AN INTRODUCTION TO THE ENVIRONMENTAL LABORATORY

Calscience

Environmental, Air & Marine Chemistry

The difference is service

WELCOME TO CALSCIENCE ENVIRONMENTAL LABORATORIES, INC.

Founded in 1986, Calscience Environmental Laboratories, Inc. is an industry leader in the environmental laboratory testing field. We provide quality analytical services to consultants, private industry and government agencies. Calscience offers a comprehensive portfolio of analytical methods and our analytical expertise encompasses all environmental matrices including air, groundwater, marine sediment, soil, solid waste, and wastewater. We are a customer-focused organization whose goal is to meet or exceed the expectations of our customers in terms of quality and service. It is through the quality of our service and the loyalty of our clients, that Calscience has grown to become one of the largest environmental testing laboratories in California.

Our Vision and Mission

Calscience Environmental Laboratories is dedicated to providing our clientele a competitive advantage by the routine provision of litigation quality analytical data. Achievement is attained through a continual process of quality improvement and advancement in all areas of our performance.

Calscience focuses on servicing the environmental engineering community, not only to provide reliable data, but to help our customers meet their goals in terms of project profitability and timely completion. Our customers know that Calscience, more than any other laboratory, will allow them to run their projects more efficiently, save labor hours, and provide data and information when they need it and how they need it. Stability, integrity, and service; these are but a few of the reasons Calscience is valued by our clients and why they keep coming back.

Who We Serve

Our clients come from a diverse range of consultants, private industry, and federal, state, and local governments. With a strong industry reputation, we provide contract analytical services to Fortune 100 Companies. Our services support various industries and project types including:

- School Districts
- Real Estate
- Refineries
- Landfills
- Oil and Gas Utilities
- Ports
- Treatment Facilities
- Petrochemical
- Department of Defense
- City, County, State Agencies
- Chemical Industry
- Aerospace Industry
- Entertainment/Movie Studios
- Railroads

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1.0 INTRODUCTION

The purpose of this manual is to provide a general overview of the processes and procedures at Calscience. The objective is to help employees understand the different operational areas of the laboratory and to highlight their importance in the analytical chain-of-custody.

The laboratory is divided into several operational groups; Sample Receiving and Disposal, Volatile Organics, Semivolatile Organics, LUFT, Air, Metals, Wet Chemistry and Project Management. Each of these groups works in concert with the other to produce legally-defensible analytical data of known quality while meeting the clients' needs. The following sections will focus on the flow of samples through the lab areas, general analytical approach and theory, instrumentation types and key terms used in the lab.

2.0 SAMPLE RECEIVING

The first step in the laboratory process is sample receiving. It is here that samples are received, reconciled against a Chain-of-Custody (COC), labeled and stored for processing by the lab.

2.1 Chain of Custody (COC)

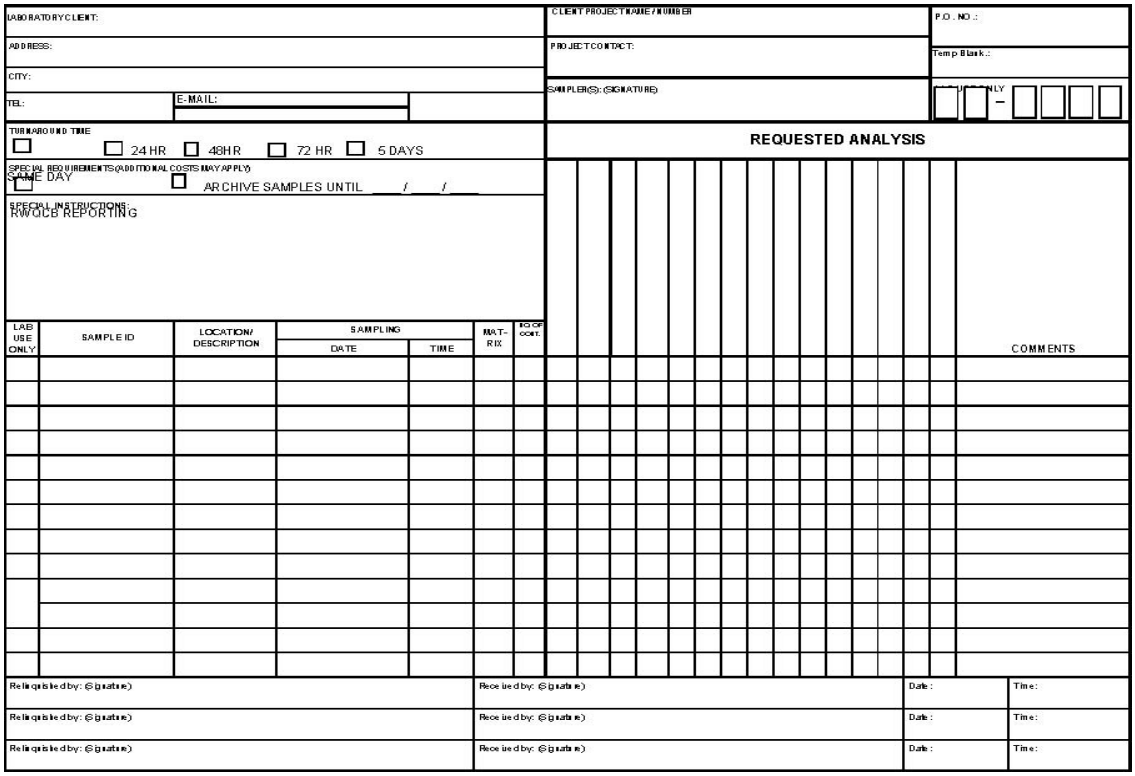
Samples are received from many different sources; shipped to Calscience via commercial carriers (FedEx, GSO, etc.), through personal delivery of samples by the clients, or by Calscience couriers picking them up from the field.

Once received by the laboratory they are checked against the COC for anomalies/discrepancies, labeled, logged into the LIMS system and stored under appropriate conditions for further processing by the analytical departments. The process of receiving samples begins with the COC. Samples should be submitted to the laboratory under standard chain-of-custody procedures and the COC should be completed in full by field personnel.

The COC is used to document all aspects of sample handling. It contains information pertaining to the client, the laboratory, the sampler, the project, the samples and their ID's, sample collection dates and times, containers and preservatives, analyses requested, turn around time required and any potential hazards or other pertinent information related to the samples, sampling event, or reporting format.

At its most basic, the COC is a legal document that presents an unbroken trail of accountability that ensures the physical security of samples, data and records. It allows the samples to be tracked from the field to the lab, and through the use of internal COC's, throughout the lab to disposal. Because of this, it is probably the single most important item, aside from the samples themselves, of the receiving process.

An example of a typical COC is pictured on the following page.



LABORATORY CLIENT:
CLIENT PROJECT NAME / NUMBER
P.O. NO.:
ADDRESS:
PROJECT CONTACT:
Temp Blank:
CITY:
SAMPLER(S): (SIGNATURE)
TEL:
E-MAIL:
TURNAROUND TIME
24 HR 48HR 72 HR 5 DAYS
REQUESTED ANALYSIS
SPECIAL REQUIREMENTS (ADDITIONAL COSTS MAY APPLY)
SAME DAY
ARCHIVE SAMPLES UNTIL ___/___/___
SPECIAL INSTRUCTIONS:
RWQCB REPORTING

LAB USE ONLY	SAMPLE ID	LOCATION/ DESCRIPTION	SAMPLING DATE	SAMPLING TIME	MATRIX	NO OF cont.	COMMENTS

Relinquished by: (Signature)	Received by: (Signature)	Date:	Time:
Relinquished by: (Signature)	Received by: (Signature)	Date:	Time:
Relinquished by: (Signature)	Received by: (Signature)	Date:	Time:

2.2 Responsibilities and Procedures

Samples are accepted at the laboratory by the Sample Receiving Group. The primary responsibilities of the Sample Receiving Group are as follows:

- Receive samples on behalf of Calscience and sign custody documents
- Unpack coolers, check temperatures, and remove/organize the samples per the COC
- Check the COC documents against the actual samples
- Check for proper sample containers, volumes and preservation
- Check for accelerated turn around times and short holding time analyses
- Complete sample receipt and anomaly documentation
- Place into and remove samples from refrigerated storage

Upon receipt, the COC is signed and dated and the sample receiving personnel visually inspect the cooler for custody seals and any obvious damage. They then open and inspect the contents of the cooler and take the cooler temperature via an IR thermometer. (The temperature upon receipt is a critical item and, for most soils and water methods, should be between >0°C and 6°C)

A Sample Receipt Form (SRF) is initiated to record the following: client, number of coolers, Calscience Work Order number, date, temperature, custody seal/sample integrity, COC integrity, sampler information, sample labels, container integrity, holding time integrity, preservative and VOC container integrity (headspace). The form is also be used to record container type received.

All documents are inspected to assure agreement between the samples received and the COC. Any discrepancy, irregularity, or compromised sample is noted on a Sample Anomaly Form (SAF) and brought to the attention of the Calscience Project Manager (PM) for resolution with the client. The COC, SRF, SAF (if used), record of client contact, and resulting instructions all become part of the project record. Once the samples have been reconciled against the COC and a Work Order Number has been assigned, the samples are labeled and placed in the appropriate storage refrigerator until retrieved by the lab for preparation and analysis.

The lab personnel retrieve the samples and, when required by project or client specifications, complete an internal chain of custody (ICOC) to track sample movement throughout the lab. After analysis the samples are returned to the appropriate refrigerator where they are maintained for 30 days. After 30 days the samples are moved to ambient storage where they are maintained for up to an additional 30 days, possibly longer depending on project requirements, and then properly disposed of.

3.0 ANALYTICAL APPROACHES

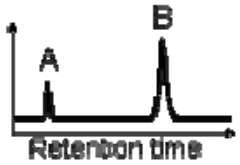
Samples may be analyzed for one or more organic or inorganic constituents. Calscience uses a myriad of analytical systems to generate, quantitate and even qualify numerous environmental constituents in varying matrices. Gas Chromatography, High Pressure Liquid Chromatography, Ion Chromatography, and Inductively Coupled Plasma, in concert with different detector types, as well as many of the manual wet chemistry methods, are used to produce analytical results. One analytical approach may be better for determining specific analytes than another. The following sections will introduce the basic instrumentation types, analytical methods, extraction/preparation methods and general chemistry approaches for sample analysis.

