

Field Sampling Reference Guide



State of Oregon
Department of
Environmental
Quality

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*DEQ is a leader in
restoring, maintaining
and enhancing the quality
of Oregon's air, land and
water.*

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Alternative formats (Braille, large type) of this document can be made available. Contact DEQ's Office of Communications & Outreach, Portland, at (503) 229-5696, or toll-free in Oregon at 1-800-452-4011, ext. 5696.

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1.0 INTRODUCTION

The goal of **DEQ LEAD's Field Sampling Reference Guide** is to provide agency staff with information they need to make knowledgeable and efficient use of the resources available from the Laboratory and Environmental Assessment Division (LEAD). Available LEAD services include: broad technical capabilities in qualitative and quantitative inorganic, organic, microbiological, microscopic, and biological sampling and analyses of air, water, soil, sediment, and tissue; quality assurance support for: data validation, statistical data analysis, analytical data interpretation and QA Plan review and approval; etc. **Note:** The user is responsible to review their specific sampling and analysis needs with the analytical chemistry managers well in advance of sample collection since methods do change and there may be specific sample matrices or analyses that require special handling not covered in this document.

The Division is divided into seven functional sections: Air Monitoring, Water Monitoring, Watershed Assessment, Inorganic Laboratory, Organic Laboratory, Technical Services, and Administration (including Quality Assurance). Each year the laboratory logs in, analyzes, and reports data on approximately 1400 separate sampling events, each consisting of from 1 to 75 samples. The DEQ Laboratory and Environmental Assessment Division is located at 3150 NW 229th, Suite 150, Hillsboro, OR 97124. It is located in the same building as the Oregon Public Health Laboratory.

Note: *Hyperlinks are inserted throughout this document to connect to other documents that may be referenced; however, they may not work on all computers. Many of the hyperlinks are to documents located on DEQ's internal network and will only work if the user of this document is connected to DEQ's intranet. There are also many hyperlinks that link to various websites and will function on any computer connected to the internet.*

2.0 INFORMATION CONTACTS

LEAD must be informed as early as possible, prior to sample collection, to insure that both your needs and the LEAD's are met in a timely manner. Contact the Sample Coordinator for information on sample requirements, sampling equipment needs and availability, field preservation (also found in DEQs' [Water Monitoring and Assessment Mode of Operations Manual](#) (MOMs)) or filtration requirements, sample transport, and scheduling. Speak to the appropriate Section Manager about analytical capabilities that are available.

For parameters having a short holding time (24 hours, or less refer to [APPENDIX I](#)), schedule sample collection thoughtfully. Inform the sample coordinator of your intent to sample, approximate number of samples, what you are looking for, and when samples will be delivered to the laboratory. Analysts need to be made aware of your needs and expectations so schedules and workloads can be adjusted to accommodate the work.

2.1 Sampling and Analytical Information

(503) 693-5700

2.1.1. SAMPLE COORDINATOR

Shannon Swantek (503) 693-5784: Type of container for specific tests, general inorganic and organic sampling inquiries, sampling supplies, sample preservation, equipment, preliminary results.

2.1.2. ORGANIC LABORATORY MANAGER

Brian Boling (503) 693-5745: Organic and Physical Test laboratory capabilities, sample requirements, methods, data interpretation.

2.1.3. **ORGANIC – LEAD CHEMIST**

Sara Krepps (503) 693-5749: Analytical methods, data interpretation, and test requirements.

2.1.4. **INORGANIC LABORATORY MANAGER**

TBD (503) 693-5757: Inorganic laboratory capabilities, sample requirements, methods, data interpretation.

2.1.5. **METALS – Metals Chemist**

Karen Yates (503) 693-5769: Analytical methods, data interpretation, and test requirements.

2.1.6. **NONMETALS – LEAD CHEMIST**

Linda McRae (503) 693-5765: Analytical methods, data interpretation, and specific test requirements.

2.1.7. **AIR MONITORING - MANAGER**

TBD (503) 693-5719: Air and meteorological monitoring procedures, capabilities, data interpretation.

2.1.8. **WATER QUALITY MONITORING MANAGER**

Aaron Borisenko (503) 693-5723: Water sampling/monitoring procedures, sampling capabilities, data interpretation, biomonitoring. Surface, soils, sediments, estuaries, lakes, streams, rivers, etc.

2.1.9. **TECHNICAL SERVICES MANAGER**

Paul Seidel (503) 693-5781. Analytical data storage and retrieval, and requests that do not fall in one of the other sections, such as sampling plan design, data management, interpretation or support on specialized project needs. For status of analytical work in progress, LEAD can provide users with a desktop application allowing you to view the status of your work directly.

2.1.10. **QUALITY ASSURANCE OFFICERS**

- **Chris Redman Laboratory and WQ (503) 693 5706**
- **Scott Hoatson Agency and LQ (503) 693-5786**
- **Chris Moore Air Quality Monitoring (503) 693-5722**

General sampling and analytical QA/QC requirements and assistance, QA project plan development and approval, data quality needs, data quality assessment, split sample results, coordination of quality requirements, etc.

