SPECIALTY ANALYTICAL

LABORATORY QUALITY ASSURANCE MANUAL

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This QA manual has been prepared to be compliant with the following standards:ANSI/ISO 17025, NELAC 2016.

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INTRODUCTION AND SCOPE

Statement of Management Philosophy

Specialty Analytical is committed to providing the highest quality services and customer service to our clients. The display of this commitment is the responsibility of each employee in the organization. The requirements of this Laboratory Quality Assurance Program apply to each member of the laboratory at every level.

The Specialty Analytical Quality Assurance Manual provides the guidance document to allpersonnel in fulfilling the responsibilities to provide the highest quality, regulatory defensible data. It serves as the statement of quality by the Laboratory and all the personnel at Specialty Analytical.

Specialty Analytical ensures that no economic incentives will compromise data quality.Management will not place any undue production pressure (including production incentives) on the staff of the laboratory that may compromise data quality.

The purpose of Data Quality Objectives (DQO) is to establish specific targets for accuracy (precision and bias). The initial DQO's for a method is to achieve improved precision and accuracy levels than specified in the method reference. Once sufficient data is produced, control charts are established for each analytical parameter appropriate, and the statistics produced are used as the new DQO's for the method, i.e., the mean and standard deviations become the DQO's for that test, provided they are better than the initial DQO's.

The Laboratory Quality Assurance Manager, with the active support of the Laboratory Management Team, is responsible for the implementation of the Quality Assurance Manual. They have the authority, and obligation, to stop work if it fails to meet the exacting criteria defined in the Manual.

Marty French
Laboratory Director
Specialty Analytical

QUALITY SYSTEM

Quality Policy

Quality Policy Statement

The objective of the quality system and the commitment of management are to consistently provide our customers with data of known and documented quality that meets their requirements. Our policy is to use good professional practices, to maintain quality, to uphold the highest quality of service, and to comply with the NELAC Standard. The laboratory ensures that personnel are free from any commercial, financial, and other undue pressures, which might adversely affect the quality of work. This policy is implemented and enforced through the unequivocal commitment of management, at all levels, to the Quality Assurance (QA) principles and practices outlined in this manual. However, the primary responsibility for quality rests with each individual within the laboratory organization. Every laboratory employee must ensure that the generation and reporting of quality analytical data is a fundamental priority. Every laboratory employee is required to familiarize themselves with the quality documentation and to implement the policies and procedures in their work. All employees are trained annually on ethical principles and procedures surrounding the data that is generated. The laboratory maintains a strict policy of client confidentiality.

Quality Manual

This Laboratory Quality Assurance Manual (QAM) details the operations to provide the highest quality and legally defensible data at Specialty Analytical. It outlines procedures with a focus on the Quality Assurance/Quality Control Procedures implemented at the Laboratory. These are based on EPA, and in some cases, State guidelines for analysis of multimedia (soil, water, air) samples for organic and inorganic contaminants.

This document specifies the operations in the Laboratory to assure the accuracy, precision, completeness, representativeness, and comparability of all analyses performed in the Laboratory. The purpose is to assure that all data being reported are of the highest quality, and all documentation supporting this is available.

Quality Assurance and Quality Control Defined

Quality Assurance

Quality Assurance is a system for the integration of planning, assessment, and improvement efforts related to quality in all sections at the laboratory. This includes all actions performed by laboratory personnel, and documentation of laboratory performance. This is designed to identify and correct problems in the analytical process or demonstrate statistical control of a process in the laboratory. The objective of a quality assurance program is to reduce measurement errors to specified limits and produce results of acceptable and consistent quality.

Quality Control

Quality Control is daily procedures and actions taken by the laboratory to assure sample integrity, performance of testing and analysis, record maintenance and data processing. It is a systematic approach to inspection, testing, and corrective actions applied to processes estimating sample quality, and to determine any changes required to maintain or achieve a specific level of quality.

A quality control program includes:

1) Strict adherence to Good Laboratory Practices (GLP).

2) Consistent use and application of Standard Operating Procedures (SOP's).

3) Adherence to specific protocols for measurement processes.

4) Use of highly trained and qualified personnel.

5) Use of reliable and maintained equipment.

6) Use of appropriate calibrations, standards, and quality control materials.

7) Close supervision of operations by senior personnel and management.

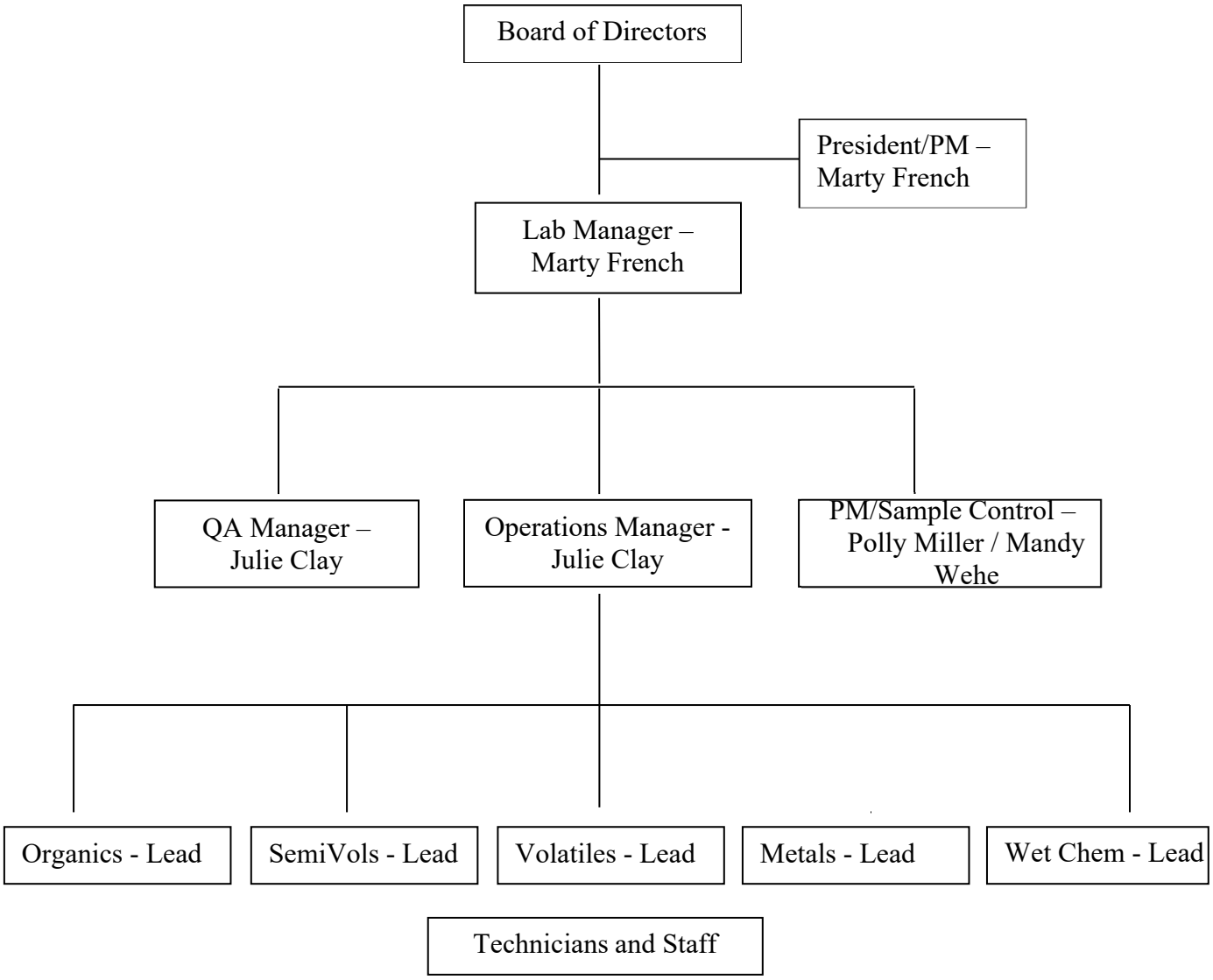
8) Specific acceptance criteria for all Quality Control parameters.

9) Procedures for identification and correction of out-of-control data.

LABORATORY ORGANIZATION - ROLES AND RESPONSIBILITIES

Specialty Analytical management reports to the Board of Directors of E.R. Analytical, Inc., through the President of Specialty Analytical. All employees of Specialty Analytical are responsible to the Laboratory Manager, who reports directly to the Board of Directors.

Laboratory Organization Chart



Roles and Responsibilities

Quality Assurance Management

Laboratory Manager - The Laboratory Manager has overall responsibility for all aspects of the Quality Assurance/Quality Control program, cost management, personnel management, and project management.

Quality Control Manager - The Quality Control Manager is responsible for adherence on a daily basis to all quality control procedures. This includes Good Laboratory Practices (GLP) compliance, maintenance of the laboratory Quality Assurance/Quality Control program, maintenance of all laboratory accreditation programs, establishment of QC procedures and control limits, and implementing corrective action reports and plans for analysis performed in the laboratory. Additionally, the Quality Assurance Manager reviews all project specific quality assurance plans (QAM).

Operations Manager - The Operations Manager is responsible daily for implementation of all quality control procedures. This includes maintaining all required training of analysts and staff, and verification of proper laboratory procedures.

Regulatory Compliance/Health and Safety Management

Laboratory Manager- The Laboratory Manager has overall responsibility for all aspects of the Regulatory Compliance program, and Health and Safety. This includes proper handling of waste, management of waste disposal, maintenance of the Health and Safety Plan, and obtaining all required licenses for laboratory operation. The Laboratory Manager is also responsible for compliance with all State and Federal Health and Safety regulations.

Client Services Management

Laboratory Manager - The Laboratory Manager has overall responsibility for all aspects of client services, including data management, project management, sample control, and invoicing/billing. The Laboratory Manager gives guidance for sample handling and storage prior to and after analysis.

Project Manager, PM- The Project Manager maintains project files and computer data entries after sample analysis. The project Manager also is responsible for interaction with clients to provide technical and sample analysis cost information, coordination with Technical Staff members to coordinate analysis types, project completion, method specific QC requirements, etc. Additionally, the Project Manager will handle review of all incoming work to determine if the laboratory has the facilities and resources

necessary to complete the job. They will notify the client in writing of any work that will be subcontracted to another laboratory. They will maintain all aspects of sample log in, adherence to proper Chain of Custody (COC) procedures, reconciliation of any COC discrepancies, and maintenance of the integrity of the samples received at the laboratory. The project manager is also responsible for dealing with client complaints. If the complaints cannot be addressed and rectified in a timely manner, the complaint must be brought to the attention of the Laboratory Manager. All communication with clients including complaints, COC reconciliation, follow up analysis requests or other pertinent information is documented in writing and included in the client file.

Technical Staff

The Technical Staff is responsible for the technical quality of the analyses being performed at the laboratory. They are required to adhere to QA procedures defined by the Quality Assurance Manual and Standard Operating Procedures, maintenance of the instruments, calibration of instruments, processing and validation of data at the bench level, reporting and correcting non-conformances, following up on corrective actions issued by the Quality Assurance Manager as necessary, and meeting client commitments to project schedules. Technical Staff members report to the Operations Manager and the Laboratory Director. The Technical Leads supervise day-to-day operations of each department in the laboratory. They are responsible for monitoring standards of performance in quality control, monitoring the validity of the analysis performed and data generated, ensuring adequate staffing of qualified personnel and provide educational training to the staff.

Minimum Education Requirements

Technical Directors

Chemical Analysis: Bachelor's degree in chemical, environmental, biological sciences, physical sciences, or engineering, with at least 24 college semester credit hours in chemistry and at least two years' experience in the environmental analysis of representative inorganic and organic analytes.

Microbiological or biological Analysis: Bachelor's degree in chemical, environmental, biological sciences, physical sciences or engineering, with at least 16 college semester credit hours in microbiology and biology and at least two years' experience in the environmental analysis of representative analytes.

Quality Assurance Manager

The Quality Assurance Manager shall: arrange for or conduct internal audits annually, notify laboratory management of deficiencies in the quality system, and monitor corrective actions. The Quality Assurance Manager must have documentation of training or experience in QA/QC procedures. The Quality Assurance Manager shall have direct access to the highest level of management at which decisions are made on laboratory policy or resources.

Technical Staff

The Laboratory Manager and/or Operations Manager will ensure new and current employees have a combination of education, prior experience, prior training, or completion of Specialty Analytical's employee validation process to ensure accurate and legally defensible data is produced by each employee.

Designated Alternates

In the event that key managers are not able to perform their duties the following alternates will be assigned to take over the responsibilities of the Lab Manager, QA Manager and Technical Director (Operations Manager).

The responsibilities of the Laboratory Manager will be assumed by the Operations Manager.

The responsibilities of the Quality Assurance Manager will be assumed by the Laboratory Manager.

The responsibilities of the Quality Assurance Manager and/or the Laboratory Manager will be assumed by the Operations Manager.

REVIEW OF REQUESTS, TENDERS AND CONTRACT

Senior staff (Project Manager, Business Development Manager or Lab Manager) will review all incoming work to determine if the laboratory has the facilities and resources necessary to complete the job.