3.1 Gas Chromatography

Gas chromatography is an instrumental method for the separation and identification of organic chemical compounds. A Gas Chromatograph (GC) usually consists of a flowing mobile phase (carrier gas), an injection port, a column containing the stationary phase, a temperature programmable column oven, a detector, and a data recording system. The general process involves a 'sample' being dissolved in a mobile phase. The mobile phase is then forced through an immobile, immiscible stationary phase, in this case an analytical column, causing separation of the components and a response which is then measured on a detector.

Typically, a small amount of sample is introduced to the GC using a syringe. The injector is set to a temperature higher than the components' boiling points allowing for the components of the mixture to evaporate into the gas phase inside the injector. A carrier gas, such as helium or nitrogen, flows through the injector and pushes the gaseous components of the sample onto the GC column, a process called elution. It is within the column that separation of the components takes place. Molecules partition between the carrier gas (the mobile phase) and the high boiling 'liquid' (the stationary phase) within the GC column and causes a response on the detector.[1]

The chromatographic separation process within the column is based on the difference in the surface interactions of the analyte and eluent molecules to the stationary phase. Those with less affinity for interaction (A) will elute first from the column whereas those with strong interactions (B) will take longer to elute.



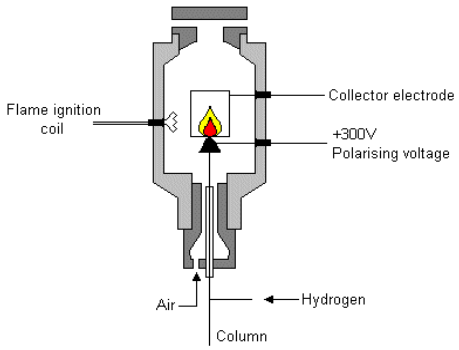
3.1.1. Detectors

There are many detectors which can be used in gas chromatography. Different detectors will give different types of selectivity. A non-selective detector responds to all compounds except the carrier gas, a selective detector responds to a range of compounds with a common physical or chemical property, and a specific detector responds to a single chemical compound.

Detector	Selectivity	General Methods
Flame ionization (FID)	Most organic compounds	8015, TO-3
Thermal conductivity (TCD)	Universal	Fixed gases
Electron capture (ECD)	Halides, nitrates, nitriles, peroxides, organometallics	8081, 8082, 8151
Nitrogen-phosphorus (NPD)	Nitrogen, phosphorus	8141
Flame photometric (FPD)	Sulphur, phosphorus,	8141
Photo-ionization (PID)	Aliphatics, aromatics, ketones	8021
Mass Spectrometer (MS)	Most volatile/semivolatile cmpds	8260, 8270, TO-14/15

Detectors can also be grouped into concentration dependant detectors and mass flow dependant detectors. The signal from a concentration dependant detector is related to the concentration of solute in the detector, and does not usually destroy the sample. Dilution of the sample with make-up gas will lower the detector's response. Mass flow dependant detectors usually destroy the sample, and the signal is related to the rate at which solute molecules enter the detector. The response of a mass flow dependant detector is unaffected by make-up gas.[2]

The Flame Ionisation Detector



As an example, the FID, which is a mass sensitive detector, creates a response by taking the effluent from the column and mixing it with hydrogen and air, and then igniting it. Organic compounds burning in the flame produce ions and electrons which can conduct electricity through the flame. A large electrical potential is applied at the burner tip, and a collector electrode is located above the flame. The current resulting from the pyrolysis of any organic compounds is measured. It is a useful general detector for the analysis of organic compounds; it has high sensitivity, a large linear response range, and low noise.[2]

3.1.2. Mass Spectrometer (MS)

Placed at the end of a chromatographic column in a manner similar to the other GC detectors, the MS detector is more complicated than, for instance, the FID because of the mass spectrometer's complex requirements for the process of creation, separation, and detection of gas phase ions.[3] This analytical technique is known as “GC/MS”.

The major components of the mass selective detector are an ionization source, a mass separator and an ion detector. The general operation of a mass spectrometer is to:

1. create gas-phase ions
2. separate the ions in space or time based on their mass-to-charge ratio
3. measure the quantity of ions of each mass-to-charge ratio[4]

The MS identifies substances by electrically charging the molecules, accelerating them through a magnetic field, breaking the molecules into charged fragments and detecting the different mass-to-charge ratios. A spectral plot displays the mass of each fragment. The compound's mass spectrum can then be used for qualitative identification. In essence these fragment masses can be used like puzzle pieces to piece together the mass of the original molecule, the "parent mass". The parent mass is analogous to the picture on top of a puzzle box, a guide to the end result obtained by putting together the fragment masses, or puzzle pieces. From the molecular mass and the mass of the fragments, reference data is compared to determine the identity of the specimen. Each substance's mass spectrum is unique. Providing that the interpretation of the output correctly determines the parent mass, MS identification is conclusive.[5]

3.2. Liquid Chromatography

Liquid chromatography is similar to gas chromatography in that both techniques use a mobile phase and a stationary phase to separate components for measurement by a detector. In this case, the mobile phase is a liquid and not a gas, and the detectors used are primarily the UV/VIS, Fluorescence or Mass Spectrometer.

The basic liquid chromatograph consists of six units; the mobile phase supply system, the pump and programmer, the sample valve/injector, the column, the detector and finally a means of presenting and processing the results.[6]

3.2.1. High Performance Liquid Chromatography

High Performance Liquid Chromatography (HPLC) utilizes a liquid mobile phase to separate the components of a mixture. These components (or analytes) are first dissolved in a solvent, and then forced to flow through a chromatographic column under a high pressure. In the column, the mixture is resolved into its components. The interaction of the solute with mobile and stationary phases can be manipulated through different choices of both solvents and stationary phases. As a result, HPLC acquires a high degree of versatility not found in other chromatographic systems and it has the ability to easily separate a wide variety of chemical mixtures.[6]

At Calscience, HPLCs are used for the analysis of Polynuclear Aromatic Hydrocarbons (PAHs), Perchlorate and Explosives – EPA Methods 8310, 6850/331.0 and 8330.

3.2.2. Ion Chromatography

Ion chromatography is used to measure the concentrations of major anions such as fluoride, chloride, nitrate, nitrite, and sulfate, by separating them based on their interaction with a resin contained in a column. Sample solutions pass through the pressurized chromatographic column where the ions are absorbed by column constituents. As an ion extraction liquid, known as eluent, runs through the column, the absorbed ions begin separating from the column and are measured on a conductivity detector.

At Calscience, Ion Chromatographs (ICs) are used for the analysis of Anions, Perchlorate and Hexavalent Chromium – EPA Methods 300.0, 314.0 and 218.6/7199, respectively.

3.3. Inductively Coupled Plasma - Atomic Emission Spectrometry / MS

Inductively Coupled Plasma - Atomic Emission Spectrometry (ICP-AES) is an emission spectrophotometric technique, exploiting the fact that excited electrons emit energy at a given wavelength as they return to ground state. The fundamental characteristic of this process is that each element emits energy at specific wavelengths peculiar to its chemical character.

Although each element emits energy at multiple wavelengths, in the ICP-AES technique it is most common to select a single wavelength (or a very few) for a given element. The intensity of the energy emitted at the chosen wavelength is proportional to the amount (concentration) of that element in the analyzed sample. Thus, by determining which wavelengths are emitted by a sample and by determining their intensities, the analyst can quantify the elemental composition of the given sample relative to a reference standard.[7]

The ICP may also be coupled to a Mass Spectrometer for more sensitive metals detection. Analyte species originating in a liquid are nebulized and the resulting aerosol transported by argon gas into the plasma torch. The ions produced are entrained in the neutral plasma gas and introduced, by means of a water-cooled interface into a mass spectrometer. The ions and ion clusters produced in the plasma are sorted according to their mass-to-charge ratios and quantified with a dual mode electron multiplier detector.

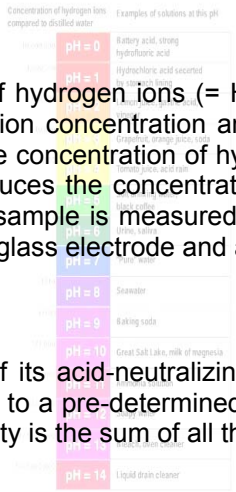
At Calscience, the ICP is used for the analysis of trace metallic elements in a solution – EPA Methods 6010, 6020, 200.7 and 200.8.

3.4. General Wet Chemistry Techniques

Where many of the above approaches are specific to a select set of analytes or compounds using more 'sophisticated' analytical equipment, the wet chemistry department encompasses a broad array of manual and instrumental techniques to measure both organic and inorganic constituents.

Common laboratory equipment such as burettes, pipettes, funnels, filters, pH meters, conductivity meters, balances and ovens are used to make routine analytical determinations in this department. Some of the determinative methods are noted below.

3.4.1. pH – 'Power of Hydrogen'



pH is a measure of the concentration of hydrogen ions (= H^+) (= protons) in a solution. Low pH corresponds to high hydrogen ion concentration and vice versa. A substance that, when added to water, increases the concentration of hydrogen ions (lowers the pH) is called an acid. A substance that reduces the concentration of hydrogen ions (raises the pH) is called a base. The pH of a sample is measured electrometrically by using a calibrated pH meter equipped with a pH glass electrode and a reference electrode.

3.4.2. Alkalinity

The alkalinity of water is an indicator of its acid-neutralizing capacity. The sample is titrated with a standardized acid solution to a pre-determined end point of pH 4.5 using a calibrated pH meter. In essence, alkalinity is the sum of all the titratable bases.

3.4.3. BOD – Biochemical Oxygen Demand

Biochemical oxygen demand (BOD) is a measure of the quantity of oxygen used by microorganisms (e.g., aerobic bacteria) in the oxidation/degradation of organic matter during a specified incubation period, usually five days. On the day of collection, the dissolved oxygen (DO) level is measured in an initial sample, and then again five days later, using a dissolved oxygen meter. The BOD is the difference between the initial and final DO levels in the sample.