2.1.11. **LEAD DIVISION ADMINISTRATOR -**

Greg Pettit (503) 693-5705: Reassignment of work priorities, budgeting, cost recovery, overall laboratory administration.

3.0 SAFETY CONSIDERATIONS

Any field activity or complaint investigation can lead into the realm of the unknown. Approach any complaint investigation as a potentially hazardous event. Obtain the proper training to recognize, deal with, and protect yourself from hazardous chemicals. If you have any questions about what you might be dealing with, utilize existing resources (e.g. MSDS, literature, and laboratory staff) and contact the appropriate authority (e.g. DEQ Health & Safety Manager, Laboratory Managers, Safety Committee,

etc.). The DEQ Safety Committee continually reviews safety and health needs. The Health & Safety Manager can recommend and supply the most appropriate Personal Protective Equipment.

3.1 DEQ Safety Committee

DEQ has 3 safety committees: The Central committee has members across all of DEQ and work with agency-wide safety concerns. The Vehicle Inspection Program (VIP) and laboratory and Environmental Assessment Division (LEAD) both have safety committees dedicated to safety concerns specific to their operations. Below is the current list of Central committee members. The most up to date list and the list of VIP and LEAD committee members can be found on DEQ's intranet (Q-Net) at:

</deq05/intranet/msd/HR/h&s/Committees/CommitteeMembers.htm>

3.2 Portable "Sniffers"

Portable photoionization detectors (PID) (MicroTip™ and HNu™) are available to screen for the presence of toxic gases. The MicroTip™ is also capable of collecting an air sample in a bag for subsequent qualitative or quantitative analysis at the laboratory. LQ has an Organic Vapor Analyzer (OVA) and Regions possess Combustible Gas Analyzers (CGA-GasTek™). These are "sniffers," used to screen for hazards prior to site entry. Though they are used for screening, they should be calibrated periodically to ensure they are performing correctly. It should be noted that they cannot always discriminate one pollutant from the next, nor are they very quantitative.

3.3 Chemical Preservatives

In order to stabilize samples for certain tests it is necessary to add a chemical preservative, in addition to immediate cooling, storage, and transport on ice. Failure to add the proper preservative, or amount, could result in samples deteriorating to the point of being useless for analysis.

The DEQ laboratory supplies sample preservative chemicals that can be picked up by agency personnel on request (**Note:** chemicals cannot be shipped). Staff requesting these chemicals are expected to be familiar with and able to implement the necessary safety precautions, or to ask for LEAD assistance.

Liquid preservatives are provided in 50 milliliter **Teflon** bottles¹ having integral dropper spouts with tethered closures. Sodium hydroxide is supplied in screw-cap plastic bottles containing less than 25 grams of pure caustic pellets. Each bottle is labeled with the appropriate hazard warnings and sealed in a **Zip-Lock** plastic bag to contain leakage, should the bottle closure fail in transit. Any liquid observed inside the plastic bag should be assumed to be leakage from the bottle, in which case do not use the bottle and the bottle and bag should either be returned to the laboratory or disposed of properly. Solid sodium hydroxide pellets are *deliquescent*, that is, they absorb moisture from the air. This chemical is capable of absorbing enough moisture to put itself into solution. Make sure that lids are screwed down tightly!

3.3.1. Preservatives commonly provided

- **Concentrated** Sulfuric Acid, H₂SO₄, 95+ %
- **Concentrated** Nitric Acid, HNO₃, 70+ % (Trace Metal grade or better)
- **Concentrated** Phosphoric Acid, H₃PO₄, 85%
- **Concentrated** Hydrochloric Acid, HCL, 37%
- **Solid** Sodium Hydroxide, NaOH, 100%

Note: At the concentration supplied for sample preservation these chemicals are **highly corrosive** and **capable of causing severe physical damage to skin, eyes, and clothing** unless the proper precautions are observed. Treated with appropriate respect, and following a few simple rules, these chemicals should not cause injury.

¹ When these Teflon bottles become empty **DON'T** throw them away! They cost nearly \$20 each. Return them to the Laboratory so they can be refilled.

- a) Transport preservative containers in a restrained, upright position.
- b) Wear appropriate protective eyewear. (Safety glasses or goggles)
- c) Wear chemically resistant synthetic gloves (Nitrile or Silver Shield). Only use powder-free gloves as the powder is a common source of contamination.
- d) Be prepared to deal with accidents

3.3.2. Safety Data Sheets (SDS)

One source of information on hazards associated with each of these chemicals, and the precautions that should be taken during use, are the **Safety Data Sheets (SDS)**. Previously, these were known as **Material Safety Data Sheets (MSDS)**. **In 2013, OSHA has implemented new regulation that change the name, content and format for the data sheets. MSDS/SDS's for each of the above chemicals may be obtained from the laboratory.** Location Specific MSDs can be found on the DEQ internal website (Q-Net) at <http://deq05/intranet/msd/HR/h&s/HazardCommunication/SiteSpecificHazComResources.htm>

When reading the MSDS or SDS keep in mind that you are dealing with less than 50 milliliters of liquid, and less than 25 grams of the solid. Many of the precautions cited (e.g. respiratory protection, self-contained breathing apparatus, etc) are intended for emergency response personnel dealing with bulk or industrial quantities of the material.