They will also evaluate, in cooperation with the QA manager, the labs ability to perform the requested analyses, their accreditation status and ability to meet required detection limit. The client will be notified of the results of this review if it indicates any conflict, deficiency, lack of accreditation or inability of the lab to complete the requested work order.

They will also notify the client in writing of any work that will be subcontracted to another laboratory.

Any discrepancies between the request, tender or contract and the lab's ability to perform the work must be resolved before any work begins.

All communication with the client and notes on the review will be maintained in a client folder.

SUBCONTRACTING OF TESTS

When subcontracting analytical services, a senior staff member (Project Manager, Business Development Manager or Lab Manager) assures that the work to be performed is placed with an appropriately accredited laboratory or one that meets applicable statutory and regulatory requirements for performing the tests.

A list of subcontractors is maintained on the LIMS server.

Clients are notified in writing of our intent to subcontract any portion of their work order.
When possible, approval of the client to subcontract their work is obtained prior to implementation, preferably in writing.

The laboratory performing the subcontracted work is identified in the final report. The laboratory assumes responsibility to the client for the subcontractor's work, except in the case where a client or a regulating authority specified which subcontractor is to be used.

PURCHASING SERVICES AND SUPPLIES

The laboratory ensures that purchased supplies and services that affect the quality of environmental tests are of the required or specified quality by using approved suppliers Purchase and control of materials related to sample analysis, including glassware, reagents, calibration standards, reference materials, instruments, solvents, and suppliesmust be approved prior to utilization in the analysis of samples.

Specifications of Materials - Increased use of analytical instruments with higher sensitivity has increased the quality and productivity of laboratory analysis. It has also required a focus on higher quality materials utilized in the laboratory, as contamination levels acceptable on instruments 5 years ago now affect quality of results.

The Lab Manager or Project Manager reviews and approves the supplier of services and supplies and approves technical content of purchasing documents prior to ordering.

Evaluation of suppliers is accomplished by ensuring the supplier ships the product or material ordered and that the material is of the appropriate quality by signing packing slips or other supply receipt documents. The purchasing documents contain the data that adequately describe the services and supplies ordered.

Required quality of materials begins with requests from the Technical Staff. These are evaluated by the Quality Assurance Manager to assure the required grade is requested. Evaluations must be made as to types and levels of impurities, tolerances of the required method, and type of supply requested. Minimum requirements are for ACS grade reagents, NBS traceable materials, or other acceptable references.

Prior to use, standards are inspected, and tested, to assure conformance with requirements of the methods. Normally, this will be an analysis to verify lack of contamination of the standard, and verification of concentration against a second standard of equal quality, purchased from an independent source. For standards made in the laboratory, comparison against a second standard, made at a separate time, is required for concentration verification.

All materials are checked for suitability as blanks and compared to a reference source prior to use in the laboratory.

Standard Control

Control of materials is the responsibility of the Technical Staff and the Quality Assurance Manager. The identity, purity, shelf life, original source, tests conducted for verification, and expiration date are all maintained and tracked to assure use of only appropriate

standards during sample analysis.

SERVICE TO THE CLIENT

The laboratory collaborates with clients and/or their representatives in clarifying their requests and in monitoring of the laboratory performance related to their work. Each request is reviewed to determine the nature of the request and the laboratory's ability to comply with the request within the confines of prevailing statutes and/or regulations without risk to the confidentiality of other clients.

Client Confidentiality

Specialty Analytical adheres to strict client confidentiality policy. This includes distribution of analytical results and client privileged information only to approved parties. No information is provided without confirmation as to the recipient's identity. Phone numbers and email addresses are confirmed on the COC or verified independently.

All electronic data (storage or transmissions) are kept confidential, based on technology and laboratory limits, as required by client or regulation.

If necessary, client specific confidentiality agreements will be arranged.

PERSONNEL, TRAINING AND DATA INTEGRITY

“No quality assurance program, whether it be voluntary or imposed, can correct frequent mistakes and unreliable performance introduced by insufficient training, inadequate laboratory environment, and poor administrative practices.” William Horwitz, Quality Assurance Practices for Health Laboratories, p.547 APHA (1978)

Training Requirements

A vital requirement in quality assurance programs is adequate training of analysts prior to sample analysis being performed. All quality related activities performed in the laboratory will be accomplished by qualified, certified analysts with documented experience, degrees, training, and education. Activities that require certification include sample receiving, sample preparation, standard or reagent preparation, calibration of instruments or tools, data validation, and record/document management. All analysts will be trained in QA/QC principles in addition to technical methodology. This will allow achievement of skill levels that meet standards of quality.

Initial Orientation

All analysts undergo initial Quality Assurance training. Included is a discussion of their ethical and legal responsibilities including potential punishments and penalties for improper or illegal actions, general training requirements of the analysts and regulatory information. All analysts are required to read the Quality Assurance Manual (QAM). Analyst signatures are required to document their understanding and adherence to all SpecialtyAnalytical Quality Assurance policies.

Certification of Analysts

Specialty Analytical allows and requires only certified analysts to perform analytical tasks in the laboratory. To become certified, analysts must demonstrate training or education, knowledge of the method or task to be performed, and performance of the method requirements including an Initial Demonstration of Capability and Method Detection Limit studies. All must be performed prior to analysis of samples in the laboratory unless analyst is under direct supervision of a trained analyst.

To maintain certification the analyst must read each revision of the QAM. This must be documented in their analyst validation file.

Training and Experience - Analysts must meet the minimum requirements in education or experience (or both) prior to beginning the certification process. This can be education, work experience, technical training or other demonstrated knowledge of the job requirements and duties. Evidence of these qualifications will be maintained as training documents by the QAM. These records will document the quality assurance and technical training received by the employee, dates of completion of qualification or renewal, and approval or certification. Recertification will be required on an annual basis.

Other documents in the training files will include:

1) Personnel resumes

2) Quality Assurance Examinations

3) Attendance records at training sessions

4) Internal training certifications

5) Results of qualifying sample sets or performance samples

6) Records of observation of sample preparation procedures

7) Professional certificates or training

Technical Training

Prior to beginning sample analysis, analysts must demonstrate knowledge of method requirements by passing certification requirements. These procedures may be specific methods, or Laboratory Standard Operating Procedures.

This demonstration includes:

1) Reading the method from a primary source document (EPA or State method, Standard Methods, etc.)

2) Reading and review of the SOP, and identification of any method exception criteria applicable.

3) Acceptable preparation and analysis of a Calibration Curve (if required by method), which must meet statistical requirements of the specific method.

4) Acceptable preparation and analysis of an Initial Demonstration of Capability (IDOC) or Method Validation Study (MVS), which must meet statistical requirements of the method.

5) Acceptable preparation and analysis of a Method Detection Limit Study (MDL), which must meet statistical requirements of the method, and provide detection limits like EPA, method, or laboratory requirements, utilizing EPA 40 CFR 136 criteria for calculations.

6) Acceptable preparation and analysis of a single- or double-blind Performance Evaluation (PE) sample. This can be purchased or prepared in the laboratory as necessary.

The Laboratory Director, Technical Director and/or Quality Assurance Manager are responsible for initial and continuing certification of analysts.

Re-qualification is required if the analyst has not performed the method regularly during the previous year or fails to show acceptable performance on PE samples. Annual

certification is required, which will include demonstration of MDL and blind PE studies with passing results, another MVS or four consecutive passing Lab Control Standards. The analyst must read the current SOP and documentation is kept in the validation file. Failure to adequately complete annual certification will result in loss of certification and removal of the analyst from performance of the method until rectified.

Quality Assurance Training

General training in requirements of the Laboratory QAM is required of all Laboratory personnel. Training will address regulatory requirements, basic and advanced quality control concepts and practices, individual responsibilities of the technical staff, reporting and correction of non-conformance, performance audits, and laboratory documentation.

Data Integrity Training

Data integrity training shall be performed on an annual basis for all employees. In addition, all employees will be required to read and sign the Specialty Analytical Ethics Agreement during their initial employment orientation and annually as part of their formal ethics training.

Data integrity training shall be documented, and copies of signed ethics agreements will be kept in each employee's personnel file.

The data integrity training shall include reasons for ethical behavior, penalties for acting unethically, specific examples of fraudulent behaviors and improper procedures, the obligations of the company and of the employees to maintain an ethical workplace, requirements for reporting unethical behavior and mechanisms for confidential reporting of ethical breaches.

Code of Ethics

Specialty Analytical maintains a program of ethical conduct. All employees must adhere to the following statement of ethical compliance, ensuring that all data is of sound quality free from any intentional bias or fraudulent activity.

I understand the high ethical standards required of me regarding the duties I perform and the data I report in connection with my employment at Specialty Analytical.

I have read and understand the Laboratory Quality Assurance Manual and will adhere to all Quality Control policies and procedures including the Specialty Analytical Code of Ethics.

I agree to the following:

I will not intentionally report data values that are not the actual values observed or measured.

I will not intentionally report dates and times of data analysis that are not the actual times the data analysis was conducted.

I will not condone any accidental or intentional reporting of invalid data by other employees and immediately report the occurrence to the QA officer.

I will immediately report any accidental reporting of invalid data to the QA officer.

I will keep all client data and information confidential.

I understand that improper, unethical or illegal actions are not permitted and possible punishments and penalties include verbal or written reprimands, unpaid time off, dismissal and potentially legal action including incarceration.

In the event Specialty Analytical is sold or goes out of business the laboratory will notify all clients and insure the transfer of all records that are requested by the client. In the case of a change in ownership, the current ownership will provide in writing acceptance of all liability of the analysis, data and reports up until the time of the legal transfer of ownership. The buyer will provide, in writing, acceptance of all liability of the analysis, data, and reports after the time of legal transfer of ownership and that all records will be maintained for five years.

ACCOMMODATIONS AND ENVIRONMENTAL CONDITIONS

Facilities

Specialty Analytical occupies 7,900 square feet of space, located in Clackamas, Oregon. Office space occupies approximately 1,000 square feet of the space, with the remainder allocated to analytical laboratories and hazardous waste management facilities.

The laboratory is planned as a “single room” facility, allowing interaction between Technical Staff in various areas. This allows peer to peer interaction and provides a forum for discussion of technical facets of jobs currently in the analytical process.

The laboratory has also been designed to minimize contamination between incompatible activities.

The volatile laboratory is provided with an enclosed, positive pressure environment, allowing detection of contaminants at low levels that are also utilized in the laboratory (i.e. methylene chloride, acetone, etc.).

Additional space, providing “clean-room” environments, is used in the laboratory for analysis of metals and volatile organics at reporting levels below current environmental needs, such as semi-conductor, “pristine” water analysis for non-impacted streams and rivers, and other low level analysis types.

The environmental conditions of the laboratory such as temperature are maintained and monitored where applicable to the performance of the test methods

The laboratory has been designed to provide adequate and unencumbered bench space for each analysis. The aisles and workspaces have been designed to provide clearance for safe conduct of samples and equipment throughout the lab.

The facility is secure and access to the building is monitored at the front entrance. The building is equipped with smoke and burglar alarm systems.

EQUIPMENT

Specialty Analytical maintains analytical equipment inventories sufficient to perform analysis requested routinely by clients.

Up-to-date instructions on the use and maintenance of equipment (including any relevant manuals provided by the manufacturer of the equipment) are readily available for use by laboratory personnel.

All equipment is calibrated or checked before being placed into use to ensure that it meets laboratory specifications and the relevant standard specifications.

Test equipment, including hardware and software, are safeguarded from adjustments which would invalidate the test results measures by limiting access to the equipment and using password protection where possible.

Equipment that has been subject to overloading, mishandling, given suspect results, or been shown to be defective or outside specifications is taken out of service, isolated to prevent its use, or clearly labeled as being out of service until it has been shown to function properly. If it is shown that previous tests are affected, then procedures for non-conforming work are followed.

Each item of equipment and the software used for testing and significant to the results is uniquely identified and records of equipment and software are maintained. This information includes the following:

a) Identity of the equipment and its software.
b) Manufacturer's name, type identification, serial number or other unique identifier.
c) Checks that equipment complies with specifications of applicable tests
d) Current location.
e) Manufacturer's instructions if available or a reference to their location.
f) Dates, results and copies of reports and certificates of all calibrations, adjustments, acceptance criteria.
g) Maintenance plan where appropriate and maintenance carried out to date. Documentation on all routine and non-routine maintenance activities and reference material verifications.
h) Any damage, malfunction, modification, or repair to the equipment.
i) Date received and date placed into service (if available).

Maintenance for Analytical Equipment

All equipment is properly maintained, inspected, and cleaned.

Maintenance of analytical instruments and other equipment may include regularly scheduled preventive maintenance or maintenance on an as-needed basis due to instrument malfunction and is documented in Instrument Maintenance Logs, which become part of the laboratory's permanent records.
For specific equipment parameters refer to applicable SOP for the method.

Support Equipment

Support equipment includes, but is not limited to: balances, ovens, refrigerators, freezers, incubators, water baths, temperature measuring devices, volumetric dispensing devices, and thermal/pressure sample preparation devices.