3.4.4. Specific Conductance

The term conductance refers to the ability of a material to carry an electric current. Liquids that carry electric current are generally referred to as electrolytic conductors. The specific conductance of a sample is measured directly on the sample at room temperature using a standardized self-contained conductivity meter that has the capability to automatically correct measurements to 25ºC.

3.4.5. TOC – Total Organic Carbon

Organic carbon in a sample is converted to carbon dioxide (CO_2) by catalytic combustion or wet chemical oxidation and the CO_2 formed is measured directly by an infrared detector. The amount of CO_2 in a sample is directly proportional to the concentration of carbonaceous material in the sample.

3.4.6. TDS – Total Dissolved Solids

This method is used to determine the total dissolved solids in aqueous samples and for soil/solid samples, after a deionized water leach. A well-mixed sample or leachate is filtered through a standard glass fiber filter. The filtrate is evaporated and dried to a constant weight at 180°C. The residue left after the drying and weighing process is equal to the amount of dissolved solids in the sample.

TDS results as compared to conductivity results can be used to help make a common sense decision about the data. The acceptable criteria for TDS vs conductivity ratios range from 0.55 to 0.7. If the ratio is outside these limits, either result may be suspect.

3.4.7. TSS – Total Suspended Solids

This method is used to determine the total suspended solids in aqueous samples. A well-mixed sample is filtered through a standard glass fiber filter. The residue retained on the filter is dried to a constant weight at 103−105°C. The difference between the initial wet weight of the solid and the final dry weight is the TSS result.

3.5 Extraction and Preparation Techniques

Where direct injection of a sample into an instrument or taking a direct reading via a meter are approaches used in the laboratory, several methods require that the samples be 'prepared' prior to analysis. This is especially true in the semivolatile and metals departments, where whole liquid and solid volume samples are extracted with a solvent and then concentrated down prior to analysis, or digested with acids, to achieve lower reporting limits. The process of extracting a sample with a solvent allows for the transfer of the constituents of concern from the sample into the solvent phase.

In addition to the common preparatory methods, there are additional preparation techniques for determining the hazardous characteristics of samples. Several of the most common techniques are introduced below.

3.5.1. EPA 3550 – Sonication

The procedure is used to extract non-volatile and semi-volatile organic compounds from solids such as soils, sludges, and wastes. The solid sample is mixed with anhydrous sodium sulfate to form a free-flowing powder and is then solvent extracted three times using ultrasonic extraction.

Sonication is the act of applying sound (usually ultrasound) energy to agitate particles in a sample, for various purposes. In the laboratory, it is usually applied using an ultrasonic bath or an ultrasonic probe, colloquially known as a sonicator.[11] The ultrasonic extraction process ensures intimate contact of the sample matrix with the extraction solvent.

The method also describes concentration techniques suitable for preparing the extract for the appropriate determinative methods including, but not limited to, EPA Methods 8081, 8082, 8141, 8270 and 8310.

3.5.2. EPA 3540 – Soxhlet Extraction

The procedure is used to extract non-volatile and semi-volatile organic compounds from solids such as soils, sludges, and wastes. The solid sample is mixed with anhydrous sodium sulfate, placed in an extraction thimble and extracted using an appropriate solvent in a Soxhlet extractor. The Soxhlet extraction process ensures intimate contact of the sample matrix with the extraction solvent.

The method also describes concentration techniques suitable for preparing the extract for the appropriate determinative methods including, but not limited to, EPA Methods 8081, 8082, 8141, 8270 and 8310.

3.5.3. EPA 3545 – Pressurized Fluid Extraction (PFE)

Similar to the Soxhlet extraction, the PFE approach describes the procedures for extracting water insoluble or slightly water soluble organic compounds from solid samples The method uses elevated temperature (100−180°C) and pressure (1500−2000 psi) to achieve analyte recoveries equivalent to those from Soxhlet extraction, using less solvent and taking significantly less time than the Soxhlet procedure.

As with EPA 3540, the method also describes concentration techniques suitable for preparing the extract for the appropriate determinative methods including, but not limited to, EPA Methods 8081, 8082, 8141, 8270 and 8310.

3.5.4. EPA 3510 – Separatory Funnel Extraction

The procedure is used to isolate and extract water-insoluble and slightly water-soluble organic compounds from aqueous samples. A measured volume of sample, usually one liter, at a specified pH, is serially extracted three times with methylene chloride using a hand-held 2-L separatory funnel.

The method also describes concentration techniques suitable for preparing the extract for the appropriate determinative methods including, but not limited to, EPA Methods 8081, 8082, 8141, 8270 and 8310.

3.5.5. EPA 3520 – Liquid-Liquid Extraction

Similar to EPA 3510, the liquid-liquid extraction technique describes the procedures for isolating water-insoluble and slightly water-soluble organic compounds from aqueous samples. A measured volume of sample, usually one liter, at a specified pH, is placed into a continuous liquid-liquid extractor, and extracted with organic solvent, usually methylene chloride, for 18−24 hours.

As with EPA 3510, the method is suitable for preparing the extract for the appropriate determinative methods including, but not limited to, EPA Methods 8081, 8082, 8141, 8270 and 8310.

3.5.6. EPA 3005 – Acid Digestion for Surface or Ground Waters

This acid digestion procedure is used to prepare surface and ground water samples for analysis by inductively coupled plasma-atomic emission spectrometry (ICP-AES), or by inductively coupled plasma-mass spectrometry (ICP-MS) using methods EPA 6010, 6020, 200.7 or 200.8. The entire sample is acidified with nitric acid at the time of sample collection. After receipt at the laboratory, the sample is then heated with acid and substantially reduced in volume. The digestate is diluted to volume prior to analysis.

3.5.7. EPA 3010 and EPA 3020 – Acid Digestion of Aqueous Samples

Similar to EPA 3005, these acid digestion procedures are used to prepare aqueous samples, mobility-procedure extracts, and wastes containing suspended solids for analysis by ICP-AES and ICP-MS, respectively.

A mixture of nitric acid and the material to be analyzed is refluxed in a covered digestion tube. This step is repeated with additional portions of nitric acid until the digestate is light in color or until its color has stabilized. After the digestate has been brought to a low volume, it is refluxed with hydrochloric acid and brought up to volume for analysis by ICP, or allowed to cool and then brought to volume for analysis by ICP-MS.

3.5.8. EPA 3050 – Acid digestion of Soils, Sediments, Sludges

EPA Method 3050B has two separate acid digestion procedures. One is used to prepare sediment, sludge, and soil samples for total metals analysis by ICP-AES and the other is used to prepare the same matrices for analysis by ICP-MS. In both, a representative 1 g (dry weight) of sample is digested with repeated additions of nitric acid and hydrogen peroxide.

The digestates from these two acid digestion procedures are not inter-changeable as hydrochloric acid is added to the initial digestate and the sample is refluxed when the analysis needed is ICP. For ICP-MS analysis, the volume of digestate is reduced while heating, and then diluted to a final volume.

3.5.9. TCLP – Toxicity Characteristic Leaching Procedure

EPA Method 1311, the toxicity characteristic leaching procedure, determines the mobility of organic and inorganic analytes present in liquid, solid, and multiphasic wastes. This leachate method is designed to simulate the leaching a waste will undergo if disposed of in a landfill. Over time, water and other liquids percolate through landfills and react with the solid waste in the landfill posing environmental health risks because of the contaminants it absorbs.

The leaching procedure involves extracting a subsample of a ‘waste’ with the appropriate buffered acetic acid solution for 18 ± 2 hours. The TCLP extract (leachate) is then digested/extracted and analyzed by one or more analytical methods to determine if any of the thresholds established for 40 Toxicity Characteristic constituents listed in 40 CFR §261.24 have been exceeded.

In general, if a “Solid Waste” fails the test for one or more of these compounds, the waste is considered to be a characteristic hazardous waste under federal guidelines.

3.5.10. STLC - Soluble Threshold Limit Concentration (“WET”)

This analysis determines the amount of each analyte that is soluble in the "Waste Extraction Test", (WET) leachate. The STLC test is similar to the TCLP test in that it simulates the conditions that may be present in a landfill.

The leaching procedure involves extracting a subsample of a waste in a citric acid solution for 48 hours. The STLC extract (leachate) is then analyzed by one or more analytical methods to determine if any of the thresholds established in Title 22 of the California Code of Regulations (CCR) has been exceeded. In general, if the waste fails the test for one or more of the listed compounds the waste is considered to be a hazardous waste under California guidelines.

4.0 QUALITY CONTROL

Quality control can be defined as the overall system of technical activities whose purpose is to measure and control the quality of a product or service so that it meets the needs of users. The following quality control principles apply, where applicable, to all testing at Calscience. The manner in which each is implemented is dependent on the types of tests performed by the laboratory and are spelled out in the applicable standard operating procedures (SOP).

Independent of the test method, all quality control measures are assessed and evaluated on an on-going basis, and defined quality control acceptance criteria are used to determine the usability of the data. All departments have detailed written procedures in place to monitor the following quality elements:

1) Positive and negative controls (blanks, spikes, etc.) to monitor tests;
2) Tests to define the variability and/or repeatability of the laboratory results such as replicates;
3) Measures to assure the accuracy of the test method including calibration and/or continuing calibrations, use of certified reference materials (CRM), proficiency test samples, or other measures;
4) Measures to evaluate test method capability, such as detection limits and quantitation limits or range of applicability such as linearity;
5) Selection of appropriate formulae to reduce raw data to final results such as regression analysis, comparison to internal/external standard calculations, and statistical analyses;
6) Selection and use of reference reagents and standards of appropriate quality;
7) Measures to assure the selectivity of the test for its intended purpose; and
8) Measures to assure constant and consistent test conditions (both instrumental and environmental) where required by the test method, such as temperature, humidity, light or specific instrument conditions.

4.1. Quality Control Samples and Requirements

Quality control (QC) samples include a variety of samples that are used to measure the accuracy and precision of an analytical process. The QC samples may be reagent blanks, spiked with target analytes, or may be duplicates of field samples depending on the parameter under consideration. Spiked samples and/or spiking solutions may be purchased through an established vendor or be prepared by the analyst. Quality control samples are prepared and analyzed concurrently with field samples.