The primary hazards posed by laboratory chemicals in small quantities are:

Chemical burns to the skin and eyes.

Chemical damage to clothing and footwear.

The LEAD will provide, on request, protective eye wear, nitrile gloves, and "pH 6.9 buffer" which is useful to neutralize skin or clothing exposed to acid or caustic. We recommend that staff engaged in sample collection carry a portable eye wash station and, at minimum, a one-gallon jug of water in their vehicle for washing in emergencies.

Below is a chemical compatibility chart with recommended gloves and eye protection.

Table 1 Chemical compatibility of gloves

Manufacturer /Chemical	Glove to be Used (Material)	Eye Protection
Mallickrodt Baker Inc./ Sulfuric Acid (50-100%)	Silver Shield – Excellent rating for total hand immersion -OR- Nitrile – Fair protection rating for accidental splash or intermittent contact	Full coverage chemical goggles
Hach/ Dissolved Oxygen 3 Powder Pillows	latex (recommended by chemical manufacturer) or Nitrile	Safety glasses with top and side shields
Hach/ Starch Indicator Solution	latex (recommended by chemical manufacturer) or Nitrile	Safety glasses with top and side shields
Hach/ Sodium Thiosulfate Standard Solution	latex (recommended by chemical manufacturer) or Nitrile	Safety glasses with top and side shields
Fisher Scientific/.02 N Sulfuric Acid Solution	Nitrile – Excellent protection rating for total hand immersion	Full coverage safety goggles
Mallickrodt Baker Inc. / Nitric Acid (50-70%)	Silver Shield (or Nitrile if contact will be limited to small volumes and short duration)	Full coverage safety goggles
VWR Intl. EMD Chemicals/ PH 4 Buffer Solution Red	Silver Shield – Excellent rating for total hand immersion -OR- Nitrile – Fair protection rating for accidental splash or intermittent contact	Safety glasses with top and side shields

4.0 INVESTIGATIONS

Look for clues that suggest abnormal conditions, e.g., stressed vegetation, dead or dying insects or animals, soil staining, fumes, crystals, oil, puddles of liquid, abandoned containers, unusual colors, odors, phase separations, etc.

Whether or not to collect a sample is your decision. You must make the judgment whether or not your observations warrant chemical characterization of the material in question. **If a sample is collected, it must represent the condition of the site as much as possible.**

If a sample is collected, fill the sample bottle. The laboratory needs a reasonable amount of sample to conduct tests. With the exception of samples for volatile organic analyses, for safety considerations, fill the bottle to the neck not to the cap (except for *volatiles which must be filled to the cap with no air space*).

Field observations should be documented, **using waterproof ink in a permanently bound notebook**. Photographs should be taken, if possible. If you believe you have discovered a violation, it must be appropriately documented according to the “elements” of the violation, and according to your program policies. Violations must be appropriately documented for the file whether or not you are issuing a

Warning Letter or a Pre-Enforcement Notice. If you have any questions about the elements of the particular violation or need assistance in determining what evidence would support an enforcement action, contact your manager or the Environmental Law Specialist assigned to your program, or call Les Carlough in the Office of Compliance and Enforcement at 503-229-5422 for assistance. If you believe the violation may have been done deliberately, deceitfully, or deliberately, call the DEQ Environmental Crimes Coordinator (currently Susan Elworth at 503-229-5152).

Note: Appearance, odor, or nearby sources may suggest what to test for.

CAUTION:

Be very cautious of unfamiliar odors

AVOID opening unlabeled containers to make a preliminary assessment of sample.

(Be especially wary of containers that exhibit crystals around the cap; they could be extremely explosive perchlorates or organic peroxides. When exposed to light or air, ethers can form peroxides that can easily detonate!). If crystals are present and you don't know what they might be, evacuate the area and contact the local fire department.....

4.1 ORGANICS

Organic substances may appear as tar, wax, oil, crystalline or amorphous solids, colored or colorless liquids. Samples may be odorless or their smell may be sweet, sour, biting, or petroleum-like. Attempt to characterize the sample appearance and odor, and describe them on the Analysis Request Form. The more information you provide the better are the laboratory's chances of identifying an "unknown" contaminant, and successfully quantifying it.

Collect samples for organic analysis in DEQ laboratory-supplied containers (see) which have been specially cleaned to eliminate interference. Because organic analytical methods are expensive it is imperative that samples be collected in acceptable containers to avoid having the analysis invalidated as a result of container contamination. If you are caught in a bind without the correct containers, call the laboratory so we can work out an acceptable alternative.

In general, plastic containers should NOT be used to collect samples for organic analysis.