Procedures for calibration and control of support equipment are contained in SOP# SPRO006 "Quality Control of Support Equipment".

All support equipment is maintained in proper working order and records are kept of all repair and maintenance activities, including service calls.

All raw data records are retained to document equipment performance. These records include logbooks, data sheets, or equipment computer files.

All support equipment is calibrated or verified over the entire range of use using NIST traceable references where available. The results of the calibration of support equipment are within specifications or (1) the equipment is removed from service until repaired, or (2) records are maintained of correction factors to correct all measurements.

Support equipment such as balances, ovens, refrigerators, freezers, and water baths are checked with a NIST traceable reference if available, to ensure they are operating within the expected range for the application for which the equipment is to be used. .

Glass micro-liter syringes have a certificate attesting to the established accuracy. If the certificate of accuracy for glass micro-liter syringes is not available, the accuracy of the syringe is demonstrated upon receipt and documented.

Equipment lists as of the date of the revision of the QAM are listed below.

Equipment List

Agilent 6890N GC
Gas Chromatograph with Flame Ionization Detector (FID) and Thermal Conductivity Detector (TCD). Utilized in the analysis of glycols, alcohols, specialty chemical analysis, and high purity solvent analysis. Purchased 2002.

Agilent 6890 Plus GC
Gas Chromatograph with Dual Flame Ionization Detectors. Equipped with dual split/splitless injectors. Utilized in the analysis of semi-volatile petroleum fractions. Purchased 1999.

Agilent 7890 GC, Tandem FID/PID, with Tekmar Stratum P&T and Archon Autosampler
Gas Chromatograph with Tandem FID/ PID detectors. Equipped with Low Dead Volume Injector. Used in the analysis of Volatile Petroleum and Aromatic Compounds. Purchased in 2008.

Agilent 7890 GC with Dual ECD detectors and CombiPal Autosampler
Gas Chromatograph with Dual ECD detectors. Equipped with Split/Splitless Injector, Deans Switch and CombiPal Autosampler. Used in the analysis of PCB's, Pesticides, Herbicides, and Low Level Chlorinated Phenols. Purchased 2008.

Agilent 6890 GC
Gas Chromatograph with Dual ECD detectors. Equipped with Split/Splitless Injector. Used in the analysis of PCB's. Purchased 1998.

Agilent 6890/5973 GC/MS
Gas Chromatograph/Mass Spectrometer used in the analysis of Semi-volatile compounds, including Polyaromatic Compounds and BNA's. The instrument includes Enviroquant Data System and 58,000 compound reference library for identification of unknown compounds. Purchased 1998.

Agilent Technologies 6890N GCMS 5975 inert source with 7873A Autosampler.
Gas Chromatograph/Mass Spectrometer used in the analysis of Semi-volatile compounds including Polyaromatic Hydrocarbons, BNA's and Organophosphorous Pesticides. The instrument includes an autosampler, Enviroquant Data System and a reference library with 115,000 compound reference spectra for identification of unknown compounds. Instrument can run in SIM (single ion monitoring)and full scan simultaneously. Purchased 2005.

Bruker 430-GC with CP-8400 Autosampler
Gas Chromatograph with FID detector. Equipped with Split/Splitless Injector. Used in the analysis of nonhalogenated volatiles. Purchased 2013.

Agilent 6890/5973 GC/MS with Tekmar Stratum P&T and EST Centurion Autosampler
Gas Chromatograph/Mass Spectrometer with Low Dead Volume Injector, and Purge and Trap Autosampler. Includes Enviroquant Data Station with 58,000 compound Reference Library. Used for the analysis of Volatile Compounds and Gasoline Additives. Purchased 1998/2008/2019.

Agilent 7890/5975C GC/MS
Gas Chromatograph/Mass Spectrometer used in the analysis of fixed gas compounds. The instrument includes Enviroquant Data System and 58,000 compound reference library for identification of unknown compounds. Purchased 2008

Agilent Technologies 1200 Series HPLC
High Performance Liquid Chromatography with binary gradient pump and diode array, multi-channel UV/VIS detection. Purchased 2007.

Thermo Instruments TSQ Quantam GC XLS
Thermo Instruments TSQ, triple-quad GC/MS/MS used for analysis of PCB Congeners and emerging contaminants.

Thermo Instruments Trace 1310/ISQ 7000
Thermo Instruments Gas Chromatograph/Mass Spectrometer used in the analysis of Semi-volatile compounds including Polyaromatic Hydrocarbons, BNA's and Organophosphorous Pesticides. Purchased 2019.

PerkinElmer NexIon 350X ICP-MS Spectrometer
Advanced ICP-MS with ESI SC2 DX Autosampler. Used in the analysis of Metals to Low ppt levels. Allows of positive determination of other metals without interference. Purchased 2014.

CETAC M-8000 Mercury System, with CETAC ASX-510
Cold Vapor Mercury analyzer for automated analysis of Hg in soil and water samples to Low PPT (parts per trillion) levels. Purchased in 2008.

Acumet 150 pH/Titration Controller
pH/Titration controller with Automatic Temperature Compensation. Used for pH analysis and Ion-Selective Electrode determinations. Purchased 1997.

Shimadzu TOC_L Total Organic Carbon Analyzer
TOC analyzer with ASI-L autosampler allowing aqueous samples to be analyzed. Purchased 2018.

OI Analytical Dual FS 3100 Flow Injection analyzer (FIA), OI 3090 autosampler
Flow Injection Analyzer used in the analysis of Total Cyanide, Available Cyanide using Ligand pretreatment, Free Cyanide, Ammonia, TKN, Nitrate, and Nitrite. Purchased 2010.

Thermo Genesys-20 UV/VIS Spectrometer
Variable wavelenth 325 to 1100nm for wet chemistry analysis. Purchased 2014.

Thermo Dionex ICS-2100
Ion Chromatograph with Autosampler, Membrane Suppression System, and Chromeleon Data System. Used for analysis of nutrients and wet chemistry parameters. Purchased 2011.

Thermo Dionex ICS-2000
Ion Chromatograph with Autosampler, Membrane Suppression System, and Chromeleon Data System. Used for analysis of cations and wet chemistry parameters. Purchased 2019.

Thermo Dionex ICS-2000
Ion Chromatograph with Autosampler, Membrane Suppression System, and Chromeleon Data System. Used for analysis of hexavalent chromium and wet chemistry parameters. Purchased 2019.

Denver Instrument Company A-160
Analytical balance with range of 0-160g.

Koehler 16200 PMCC Flash Point Tester
Pensky-Martens closed cup flash point tester. Purchased 1998.

Metrohm-888 Titrando
Automated titrator. Used for Karl Fisher, Alkalinity, Fluoride and Chloride by ISE.
Purchased 2018.

YSI-5100-115V
Benchtop dissolved oxygen meter. Used for BOD and dissolved oxygen testing.
Purchased 2007.

Fisher Isotemp Incubator (2)
Incubators set and monitored at a constant 35°C and 44.5°C for microbiological testing.
Purchased 2000.

Nor-Lake Scientific BOD Incubator
Incubator set and monitored at a constant 20.5°C for BOD testing.
Purchased 2007.

Parr 1341EB Bomb Calorimeter
Used to determine the calorific value of solid and liquid samples. Purchased 2002.

All data stations have received upgraded software or have GLP compliant systems in place to ensure proper reporting of data provided.

Equipment Calibration

Calibration is the standardization of a measurement or instrument by use of a standard or standards, or another instrument to adjust to any variances in accuracy of measurement.

Calibration Procedures

Calibration of instruments is performed using either Internal or External Standard Calibration. See glossary for definitions of calibration types. In each case, the following process occurs:

1) Calibration standards are prepared, as discussed above, to encompass the linear range of the instrument, or the working range for the analysis being performed. The low standard is at or below the reporting limit, unless defined in the method or SOP. Multiple points, covering the range selected, are analyzed. Calibration acceptance criteria are defined in each method or SOP, but typically require a 3 or 5 point calibration curve, which may include the blank for certain analysis. The curve must have a RSD better than or equal to that required in the method, or have a correlation coefficient (r) of 0.990 or greater (some analysis require 0.995 as defined in the SOP). If the RSD or r criteria are not met, the linear range is too large or re-calibration is required.

2) A second source, or a standard of known value, prepared or purchased from a source different from the calibration standards, must be analyzed. Its value must be within 20% of true value, or within the acceptance range given on the standard or within method acceptance criteria.

3) Continuing Calibration Verifications must be analyzed at the frequency required by the method or the SOP. At a minimum, one must be run prior to analyzing a sample batch, and must be re-analyzed in accordance with the method. With the exception of Internal Standard calibrations, they must be analyzed at the end of the run batch to verify instrument response stability. Internal Standard calibrations must be analyzed at least every 12 hours.

4) If CCV analysis does not meet method criteria, instrument maintenance may be performed. After maintenance, the CCV may be re-analyzed. If it fails to meet acceptance criteria again, the instrument must be calibrated again, and all samples analyzed from the last passing CCV must be re-analyzed.

TEST METHODS AND METHOD VALIDATION

Reference Methodologies

For organic and inorganic analysis, Specialty Analytical utilizes procedures provided by the EPA and state regulatory agencies. Wastewater samples are analyzed using methods from 40 CFR part 136, as published and revised in the Federal Register; Methods for Chemical Analysis of Water and Wastes, EPA-600/4-79-020; and Standard Methods for the Examination of Water and Wastewater, 19th and 20th Edition. Soil, water, and hazardous waste samples are analyzed using Test Methods for Evaluating Solid Waste (SW-846), 3rd Edition, and updates. Methods proposed for inclusion in SW-846 may be utilized by the laboratory upon notification and approval of the client. Drinking water samples will be analyzed using 40 CFR 136 methods, Methods for Chemical Analysis of Water and Wastes, Methods for the Determination of Organic Compounds in Drinking Water, and Standard Methods for the Examination of Water and Wastewater. Underground Storage Tank contamination will be analyzed using Northwest Total Petroleum Hydrocarbon Methods and ADEC Analytical Methods No. 101, 102, 103, Alaska Department of Environmental Conservation. Additional test methods for UST analysis may be utilized (EPA or ASTM methods). Air analysis will be accomplished using Methods for the Determination of Toxic Organic Compounds in Ambient Air, EPA/600/4-89/018 (and supplements); State of California Air Resources Board (CARB) Source Test Methods, 1990; and NIOSH Manual of Analytical Methods, US Dept. of Health and Human Services, NIOSH, Cincinnati, OH. and ASTM or OSHA methods upon determination of matrix, use of data, and requirements.

Method Exceptions

Any deviations from published methodologies must be documented. Changes that will be performed on a routine basis will be included in the Laboratory Standard Operating Procedure (SOP), approved, and signed by the Quality Assurance Manager, and the Laboratory Director.

Method Variances

Occasionally, due to matrix interference or other influences, typical methods may be ineffective in providing acceptable quality data. Parameters such as detection limits, precision, specificity of individual components, etc., may be affected. In order to provide data meeting client specifications, variances to the method may be proposed by the laboratory. In this case, the client's Project Manager will notify the client of the proposed variance, and a copy of the proposed variance may be sent to the client upon

request. This will describe the variance, document the reasons for the variance, and show that the conditions for the laboratory variance are similar to the expected conditions in the approved method.

Method Validation

A method is validated before it is put into use. Procedures for method validation are found in SOP# SPRO014 "Method, Calibration and Quality Control Parameter Development". In general, the procedure is summarized below.

Demonstration of Capability (DOC)

A Demonstration of Capability (DOC) is a procedure to establish the ability of the analyst to generate data of acceptable accuracy and precision.

The laboratory confirms that it is capable of generating data of acceptable accuracy and precision on all methods before employing them.

The IDOC is documented on the form in Appendix C of the 2016 NELAC Standard, and these completed forms are kept in the training files for each analyst.

A DOC is performed for each analyte whenever the method, analysts, analytes, or instrument type is changed.

The Technical Director certifies that technical staff members in their area of expertise are trained and authorized to perform all tests for which we are accredited by signing the DOC form.

The process for IDOC is documented in SOP# SPRO014.

On-Going (or Continued) Proficiency

After the demonstration of capability is completed, on-going proficiency is maintained and demonstrated at least annually through the analysis of either single-blind samples, performing another DOC, or use of four consecutive laboratory control samples compared to pre-determined acceptance limits for precision and accuracy. This is documented in the training file of each analyst.

Estimation of Uncertainty

ESTIMATION OF UNCERTAINTY consists of the sum (combining the components) of the uncertainties of the numerous steps of the analytical process, including, but not limited

to, sample plan variability, spatial and temporal sample variation, sample heterogeneity, calibration/calibration check variability, extraction variability, and weighing variability.

The laboratory estimates uncertainty using the standard deviation calculated from routine quality control samples.

Laboratory-Developed or Non-Standard Method Validation

Laboratory developed, modified standard methods, and non-standard methods require method validation.

Refer to SOP# SPRO014 “Method, Calibration and Quality Control Parameter Development” for specific procedures.

Method Validation is the confirmation by examination and the provision of objective evidence that the requirements for a specific intended use are fulfilled (NELAC2016).