Project-specific Quality Assurance Project Plans (QAPP) and governmental agency Quality Assurance plans normally include a requirement for the number and type of QC samples to be included with each group of field samples. In the event that this guidance is not available, laboratory SOP's, both method-specific and QC-specific (such as SOP-T020), define the minimum required QC per batch of field samples.

Typically, at least one Method Blank and one Laboratory Control Sample (LCS), or QC sample such as a Sample Duplicate, must accompany each group of field samples at a rate of one per twenty. Certain analytical methodologies or contracts may present different requirements for QC sample analysis.

4.1.1. Sample Batching

The basic definition of a batch is as follows: environmental samples plus associated QC samples, which are prepared and/or analyzed together with the same process and personnel, using the same lot(s) of reagents. Samples are 'batched' together for analysis in two ways; by preparation or by analysis.

The type of batch that is required is based upon the analytical methodology being used for the samples. Samples are either prepared via an extraction or digestion procedure prior to analysis, or they are analyzed directly on an instrument.

- A preparation batch is composed of one to twenty environmental (client) samples plus batch QC, of the same matrix (aqueous or solid), that are prepared using the same reagents and standards, at the same time by the same personnel. At a minimum, batch QC consists of a Method Blank and an LCS. But, more often, the batch QC consists of a Method Blank, LCS, LCS Duplicate, Matrix Spike and a Matrix Spike Duplicate. Specific methods may require that a Sample Duplicate (SD) be used in place of a spiked sample (LCS/MS/MSD). In all cases, the batch QC samples must be treated in the same manner as the client samples.

  Samples that are batched together at the preparation stage may not necessarily be analyzed together on the same instrument. In the instance where the sample extracts are split up and analyzed on multiple instruments, the method blank should also be analyzed on each instrument. At a minimum, an instrument blank must be analyzed on each instrument.

- An analytical batch is composed of prepared environmental samples, extracts, digestates or concentrates that are analyzed together as a group. In general, this is applied to the analysis of volatile samples by EPA 8015 or 8260, but may also apply to any sample that is directly injected onto or measured by an instrument or manual technique for which there is no associated preparation step. In these cases, a method blank and LCS/LCSD are analyzed and, if volume permits, also an MS/MSD.

4.1.2. Blanks

Blank samples; Method Blanks, Instrument Blanks or Preparation Blanks, are utilized at a rate of at least 1 blank per 20 client samples to detect the possible presence of contaminants in an analytical procedure. Any blanks that are submitted by the client are treated as field samples and can include trip blanks, rinse blanks, equipment blanks, and field blanks.

A Method Blank (MB) is made from a reagent matrix that is similar to the batch of associated samples (when available) that is free from the analytes of interest and is processed simultaneously with and under the same conditions as samples through all steps of the analytical procedures, and in which no target analytes or interferences are present at concentrations that impact the analytical results for sample analyses. Not all methods, such as pH, require the analysis of Method Blanks with the samples, but for a majority of laboratory procedures, the Method Blanks are an important QC component.

An Instrument Blank is basically a target analyte-free standard made from a clean reagent source, most often sand or water. These blanks are used to verify the absence of contamination in the analytical instrumentation system and that the system is not producing excess instrument noise. Instrument blanks are not prepared with field samples and any analytes of interest should not be present at a level greater than the reporting limit, or greater than ½ the reporting limit if so defined by the project.

If the level of any analyte exceeds this criterion, the source of contamination must be identified. If sufficient sample volume is available and holding times have not expired, the blank and all associated samples and QC must be re-prepared and/or re-analyzed depending on the method. If sufficient volume is not available and/or holding times have expired, the associated results must be flagged on the final report. It is never appropriate to subtract blank results from sample results unless specifically allowed by method.

4.1.3. Laboratory Control Samples (LCS)

A Laboratory Control Sample (LCS) is a sample composed of a known clean matrix similar to the field samples that is spiked with a compound or compounds representative of the target analyte(s), and surrogate compounds if required by the method. The LCS is used to document laboratory performance for a given method in that it provides information regarding the “best case” scenario for the analytical process negating the factors of matrix. As with the MB, not all methods require the use of an LCS, but when used they provide critical QC information about the analytical process.

In general, at least one LCS sample is prepared and analyzed along with each batch of up to 20 client samples of a similar matrix, if required by the specific analytical methodology. A duplicate LCS (LCSD) may also be prepared and must be evaluated against the same criteria as the LCS. In the event that there is insufficient sample volume to prepare an MS/MSD, an LCS/LCSD should be prepared.

The LCS recovery values should be within laboratory control limits. If the LCS recovery is beyond control limits, the analytical process may be considered "out of control" and corrective action steps should be taken to return the analytical system to a controlled status. The corrective action may include re-preparation and analysis of samples, re-calibration of the instrument and re-analysis of samples, or possibly, data qualification.

There are possible cases when LCS limits are beyond control criteria and the analytical results may still be considered valid. If there are high LCS recoveries but the analytes of interest are not detected in the samples, then that may indicate a high quantitation bias. The high bias would also be applicable to the samples but, since the analyte is not present in the sample, the high recovery of the LCS is noted, but requires no further action on those samples analyzed concurrently.

Whenever there are signs that the system is not 'in control', whether the results are bias low or bias high, corrective action should be taken to prevent the reoccurrence of the failure. In all cases, when the results for the laboratory QC samples are below acceptance criteria, steps must be taken to bring the system back into control, and any positive samples, analyzed when the system is not demonstrated to be in control, should be re-analyzed.

4.1.4. Matrix Spike/Matrix Spike Duplicate Samples (MS/MSD)

Matrix Spike (MS) samples are quality control (QC) samples employed to evaluate the effect a particular sample matrix has on the accuracy of a measurement. A Matrix Spike Duplicate (MSD) sample is a duplicate of the MS sample and provides a means of measuring method precision. As with the MB and LCS, not all methods require the use of an MS/MSD, but when used they provide valuable QC information about the sample.

The MS and MSD are prepared at a rate of 1 per not more than 20 field samples as the analytical methodology requires, providing that sufficient sample volume is available. Although the MS and MSD recoveries do provide information on the overall efficiency of the analytical methods used, true method efficiency is better evaluated with the LCS.

The MS and MSD samples are prepared by adding a known amount of the target analyte(s), and surrogate compounds, if required by the method, to a sample aliquot. These aliquots are separate from (but theoretically equal to) the aliquot to be used to report the target analyte concentrations in the sample. The spike recovery provides an indication of how efficient the analytical procedure was for the particular sample used for the MS. If the MS recovery does not fall within the method acceptance criteria, it may be an indication of sample matrix interferences.[8]

4.1.5. Surrogates

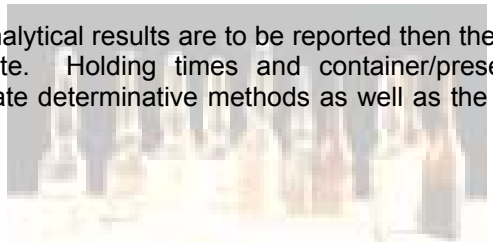
Surrogates are organic compounds that are similar in chemical composition and behavior to target analyte(s) but are not expected to be present in the environment. As applicable to the method, surrogates are added to all field samples, blanks, and QC samples prior to all sample preparation and/or analytical processing. Most organic methods (8015, 8081, 8260, 8270, etc.) use surrogates to help measure the efficiency of the analytical process. Surrogates are calibrated and quantitated using the same process as the target analytes within the analytical procedure.

Surrogate recoveries, in the form of percentages, are calculated by spiking a known amount of surrogate into a sample prior to processing and monitoring the amount of recovered surrogate during analysis. This process provides a simulation of the efficiency of the entire analytical process for the compounds of interest.

4.1.6. Holding Times

Published methods may specify that field samples be extracted and analyzed within certain time limitations. Field sample results must meet holding times in order to be in compliance with the specific method requirements. Upon receipt, field sample documentation is reviewed to assure that proper holding time limitations are met. If the holding times have expired, the client is contacted to determine a course of action.

If the holding times have expired and analytical results are to be reported then the data is flagged as being a minimum estimate. Holding times and container/preservation requirements are noted in the appropriate determinative methods as well as the sample receiving SOP-T100.



4.1.7. Standards and Reagents

Reference standards are purchased from reputable sources and are traceable to an agency standard or are certified by the vendor. The certificates of analysis are maintained in the laboratory and sufficient documentation for traceability is accomplished through the use of standard preparation logbooks with unique solution identification numbers. Standards are verified prior to use to assure that the concentration of a standard or spiking solution is within acceptance criteria.

An independent second-source standard solution (ICV, section 4.3.3) is used to verify initial calibrations and may be used for sample spiking. Documentation of all standards preparation is accomplished by recording data directly into logbooks maintained within the laboratory and all standards are frequently checked for degradation and evaporation and are replaced if a change has occurred. Solutions that have expired shall not be used and should be identified as ‘expired’ and properly disposed of.

4.1.8. Additional Quality Control Samples

Other samples may be utilized to monitor the quality of an analytical procedure. These samples include replicates, duplicates, proficiency testing materials and specially prepared quality control samples. A replicate is a second analysis of the prepared sample used to demonstrate the reproducibility of the analytical process. Replicates do not demonstrate the precision of the entire analytical method, but may be used to demonstrate precision of the instrumental analysis portion.

A duplicate is a second preparation and analysis of a field sample. Being essentially identical, the two samples can, when processed and analyzed together, demonstrate the precision of the entire analytical method or may be used to demonstrate the homogeneous distribution of analyte throughout the sample matrix. The utilization of duplicates to demonstrate precision is limited only by the degree of homogeneity between the duplicate samples. Duplicate analyses are performed at a rate of one pair per analytical batch (usually one in twenty) as required by the determinative method.

4.2. Detection Limits

Method detection limits (MDL) and method reporting limits (RL) are slightly different values used when calculating a sample result. The RL is a legally defensible, quantitative value based upon the lowest calibration standard, whereas the MDL is determined by statistical analysis and is the lowest theoretical level of detection, which is an estimated value and is not as legally defensible.