4.1.1. VOLATILE ORGANIC COMPOUNDS (VOCs)

VOCs are a large group of organic solvents, cleaners and degreasers that evaporate at ambient temperature. Although they can volatilize quickly, trace amounts tend to persist in soils and water. This persistency, coupled with their mobility, makes groundwater contamination a primary concern following a spill or release of VOCs into the soil. When requesting VOC analysis, it is important to convey the type of situation you are investigating (dry cleaner, UST, spill, etc.) and whether or not you are interested in specific chemicals or materials such as gasoline, kerosene, trichloroethylene or benzene. This information will help the laboratory choose an analytical method appropriate for your situation. The default VOC method is Method 8260, a GC/MS procedure that analyzes for roughly 65 volatile organics plus Tentatively Identified Compounds (TICs). Unfortunately not all VOCs can be analyzed using this method; hence our need to know as much as possible about the possible contaminant source.

4.1.2. SEMI-VOLATILE ORGANIC COMPOUNDS (SVOCs)

Semi-Volatile organic compounds (SVOC) (comprise a large group of chemicals that include several categories: phenols, chlorophenols, phthalates, pentachlorophenol, polynuclear aromatic hydrocarbons (PAH) , organophosphorus pesticides, etc. The phrase SVOC is also generically used to refer to GCMS methods for the analysis of Base Neutral and Acid (BNA) compounds (EPA 8270C, EPA 625, EPA525.2) approximately 85 specific analytes, and also provides provisional identification of additional analytes as Tentatively Identified Compounds (TIC's). As with VOCs the list of analytes is large, but not universal. If

the question arises whether or not a particular chemical can be tested for by this method, call the Organic Section at the laboratory.

4.1.3. **CYANIDES**

There are three basic forms of Cyanide analyses Total, Available, and Free Cyanide. Total cyanide analysis captures all forms of cyanide, where the available cyanide analysis captures only the easily dissociated forms and free cyanide is only the CN⁻ and HCN forms of cyanide. In many instances “free cyanide” is the regulated analyte, where this is the case, in addition to the free cyanide test methods DEQ also recognizes the available cyanide test methods for compliance purposes. Total Cyanide may also be used to demonstrate compliance if the results are below the needed action level.

Comment on “Free Cyanide”: Technically, “Free” Cyanide is only the CN⁻ and HCN forms of Cyanide and there are available approved methods in 40 CFR part 136. However the term “Free” Cyanide” has been used to imply any of the following variations: Cyanide Amenable to Chlorination, Weak Acid Dissociable Cyanide, Reactive Cyanide as well as Free Cyanide. Amenable and weak acid dissociable cyanide are commonly used methods for the analysis of “free” cyanide as they are readily available and are less costly than the true free cyanide methods. **To avoid confusion, be specific**, ask for the Cyanide form you need either amenable cyanide or weak acid dissociable cyanide, or free cyanide.

It is usually less expensive to request a total cyanide method to demonstrate compliance to the Amenable, or WAD, or Free CN action levels (with Free Cyanide the most expensive). As long as the total cyanide results are below the action level, compliance has been demonstrated for the other forms. The analysis of the other forms is only necessary if the total cyanide is high.

Note: *the “Reactive” Cyanide method has been removed by EPA and is not available at DEQ.* Please request **Total Cyanide or Cyanide Amenable to Chlorination** when seeking to determine characteristic waste criteria under RCRA.

4.1.4. **DIOXINS and FURANS**

The analysis of these analytes requires the use of high-resolution gas chromatography and high resolution mass spectrometry (HRGC/HRMS) on purified sample extracts. The procedure provides the detection and quantitative measurement of polychlorinated dibenzo-p-dioxins (tetra- through octachlorinated homologues; PCDDs), and polychlorinated dibenzofurans (tetra- through octachlorinated homologues; PCDFs) in a variety of environmental matrices and at part-per-trillion (ppt) to part-per-quadrillion (ppq) concentrations

Safety Note:

Because of the extreme toxicity of these compounds, the analyst and sampler must take necessary precautions to prevent the exposure of laboratory and field personnel or others to materials known or believed to contain PCDDs or PCDFs

4.1.5. **POLYCHLORINATED BIPHENYLS (PCBs) & CHLORINATED PESTICIDES**

Both extracted by the same method, therefore a single container may be used to sample for these two analyses. Other organic tests require a separate container for each method of extraction. Analyses can be performed by Gas Chromatography (GC) or by GC Mass Spectrometry (GCMS)

4.1.6. **EMERGING CONCERN CHEMICALS**

There are several groups of emerging compounds (Semi-volatile organics) that have recently become of interest to the EPA and DEQ as they have been found in wastewater and surface waters. The DEQ laboratory has the capability to analyze for the following emerging chemical classes. Contact the Organic Manager at the laboratory for specific analyte lists.

- Flame Retardants (polybrominated diphenyl ethers, PBDEs)
- Pharmaceuticals, Steroids and Personal Care Products.
- PCB Congeners

- Emerging Pesticides (not on standard Organochlorine pesticide list)

4.1.7. **PETROLEUM IN SOILS**

Methods for analysis are designated in Oregon's Soil Matrix Rules for Petroleum Underground Storage Tank Cleanups (OAR 340-122-350). Petroleum products in soil are first identified and then quantified by the Total Petroleum Hydrocarbon (TPH) methods:

- NWTPH-HCIDis a qualitative screen to determine which petroleum products (if any) are present, and what subsequent quantitative methods may be required.
- NWTPH-Gx is the quantitative method for gasoline.
- NWTPH-Dx is the quantitative method for diesel and other heavy oils.