Where applicable, the laboratory validates non-standard methods, laboratory-designed/developed methods, standard methods used outside their published scope, and amplifications and modifications of standard methods to confirm that the methods are fit for the intended use.

Initial Test Method Evaluation

For chemical analyses, the INITIAL TEST METHOD EVALUATION involves the determination of the Limit of Detection (LOD), confirmation of the Limit of Quantitation (LOQ), an evaluation of precision and bias, and an evaluation of the selectivity of the method.

Control of Data

All calculations and all relevant data are subject to appropriate checks in a systematic manner.

Commercial off-the-shelf software (e. g. word processing, database, and statisticalprograms) used within the designed application range is considered sufficiently validated when in-house programming is not used.

The laboratory assures that computers and software are protected, maintained, and secure through measures such as documentation, locked access, and control of the laboratory environment.

SOP # SPRO005 “Data Review and Approval, Report Generation, Data Storage and Handling” outlines procedure to insure that reported data are free from transcription and calculation errors, and that all quality control measures are reviewed and evaluated before data are reported.

SOP # SPRO005 also addresses manual calculations, including manual integrations.

The laboratory assures that computers, user-developed computer software, automated equipment, or microprocessors used for the acquisition, processing, recording, reporting, storage, or retrieval of environmental test data are:

a) documented in sufficient detail and validated as being adequate for use.
b) protected for integrity and confidentiality of data entry or collection, data storage, data transmission and data processing.
c) maintained to ensure proper functioning and are provided with the environmental and operating conditions necessary to maintain the integrity of environmental test data; and
d) held secure including the prevention of unauthorized access to, and the unauthorized amendment of, computer records.

Method Detection, Quantification, and Reporting Limits

Limit of Detection (LOD)

The LIMIT OF DETECTION (LOD) is an estimate of the minimum amount of a substance that an analytical process can reliably detect. An LOD is analyte-and matrix specific and may be laboratory-dependent. (NELAC Glossary 2016)

Limit of Quantitation (LOQ)

The LIMIT OF QUANTITATION (LOQ) is an estimate of the minimum amount of a substance that can be reported with a specified degree of confidence. (NELAC Glossary 2016)

Method Detection Limit (MDL)

Method Detection Limits (MDL) are defined as the minimum concentration of an analyte that can be identified, measured, and reported with 99% confidence that the analyte

concentration is greater than zero. It also refers to the minimum concentration of an analyte that a method can detect reliably in either a given matrix or blank.

MDL is calculated as:

$$MDL = t(99\%)\ (S)$$

where: t(99%) = students t value for 99% confidence limit, and n-1 degrees of freedom.

(S) = standard deviation of replicate analysis.

The standard deviation is based on a minimum of 7 replicate analysis.

Quantification Limits, either Practical Quantification Limits (PQL) or Limits of Quantification (LOQ) are determined after MDL calculations are performed. Limits of Quantification are defined as the concentration of an analyte where quantification within specified limits of accuracy can occur. This is normally a multiple of the MDL (2.5 to 10X) and is commonly rounded for ease of reporting and use. PQL's are "assumed" factors applied to the MDL's to simulate real world matrix effects. These vary by matrix from 10X for groundwater to 10,000X for sludge and Hazardous Waste.

Reporting Limit (RL)

Reporting Limits are the standard concentration level at which the laboratory provides "less than" or Not Detected (ND). These levels are above the MDL, and represent the concentration that meets the laboratories LOQ, and accuracy requirements. These limits are commonly grouped into ranges for ease of use of the data. Reporting limits are always above the MDL.

Specialty Analytical can provide MDL information upon request for any appropriate analyte.

Frequency of Method Detection Limit Studies

MDL determinations, and evaluation of LOQ and Reporting Limits, are performed at:

1) Initial validation of a method in the laboratory, or upon validation of a specific instrument in the laboratory.

2) Upon validation of an analyst utilizing that instrument or performing a specific method.

3) Anytime there is a change in instrument sensitivity (normally defined as 35% change in sensitivity or response of an instrument).

4) At a minimum, 2 MDLs per quarter that samples are analyzed.

These determinations can be simultaneous, i.e., both an analyst and an instrument can be validated at once. MDL's must be determined on each instrument the method will be performed on.

Procedures to be followed for the determination of MDL's are found in 40 CFR part 138 App. B. This procedure specifies the level for spiking MDL's at, and allows accurate determination of signal/noise levels. This determination is critical to accurate determination of MDL's. Spiking concentrations that are too large will lead to artificially low MDL's due to higher precision at levels above the signal noise. This is one of the most common reasons for non-reproducible MDL determinations.

Spiking levels for MDL's should be 0.5-5X the calculated MDL and should never exceed 10X the calculated MDL. Calculated MDL's exceeding the above guidelines need re-determinations prior to determinations of PQL or RL's.

Process for Performing Method Detection Limit Studies

1) Determine the required reporting limit - normally the regulatory defined limit.

2) Perform a minimum of 7 replicate analysis of blank matrix spiked at ½ to 1 of the required reporting limits and seven method blanks. These replicates must be prepared inat least three batches on three separate calendar dates and analyzed on three separatecalendar dates.

3) Using provided spread sheet, input, date run, spike level, parameter, and values for results of replicate analysis.

4) Evaluate spreadsheet for outliers in results, using Grubs criteria.

5) Remove any outliers that are not part of the statistical variance, calculate the standard deviation, MDL, and LOQ.

6) Evaluate signal to noise ratio, verify that the level is in the noise range approved, and that the calculated MDL is not less than 10 times the spike level or greater than the spike amount. This is to verify appropriate spike levels.

7) If s/n is not reached, re-analyze minimum 7 replicates at lower spiking level.

8) If value check or standard deviation is too high, perform 7 replicate analysis at higher spike level.

9) When all criteria are met, calculate MDL from spreadsheet using the Standard Deviation times the student t value for proper number of replicate analysis.

10) Calculate LOQ as at least 2.5 times MDL as calculated above.

11) Set reporting limits at or above the calculated LOQ.

12) In some cases, mostly for UST analysis, performing MDL determinations is not as important as precision studies at the reporting limits. In this case, performing the minimum 7 replicate analysis at the regulatory required reporting limit will replace the requirement for MDL determination by verifying precision and accuracy at the reporting limit. In no case will results be reported below this verified limit without performance of a MDL study.

MEASUREMENT TRACEABILITY

Calibration is the standardization of a measurement or instrument by use of a standard or standards, or another instrument to adjust to any variances in accuracy of measurement. Procedures for documentation of traceability of analytical standards are in SOP# SPRO004 “Standards and Reagents Documentation”.

Analytical Standards

Analytical Standards are utilized for calibration and preparation of quality control samples. These standards must be traceable to standard reference materials. Acceptable materials are:

1) EPA CRADA traceable materials

2) NIST reference standards

3) Standards made by vendors traceable to one of the above.

If none of the above are available, duplicate analysis of standards prepared separately is acceptable.

All standards must be stored under conditions that provide the largest amount of protection from deterioration and change.

Traceability of the standards is established through statistical evaluation of the control sample, or analytical standard relative to NIST or CRADA reference materials.

Criteria for acceptability of a new standard solution is: Triplicate analysis of a standard yields a Relative Percent Difference (RPD) of less than 15% when quantified against a SRM from one of the above sources.

Expiration Dates of Standards

All standards obtained from commercial sources as well as SRM's are dated upon receipt. This will be written in indelible ink upon the container received, as well as any container the standard may be transferred to. This information will also be recorded in the standards logbook for that department. The expiration date of the standard is also noted and recorded as above. If no information as to expiration date is available, establishment of an expiration date based upon working knowledge of the standard is acceptable.

Standards must be protected from degradation, deterioration, and contamination by strict adherence to proper storage and handling procedures.

Stock and working standards are prepared fresh as required to maintain stability and are checked prior to every use for signs of contamination or deterioration (color formation, precipitation, concentration changes, additional peaks, or leakage).

Standards prepared as stock or working standards are labeled with name of compound or mixture, concentration, solvent, date, preparer, and expiration date. This information will also be recorded in the working or stock standards log for that department. All requirements for storage of the primary standard will be adhered to with the working or stock standard.

A LCS or check standard from a certified, independent source will be used to monitor for degradation or concentration changes. Results should fall within the 95% confidence limit of the given target range values.

Inorganic Standards

Stock standards shall conform to the expiration date of the commercial standard. These expiration dates are checked daily and recorded on the daily bench sheet.

Working Standards are prepared at a minimum of every 6 months, when standards show signs of degradation, or when other factors require preparation.

Organic Standards

Liquid or Solid stock standard holding times are the manufacturers recommendation, or 6 months if no expiration date is available.

Commercially prepared standard holding time is the manufacturers recommendation, or 1 year if no expiration date is available.

Standard Preparation Equipment

Only Class A volumetric glassware, certified or calibrated pipets, and ACS grade solvents at a minimum, will be used for dilutions of primary standards.

Guidelines for Standard Preparation

1) Laboratory Technicians or Chemist experienced in calibration and use of analytical measurement tools are assigned to standard preparation.

2) Analytical reagent grade materials, in solution or neat, are utilized in the preparation of analytical and control standards. When possible, guaranteed assay materials with supporting documentation (chromatograms, assay results, etc.) are requested and filed with QAM.

3) Solvents utilized for dilution of standards are checked for contamination.

4) Analytical measuring tools (balances, pipettes, syringes, etc.) will be calibrated prior to use to assure accurate and acceptable measurements.

5) All data generated is documented immediately in the Lab Information Management System (LIMS) and in standard preparation logbooks for each department.

6) A sequential log number is assigned to the newly prepared standard. This number must be noted in the standard log, in preparation books, and on any storage, vessels used for any aliquot.

7) Standards are analyzed prior to use on the instrument the standard is to be used on.

8) A standard obtained from another approved source is used as the traceability standard.

9) To maximize the precision of the result, both the new standard and the reference standard are analyzed on the same instrument, within the same batch, and as close in time as feasible.

10) Once the standard has passed QC evaluation, it is stored at the required temperature and condition until required for use.

QUALITY OF TEST RESULTS

Quality Assurance Objectives

The purpose of Data Quality Objectives (DQO's) is to establish specific targets for accuracy (precision and bias). The initial DQO's for a method is to achieve improved precision and accuracy levels than specified in the method reference. Once sufficient data is produced, control charts are established for each analytical parameter appropriate, and the statistics produced are used as the new DQO's for the method, i.e., the mean and standard deviations become the DQO's for that test, provided they are better than the initial DQO's.

Specialty Analytical's Quality Assurance Manual has the objectives to verify the productionof high quality, defensible, and cost-effective data to its clients. While specific quality assurance plans and items will be required for projects, general guidelines are:

1) Data should be accurate within method specifications and agree within limits with reference values.

2) Data should agree among individual measurements under similar conditions (comparability).

3) Data should be complete in terms of valid data produced as compared to planned (completeness).

4) Data should be comparable to prior data for evaluation, whether produced by Specialty Analytical or not.

5) Data should be representative of the population of parameters measured.

6) Data should be reproducible under similar conditions at any location.

7) Quality Assurance programs should continually upgrade the quality of the laboratory performance.

The goal of the laboratory QAM is to produce data of defined and consistent quality. Guidelines are provided for the assessment and reporting of data quality parameters, and for the incorporation of such assessments into major databases for evaluation and tracking.

Control of sample receiving, login of samples, and tracking of samples throughout the analytical process is maintained to ensure the integrity of the sample. Documentation of instrument performance and preventative maintenance is maintained to allow review of factors effecting data produced during sample analysis.

Determinations of the quality of analytical work through analysis of quality control reference samples, duplicate analysis, matrix spike/spike duplicate, and laboratory control samples are routinely performed, in compliance with method or laboratory requirements.

Maintaining accreditation through efficient state sponsored programs including a proficiency testing program and external audits.

Quality Control Definitions

Accuracy - Accuracy is evaluated in two fashions, either as measurement accuracy or analytical accuracy. Measurement accuracy is measured by analysis of QC reference materials, and analytical accuracy is determined by spiking known concentrations into samples prior to preparation and analysis. Measurement accuracy is independent of matrix effects, while analytical accuracy includes any effects from sample matrices upon the recoveries of the analytes. Accuracy is affected by both systematic and random errors.

Measurement data is evaluated by comparing the percent recoveries of QC reference materials of known and evaluated concentration, independent of routine calibration. These reference materials can be used as prepared or diluted into an inert matrix. Control limits are determined by control charting the recoveries and performing statistical analysis upon the results. Laboratory Control Samples (blank spikes) are also utilized to evaluate laboratory performance of methods. This is also independent of matrix effects.

Analytical accuracy is evaluated by comparing the percent recoveries of analytes, which have been added to samples at a known concentration. This is evaluated using matrix spikes and spike duplicate samples. Spike samples are analyzed at 5% frequency (1 in 20 samples), or per analytical batch, whichever is more frequent. Recoveries are assessed to determine the method efficiency and matrix interference effects upon analyte recovery.