4.2.1. Method Detection Limits

In general, the MDL is the minimum concentration of an analyte that can be detected in a 'blank' sample measured with 99% confidence that the analyte concentration is greater than zero and determined from analysis of sample in a given matrix containing the analyte. (40 CFR Part 136, Appendix B)

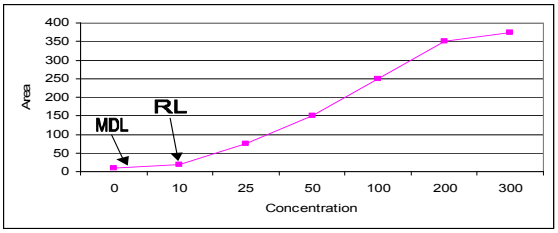
The MDL process involves the analysis, or preparation and analysis, of seven replicate samples that are spiked with all the target analytes, at the same concentration. Once the 7 replicate samples have been analyzed, the standard deviation across all seven values is calculated and using the associated t-value (1% confidence level) the MDL is calculated.

4.2.2. Reporting Limits

The RL is the lowest concentration at which an analyte can be detected in a sample whose concentration can be reported with a reasonable degree of accuracy and precision. In most cases, the RL is based upon two things; the calculated MDL and the lowest standard used for instrument calibration. For most methods, the RL is set somewhere between 2 and 5 times the calculated MDL. But, this value must be supported by the lowest calibration standard for that method. Or stated another way, the lowest calibration standard must be analyzed at or below the RL.

4.2.3. Dilutions – Raising the RL and MDL

When a sample has to be diluted before analysis, either because of matrix problems or to bring the result within the linear range, the RL and MDL must be raised by a factor corresponding to the dilution factor.



4.3 Calibration and Calibration Verification

Calibration is a measurement process that assigns values to the property of an artifact or to the response of an instrument relative to reference standards or to a designated measurement process. The purpose of calibration is to eliminate or reduce bias in the user's measurement system relative to the reference base.

The calibration procedure compares an 'instrument' response with reference standards according to a specific algorithm. The levels of the applied calibration standard should bracket the range of planned or expected sample measurements. Initial instrument calibration is directly used for the quantitation of sample results and continuing instrument calibration verification is used to confirm the continued validity of the initial calibration. The following information outlines basic calibration procedures when instrumentation is involved in the process and does not necessarily apply to many of the manual technique methods used in the lab.

4.3.1. Initial Calibration (ICAL)

For each analyte and surrogate (if applicable) of interest, calibration standards are prepared at the minimum number of concentrations as stated in the appropriate determinative SOP or mandated method. If a reference or mandated method does not specify the number of calibration standards, usually five, the minimum number is three, not including blanks or a zero standard. The lowest concentration standard must be at or below the stated reporting limit for the method based on the final volume of extract (sample). The highest standard normally defines the upper portion of the detector's linear range.

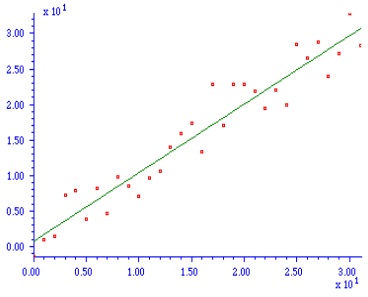
Due to instrument and analyte responses, it may be useful to prepare six or maybe even seven standards for calibration. The lower five standards could be used for most analytes and the upper five for analytes that have poor instrument response. In situations such as these, the reporting limit or linear range will need to be adjusted to account for the standards used in generating the calibration. The analysis of six or more standards also allows for the generation of a least squares regression (quadratic curve) for those analytes that do not have a linear response.

Establish operating parameters based upon method and determinative SOP guidance. Select appropriate columns, injection volumes, detectors, temperatures, temperature programming and flow rates to optimize chromatographic resolution. Once the operating parameters have been established according to the determinative SOP, each instrument may then be calibrated for sample analysis.

Before initial calibration, make sure the system is 'clean' (no results or contamination greater than the reporting limit or ½ the reporting limit depending on the project) by injecting a solvent or instrument blank into the system. If the system is not clean, inject another blank. If contamination is still an issue, perform system maintenance to eliminate the source of the problem before proceeding with the calibration.

Once it is determined that the system is ready for calibration, inject the calibration standards sequentially into the instrument, beginning with the lowest standard first and working up to the highest. An example (ex.1) follows:

Cal 1 - 0.05ppb
Cal 2 - 1.0ppb
Cal 3 - 10ppb
Cal 4 - 20ppb
Cal 5 - 50ppb
Cal 6 - 100ppb *
Cal 7 - 200ppb *
ICV – 2nd Source



* may not be required but may be needed if it is a multiple-analyte method

Do not inject multiple standards of the same calibration level. The process of injecting multiple standards of the same value and then picking the "best" standard is considered "cherry picking' and is not good laboratory practice. A properly maintained system operating at optimal levels should not be difficult to calibrate.

If there are issues with low level standard response then perhaps the standard is 'bad', the column needs trimming or replacing, the system needs to be re-tuned, flows need adjusting, or there are other maintenance-related issues that need addressing before calibration can proceed. Fix the system first. It will save you time in the long run and make the data you produce much more reliable.

Policies regarding the use of calibration standard results for creating the curve are:

- It is never appropriate to remove points from a calibration curve unless they are the highest or the lowest point and the reporting limit and/or linear range is adjusted accordingly. A minimum of 5 points (unless otherwise stated in the method) must remain in the curve for it to be valid.
- A low calibration standard may be excluded from the calibration if the signal-to-noise ratio or spectral criteria are not suitable. The reporting level must be elevated to be the lowest calibration standard used for calibration.
- The upper calibration standard may be excluded if it saturates the detector or is obviously becoming non-linear. Any sample exceeding the upper standard used in the calibration must be diluted and re-analyzed.
- Mid-calibration standards may not be excluded unless an obvious reason is found. In the instance where an analyst can clearly document the reason for removing a middle point (not the highest or lowest) from the curve – bad injection, broken vial, no injection, etc., a single point may be reanalyzed once and the reanalysis used for the calibration. This may only happen if the re-analysis of the point occurs before any QC or client samples are analyzed or before the analytical shift or tune cycle (12 or 24 hours as applicable to the method) has ended. If not useful, or if anything else is run on the instrument, recalibration is required.

4.3.2. Curve Fitting

Once the standards have been analyzed, it is time to pick a calibration curve model from which sample and QC results will be calculated. The determinative SOPs will dictate which model(s) can/should be used for data calculation. The objective of curve fitting is to find a mathematical equation that describes a set of data and that is minimally influenced by random error.

Two common approaches are Calibration Factor (CF) or Relative Response Factor (RF) and "linear least squares", a well-known mathematical procedure that is capable of finding the coefficients of polynomial equations that are a "best fit" to the data. A polynomial equation expresses the dependent variable Y as a polynomial in the independent variable X. In general, there are three main curve models used in the laboratory; 2 linear curve models, relative response factor and least squares regression, and a 2^{nd} order curve model, quadratic.

Once the calibration curve has been generated, carefully review it for compliance with the associated SOP criteria (%RSD, correlation coefficient, etc.). If there is a problem with the calibration using average calibration or response factors, you may need to utilize a linear regression model (1^{st} order), or perhaps quadratic (2^{nd} order) if sufficient standards ($\geq$6) were analyzed. For linear models, a correlation coefficient (r) of 0.995 is required before proceeding with sample analysis. (Please refer to the appropriate determinative SOP for the initial calibration equations.)

4.3.2.1 Linear Calibration using Calibration or Response Factors

Both calibration factors (CF) and response factors (RF) are measures of the slope of the calibration relationship. Each calibration or response factor represents the slope of the line between the response for a given standard and the origin. Under ideal conditions, the factors will not vary with the concentration of the standard. In practice, some variation is to be expected.

However, when the variation, measured as the relative standard deviation (RSD) of the factors, is less than or equal to 20% (or as defined by the applicable method), then the slopes of the lines for each standard are sufficiently close to one another that the use of the linear model is generally appropriate over the range of standards that are analyzed and the average calibration or response factor may be used to determine sample concentrations.

To evaluate the linearity of the initial calibration, calculate the CF (external standard calibration) or RF (internal standard calibration), the standard deviation (SD), and the RSD using the equations found in the appropriate determinative SOPs. For external standard calibration, x is the mass of the analyte in the sample aliquot introduced into the instrument and y is the area (or height) of the response. For an internal standard calibration, x and y can be assigned in various ways where x contains the amount of the analyte introduced into the instrument and y contains the instrument response to that analyte.

Given the potentially large numbers of analytes that may be analyzed in some methods, it is likely that some analytes may exceed the %RSD criteria for a given calibration. In those instances, it is recommended that corrective action is initiated or that a linear calibration model based on a least squares regression be used for those analytes not meeting %RSD criteria. [10]

4.3.2.2 Linear Calibration using Least Squares Regression

A linear least squares regression attempts to construct a linear equation by minimizing the differences between the observed results (the instrument response) and the predicted results. The regression calculation attempts to minimize this sum of the squares, hence the name "least squares regression."

A linear calibration model based on a least squares regression may be employed based on past experience or a priori knowledge of the instrument response. Further, at the discretion of the analyst, this approach also may be used for analytes that do not meet the RSD limits specified in the determinative methods.

The mathematics used in least squares regression has a tendency to favor numbers of larger value over numbers of smaller value. Thus the regression curves that are generated will tend to fit points at the upper end of the curve better than those at the lower end. Weighting the sum of the squares of the differences may significantly improve the ability of the least squares regression to fit the linear model to the data. The weighting of data must be allowed in the SOP.

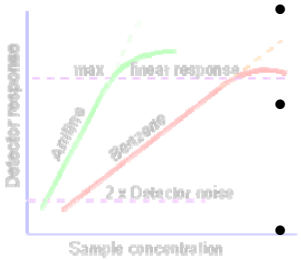
Unless specified in the method to include a 'zero' point, do not force the curve through the 'origin'. Forcing through the origin is not the same as including the origin as a point in the calibration. In essence, if the curve is forced through zero, the intercept is set to zero before the regression is calculated, thereby setting the bias to favor the low end of the calibration range by "pivoting" the function around the origin to find the best fit thus resulting in one less degree of freedom.