4.1.8. **HEXANE EXTRACTABLE MATERIAL (HEM)**

The HEM procedure has replaced the classical "Oil & Grease" method which measured the polar (animal fats, vegetable oils, etc.) and nonpolar (petroleum fuels, mineral oils, etc.) using a Freon™ extraction of the sample. It was sometimes used in conjunction with EPA's Total Recoverable Petroleum Hydrocarbon method to distinguish between polar and nonpolar components. These Freon based methods have been replaced by the more eco-friendly hexane based extraction method. The DEQ laboratory is using EPA Method 1664, n-Hexane Extractable Material (HEM), which can be used with silica gel cleanup to determine TPH or NWTPH Methods as appropriate.

4.1.9. **OTHER ORGANICS**

Other common analyses used to identify or characterize unknown organic contaminants include MBAS, Glycol/Fluorescein, Lignin-Tannin, Total Organic Carbon (TOC), Chemical Oxygen Demand (COD), Total Organic Halogens (TOX), and Formaldehyde.

Methylene Blue Active Substances (MBAS)

A nonspecific test for Methylene Blue Active Substances. Anionic surfactants, which may include LAS (Linear Alkylbenzene Sulfonates), other sulfonates and sulfate esters (colorimetric procedure). MBAS can detect the presence of detergents. This test is not performed by the DEQ laboratory.

Phenolics

A nonspecific test for phenolic-like compounds (colorimetric procedure in which sensitivity is inversely related to the number of substitutions on the aromatic ring). If specific phenols are required, see semi-volatile organic extractables. **This request will not measure pentachlorophenol.**

Fluorescein/Glycol (Antifreeze)

Fluorescein is the ingredient responsible for antifreeze's vibrant green color, and is also a dye used to monitor water flow. Unlike glycol, fluorescein is not considered harmful to the environment. Fluorescein is analyzed by HPLC with a UV/Vis detector.

Lignin Tannin

A non specific test for hydroxylated aromatic compounds found in lignin and tannin compounds (colorimetric procedure) characteristic of tree bark leaching.

Chemical Oxygen Demand (COD)

A nonspecific test to measure oxygen depletion potential of a water sample assuming all materials are chemically oxidized to their highest oxidation state: reported as mg oxygen per liter.

Total Organic Carbon (TOC)

A nonspecific test that measures organic carbon content of a water sample as milligrams of Carbon per liter.

Total Organic Halogens (TOX)

Measurement used to estimate the total quantity of dissolved halogenated (containing fluorine, chlorine, bromine, iodine) organic material in a water sample which is indicative of contamination by synthetic chemicals. This test is not performed by the DEQ laboratory.

4.2 INORGANICS

Inorganic contaminants tend to be crystalline, dissolved salts, or suspended solids in water. They include all combinations of elements, with the exception of compounds containing carbon, which are defined as Organics. Inorganic acids and some caustics have a sharp, irritating odor. Collect inorganic samples in laboratory supplied plastic containers described in .

Characterization of unknown inorganic contaminants includes physical measurements (Total Suspended Solids, Total Solids, Turbidity, Conductivity, and pH), metals, and nutrient analysis. Turbidity, pH and orthophosphate samples have very short holding times, thus these analyses must be started immediately upon sample receipt.

4.2.1. METALS

The choice of metals analyses is dependent upon the purpose of the sampling event and the data quality objectives (DQOs). **Dissolved metals** are water soluble, defined as passing through a 0.45 micron filter. **Total recoverable metals** include dissolved metals plus those metals that are more strongly attached to particulate matter and thus less available to the environment. Total Recoverable metals require an acid digestion to release the metals for analysis.

Dissolved metals samples must be filtered in the field with a 0.45 micron membrane filter, immediately after collection and before adding acid preservative ([APPENDIX C](#)). Total Recoverable metals may be either preserved in the field or delivered to the laboratory on ice and the laboratory can preserve the samples. If a request for analysis does not specify Dissolved or Total; Total Recoverable metals will be the test assigned.

Coordinate with laboratory staff prior to sample collection to select the appropriate analytical method for the project. There are three choices: Inductively Coupled Plasma (ICP), Inductively coupled Mass Spectrometry (ICPMS) and occasionally Graphite Furnace Atomic Absorption (GFAA). Though the ICP is able to obtain relatively low detection limits for a variety of metals, ICPMS is able to obtain even lower detection limits for trace metals and has almost entirely replaced the need for GFAA analyses. Reserve requests for GFAA to those situations where the method is specially required by the program such as a few drinking water metals as it is slower and more costly on a per/metal basis.

The analysis for Mercury uses a different digestion and analytical procedure than the other metals and a separate sample must be collected. Requests for mercury analysis must be specifically identified on the COC form, and separate samples collected, or it will not be performed.