The equation used to calculate percent recovery of an analyte is:

$$\% \text{ Recovery} = \frac{\text{(Spike Sample Result - Sample Result)}}{\text{Amount of Sample Spiked}} \times 100$$

Analytical precision is evaluated as the percentage difference between results of matrix spike and spike duplicate results. Results of sample and duplicate results can additionally be used for evaluation of precision, depending upon method requirements. Relative percent difference is used for this evaluation, and is calculated using the formula:

$$\text{RPD} = \frac{\text{(MS or original result) - (MSD or duplicate result)}}{\text{Mean of MS (or original) and MSD (or duplicate) results}} \times 100$$

Matrix spike/spike duplicate analysis will be performed at a minimum of 5% frequency (1 in 20), or as required by method specifications. Control charts can be utilized to determine the statistical limits for control limits. Please see the section on control charting in the QAM for details on the statistical analysis.

Representativeness of data produced by the laboratory shall be representative of the overall population of samples collected and analyzed. It will be representative of the laboratory database of accuracy and precision measurements of that parameter, matrix, and analytical method. If the results are reproducible, then the data can be stated to represent the environmental condition at the site of sampling.

Precision is a qualitative term used to denote the scatter of results. Precision is said to improve as the scatter among results becomes smaller. Random error can be referred to as imprecision. This parameter is usually measured as standard deviation.

Bias reflects the inaccuracy of an analytical result due to systematic error.

The most significant aspect of representativeness is sampling methodologies and programs. While representativeness expresses the degree to which sample data accurately represent a characteristic of a population, parameter variations at sampling point, or environmental condition, it is a qualitative parameter which depends on the proper design of a sampling program. Sampling locations and number of samples collected must be selected carefully to provide sufficient information and accurate results in identification of site conditions.

Representativeness is evaluated by describing sampling techniques and the rationale used to select sampling locations. These locations can be biased (based on information from existing data points, instrument surveys, or site conditions), or unbiased. Regardless of approach, the decision-making process to determine sampling locations must be specified. If sampling grids are used, it should be shown on a site map. The type of sample (grab or composite), as well as the SOP for sampling, should be specified.

Assessments of representativeness are determined by use of collected samples. These are samples collected so they are equally representative of a given point in space and time. Thus, information on both precision and representativeness are provided.

Comparability is achieved by using standardized sampling and analysis techniques. The goal is to provide data that is reproducible under similar conditions, whether generated by Specialty Analytical, or other firms. Comparability can be verified by using split samples, and having analysis performed at several laboratories using the same methods. Intra-laboratory comparability can be verified by comparisons of historical data from the site, if available. Evidence of comparability can be found by results of inter-laboratory Performance Evaluation (PE) samples administered by a NELAP certified private vendor.

Quality Control Samples

Various types of quality control samples generated and used by Specialty Analytical in addition to samples submitted by clients are described in this section.

Matrix Spike/Matrix Spike Duplicate (MS/MSD)

MS/MSD is used to check for precision and accuracy. These are replicate portions of a sample spiked with a known amount of a compound (or compounds) of interest, that are taken through the entire analytical process (sample preparation, analysis, data reduction). These are performed on each matrix analyzed and are performed at a frequency of 1 MS/MSD per analytical batch (10 or 20 samples, depending on the method). The sample analysis process and the spiked sample process differ only in the

adding of known amounts of compounds to the replicate MS/MSD samples. The quantity of spike varies according to the linear range and detection limit of the method.

Should the native sample contain the compound of interest also, the concentration of that analyte in the native sample is subtracted from the value of the spiked sample, and the percent recovery (%R) is calculated using:

$$\%R = \frac{\text{(Spike Sample Result- Sample Result)}}{\text{Spike Added}} \times 100$$

The sample value may be outside the linear range of the instrument utilized in the analysis. In this instance, there is no way to determine the concentration of the analytes prior to spiking the samples. Due to dilution required to analyze the sample accurately and within the linear range of the instrument, the spike added may be diluted to below reporting limits. In this case, no information on spike recovery can be determined. In this case, the use of the Laboratory Control Standard (LCS or Blank Spike) is used to determine that the analytical process is in control and the data is flagged accordingly.

The calculated percent recoveries from the above calculation are used to assess the data precision as Relative Percent Difference (RPD). This is calculated using:

$$\%RSD = \frac{\text{(MS Result - MSD Result)}}{\text{Mean of MS and MSD Result}} \times 100$$

where MS and MSD Results are the % recoveries from the analysis.

In cases where the percent recoveries or RPD values fall outside acceptance limits, case narratives, data qualifier flags, or corrective actions may be required.

Method Blanks and Reagent Blanks

Method blanks are analyzed for each matrix type and batch of analytical samples (up to 20 samples). This must be prepared at the same time as the samples in the batch. An aliquot equal to the volume or weight of sample used in sample preparation is used for the method blank analysis. The method blank is treated identically to a sample, being taken through the same sample preparation and analysis. The method blank must be free of substances and interference at the reporting limit or must have less than 10 times the contamination level found in the sample. Method blanks are typically run immediately after the CVS (CCC) standard to show system cleanliness and lack of carry-over on the instrument.

Holding Blanks

Holding blanks are aliquots of VOA free materials (normally water or methanol) that are stored in headspace free VOA vials in the refrigerator volatile samples are stored in. It is analyzed identically to samples to determine sample cross contamination during sample storage. Presence of volatile compounds (especially methylene chloride or acetone) indicates some migration of compounds through the septum seals of the VOA vials. Holding blanks are analyzed twice monthly as part of routine quality control, upon client request, or upon some indication of sample contamination discovered by the laboratory.

Calibration or System Blank

A calibration blank is used to establish the analytical curve “zero” point in some methods, taking background responses into account. It is identical to the matrix being analyzed for in the method. System blanks are utilized to check for system contamination, after standard analysis or high-level samples have been analyzed to verify system cleanliness.

Filtration Blank
A filtration blank is used to establish that the specific Lot# filters are free of contamination. One filtration blank is run every 24 hours of analysis per Lot# and is batched with the sampled analyzed.

Laboratory Control Samples

Sometimes called a blank spike, the Laboratory Control Sample (LCS) is a standard of known concentration spiked into a consistent matrix (water or sand), prepared and analyzed identically to samples, and is part of the QC for an analytical batch. The spikes are compounds representing the analytes to be quantified by the method.

LCS's provide information on the accuracy of the analytical batch, and establish the efficiency of the sample extraction, digestion (if part of the method), and analysis. The LCS is also used to verify matrix interference if MS/MSD recovery or precision control limits are exceeded, as it is usually an identical spike in concentration and analytes to the MS and MSD but is not impacted by sample specific matrix effects.

Surrogate Spikes

For chromatographic analysis (GC, GC/MS, LC), surrogates are used to determine acceptable extraction and analysis efficiencies. Unlike spikes and LCS samples, surrogates are added to each sample in the analytical batch, and thus provide an extraction and instrumental analytical efficiency for each sample.

Surrogates are added prior to extraction of the sample and are spiked directly upon the sample being analyzed. Surrogates are analyte compound substitutes, i.e. they are not specifically requested analytes of interest, and are normally not naturally occurring compounds. Surrogates should not interfere with the quantification and identification of the target compounds and must be chemically similar to the compounds of interest.

Surrogate spike calculations for recovery are performed on all samples, blanks, LCS and MS/MSD.

Control limits are established for surrogates. Exceeding the control limits requires re-analysis to verify any matrix effects upon the surrogate, which may indicate effects upon the target compounds.

Data Review

During data review, data is compared to historical data, review of sample preparation and data from sample analysis is evaluated. Corrective actions are minimized by implementation of routine system controls. Analysts are provided with, and comply with, specific criteria for procedure, operation, and measurement systems.

Data review contains several levels. Analysts are responsible for initial data review, check compliance with QC criteria, and checking instrument calibration, blank reviews, review of raw data (including peak identification), and calculations of raw data. Analysts review 100% of data, whether hand or instrumental calculations are performed. If for any reason the sample(s) were re-analyzed/ re-run or re-extracted, internal comments are recorded within the Khemia Omega 12 Lims Example: "RR1 Failing QC" or "RE Failing QC"

Upon completion of this step, secondary review is performed at a minimum of 20%.

This step emphasizes data acceptability relative to QC indicators and acceptance criteria, and on accuracy of data summaries. A final review is performed for data consistency and completeness.

Relative Error

All calibration curves shall be evaluated for %RE by the analyst before running samples. All %RE results shall be submitted with all calibration curves for QA review in one of the following:

1) Printed out with raw data as generated by the instrument software.
2) Provided in an Excel sheet as generated by the instrument software.
3) Inputted into the LIMS

Relative Error is calculated using the following equation:

$$\% \text{ Relative Error} = \frac{x - x_i}{x_i} \times 100$$

x_i= True value for the calibration standard
x= Measured concentration of the calibration standard

This calculation shall be performed for a minimum of two calibration levels: the standard at or near the mid-point of the initial calibration and the standard at the lowest level.

PERCENT RELATIVE ERROR ACCEPTANCE CRITERIA:

NWTPH: +/-20%

Organics by 8000 & 600: Percent error between the calculated and expe4cted amounts of an analyte should be ≤ 30% for all standards. For some data uses, ≤50% may be acceptable for the lowest calibration point

Inorganics: +/- 25%

DOCUMENT MANAGEMENT

This Section describes procedures for document management, which includes controlling, distributing, reviewing, and accepting modifications. The purpose of document management is to preclude the use of invalid and/or obsolete documents.

Documentation

The laboratory manages three types of documents, 1) controlled, 2) approved, and 3) obsolete.

A CONTROLLED DOCUMENT is one that is uniquely identified, issued, tracked, and kept current as part of the quality system.

APPROVED means reviewed, and either signed and dated, or acknowledged in writing or secure electronic means by the issuing authority(ies).

OBSOLETE DOCUMENTS are documents that have been superseded by more recent versions.

All documents are dated, and a log is kept by the Technical Director to indicate the timeperiod during which the procedure or document was in force.

Document Control and Distribution

The Quality Assurance Manual (QAM) is maintained as a controlled document internally. Assuch all hard copies are uniquely identified with date of issue, revision identification, page number, the total number of pages, and the signatures of the issuing authority.

The QAM controls the QAM. Document control numbers unique to each copy areincluded and a list of where the copies are located is kept by the QAM.

External copies of the QAM may be issued as uncontrolled copies. All hardcopies andelectronic copies of the QAM that are issued as uncontrolled copies will be clearly marked as "uncontrolled" and will not be issued document control numbers.

All laboratory personnel are required to read any revisions of the QAM. Signed documentation is placed in their training file to indicate that they read and understood the revision.

Laboratory Standard Operating Procedures (SOPs) are maintained as approved documents.

The Technical Director controls revision, storage, and availability of all SOPs.

All original documents are signed and dated by the Laboratory Director and QAM and scanned into read only electronic (.pdf) files. These files are placed on the laboratory LIMS server and are made accessible by all staff members. They are also printed and kept in binders in each department.

It is the responsibility of each employee to verify that any SOP in their possession, either paper copy or electronic copy is the most current revision available on the LIMS Server.

Any obsolete copies in their possession should be handed back to management and replacedwith the most current version.

The Technical Director will maintain master copies of all SOPs in WORD format in a secure location or file folder.

Suggested revisions to electronic documents are presented to the Technical Director for review and approval. The Technical Director will issue a draft copy of the SOP file in WORD to facilitate the revision. Changes to electronic documents are approved through electronic means (such as email, change tracking functions, or memoranda).

Where practicable, the altered text or new text in the draft is identified during the revision or review process to provide for easy identification of the modifications.

Once the revision is complete an original paper copy with signature page will be approved and scanned into the LIMS Server as a read-only(.pdf) file.

Electronic copies of obsolete SOPs will be maintained in an archive folder for future reference.

The QAM and SOPs are reviewed annually to ensure their contents are suitable and incompliance with the current quality systems requirements, and accurately describe current operations.

STANDARD OPERATING PROCEDURES (SOPs) are used to ensure consistency of application of common procedures, are written procedures that describe in detail how to accurately reproduce laboratory processes and are of two types: 1) test method SOPs, which have specifically required details, and 2) general use SOPs which document the more general organizational procedures. SOPs do not have to be formal documents with predefined section headings and contents. They can be fewer formal descriptions of procedures described in the QualityManual or other documents. Copies of all SOPs are accessible to all personnel.

Each SOP indicates the effective date, the revision number, and the signature(s) of the QAM and Lab Director.

Test Method SOPs

The laboratory has SOPs for all test methods within its scope and for procedures that are part of the Quality System that accurately reflect how the analytical process is performed. Where equipment manuals or published methods accurately reflect

laboratory procedures in detail, a separate SOP is not required.

Any deviation from a test method is documented, including both a description of the change made and a technical justification. The deviation from a test method is reported to the client.

Each Test Method SOP includes or references (as applicable) the following:

a) identification of the test method;
b) applicable matrix or matrices;
c) detection limit;
d) scope and application, including components to be analyzed;
e) summary of the test method;
f) definitions;
g) interferences;
h) safety;
i) equipment and supplies;
j) reagents and standards;
k) sample collection, preservation, shipment and storage;
l) quality control, including acceptance criteria (5.4.10.6);
m) calibration and standardization;
n) procedure;
o) data analysis and calculations;
p) method performance;
q) pollution prevention;
r) data assessment and acceptance criteria for quality control measures;
s) corrective actions for out-of-control ;
t) contingencies for handling out-of-control or unacceptable data;
u) waste management;
v) references; and,
w) any tables, diagrams, flowcharts and validation data.

