser Operation, Calibration and Maintenance
EFSR-P-SH-SOP2712 Refrigerator and Freezer Temp Monitoring and Maintenance
EFAFS-S-SB-SOP5132 Cleaning of Sampling Equipment and Bottles for Mercury Analysis
EFAFS-T-AFS-SOP2797 Distillation of Aqueous Samples for Methyl Mercury Analysis
EFQA-P-DR-SOP2801 Data Review and Validation and Monthly Logbook Reviews
EFQA-Q-QD-SOP2835 Incident and OOS Reports
EFQA-Q-QD-SOP2989 Internal Quality Assurance Audit Procedures
EFQA-Q-QD-SOP2804 Creation and Control of Standard Operating Procedures
EFAFS-S-T-SOP2806 Preparation of Carbo-Traps for Methyl Mercury Analysis
EFAFS-T-AFS-SOP2808 Determination of Methyl Mercury in Various Matrices
EFQA-S-P-SOP2809 Ordering Laboratory Supplies/Services and Testing Lots
EFTM-T-TM-SOP2839 Stock and Working Standards for Trace Metals Analysis
EFQA-R-EQ-SOP2990 Documentation of Equipment Maintenance
EFQA-Q-QD-SOP2812 Procedures for Departures from Laboratory Policy
EFHS-S-HS-SOP2991 Waste Disposal Procedures for Client Sample Waste
EFQA-Q-QD-SOP2817 Traceability Protocols