4.2.2. NUTRIENTS

Nutrients include Total Organic Carbon (TOC), Chemical Oxygen Demand (COD), Nitrate-Nitrite(NO₃+NO₂), Total Kjeldahl Nitrogen (TKN), Ammonia (NH₃), Dissolved (OPO₄) and Total (TPO₄) Phosphate. Depending on the requirements, Total Nitrogen (TN) is also sometimes included with the Nutrients). These analytes can occur naturally at low levels in the aquatic environments. Elevated values are frequently indicative and a source of problems. Discharges from industry, municipal wastewater treatment plants, municipal storm water and agricultural operations can increase certain nutrient levels. Nitrogen nutrients are reported as mg/L of equivalent nitrogen, and phosphate nutrients are reported as mg/L equivalent phosphorous.

There are a wide variety of chemical forms that phosphate can exist in the environment, and be analyzed². Forms include Total, Total Reactive, Total Acid Hydrolyzable, Total Organic, Dissolved Reactive, Total Dissolved, Dissolved Acid Hydrolyzable, and Dissolved Organic. Notify the laboratory well in advance if any form other than Total or Dissolved Reactive phosphates are of interest. DEQ laboratory

² Table 4500-P1, Standard Methods for the Analysis of Water and Wastewater, APHA-AWWA, 21st Edition , 2005

uses the Molybdate-Ascorbic acid for Dissolved Reactive Phosphate (OPO₄) on filtered samples, and Molybdate-Ascorbic acid proceeded by persulfate-autoclave digestion for Total Phosphate (TPO₄). Methods used by other labs may vary, so data comparison could be difficult.

Total Organic Carbon (TOC)

A nonspecific test measuring organic carbon content of a water sample. Reported as milligrams of carbon per liter.

Chemical Oxygen Demand (COD)

The measure of the oxygen equivalent of organic matter contained in a sample that is subject to oxidation by a strong chemical oxidant.

4.2.3. BIOCHEMICAL OXYGEN DEMAND (BOD)

Biochemical Oxygen Demand (BOD) is a nonspecific test used to evaluate consumption of Dissolved Oxygen (DO) by biochemical processes when incubated at 20°C over a period of 5-days. It is a bioassay procedure that is normally applied to water samples, although it can be applied to sediment as well. BODs conventionally are done over a 5-day period, however longer time periods are possible (e.g. 14-, 28-day). Special requests are necessary for any but the 5-day test.

BODs can evaluate the oxygen depletion caused by carbonaceous material and nitrogen species (BOD), such as TKN and ammonia, carbonaceous material alone (CBOD), or nitrogen species alone (NBOD). Special requests are necessary for any test other than BOD or CBOD.

Collect liquid samples in specially cleaned DEQ containers (refer to Table I). The laboratory must be notified prior to sampling. BOD samples for surface and receiving waters low in BOD are collected in special glass BOD bottles. It is desirable to determine Field DO on these samples, prior to sending to laboratory (see procedure in Appendix B). When Field DO measurements aren't made, DO is less than 5 mg/L, or DO exceeds 120% Saturation, specify the BODS2 test on the COC form to inform the laboratory analyst of the need to perform the initial DO or %Sat test.

The regulatory holding time for BOD is 48 hours between the time of collection and the start of the analysis. Make every effort to deliver the samples to the laboratory within 24 to give them time to start the analysis. BOD samples will be accepted between 08:00 AM Tuesday and 12:00 AM Friday. Accepting BOD samples at any other time will require overtime work, and must be pre-approved by the Inorganic Section Manager. During the period following collection, and including transport to the lab, biological samples must be kept cool on ice (4°C).

4.3 MICROBIOLOGICALS

Bacteria (Coliform, E. Coli, Enterococcus etc.). samples are analyzed by the DHS Public Health laboratories' microbiological laboratory. Collect liquid samples in sterile bottles (refer to Table I). DEQ laboratory must be notified prior to sampling.

Bacterial samples will be accepted Monday through Thursday before 4pm. Bacterial samples may be held for a maximum of 24 hours (time elapsed from collection to analysis) as long as they are kept on ice (and not frozen). Once samples exceed the 24hr holding time the sample results are then qualified. Since PHL performs the micro analyses for DEQ, if other sampling days are needed, contact the DEQ laboratory for scheduling.

Note: The holding time for regulatory purposes varies based on the regulations. Example: the holding time on bacterial samples is 8 hours (NPDES) or (30 hours (SDWA) or 8 Hours (surface water treatment rule)! ***If a holding time of less than 24 hours is required for a project, it must be noted on the COC.***

4.4 TOXICITY CHARACTERISTIC LEACHING PROCEDURE (TCLP)

TCLP extraction is used in RCRA to evaluate hazardous waste characteristics. It is used to assess the leachability of metals and certain organics under precisely controlled conditions. TCLP procedures are also used in DEQ's Soil Cleanup Rules. ***The Hazardous Waste Characteristic of TCLP is a method***

defined regulation, meaning that the regulation is dependent on the TCLP procedure being followed prescriptively.