CONTROL OF RECORDS

The purpose of records management is to ensure all records for the laboratory are accountable and traceable, legible, retrievable, and protected from deterioration or damage including COC records, logbooks, graphs, raw data, and other required items for verification of data.

The laboratory maintains a record management system for control of laboratory notebooks, instrument logbooks, standards logbooks, and records for data reduction, validation, storage, and reporting.

Archived information and access logs are protected against fire, theft, loss, environmental deterioration, vermin, and in the case of electronic records, electronic or magnetic sources.

In the event that the laboratory transfers ownership or goes out of business, records are maintained or transferred according to the clients' instructions.

Documentation of client samples begins with Project Management, where samples are received, assigned unique laboratory numbers, and reconciled with COC forms. Each shipment has a unique number (laboratory job number) that is carried throughout the sample analysis and reporting process.

The information about sample receipt is contained in the Sample Receipt Checklist, which contains information on sample receipt, matrix, containers, and documentation.

Additional required information is contained in the instrument maintenance logs, standard logbooks, chemist's notebooks and bench sheets, and instrument run logs.

All electronic records are backed-up periodically. Access to protected records is limited to laboratory management or their designees to prevent unauthorized access or amendment.

Procedures for storage and handling of records and data can be found in SOP# SPRO005 "Data Storage and Handling".

All sample and analytical data is retained for a minimum of five years.

Logbook Requirements

Requirements for logbooks used in the laboratory are as follows:

1) Bound logbooks with numbered pages are preferred over loose sheets. Sheets must be affixed to a logbook page, signed, and dated both on the sheet and running onto the logbook.

2) Only assigned logbooks can be used for record keeping. Contract required logs will be maintained as part of contract requirement in project files.

3) All writing must be legible and in black ink. Corrections can only be made by drawing a single line through the entry, initialing, and dating the change.

4) Complete information must be entered.

5) Any data invalidated must be noted as to the cause.

6) All data relevant to the information must be included. This may include reagent lots, preparation information, instrument conditions, etc.

7) When work is continued to another log, the number of the first log is entered on the first page of the second log.

Logbooks must contain traceability measures when applicable including but not limited to:

1) Analytical Batch ID.

2) Thermometer & balance IDs

3) Standard, reagent, surrogate, and chemical IDs.

4) Water bath & incubator IDs.

CONTROL OF NON-CONFORMING WORK

Out of Control Events

Out of control events are defined as any occurrence failing to meet pre-established criteria.

Non-conformances

A non-conformance is a deficiency in characteristic, documentation, or procedure sufficient to make the quality indeterminate or unacceptable. An out-of-control event is a sub-category of non-conformance.

When either a non-conformance or out of control event occurs, it will be categorized as either a deficiency, or an observation.

Deficiencies are defined when a specific requirement of a program, process, or procedure has been violated.

Observations are defined when there is recognition of an activity or action that may be improved but is not in violation of a specific requirement. Observations may degrade to a deficiency if not corrected.

Criteria for Out-Of-Control Events

Factors that affect data quality (failing calibration, improper sample storage, improper preservation of samples, failure to meet hold time, inadequate record keeping, etc.) require investigation and corrective actions. Some factors can be assessed through use of control charts. Control charts can yield information on trends, biases, and shifts in the operation of the analytical system.

The detection of one of these conditions is an indication the analytical system is out-of-control. The out-of-control value is placed on a control chart, circled, and documented using a non-conformance form. The Laboratory QAM, Technical Director and/or Laboratory Director are notified, and determine whether the incident is random,or reflects a process that is out-of-control.

Control Charts

Measurement system performance can be demonstrated by the measurement of homogeneous and stable control systems. The data compiled is plotted, and a control chart generated to indicate whether the system is providing process control. It is also used to notify the laboratory of developing method performance issues, by identifying systematic errors, drifts, or other problems.

Use of control charts are:

1) Provide graphical assessment of accuracy and precision for the measurement of each analyte.

2) Allow observations of recovery trends for individual analytes and allow self-evaluation of analytical output.

3) Provide assessments of analytical capability of the technical staff.

A system must be verified as being in control, in order to be maintained in control. A system is not in control if it is observed to produce unexpected data more than once

every 20-25 runs. Control limits usually become tighter once a process is under a controlled protocol.

Control Chart Types

Control charts that are used to monitor the performance of the methods are:

1) Surrogate Percent Recoveries

2) MS/MSD Recoveries per matrix type

3) LCS Percent Recoveries

Preparation of Control Charts

For methods which quality control acceptance limits are not specifically determined (either internally or by QAM), control charts are used to evaluate lab performance. Control charts, when used, will be prepared for each analysis and matrix type separately. The charts will consist of a centerline, two warning limits, and two control limits. This process is described in HAZWRAP DOE/HWP-65/R1 and EPA Handbook on Analytical Quality Control in Water and Wastewater Laboratories. Control charts require a minimum of 20 points to begin charting, so method recommended recoveries shall be used until sufficient data points have been gathered.

Interpretation of Control Charts

Representative concentrations of compounds as described in the method or SOP are spiked into the required percentage of samples (method required frequency for LCS and MS/MSD, all samples for surrogates when utilized). Recovery information is gathered for these points. The mean (x) and standard deviation (s) is calculated, and from this information warning and control limits are calculated. These are:

1) Warning limits are defined as x +/- 2s

2) Control limits are defined as x +/- 3s.

The %R of each surrogate recovery, MS/MSD recovery, or QC sample is plotted on a control chart and compared with the control and warning limits.

Control charts can also be used to evaluate data precision (RPD) on duplicate or spike duplicate samples. The calculations for warning and control limits are the same as above.

Any of the following incidents causes an out-of-control corrective action to be performed:

1) One or more points outside the control limit (3s).

2) Two or more consecutive points outside the warning limit (2s).

3) Seven or more consecutive points on same side of mean recovery bar (x), indicating data trends or shifts.

4) Cyclic or non-random patterns occurring in the data points plotted.

5) Runs of 6 or more points in the same direction.

Procedures for Corrective Action

When an out of control event occurs, the analyst will consult the appropriate Method SOP for proper corrective action. If corrective action is not possible (i.e. insufficient sample volume for reanalysis or suspect matrix interference) and the data is out of control or nonconforming, the analyst writes a case narrative describing the conditions of data analysis. A form is filled out and the situation is brought to the attention of the Laboratory Director, Technical Director or QAM for evaluation. The QAM will determine the validity of the data and the Laboratory Director or Project Manager will contact the client to determine if data is acceptable to the client or additional action is necessary (i.e. re-sampling). All conversations with the client are documented. A case narrative is written to describe the nonconformity and is included with the final report. All associated data must be flagged with a CN and all QC parameters that are out of control and associated with the data are flagged and included in the final report.

The Technical Director is responsible for review of corrective action reports and will suggest improvements, alternative approaches, and procedures where needed, documenting the actions taken in response to the nonconformity.

The discovery of a non-conformance for results that have already been reported to the client must be immediately evaluated by the QAM for significance of the non-conformance, its acceptability to the client, and determination of the appropriate corrective action.

The QAM will also determine if the nonconformance will trigger a data recall.

Exceptionally Permitting Departures from Documented Policies and Procedures

Under certain circumstances the Laboratory Director or the QAM may permit departures from the documented policies or procedures. These departures are documented by the QAM, Laboratory Director, or Project Management and communicated to the client via a report case narrative.

Procedures for Halting Analysis

When the analytical system is out-of-control corrective action processes are initiated by the analyst and QAM.

If the problem is instrumental or specific to a particular batch analysis, any samples prepared after the event are re-processed after re-calibration, providing holding times are met.

If the batch or sample is still out-of-control after re-analysis, all method related activities are stopped. A detailed investigation to the causes is conducted, and any process problems are corrected. All actions taken are documented in the project file related to the sample(s).

Only after the corrective action is performed is the analysis placed on-line at the laboratory again.

CORRECTIVE ACTION

Need for corrective actions come from several sources, equipment malfunction, failure of QA/QC checks, follow-up of performance or system audit findings, and QA non-compliance.

When instrumental or analytical methods fail QA/QC, the failure will be brought immediately to the attention of the Technical Director, QAM and/or the Laboratory Director. Corrective measures taken will depend on the analysis, the type of error, and the extent of the failure. If an analysis fails batch QA/QC (MS, LCS, Blank or duplicate) criteria and the batch cannot be reanalyzed (due to insufficient sample) or matrix interference is suspect all failures will be flagged as out of control and reported to the end data user.

Corrective actions can be extensive or simple. Each case requiring a corrective action will be determined on a specific basis.

Equipment malfunctions will be handled by segregating the equipment, until repairs can be performed, and precision and accuracy on the instrument restored.

Failure of performance audits will require Specialty Analytical to identify the problem in analysis or document traceability. A step-by-step analysis of the root causes of the analytical problem is instituted, and corrective actions are taken.

All incidents of QA failure and corrective actions will be documented fully. Any actions will be taken quickly, and the process will include actions to prevent re-occurrences of the event.

PREVENTIVE ACTION

Preventive action, rather than corrective action, aims at minimizing or eliminating inferior data quality or other non-conformance through scheduled maintenance and review, before the non-conformance occurs.

Preventive action includes, but is not limited to, review of QC data to identify quality trends, discussion of quality trends and issues at daily staff meetings, annual budget reviews and annual managerial reviews.

Routine and periodic maintenance schedules for equipment and instruments are followed in the lab.

All employees are encouraged to make suggestions to improve processes throughout the lab and are empowered by management to implement those improvements.

Trend analysis is used throughout the laboratory to identify systematic and systemic issues that may affect the quality of results or efficiency of processes. Examples of types of systemic issues and measurement tool and useful records that are employed in trend analysis include but are not limited to audit reports, control charts, maintenance records, LIMS reports, COC discrepancies, hold time violations, on time percentage, calibration reports and method QC failures.

REPORTING OF RESULTS AND DATA VALIDATION

Reporting of Results

The result of each test carried out is reported accurately, clearly, unambiguously, and objectively and complies with all specific instructions contained in the test method.

Data are reported without qualification if they are greater than the lowest calibration standard, lower than the highest calibration standard, and without compromised sample or method integrity.

Test Reports

The report format has been designed to accommodate each type of test performed and to minimize the potential for misunderstanding or misuse.

Each test report generated contains the following information (unless not required by the client):

a) a title
b) the name and address of the laboratory, the location of the laboratory if different from the address, and the phone number and name of a contact person;
c) unique identification of the test report, such as a serial number, on each page and a pagination system that ensures that each page is recognized as part of the test report and a clear identification of the end of the report, such as 3 of 10;
d) the name and address of the client if applicable;
e) the identification of the test method used;
f) an unambiguous identification of the sample(s), including the client identification code;
g) the date of sample receipt when it is critical to the validity and application of the results, date and time of sample collection, dates the tests were performed, the time of sample preparation and analysis if the required holding time for either activity is less than or equal to 72 hours;
h) reference to the sampling plan and procedures used by the laboratory where these are relevant to the validity or application of the results;
i) the test results with failures identified, units of measurement, an indication of whether results are calculated on a dry weight or wet weight basis;
j) the name, function, and signature or an equivalent electronic identification of the person authorizing the test report, and the date of issue;
k) a statement to the effect that the results relate only to the samples;

l) at the laboratory's discretion, a statement that the report shall not be reproduced except in full without written approval of the laboratory;
m) certification that the results are in compliance with the NELAC Standards if accredited to be in compliance or provide reasons and/or justification if they do not comply.

Supplemental Test Report Information

When necessary for interpretation of the results or when requested by the client, test reports include the following additional information:

a) a Case Narrative with deviations from, additions to, or exclusions from the test method, information on specific test conditions, such as environmental conditions, and any non-standard conditions that may have affected the quality of the results, and any information on the use and definitions of data qualifiers;
b) a statement of compliance/non-compliance when requirements of the quality systems are not met, including identification of test results that did not meet NELAC sample acceptance requirements, such as holding time, preservation, etc.;
c) where applicable and when requested by the client, a statement on the estimated uncertainty of the measurement;
d) where appropriate and needed, opinions and interpretations
e) when opinions and interpretations are included, the basis upon which the opinions and interpretations are documented. Opinions and interpretations are clearly marked as such in the test report.
f) additional information which may be required by specific methods or client;
g) qualification of results with values outside the working range.

For test reports that contain the results of sampling, the following is provided if necessary for the interpretation of the results:

a) the date of sampling;
b) unambiguous identification of the material sampled;
c) the locations of the sampling, including diagrams, sketches, or photographs;
d) a reference to the sampling plan and procedures used;
e) details of any environmental conditions during sampling that may affect the interpretations of the test results;
f) any standard or other specification for the sampling method or procedure, and deviations, additions to or exclusions from the specification concerned.

Environmental Testing Obtained from Subcontractors

Test results obtained from test performed by subcontractors are clearly identified on the test report by subcontractor name and/or accreditation number.