For the general case of an unweighted linear least squares regression, i.e., a regression that varies both x and y and with a weighting factor equal to one, the correlation coefficient (r) can be used to measure the "goodness of fit." The data system will typically calculate the correlation coefficient (r). Where an 'r' value of 1.00 indicates a perfect fit for these conditions the minimum criteria using a least squares equation is 0.995.

If other conditions for x and y are used, or the weighting factor is variable, then the coefficient of determination (COD) or r^2 should be used to measure the "goodness of fit" assuming a sufficient number of points are in the curve. [10]

4.3.2.3 Coefficient of Determination (r^2 or Quadratic)

Instead of a linear curve model (Average RF or least squares regression), a second order curve (Quadratic) may be used as long as it contains at least six data points. As a rule of thumb, if there is a consistent trend in RFs (or CFs) in the calibration curve, either up or down, then quadratic curve fit may be indicated as the preferred calibration routine for that analyte. The coefficient of determination (r^2) for the quadratic curve must be at least 0.990 for it to be considered acceptable. Some limitations on the use of Quadratic Curve fits;



- Care MUST be exercised to assure that the results are real, positive, and fit the range of the initial calibration.
- They may not be used to mask instrument problems that can be corrected by maintenance. (Not to be used where the analyte is normally found to be linear in a properly maintained instrument).
- They may not be used to compensate for a saturated detector. If it is suspected that the detector is being saturated at the high end of the curve, remove the higher concentration standards from the curve, leaving at least five, and try a 1st order fit or average RF. [10]

4.3.3. Initial Calibration Verification (ICV)

The ICV is also known as the Second Source Standard because it is acquired from a source other than that used to make up the initial calibration standards. Each new ICAL must be verified using a second-source standard that contains all of the analytes of interest for the method being analyzed.

The ICV is normally prepared at a level that represents the mid-point of the calibration curve and is analyzed immediately after the calibration curve has been analyzed and before the analysis of samples or batch QC begins. (refer to ex.1)

The ICAL is deemed valid if the percent difference (%D), or % drift for the ICV is within the criteria specified in the appropriate determinative SOP. As a general rule the acceptance limits for calibration verifications (%D) are; GCMS + 20%, GC and HPLC + 15%, Inorganics: + 10 or 15%. Actual methods may have wider or tighter limits. Refer to the appropriate determinative SOPs for specifics on acceptance criteria and the associated equations.

Some methods, such as EPA 8260 and EPA TO-15, may use a second source standard for the LCS. In this case, as long as the 'LCS' is analyzed immediately following the calibration standards it is treated as an ICV and must be verified against the ICV criteria.

If the initial calibration has been verified by the ICV then the analysis of samples may proceed. If the ICV does not validate the initial calibration then:

- reanalyze the ICV once,
- reprepare or acquire new standard and analyze once,
- evaluate instrument conditions, and
- regenerate a new ICAL.

4.3.4. Continuing Calibration Verification (CCV)

The CCV is usually made from the same source as the calibration standards but may be from a different source. Once established, every ICAL must be verified periodically by the analysis of a CCV. The process of calibration verification applies to both external standard and internal standard calibration techniques, as well as to linear and non-linear calibration models.

In general, the ICAL must be verified at the beginning of each 12-hour analytical shift during which samples are analyzed. The analytical shift begins with the injection of the CCV (or the tuning standard then the CCV in GCMS). A CCV must be repeated at the beginning and, for methods that use external calibration models, at a minimum, the end of each analytical batch. Some methods or client/project directives may have more frequent CCV requirements; opening CCV, closing CCV and every 10 or 20 samples, and many inorganic methods require the CCV to be analyzed after ever 10 samples.

> It is recommended that only one CCV be analyzed at a time. The process of routinely analyzing 2 CCV's in a row creates the potential for confusion regarding acceptance of the sequence. If both pass, or both fail, then the acceptability or the corrective action needed is clear. But, if one passes and one fails then the analyst is left to decide the validity of the associated data.

If the initial CCV passes but the second CCV fails then any data previous to the initial CCV has been successfully bracketed, but any data following the failed CCV has not been, (the 'opening' CCV has failed). Corrective action for the later samples would need to be effected (possibly involving recalibration) and the samples re-analyzed. The opposite may be true for an initial CCV that failed but the second CCV passed.

The ICAL is deemed valid if the percent difference (%D), or % drift for the CCV is within the criteria specified in the appropriate determinative SOP. As a general rule the acceptance limits for calibration verifications (%D) are; GCMS + 20%, GC and HPLC + 15%, Inorganics: + 10 or 15%. Actual methods may have wider or tighter limits; refer to the appropriate determinative SOPs for specifics on acceptance criteria and the associated equations.

If the response (or calculated concentration) for an analyte is within the acceptance limits of the response obtained during the initial calibration process, then the ICAL is considered still valid, and the analyst may continue to use the CF, RF or % drift values from the ICAL to quantitate sample results.

If the response (or calculated concentration) for any analyte varies from the mean response obtained during the initial calibration process by more than the acceptance criteria, then the initial calibration relationship may no longer be valid.

If routine corrective action procedures fail to produce a second consecutive CCV within acceptance criteria, then either the laboratory has to demonstrate performance after corrective action with two consecutive successful CCVs (one below the mid-point of the curve), or a new ICAL must be performed.

A second CCV may be analyzed following a failing CCV if no samples / QC have been analyzed and the analytical tune or batch period has not expired. The reason for the analysis of the additional CCV must be documented in the run log - i.e., note why the initial CCV failed and what corrective action was taken.

However, sample data associated with an unacceptable CCV may be reported as qualified data under the following special conditions:

When the acceptance criteria for the CCV are exceeded high, i.e., high bias, and there are associated samples that are non-detects, then those non-detects may be reported. Otherwise, the samples affected by the unacceptable CCV shall be reanalyzed after a new ICAL has been established, evaluated and accepted.

When the acceptance criteria for the CCV are exceeded low, i.e., low bias, those sample results may be reported if they exceed a maximum regulatory limit/action level. Otherwise, the samples affected by the unacceptable CCV shall be reanalyzed after a new ICAL has been established, evaluated and accepted.

4.4. GC/MS Tuning

GC/MS has become a routine analytical technique for the analysis of environmental pollutants and methods have been developed for the analysis of both volatile and non-volatile components in a variety of matrices. In an attempt to standardize and monitor the performance of GC/MS systems throughout the environmental laboratory community, the Environmental Protection Agency (EPA) has developed the use of tuning compounds.

Bromofluorobenzene (BFB) and decafluorotriphenylphosphine (DFTPP) are routinely used as the instrument tuning compounds for the analysis of volatile and semi-volatile organic compounds, respectively. The tuning compounds are injected into the GC/MS under standardized conditions and the resulting spectra are examined to determine the performance characteristics of the system which include mass resolution, relative ion abundance, and mass accuracy over the mass range.

The mass spectrometer is tuned to obtain a spectrum for BFB or DFTPP that meets all the criteria as specified in the determinative method being used. After the GC/MS system meets the tuning criteria, it can be used for sample analysis.[9]

4.5 Data Integrity and Ethics

Data Integrity is a comprehensive program of behavioral, administrative, management, operational expectations and procedures to ensure that produced analytical data is technically sound and legally defensible. It relies on the adherence of all employees to the policies and procedures maintained by the laboratory with regards to the production of analytical results.

A cornerstone of the data integrity policy is the understanding that all employees will act in an ethical manner and will not alter data such that the data are unauthentic or untrue representations of the test performed. To reinforce this philosophy, employees are to read and understand the Data Integrity SOP-T065, undergo annual integration and ethics training and follow the processes as outlined in the training.

Another important aspect of data integrity is the traceability of that which may impact the sample results, in other words, documentation. Procedures for documenting sample receipt, preparation of samples and standards, extraction and environmental conditions in the laboratory, recording of analytical results, etc. are all outlined in the SOPs. But, there are times when data is recorded incorrectly and laboratory personnel are expected to address the errors quickly and correctly.

When a documentation error is noted the appropriate correction technique is to draw a single line through the error such that the item identified as incorrect can still be seen. (No obliterating of data or scribbling over the error). Once a line has been drawn through the error, write the corrected value down close to the original value, but not over it, and then initial and date the correction. Data errors are not the only items that need correcting in the laboratory. Often times, chromatographic data will need to be corrected due to any one of many factors. The correction of baseline integration on a data system is called manual integration and is addressed below.

4.5.1 Manual Integration

Manual integration (MI) is employed to correct an improper integration performed by the data system. Efforts should be made during method development to define the most optimum instrument parameters that, in most cases, allows for automatic integration by the data system. However, regardless of the sophistication of the software, instances occur when the software does not integrate a peak correctly.

The failure of the software to appropriately integrate a peak is usually obvious from visual inspection of the chromatogram (at an appropriate scale). Various errors occur which include, but are not limited to, peak splitting, adding area due to a coeluting interferant, failure to detect a peak, excessive peak tailing due to failure of the instrument response to return to baseline or a rise in the baseline, and failure to separate peaks.

The software packages invariably provide a procedure where by the analyst can review the individual data file and provide peak specific instructions on integration to correct these problems. This procedure is referred to as "manual integration" and relies solely upon the experience of the analyst to determine proper integration for each peak. All data must be integrated consistently in standards, samples and QC samples. Integration parameters, both automated and manual, must adhere to valid scientific chromatographic principles and be fully documented.

Manual integration is employed to correct an improper integration performed by the data system and must always include documentation clearly stating the reason the manual integration was performed, who completed the work, and the initials of the reviewer approving the MI. Under no circumstances should manual integration be performed solely for the purpose of meeting quality control criteria. In other words, peak shaving, peak enhancing, or manipulations of the baseline to achieve these ends must never occur as this results in an improper integration rather than correcting a data system error.

4.6. Data Review

Once data has been analyzed it must be reviewed before it can be reported. Data review occurs at three different levels, two in the laboratory and one in project management. A data review checklist is used to document the initial part of the review.