When asking for TCLP on samples, be aware of the method's limitations. TCLP extraction is an involved and relatively expensive method to run, depending on the nature of the samples.

Samples having multiple phases (water-oil, water-solid, oil-solid, water-oil-solid) are difficult to deal with under this protocol. Sufficient sample must be collected to provide an adequate quantity of each phase for both inorganic and organic TCLP extractions: approximately 500 grams of solid, or at least 1 liter of liquid. More sample material is required as the ratio of aqueous to solid (other) phase increases. If a sample contains less than 150 grams solid (other) phase in the conventional sample size, a larger quantity of sample is necessary just to run the TCLP inorganic tests. If TCLP organics, duplicate, matrix spike, or matrix spike duplicate analyses are requested considerably more sample is needed. According to the TCLP protocols the laboratory needs to have 100 grams of solid to perform the extraction. As a guideline, see the following table.

% Solid (other)	Desirable Sample
2%	7.5 liters
5%	3.0 liters
10%	1.5 liters
15%	1.0 liters (≈ quart)
20%	0.5 liters (≈ pint)

In general, a 1 gallon sample (≈ 3.8 liters) is a practical limit.

When requesting TCLP on a sample it is more efficient for the Organic section to analyze for Totals (e.g. Total VOCs) first, and then to perform TCLP on any sample where a Total is observed that exceeds the TCLP Regulatory Limit. It serves no purpose to do a TCLP extraction, followed by analysis of the TCLP extract, if there isn't enough regulated substance in the sample in the first place to exceed the TCLP standard. On the other hand, it is more efficient for the Inorganic section to perform the TCLP extraction right away. Submitting samples for inorganic Total Metals, with the instruction that samples having high levels of regulated metals should be subjected to TCLP, will take considerably longer than simply requesting TCLP.

4.5 FIELD ANALYSES FOR WATER SAMPLES

4.5.1. DISSOLVED OXYGEN (DO)

A significant indicator of stream health, low DO is frequently the primary cause for fish kills. Refer to [APPENDIX B SAMPLING PROCEDURE FOR DISSOLVED OXYGEN](#) for field analytical procedure. This analysis should be performed in the field because DO samples are not stable. Dissolved oxygen can change rapidly after sample collection, depending on temperature, barometric pressure, and other dissolved gases.

4.5.2. Alkalinity, Conductance, pH, etc

Other field analyses include alkalinity, conductance, pH, temperature, turbidity. Obviously sample temperature will change, but pH may also change over short periods of time. It is recommended that pH be measured in the field whenever possible. Meters must be calibrated and the calibration verified prior to use.

5.0 COMPLIANCE

5.1 NPDES³, WPCF⁴, Solid Waste Landfills,

Refer to DEQ or EPA inspection procedures or guidelines, and review Permit requirements. In addition, a Sampling and Analysis Plan (SAP) should be created for the sampling event using the template [DEQ08-LAB-0009-TMPL](#) as a guide. If the source is required to report self-monitoring test data to the agency, and you intend to investigate the source's analytical capability, collect split samples. Samples will be analyzed by both the source laboratory and DEQ laboratory, resulting in an inter-laboratory comparison. **Leave a split-sample analysis form (Form I: [DEQ10-LAB-0002-FORM](#) or Form II: [DEQ10-LAB-0003-FORM](#))⁵ with the source.** Request that they fill in their test results and return it to the DEQ QA chemist at the laboratory as soon as possible. It is not necessary to limit requested analyses to the parameters in permits. The DEQ laboratory will perform any reasonable analyses and prepare a split comparison report.

The laboratory would appreciate advance notice of split sampling. Inter-laboratory splits are a useful QC measurement for the DEQ lab, as well as the source's laboratory. The field inspector is responsible for insuring that the source reports their results to the laboratory QA chemist as quickly as possible, particularly when a comparison is wanted. LEAD would appreciate receiving a monthly forecast of compliance split sampling schedules.

5.1.1. SPLIT SAMPLING OF STPs FOR COMPLIANCE INSPECTIONS

Split samples are a special type of sample where two laboratories independently analyze the same sample. It is often done when sewage treatment plants maintain their own laboratories, or where landfill operators use contractor labs for leachate analysis. Split samples for sewage treatment plants (STPs) should be collected in a single large container, homogenized, and then poured into separate, clean sample bottles: one portion for the non-DEQ laboratory and one portion for the DEQ laboratory. Collecting one grab sample for the non-DEQ laboratory and a second, separate grab sample for the DEQ laboratory is **not** the same thing. To limit variance to analytical technique alone, it is imperative that both of the laboratories analyze samples that are as near identical as possible.

Common sewage treatment plant (STP) Split Sample parameters are Fecal Coliform (FC), E.Coli, Total Suspended Solids (TSS), pH, and Biological Oxygen Demand (BOD). Depending on the source's Permit, a sample for CBOD may be split also. The DEQ laboratory measures pH on all BOD samples, even though not all STP's follow this procedure. Because of limited holding time requirements these samples must be received at the laboratory as follows.