The test results from subcontractors are reported in writing or electronically. A copy of the subcontractors report is be made available to the client if requested.

Electronic Transmission of Results

All test results transmitted by telephone, fax, e-mail, or other electronic means comply with the requirements of this Quality Manual and associated procedures to protect the confidentiality and proprietary rights of the client.

Amendments to Test Reports

Material amendments to a test report after it has been issued are made only in the form of another document or data transfer. All supplemental reports meet all the requirements for the initial report and the requirements of this Quality Manual.

Amended test reports are titled to assure they can be differentiated from other test reports.

When it is necessary to issue a complete new report, the new report is uniquely identified and contains a reference to the original that it replaces.

Data in the Lab

Technical staff will document sample preparation activities in prepared laboratory notebooks or bench sheets. These are the primary record of preparation activities in the laboratory, and for data reduction occurring after analysis. Data for GC/MS, GC, ICP, and LC methods are generated by stand-alone computer systems. Results from each analysis are transferred only to analytical forms specific to the particular analysis. If for any reason the sample(s) were re-analyzed/ re-run or re-extracted, internal comments are recorded within the Khemia Omega 12 Lims Example: “RR1 Failing QC” or “RE Failing QC”. This data is not on the final report unless specifically requested.
Data is checked for accuracy and precision at the bench and instrument analyst level, by secondary review process, and by the Project Manager and Technical Reviewer prior to release of the data to the client. Data will be supported by the following to be valid:

1) Description of calibration.

2) Description of instrument checks (noise levels, drift, and linearity).

3) Documentation of traceability of instrument standards, samples, and data

4) Documentation of analytical methodology and QC requirements.

5) Description of control taken to minimize and verify interference in the method (method blanks and LCS analysis for accuracy and precision).

6) Description of routine maintenance.

7) Documentation of sample preservation and transport.

Laboratory Data Validation and Data Reporting

Data validation is the process of reducing, reviewing, and accepting (or rejecting) data produced by the laboratory against a set of criteria. It is a systematic procedure of reviewing a body of data against the criteria to provide assurance of validity prior to use by a client or regulatory agency.

Data validation is performed by the analyst and a second, trained analyst, QAM, or Technical Director. A minimum of two reviews is required to validate a package. The Project Manager or Technical Reviewer will perform a data package review for completeness and consistency, and to assure compliance with customer deliverables requirements.

Validation is accomplished through routine audits of data collection and flow procedures, and by monitoring QC sample results. Data validation includes signed and dated entries on worksheets and in logbooks used for all samples, the use of sample tracking and numbering systems, and the use of quality control criteria to reject or accept data.

The process consists of data editing, screening, checking, auditing, verification, certification, and review. Specialty Analytical will certify in writing that the data has been validated in accordance with defined laboratory processes.

Minimum Validation Requirements

1) A minimum 3 point calibration, not including the isoelectric point or calibration blank

2) Laboratory Control Samples/QC samples included for the analysis

3) One method blank per matrix and per concentration level for each batch or day

4) Matrix Spike/Matrix Spike Duplicate per concentration level and per matrix at the regulatory required frequency, or per batch.

Data checks used to validate precision and accuracy of parameters measured, and to support the representativeness, comparability, and completeness include:

1) Correlation coefficient of > or = 0.990, or a RSD of < 20% for the calibration curve.

2) Documentation of traceability of the instruments standards

3) Documentation of methodology (analytical and QC) from the SOP.

4) Routine maintenance performed

5) Documentation of sample transportation and preservation.

Review of QC Data

When an analytical data set is complete, the results will be reviewed to establish the validity of the data. General principles are:

1) Blank Evaluations - Review of method, instrument, and rinse blanks for background contamination.

2) Field/Trip Blank Evaluation - Review of field or trip blanks to check for background, instrument, and carry-over or transport contamination.

3) Matrix Spike/Duplicate Evaluation - Review of precision and accuracy for the spike and duplicate results.

4) Calibration Standards Evaluation - Review to determine linearity, range, and verify sample values within calibration ranges.

5) Duplicate Sample Evaluation - Review of precision using RPD criteria for duplicates within set, if used in method.

6) LCS Evaluation - Results of LCS or Blank Spike are evaluated for % Recovery values,

and compared with acceptance criteria.

7) Surrogate Spike Evaluation - Review of surrogate percent recovery to determine compliance with acceptance criteria.

Data Collection and Flow

Audits and review of data collection and flow will include:

1) Review of sample documents for completeness, with review by second analyst or technical reviewer.

2) Daily review of instrument logs, performance results, and analyst performance by QAM.

3) Daily review of performance indicators by analysts and QAM.

4) Random calculation checks at a minimum of 20%.

5) Review of all reports prior to and subsequent to data entry.

Signatories & Electronic Signatures

Specialty Analytical has laboratory approved signatories. Signatories are granted permission to sign off on all controlled documents including, but not limited to, reports, SOP's, corrective/preventative actions and initial demonstration of capabilities.
Positions that are considered for signatory responsibilities include the Lab Director, Technical Director, QA/QC Manager, and Project Manager.
Current approved signatories: Marty French, Julie Clay, and Polly Miller.

Specialty Analytical' s Electronic Signature Policy is that all electronic signatures are maintained in the LIMS. Final reports are signed electronically before being released from the laboratory.

AUDITS AND MANAGEMENT REVIEW

System and Performance Audits of Specialty Analytical will be performed on a periodic, on-going basis. These audits will be included in all Laboratory Audit files, and analyst specific information will be contained in the Analyst Certification/Training Files. The procedures for internal audits are found in SOP# SPRO011 "Quality Systems, Internal Audits and Corrective Actions".

System Audits would include audits performed by outside agencies. These may include

regulatory agencies, clients, or internal system audits. Internal system audits would be performed similarly to outside audits, but be performed by Laboratory Management. Each department will be audited at a minimum of yearly, and include Sample Management and Document control as well as analytical departments.

Performance Audits would include any Performance Evaluation Samples analyzed by Specialty Analytical. These will be documented, and copies of relevant information will be included in Analyst Certification/Training Files. These will include, as a minimum, all Performance Evaluation Samples analyzed by the laboratory, including single-blind samples initiated by the Laboratory Quality Assurance Manager. They would also

include all double-blind PE samples analyzed as part of maintenance of accreditation by regulatory agencies. Information on corrective actions should be included. All analysis performed by the laboratory, for which a PE sample can be obtained, will be analyzed on a twice-yearly basis. This information will be recorded in the annual QA/QC report, and relevant analyst training files.

QA/QC audits will summarize information on laboratory performance, and will contain relevant information from the above reports. They will also include periodic audits performed by Laboratory personnel, including departmental audits. They will include laboratory performance on PE samples, both internal and external, information on activities to improve precision and accuracy of the analysis, and summary information on regulatory and client audits. They should include responses to PE results exceeding acceptance limits, and responses to comments contained in internal and external audits. QA/QC audits will be performed at a minimum of annually. Any methods, which have been halted due to QA/QC issues, will be reviewed in a QA/QC report prior to validating the analysis again.

ACRONYMS

A list of acronyms used in this document and their definitions are:

ANSI	–	American National Standards Institute
ASTM	–	American Society for Testing and Materials
°C	–	degrees Celsius
CCV	–	Continuing calibration verification
CN	–	Case Narrative
COC	–	Chain of custody
DO	–	Dissolved oxygen
DOC	–	Demonstration of Capability
EPA	–	Environmental Protection Agency
GC/MS	–	gas chromatography/mass spectrometry
ICP-MS	–	inductively coupled plasma-mass spectrometry
ICV	–	Initial calibration verification
IDOC	–	Initial Demonstration of Capability
ISO/IEC	–	International Organization for Standardization/International Electrochemical Commission
LCS	–	Laboratory control sample
LFB	–	Laboratory fortified blank
MDL	–	method detection limit
mg/Kg	–	milligrams per kilogram
mg/L	–	milligrams per liter
MS	–	matrix spike
MSD	–	matrix spike duplicate
NELAC	–	National Environmental Laboratory Accreditation Conference
NELAP	–	National Environmental Laboratory Accreditation Program
NIST	–	National Institute of Standards and Technology
PT	–	Proficiency Test(ing)
QAM	–	Quality Assurance Manual
QAPjP	–	Quality Assurance Project Plan
QC	–	Quality Control
QAM	–	Quality Assurance Manager
RL	–	Reporting level
RPD	–	Relative percent difference
RSD	–	Relative standard deviation
SOPs	–	Standard operating procedures
TNI	-	The NELAC institute
ug/L	–	micrograms per liter
UV	–	Ultraviolet
VOC	–	Volatile organic compound

GLOSSARY

Accuracy. The degree of agreement between an analytical result and an accepted reference value or true value. The accuracy of a result is affected by both systematic errors (i.e., bias) and random errors (i.e., imprecision). Some analysts improperly use accuracy to denote only systematic error. (See "bias" and "precision.")

Action limit. A control limit on a control chart, which, if exceeded, requires corrective action to be taken. Action limits are usually placed at +3 standard deviations from the expected or mean value. (See "Control Limit" and "Warning Limit" below.)

Analyte. That which is analyzed for in chemical, but not physical or biological, determinations.

Analytical error. The error, E, of an analytical result, R, is defined as:

$$E = R - T$$

where T is the true value.

Analytical Method. Written instructions describing an analytical procedure followed to obtain a numerical estimate of the concentration of a determinant (analyte) in a sample or samples.

Analytical Response. A numerical observation obtained when a sample is presented to a measurement sub system (e.g., spectrophotometric measurement of the absorbance of a solution). The magnitude is related to the concentration of the determinant (analyte) in the sample.

Analytical Result. A numerical estimate of the concentration of a determinant (analyte) in a sample, obtained by carrying out once the procedure specified in an analytical method. A method may specify analysis of more than one portion of a sample to produce one analytical result. The result can also be thought of as the final value reported to the user.

Analytical System. A combination of analyst, analytical method, equipment, reagents, standards, laboratory facilities, any other components involved in carrying out an analytical procedure.

Batch. A group of environmental samples prepared and/or analyzed together using the same process, personnel and reagents.

Prep Batch. A batch of samples from 1-20 of the same matrix that are processed together. The maximum time between the start of processing the first and last sample in the batch is 24 hours.

Analytical Batch. A batch of prepared samples (extracts, digestates or concentrates) which are analyzed together as a group. The analytical batch may contain samples derived from more than one matrix and more than one prep batch.

Bias. That part of inaccuracy of an analytical result caused by systematic error.

Blank. A sample used to estimate the analytical response attributable to all factors other than the determinant (analyte) in the sample. Blanks are analyzed identically to samples, but do not contain the determinant (analyte) (e.g., in water analyses, pure water would be analyzed to determine the blank). Some analysts use the term method blank with the same meaning given here.

Calibration Standards. Solution(s) of known analyte concentration, used in the calibration (standardization) procedure to determine the relationship between concentration and analytical response.

Certified Reference Material. A substance, one or more property values of which are certified by a technically valid procedure accompanied by or traceable to a material certificate or other document issued by a certifying body.

Check Standard. A solution of known concentration used to check the precision analyses (and bias due to calibration). When used in conjunction with a control chart, the check standard is called a control standard.

Control Limit. A value on a control chart used to make decisions as to whether the results or control tests are acceptable. Two kinds of control limits are usually used: warning limits and action limits.

Criterion of Detection. The smallest concentration, which can be distinguished from a blank with no more than a 5% chance of reporting a false positive.

Data Quality Objectives. Qualitative and quantitative statements of the quality of data. Qualitative statements may be made concerning completeness, defensibility, representativeness, and accuracy, as well as other factors. Quantitative statements are usually made concerning acceptable bias and precision (which together, influence accuracy).

Degrees of Freedom. A whole number expressing the amount of information available for an estimate. The whole number is generally the number of independent results less the number of constraints. Constraints are what else needs to be estimated from the same set of results.

Determinant. That which is to be determined. Covers chemical, physical, biological, or other analytical determinations. (See "Analyte" above.)

External Standard Calibration. The use of independently prepared standards to determine the relationship between response and concentration, run separately from the sample(s). Also called external standardization.

Interference. Systematic error (bias) in the analytical result caused by the presence of a substance in the environmental sample (or added to the sample during analysis).

Internal Standardization. A calibration and analysis procedure in which the responses of analytes are determined relative to an internal standard(standards added to every sample). Two solutions calibration and spiking are required.

Isotope Dilution. An internal standardization procedure in which the internal standards are isotopically labeled analogs of target analytes. Isotope dilution techniques are characterized by very low bias due to calibration.

Laboratory Control Standard (LCS). See Check Standard.

Limit of Detection. The smallest concentration of an analyte for which there is at least a 95% chance the analyte, if present, will be detected (i.e., there is only a 5% chance of obtaining a false negative).

Matrix Spike/Matrix Spike Duplicate (MS/MSD). See Spike.

Percent Recovery. That percent of a known amount of material 'spiked" or added to a sample being analyzed which is reported at the end of the analysis. See "spike," "analytical recovery," and "physical recovery."