4.6.1 Bench Level Review

The first round of data review occurs at the bench level. Analysts are expected to review the initial and continuing calibrations for compliance with the criteria specified in the associated SOPs. Method blanks and instrument blanks are evaluated for contamination and LCS/MS/MSD's are evaluated against control limits established for each matrix. Surrogates and internal standards, if used, are also evaluated against acceptance criteria. In the instance where a quality control parameter does not meet criteria, the bench level chemist should effect appropriate corrective actions as outlined in the determinative or QC-based SOPs.

4.6.2. Second Level Review

After the bench level review is completed, the data is reviewed by a second person. The data is reviewed for the same items as are done at the bench level. It is important for the second level reviewer to confirm the appropriateness of any manual integrations performed on the data.

4.6.3. Completeness Review

After the data has been released by the laboratory, it is sent to the appropriate Project Manager for reporting to the client. At this stage, the data is reviewed for completeness with the associated project objectives and any outliers, discrepancies or anomalies with the data are noted in a narrative.

5.0 PROJECT MANAGEMENT

Project Management is a systemic process of understanding a client's requirements, developing guidelines for implementation and adherence of those requirements by the laboratory, review of final reports to ensure those requirements have been met, and any follow up work based upon client feedback after receipt of the analytical report.

The process entails (but is not limited to) project inquiries, project development, certifications, quotations, regulatory guidance, analytical advisement, sample containers, holding times, shipping, subcontracting, sample receipt, work order log-in, QA/QC issues, EDD generation, invoicing and final report review.

Analytical reports are a compilation of practices, procedures and employee work product that are designed to provide a final report that meets the client's requirements. It is incumbent for the Project Manager (PM) to efficiently review all data packages so that any remedial analysis, if so needed, can be performed within analytical holding time. Proper ongoing project management will be helpful in precluding "surprises" as the report comes due.

Assuming all data packages are complete the (PM) assembles the final report package in LIMS, generates any needed EDDs and invoices the work. Most analytical reports are sent to the client in a pdf format where others may require a wet signature or may need to be assembled in a Level III or Level IV data package format. If a Level III or IV package is required, associated checklists are available for use by the bench level chemists and the PM to ensure completeness of the data.

6.0 HEALTH AND SAFETY

Health and Safety is important to the productive operation of the laboratory. Employees are expected to comply with all internal health and safety and accident prevention policies and procedures as outlined in the routine SOPs as well as the Health, Safety and Respiratory Protection Program Manual. Personnel are furnished with the applicable controls and personal protective equipment as needed for the material they are handling and it is expected that they will use the equipment as prescribed.

Employees are required to read product, standard and reagent labels and the associated material safety data sheets (MSDS's) to determine safe usage procedures and what safety equipment needs to be on hand prior to commencing any task. MSDS sheets should be readily available in the laboratory areas. Employees are also expected to report all accidents, dangerous occurrences, or potentially hazardous situations to their immediate supervisor and to complete any needed paperwork associated with the accident, incident or near-miss.

7.0 TERMS AND DEFINITIONS

The following definitions are used in the text of Quality Systems. The following hierarchy of definition references was used: ISO 8402, ANSI/ASQC E-4, EPA's Quality Assurance Division Glossary of Terms, and finally definitions developed by NELAC. The source of each definition, unless otherwise identified, is the Quality Systems Committee.

Acceptance Criteria: Specified limits placed on characteristics of an item, process, or service defined in requirement documents.[1]

Accuracy: The degree of agreement between an observed value and an accepted reference value. Accuracy includes a combination of random error (precision) and systematic error (bias) components which are due to sampling and analytical operations; a data quality indicator.[7]

Analysis Duplicate: The second measurement of the target analyte(s) performed on a single sample or sample preparation.

Analyst: The designated individual who performs the "hands-on" analytical methods and associated techniques and who is the one responsible for applying required laboratory practices and other pertinent quality controls to meet the required level of quality.[6]

Analytical Reagent (AR) Grade: Designation for the high purity of certain chemical reagents and solvents given by the American Chemical Society.[7]

Assessment: The evaluation process used to measure or establish the performance, effectiveness, and conformance of an organization and/or its systems to defined criteria (to the standards and requirements of NELAC).[6]

Audit: A systematic evaluation to determine the conformance to quantitative and qualitative specifications of some operational function or activity.[8]

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

Blank: A sample that has not been exposed to the analyzed sample stream in order to monitor contamination during sampling, transport, storage or analysis. The blank is subjected to the usual analytical and measurement process to establish a zero baseline or background value and is sometimes used to adjust or correct routine analytical results.[1]

Calibration: To determine, by measurement or comparison with a standard, the correct value of each scale reading on a meter or other device. The levels of the applied calibration standard should bracket the range of planned or expected sample measurements.[6]

Certified Reference Material (CRM): A reference material one or more of whose property values are certified by a technically valid procedure, accompanied by or traceable to a certificate or other documentation which is issued by a certifying body.[4]

Chain of Custody Form: A record that documents the possession of the samples from the time of collection to receipt in the laboratory. This record generally includes: the number and types of containers; the mode of collection; collector; time of collection; preservation; and requested analyses.[6]

Compromised Sample: a sample received in a condition that jeopardizes the integrity of the results. Samples are considered "compromised" if the following conditions are observed upon receipt:

- Cooler and/or samples are received outside of temperature specification
- Samples are received broken or leaking
- Samples are received beyond holding time
- Samples are received without appropriate preservative
- Samples are received with inadequate volume to perform the analysis
- Samples are received in inappropriate containers
- COC does not match samples received
- COC is not properly completed or not received
- Breakage of any Custody Seal or apparent tampering with cooler and/or samples
- Headspace (>6mm) in volatiles samples
- Seepage of extraneous water or materials into samples
- Illegible, impermanent, or non-unique sample labeling

When "compromised" samples are received, it is documented on the sample receipt form and the client is contacted for instructions. If the client decides to proceed with analysis, the project report narrative and/or qualifiers within the analytical report should clearly indicate any of the above conditions and the resolution.

Confidential Business Information: information that an organization designates as having the potential of providing a competitor with inappropriate insight into its management, operation or products.

Confirmation: Verification of the identity of a component through the use of an approach with a different scientific principle from the original method. These may include, but are not limited to:

- Second column confirmation;
- Alternate wavelength;
- Derivatization;
- Mass spectral interpretation;
- Alternative detectors; or
- Additional cleanup procedures.[6]

Conformance: An affirmative indication or judgment that a product or service has met the requirements of the relevant specifications, contract, or regulation; also the state of meeting the requirements.[1]

Corrective Action: The action taken to eliminate the causes of an existing nonconformity, defect or other undesirable situation in order to prevent recurrence. (ISO 8402) Deviations from Calscience's quality assurance manual, controlled SOP's, deficiencies, errors, or out-of-control situations require corrective action. Documentation of the problem, identification of the cause and follow-up action to prevent recurrence is accomplished using a Corrective Action Form.

Data Audit: A qualitative and quantitative evaluation of the documentation and procedures associated with environmental measurements to verify that the resulting data are of acceptable quality (i.e., that they meet specified acceptance criteria).[6]

Data Reduction: The process of transforming raw data by arithmetic or statistical calculations, standard curves, concentration factors, etc., and collation into a more useable form.[8]

Deficiency: An unauthorized deviation from acceptable procedures or practices, or a defect in an item.[1]

Demonstration of Capability: A procedure to establish the ability of the analyst to generate acceptable accuracy.[6]

Detection Limit: The lowest concentration or amount of the target analyte that can be identified, measured, and reported with confidence that the analyte concentration is not a false positive value. See Method Detection Limit.[6]

Document Control: The act of ensuring that documents (and revisions thereto) are proposed, reviewed for accuracy, approved for release by authorized personnel, distributed properly and controlled to ensure use of the correct version at the location where the prescribed activity is performed.[3]

Duplicate Analyses: The analyses or measurements of the variable of interest performed identically on two subsamples of the same sample. The results from duplicate analyses are used to evaluate analytical or measurement precision but not the precision of sampling, preservation or storage internal to the laboratory.[8]

Equipment Blank: a portion of the final rinse water used after decontamination of field equipment; also referred to as Rinsate Blank and Equipment Rinsate.

Field Blank: a blank matrix brought to the field and exposed to field environmental conditions.

Good Laboratory Practices (GLP): formal regulations for performing basic laboratory operations outlined in 40 CFR Part 160 and 40 CFR Part 729.

Holding Blank: a blank matrix stored with field samples of a similar matrix.

Holding Times (Maximum Allowable Holding Times): The maximum times that samples may be held prior to analysis and still be considered valid or not compromised.[9]

Integration: The determination of the area under a curve. Usually an automated process performed by the instrument software to determine peak area, or height, for the purpose of quantitation.

Internal Chain of Custody (ICOC): an unbroken trail of accountability that ensures the physical security of samples, data and records. Internal Chain of Custody refers to additional documentation procedures implemented within the laboratory that includes special sample storage requirements, and documentation of all signatures and/or initials, dates, and times of personnel handling specific samples or sample aliquots.

Instrument Detection Limit (IDL): the minimum amount of a substance that can be measured with a specified degree of confidence that the amount is greater than zero using a specific instrument. The IDL is associated with the instrumental portion of a specific method only, and sample preparation steps are not considered in its derivation. The IDL is a statistical estimation at a specified confidence interval of the concentration at which the relative uncertainty is +100%. The IDL represents a range where qualitative detection occurs on a specific instrument. Quantitative results are not produced in this range.

Internal Standard: A known amount of standard added to a test portion of a sample as a reference for evaluating and controlling the precision and bias of the applied analytical method.[6]

Instrument Blank: A clean sample (e.g., distilled water) processed through the instrumental steps of the measurement process; used to determine instrument contamination.[8]

LIMS: Laboratory's information and data management system.