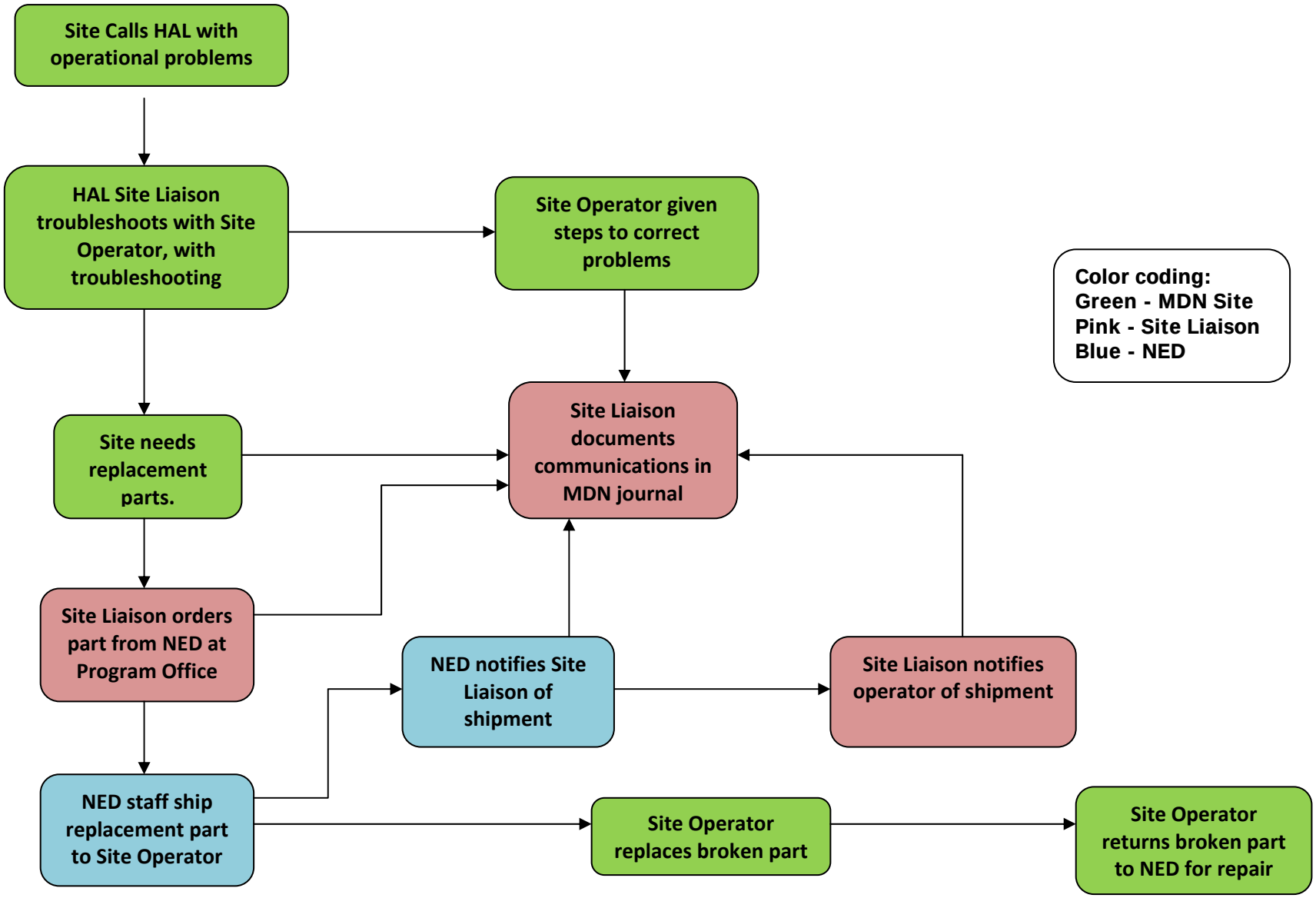
Table 4: Supplemental SOP List

MDN Precipitation Sample Analysis Lab Sheet						Dataset Code				
Analysis Date						Dataset ID				
Instrument						Analyst				
Run	Tp	Bub	HalCode	SampleID	BrCl	PA	Aliquot Volume	THg per Aliquot	THG Conc. (NET)	Remarks
1	1	1		4.00	0					
2	2	2		2.00	0					
3	3	3		1.00	0					
4	4	4		0.5	0					
5	5	1		0.05	0					
6	6	2		BB-1	1		100			
7	7	3		BB-2	1		100			
8	8	4		BB-3	1		100			
9	9	1		NIST1641d	1		0.2			% Rec
10	10	2		BrCl-1	1		100			
11	1	3		BrCl-2	1		100			
12	2	4		BrCl-3	1		100			
13	3	1		BB-4						
14	4	2		Sample # O1	1		100			
15	5	3		Sample # D1	1		100			Mean, RPD
16	6	4		Sample # S1	1		100			% Rec
17	7	1		Sample # 2	1		100			
18	8	2		Sample # 3	1		100			
19	9	3		Sample # 4	1		100			
20	10	4		Sample # 5	1		100			
21	1	1		Sample # 6	1		100			
22	2	2		Sample # 7	1		100			
23	3	3		Sample # 8	1		100			
24	4	4		Sample # 9	1		100			
25	5	1		Sample # 10	1		100			
26	6	2		CCV1 (1.00 ng/L)	0		100			% Rec
27	7	3		CCB-1	0		100			
28	8	4		Sample # O2	1		100			
29	9	1		Sample # 12	1		100			
30	10	2		Sample # 13	1		100			
31	1	3		Sample # 14	1		100			
32	2	4		Sample # 15	1		100			
33	3	1		Sample # 16	1		100			
34	4	2		Sample # 17	1		100			
35	5	3		Sample # 18	1		100			
36	6	4		Sample # 19	1		100			
37	7	1		Sample # 20	1		100			
38	8	2		Sample # D2	1		100			Mean, RPD
39	9	3		Sample # S2	1		100			% Rec
40	10	4		CCV2 (1.00 ng/L)	0		100			% Rec
41	1	1		CCB-2	0		100			
42	2	2		NIST1641d	1		0.2			
43	3	3		Sample # O3	1		100			
44	4	4		Sample # 22	1		100			
45	5	1		Sample # 23	1		100			
46	6	2		Sample # 24	1		100			
47	7	3		Sample # 25	1		100			
48	8	4		Sample # 26	1		100			
49	9	1		Sample # 27	1		100			
50	10	2		Sample # 28	1		100			
51	1	3		Sample # 29	1		100			
52	2	4		Sample # 30	1		100			
53	3	1		Sample # D3	1		100			Mean, RPD
54	4	2		Sample # S3	1		100			% Rec
55	5	3		CCV3 (1.00 ng/L)	0		100			% Rec
56	6	4		CCB-3	0		100			

Table 5: Example of an Analytical Run Sequence


Ship to site
MDN site sample collection
Coolers
Receive
HAL sample receipt
Bottles
Bottle/sample check-in receipt data entry 1
MOF
Site liaison field data review
Funnels and thistle tubes soak
MMHg Bottles
and MOFS
THg bottles
Site liaison contacts regarding problems
Acid vats
MMHg preservation
THg batch
2 Days
MMHg data entry
MMHg fridge
THg preservation
Problem mitigated
Communications documented in MDN journal
Cleaning process
24 hour
Storage
Sit 24 hrs
Analysis
Clean glass storage
Split/comp taken
Color coding:
Blue - Bottle cleaning and S&R
Yellow - Preparation and analysis at HAL
Pink - Site Liaison
Green - Shipping and sample collection
MMHg fridge split/comp storage
From bottles released for cleaning on Lab Data Management Flowchart
20 splits/
Comps
See Lab Data Management Flow Chart
Accounting sheet generation
Distillation


Analysis
MOFs/dataset
Initial review by analyst
MOFs Released
Verify rain gauge data
HAL data validation and coding
From HAL Sample Flow Chart
Review by 2nd analyst
Receipt data entry 2
Access to Excel conversion
MOF final entry 1
Preliminary monthly reports to site operator
Tertiary review by MDN PM
MOF final entry 2
Data posted to HAL website
Bottles released for cleaning
Compare first and second entries
Updates if necessary
Data import by MDN PM
First transferred and deleted
Program Office
From bottles released for cleaning on Lab Data Management Flow Chart
Color coding:
Yellow - Preparation and analysis at HAL
Orange - Review process
Blue - Bottle cleaning and S&R
Pink - Site Liaison
Purple - Data Storage
Red - Program Office
Originals
Datasets hard copy storage
MOFs copies
Copies
Storage


Site Calls HAL with operational problems
HAL Site Liaison troubleshoots with Site Operator, with troubleshooting
Site Operator given steps to correct problems
Color coding:
Green - MDN Site
Pink - Site Liaison
Blue - NED
Site needs replacement parts.
Site Liaison documents communications in MDN journal
Site Liaison orders part from NED at Program Office
NED notifies Site Liaison of shipment
Site Liaison notifies operator of shipment
NED staff ship replacement part to Site Operator
Site Operator replaces broken part
Site Operator returns broken part to NED for repair