% Recovery = 100(R2 Rl)/A

Where R, is the result for the sample without the spike, and R2, the result for the spiked sample, and A is the equivalent concentration added in the spiked sample.

Population. The collection of all possible analytical results.

Precision. A qualitative term used to denote the scatter of results. Precision is said to improve as the scatter among results becomes smaller. Random error is also referred to as imprecision. Usually measured as standard deviation.

Quality Assurance. The total integrated program for assuring the reliability of monitoring and measurement data.

Analytical Quality Control. The routine application of statistically based procedures to evaluate and control the accuracy of results from analytical measurements.

Random error. Errors indicated when repeated analyses of identical portions of a homogeneous sample do not give identical results. Random results differ among themselves and are more or less scattered about some value. They are termed random because the sign and magnitude of the error of any particular result vary at random, and cannot be predicted with confidence.

Analytical Recovery. An estimate, usually expressed in percent, of an analytical result in comparison with a true or reference value for the analyte, (e.g., an analytical recovery of 95% for compound X means that the result was 95 percent of the true or reference value for X in the sample).

Physical Recovery. An estimate, usually expressed in percent of the amount of standard or analyte present at the final stage of analysis (e.g., final extract), compared with the amount present in the original sample. Physical recovery can be an indirect indication of analytical performance.

Reference Material. A material or substance, one or more properties of which are sufficiently well established to be used for the calibration of an apparatus, the assessment of a measurement method, or for assigning values to materials.

Relative Percent Difference (RPD). The difference between duplicate results for analyses of a sample, relative to the mean value of those results and expressed as a percent.

RPD = 100(di d2)/E(dl + d2)/2
= 200(d1 d2)/(dl + d2)
where d1, is the result of the first analysis, and d2 the second.

Relative Standard Deviation (RSD). The standard deviation relative to the mean; also called coefficient of variation." RSD is calculated as either s/x bar or lOOs/x bar. The latter is sometimes referred to a percent relative standard deviation, or %RSD. RSD indicates the magnitude of imprecision.

Spike. A known amount of analyte added to a sample for the purpose of judging, from the analytical percent recovery, whether there is bias due to interference present in the sample; also referred to as fortification of the sample. See "percent recovery."

Standard. A solution of known concentration. There are two types of standards: (1) check (or control), and; (2) calibration.

SOP. A standard operating procedure, or detailed, written description of a procedure designed to systematize the performance of the procedure.

Standard Deviation. A statistical constant which describes the width of the normal distribution or spread of results. An actual standard deviation is denoted by "a" whereas an estimate of the standard deviation is denoted by "s". For "n" replicate results for a sample of known concentration, the estimate of the standard deviation (s) is:

where "x" is a result and "x" is the mean of "n" results.

For duplicate analyses of "in" pairs of unknown samples, the estimate of the standard deviation of the difference (d) for the two samples in each pair is:

For spike recoveries on “in" samples, the estimate of the standard deviation of the percent recovery (p) is:

Standard Reference Material (SRM). A sample of known concentration, also called a certified reference material, issued by the National Institute for Standards and Technology (NIST).

Statistical Sample. The results of one or more determinations from the sample population of all possible results.

Statistics. Certain single values computed from the results, which characterize the distribution of the results. Each statistic his its own frequency distribution which is defined by a particular mathematical function.

Surrogate Standard. A type of check standard added to each sample for certain types of analyses (e.g., trace organic), in a known amount and at the start of processing. The surrogate is not one of the target compounds for the analysis, and is not expected to be present in environmental samples, but should have analytical properties similar to those compounds.

Systematic Errors. Errors indicated by a tendency of results to be greater or smaller than the true value. Usually bias can be considered to be equivalent to systematic error.

Target Compound. A compound which is expected to be in an environmental sample or for which the analysis is being conducted.

Warning Limit. A control limit on a control chart, usually +2s distant from the expected or mean value. Action is required when results fall outside the warning limits too frequently. A single value outside a warning limit does not necessarily require action, but should alert one to a possible problem.

Methods Performed at Specialty Analytical

Conventional Methods

*** = Accredited through ORELAP**

Alkalinity by EPA 310.1*/2320B*/310.2*
Ammonia by EPA 350.1*/SM4500 NH3H*
Chemical Oxygen Demand by EPA410.4*/SM5220D*
Chloride by EPA 9056A*/EPA 300.0*/SM 4100B*/ASTM D512-04C*
Chlorine, Residual by SM4500 Cl G*
Conductivity by EPA 120.1*
Cyanide by SM4500 CN*/OIA-1677*
Dissolved Oxygen by SM4500-O G*
Inorganic Anions by EPA300.0*/EPA 9056A*/SM4110B*
Flashpoint by EPA 1010*
Fluoride by EPA300.0*/9056A*/SM4500-F-C*/SM4110B*
Hardness by SM 2340B*
Calculated Hardness by SM 2340B*
Hexavalent Chromium by EPA 7196A*/EPA 7199*/EPA 218.6*/ SM3500Cr B*/SM 3500-Cr C*
Nitrate/Nitrite by EPA 353.2*/EPA 300.0*/EPA 9056 A*/SM 4110 B*
Total Kjeldahl Nitrogen by EPA 351.2*/SM4500-Norg C*/ SM4500-Norg D*
Oil and Grease by EPA 1664B*
Oxidation-reduction potential EPA 2580 B/ ASTM G200/ ASTM D1498
Paint Filter Liquids Test by EPA 9095A
pH of Water by EPA 9040/150.1/SM4500-H+ B*
pH of Soil by EPA 9045D*
Phenolics, Total Recoverable by EPA 420.1*/SM 5530 D*
Phosphorus, total by EPA 365.2*/SM4500-P E*
Phosphorus, ortho Waters by EPA 300.0/EPA 9056A/SM4100B/SM4500-P E*
Phosphorus, ortho Solids by EPA 9056A*
Total Dissolved Solids by EPA 160.1/SM2540 C*
Total Suspended Solids by EPA 160.2/SM2540D*
Total Solids by EPA 160.3/SM2540B*
Volatile Solids by SM 2540E*
Settable Solids by EPA 160.5/SM2540F*
Specific Conductance by EPA 120.1/SM2510B*
SPLP Extraction by EPA 1312
Sulfate by EPA 375.4*/EPA 9056A*/EPA 9038*/EPA 375.3*/EPA 300.0*/SM 4110B*/SM4500-SO4 E
Sulfide by EPA 376.1*/EPA 9030B*/EPA 9034*/SM4500-S2 F*
Sulfite by EPA 377.1/SM4500 SO3-B
Tannin and Lignin by SM 5550B*
TCLP Extraction by EPA 1311
Total Organic Carbon by EPA 415.1/EPA 9060/SM 5310 B*
Turbidity by EPA 180.1*/SM 2130 B*
Water content, Karl Fischer by ASTM D4377

Microbiology Methods

***= Accredited through ORELAP**

Total Coliform by SM 9222B/9223B/Colilert-18 Fecal Coliform by SM 9222D/Colilert-18
Biochemical Oxygen Demand by EPA 405.1/SM5210B*

Metals Methods

*** = Accredited through ORELAP**

ICP-MS Metals by EPA 6020B*/EPA 200.8*
Mercury in solids by EPA 7471B
Mercury in water by EPA 245.7

Organics Methods

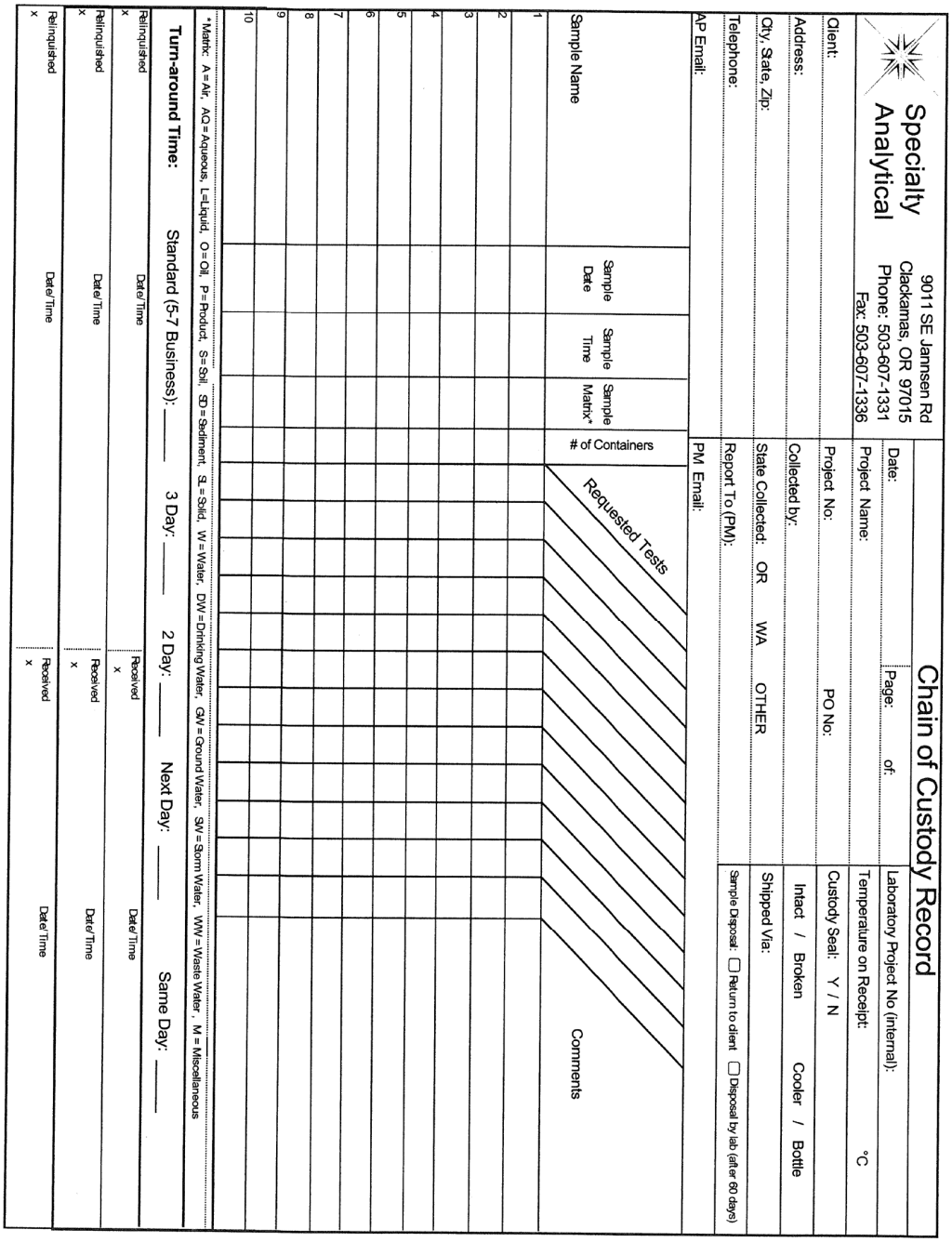
*** = Accredited through ORELAP**

Volatiles by EPA 8260D*
PCBs by EPA 8082A*
Organochlorine Pesticides by EPA 8081B*
BTEX by EPA 8021B
Volatile Petroleum Products by NWTPH-Gx*
Semi-Volatile Petroleum Products by NWTPH-Dx*
Petroleum Hydrocarbon ID by NWTPH-HCID*
Glycols by EPA 8015B*
Alcohols by EPA 8015B*
Volatile organics by EPA 8260D/EPA 624*
Chlorinated Herbicides by EPA 8151A
Semi-volatile organics by PMI 1671*
Volatile organics by PMI 1666*
Pesticides/PCB by EPA 8081B*/EPA 8082A*/EPA 608.3*
EPH – Extractable Petroleum Hydrocarbons*

Note: All methods certified through ORELAP have asterisk next to method.
Consult current scope of accreditation for specific analytes.

Figure 1

Chain of Custody



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Chain of Custody Record

Client:
Address:
City, State, Zip:
Telephone:
AP Email:

Date:
Page: of:
Project Name:
Project No: PO No:
Collected by:
State Collected: OR WA OTHER
Report To (PM):
PM Email:

Laboratory Project No (internal):
Temperature on Receipt: °C
Custody Seal: Y / N
Intact / Broken Cooler / Bottle
Shipped Via:
Sample Disposal: ☐ Return to client ☐ Disposal by lab (after 60 days)

Sample Name	Sample Date	Sample Time	Sample Matrix*	# of Containers	Requested Tests	Comments
1						
2						
3						
4						
5						
6						
7						
8						
9						
10						

*Matrix: A = Air, AQ = Aqueous, L=Liquid, O = Oil, P = Product, S = Soil, SD = Sediment, SL = Solid, W = Water, DW = Drinking Water, GW = Ground Water, SW = Storm Water, WW = Waste Water, M = Miscellaneous

Turn-around Time: Standard (5-7 Business): ____ 3 Day: ____ 2 Day: ____ Next Day: ____ Same Day: ____

Relinquished	Date/ Time	Received	Date/ Time
x		x	
Relinquished	Date/ Time	Received	Date/ Time
x		x	
Relinquished	Date/ Time	Received	Date/ Time
x		x	