Laboratory Control Sample (however named, such as laboratory fortified blank, spiked blank, or QC check sample): A sample matrix, free from the analytes of interest, spiked with verified known amounts of analytes or a material containing known and verified amounts of analytes. It is generally used to establish intra-laboratory or analyst-specific precision and bias or to assess the performance of all or a portion of the measurement system.[6]

Laboratory Duplicate: Aliquots of a sample taken from the same container under laboratory conditions and processed and analyzed independently.[6]

Limit of Detection (LOD): The lowest concentration level that can be determined by a single analysis and with a defined level of confidence to be statistically different from a blank. See also Method Detection Limit, Detection Limit, and Quantitation Limit. (Analytical Chemistry, 55, p. 2217, December 1983, modified)

Limit of Quantitation (LOQ): the minimum amount of a substance that can be quantitatively measured with a specified degree of confidence and within the accuracy and precision guidelines of a specific measurement system. The LOQ can be based on the MDL, and is generally calculated as 3-5 times the MDL. Also referred to as Reporting Limit (RL), Practical Quantitation Level (PQL) and Estimated Quantitation Level (EQL)

Manual Integration: Manual Integration is defined as the process by which an analyst can reset the baseline of a peak during reprocessing and quantitation such that the baseline is different from the original automated process set by the instrument run parameters. It is used to provide accurate quantitation of peak areas where the original integration provided by the data system is in error. Additionally, in some cases the data system might misidentify the peak; this situation must be corrected by manual integration.

Matrix Spike (spiked sample or fortified sample): A sample prepared by adding a known mass of target analyte to a specified amount of matrix sample for which an independent estimate of target analyte concentration is available. Matrix spikes are used, for example, to determine the effect of the matrix on a method's recovery efficiency.[7]

Matrix Spike Duplicate (spiked sample or fortified sample duplicate): A second replicate matrix spike prepared in the laboratory and analyzed to obtain a measure of the precision of the recovery for each analyte.[7]

May: Denotes permitted action, but not required action.[6]

Method Blank: A sample of a matrix similar to the batch of associated samples (when available) that is free from the analytes of interest and is processed simultaneously with and under the same conditions as samples through all steps of the analytical procedures, and in which no target analytes or interferences are present at concentrations that impact the analytical results for sample analyses.[6]

Method Detection Limit: The minimum concentration of a substance (an analyte) that can be measured and reported with 99% confidence that the analyte concentration is greater than zero and is determined from analysis of a sample in a given matrix containing the analyte.[9]

Must: Denotes a requirement that must be met. (Random House College Dictionary)

National Environmental Laboratory Accreditation Program (NELAP): The overall National Environmental Laboratory Accreditation Program of which NELAC is a part.[6]

Negative Control: Measures taken to ensure that a test, its components, or the environment do not cause undesired effects, or produce incorrect test results.[6]

Nonconformance: an indication, judgement, or state of not having met the requirements of the relevant specifications, contract, or regulation. A nonconformance is typically defined as an unplanned deviation from an established protocol. An occurrence of a nonconformance may be the result of Calscience's actions, which would be rendered as a deficiency, or the result of events beyond Calscience's control, which would be termed an anomaly. In either case the non-conforming event needs to be documented and any needed corrective action commenced to prevent a reoccurrence.

Performance Audit: The routine comparison of independently obtained qualitative and quantitative measurement system data with routinely obtained data in order to evaluate the proficiency of an analyst or laboratory.[6]

Performance Based Measurement System (PBMS): A set of processes wherein the data quality needs, mandates or limitations of a program or project are specified and serve as criteria for selecting appropriate test methods to meet those needs in a cost-effective manner.[6]

Positive Control: Measures taken to ensure that a test and/or its components are working properly and producing correct or expected results from positive test subjects.[6]

Precision: The degree to which a set of observations or measurements of the same property, obtained under similar conditions, conform to themselves; a data quality indicator. Precision is usually expressed as standard deviation, variance or range, in either absolute or relative terms.[6]

Preservation: Refrigeration and/or reagents added at the time of sample collection (or later) to maintain the chemical and/or biological integrity of the sample.[6]

Proficiency Testing Program: The aggregate of providing rigorously controlled and standardized environmental samples to a laboratory for analysis, reporting of results, statistical evaluation of the results and the collective demographics and results summary of all participating laboratories.[6]

Proficiency Test Sample (PT): A sample, the composition of which is unknown to the analyst and is provided to test whether the analyst/laboratory can produce analytical results within specified acceptance criteria.[7]

Protocol: A detailed written procedure for field and/or laboratory operation (e.g., sampling, and analysis) which must be strictly followed.[8]

Pure Reagent Water: Shall be water (defined by national or international standard) in which no target analytes or interferences are detected as required by the analytical method.[6]

Quality Assurance: An integrated system of activities involving planning, quality control, quality assessment, reporting and quality improvement to ensure that a product or service meets defined standards of quality with a stated level of confidence.[7]

Quality Assurance (Project) Plan (QAPP): A formal document describing the detailed quality control procedures by which the quality requirements defined for the data and decisions pertaining to a specific project are to be achieved.[8]

Quality Control: The overall system of technical activities whose purpose is to measure and control the quality of a product or service so that it meets the needs of users.[7]

Quality Control Sample: An uncontaminated sample matrix with known amounts of analytes from a source independent from the calibration standards. It is generally used to establish intra-laboratory or analyst specific precision and bias or to assess the performance of all or a portion of the measurement system.[8]

Quality Assurance Manual: A document stating the management policies, objectives, principles, organizational structure and authority, responsibilities, accountability, and implementation of an agency, organization, or laboratory, to ensure the quality of its product and the utility of its product to its users.[6]

Quality System: A structured and documented management system describing the policies, objectives, principles, organizational authority, responsibilities, accountability, and implementation plan of an organization for ensuring quality in its work processes, products (items), and services. The quality system provides the framework for planning, implementing, and assessing work performed by the organization and for carrying out required QA and QC.[3]

Quantitation Limits: Levels, concentrations, or quantities of a target variable (e.g., target analyte) that can be reported at a specific degree of confidence.[6]

Range: The difference between the minimum and the maximum of a set of values.[8]

Raw Data: Any original factual information from a measurement activity or study recorded in a laboratory notebook, worksheets, records, memoranda, notes, or exact copies thereof that are necessary for the reconstruction and evaluation of the report of the activity or study.

Raw data may include photography, microfilm or microfiche copies, computer printouts, magnetic media, including dictated observations, and recorded data from automated instruments. If exact copies of raw data have been prepared (e.g., tapes which have been transcribed verbatim, data and verified accurate by signature), the exact copy or exact transcript may be submitted.[8]

Reagent Blank (method reagent blank): A sample consisting of reagent(s), without the target analyte or sample matrix, introduced into the analytical procedure at the appropriate point and carried through all subsequent steps to determine the contribution of the reagents and of the involved analytical steps.[7]

Reagent Water: water (defined by national or international standard) in which no target analytes or interferences are detected as required by the analytical method.

Record Retention: The systematic collection, indexing and storing of documented information under secure conditions.[8]

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.[4]

Reference Method: A method of known and documented accuracy and precision issued by an organization recognized as competent to do so. [6]

Reference Standard: A standard, generally of the highest metrological quality available at a given location, from which measurements made at that location are derived.[5]

Replicate Analyses: The measurements of the variable of interest performed identically on two or more sub-samples of the same sample within a short time interval.[6]

Requirement: Denotes a mandatory specification; often designated by the term "shall".[6]

Sampling and Analysis Plan (SAP): a formal document describing the detailed sampling and analysis procedures for a specific project.

Sampling Media: Material used to collect and concentrate the target analytes(s) during air sampling such as solid sorbents, filters, or impinger solutions.

Selectivity: (Analytical chemistry) The capability of a test method or instrument to respond to a target substance or constituent in the presence of non-target substances.[8]

Sensitivity: The capability of a method or instrument to discriminate between measurement responses representing different levels (e.g., concentrations) of a variable of interest.[6]

Shall: Denotes a requirement that is mandatory whenever the criterion for conformance with the specification requires that there be no deviation. This does not prohibit the use of alternative approaches or methods for implementing the specification so long as the requirement is fulfilled.[2]

Should: Denotes a guideline or recommendation whenever noncompliance with the specification is permissible.[2]

Spike: A known mass of target analyte added to a blank sample or sub-sample; used to determine recovery efficiency or for other quality control purposes.[6]

Standard: The document describing the elements of laboratory accreditation that has been developed and established within the consensus principles of NELAC and meets the approval requirements of NELAC procedures and policies.[3]

Standard Operating Procedure (SOP): A written document which details the method of an operation, analysis or action whose techniques and procedures are thoroughly prescribed and which is accepted as the method for performing certain routine or repetitive tasks.[6]

Standardized Reference Material (SRM): A certified reference material produced by the U.S. National Institute of Standards and Technology or other equivalent organization and characterized for absolute content, independent of analytical method.[8]

Surrogate: A substance with properties that mimic the analyte of interest. It is unlikely to be found in environment samples and is added to them for quality control purposes.[7]

Systems Audit (also Technical Systems Audit): A thorough, systematic, qualitative on-site assessment of the facilities, equipment, personnel, training, procedures, record keeping, data validation, data management, and reporting aspects of a total measurement system.[8]

Test: A technical operation that consists of the determination of one or more characteristics or performance of a given product, material, equipment, organism, physical phenomenon, process or service according to a specified procedure. The result of a test is normally recorded in a document sometimes called a test report or a test certificate.[4]

Test Method: An adoption of a scientific technique for a specific measurement, as documented in a laboratory SOP. [6]

Testing Laboratory: Laboratory that performs tests.[4]

Traceability: The property of a result of a measurement whereby it can be related to appropriate standards, generally international or national standards, through an unbroken chain of comparisons.[5]

Trip Blank*:* a blank matrix placed in a sealed container at the laboratory that is shipped, held unopened in the field, and returned to the laboratory in the shipping container with the field samples.

Validation: The process of a second party performing a systematic review of the raw and final data produced by a laboratory using predetermined criteria to ascertain the validity of the data with respect to the criteria.

Verification: confirmation by examination and provision of evidence that specified requirements have been met. [6]

> In connection with the management of measuring equipment, verification provides a means for checking that the deviations between values indicated by a measuring instrument and corresponding known values of a measured quantity are consistently smaller than the maximum allowable error defined in a standard, regulation or specification peculiar to the management of the measuring equipment. The result of verification leads to a decision to restore in service, to perform adjustment, to repair, to downgrade, or to declare obsolete.

Work Cell: A well-defined group of analysts that together perform the method analysis. The members of the group and their specific functions within the work cell must be fully documented.[6]

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