BACTERIAL samples (*fecal coliform, E.Coli*) must be received no later than 4pm on Thursday. For permit compliance samples and some LEGAL samples, the maximum holding time of 8 hours **must be adhered to**, whereas routine monitoring samples can be held up to a maximum of 24 hours. Bacterial samples are routinely analyzed using the Colilert® Quantitray or Membrane Filtration (MF) methods. When analysis for a "non-routine" organism (e.g. fecal strep) is required, notify the laboratory in advance so that special media can be prepared⁶ for the test. **If a holding time of less than 24 hours is required for a project, it must be noted on the COC.**

BOD samples must be received no later than NOON on Friday. If it is necessary to sample between noon Friday and noon Monday, the laboratory will have to schedule someone to come in the following weekend to complete the test. Special authorization is required, prior to collection and shipment of

³ National Pollution Discharge Elimination System

⁴ Water Pollution Control Facility

⁵ Blank forms are available on Q-Net or through the QA section, DEQ LEAD.

⁶ Microbiological analysis is performed by the Oregon Department of Human Services, Public Health Laboratory. Without advanced notice, when special media is required, the test may not be performed.

samples, which will be received by the laboratory after 12:00 on a Friday. Unauthorized samples that do not fit the routine time frame may not get analyzed.

Normal procedure is to collect a 24-Hr composite sample of the influent and a 24-Hr composite of the effluent for BOD₅, TSS, and pH. A grab sample of the effluent is taken, **after the Chlorine Contact Chamber**, for Fecal Coliform (FC) and E.Coli. The Composite influent and effluent samples should be collected in 1000 mL polys, and the FC/EC grab sample should be collected in a sterile wide-mouth 125 mL sterile bottle containing sodium thiosulfate to destroy excess chlorine. Composite effluent samples cannot be used for FC/EC samples because of their lengthy chlorine exposure (up to 24 hours).

The STP staff should take their own samples from the *same* composite and grab sample as the DEQ inspector. The DEQ laboratory and the STP laboratory should begin testing at approximately the same time. Agree upon a time for test set-up, and make a note of it on the field sheet. Usually the inspector will arrange for test set-up at ten o'clock A.M. on the day following the sampling.

**Write "Split" on the top of Chain of Custody form,
along with agreed upon set-up time.**

Leave a copy of the "Split Sample Results Report" form (Form I or II) with the STP staff, with instructions to mail the completed form to DEQ laboratory QA Section as soon as they have completed their analyses.

Time of sampling must be noted on the Chain of Custody form for all samples, along with residual chlorine measurement.

5.2 SAMPLING AND ANALYSIS PLAN

A Sampling and Analysis Plan (SAP) is a combination laboratory analysis and field sampling plan. Basically, it should describe what you are going to do, who will do it, how many samples will be collected, where and when you'll do it, and how it will be done. A SAP should be developed before any samples are submitted to the lab, with the exception of complaint and routine compliance inspections. SAPs for projects on which DEQ laboratory performs analytical work must be approved and signed by a QA Officer. Contact the QA Section for assistance.

Note: To plan workloads the laboratory Sample Coordinator must receive a Sampling and Analysis Plan when:

- ***Sampler is requesting a complicated suite of tests on more than five samples,***
- ***Sampling event involves collection and analysis of more than ten samples, or***
- ***Multiple sampling events planned over a period of time (e.g. quarterly, monthly, etc.).***

At a minimum, the laboratory should be provided with a list of how many sampling sites there will be, what type of sample will be collected (e.g. soil, groundwater, sediment, etc), analyses that will be requested (e.g. VOCs, NO₃, TKN, TCLP, Total or Dissolved metals, TPH-?, BOD₅, etc.), when samples will be collected and when delivered to lab, Quality Assurance samples that are necessary, and when you need to have the data (e.g. ASAP, 2 weeks, 4 weeks, 6 weeks, etc.). The name of the Project Manager, phone number, and a Q-Time Fund Code assigned by the Business Office for the laboratory to charge work on the project must be included.

This can be accomplished in the form of either a single narrative page or a simple table, as in the example that follows.

Project:	FRED'S Koi Emporium
Project Manager:	Shirley U.Geste [503-693-57XX]
QTime-Fund Code:	42999
Sampling Date(s):	May 15-25, 2013

Delivery to laboratory: May 15, 2013 late in afternoon

Desired Turnaround: 4 weeks

Matrix	# Sample sites	Analytes	+ QA
Surface Water	3	BOD ₅ , NH ₃ , VOC, Pesticides, pH, Alkalinity, Conductivity	1 Duplicate 1 matrix spike transport blank
Sediment	4	Pesticides, VOC, TKN Total Arsenic & Lead	1 duplicate transport blank
Groundwater	5	VOC, pH, Dissolved Arsenic & Lead, pesticides	1 duplicate 1 matrix spike transport blank
Soil	3	Pesticides, TCLP and Total metals (As & Pb)	1 duplicate + 1 matrix spike for each TCLP

When the laboratory receives this information they can see what their schedule is like, and plan to accommodate your samples. The SAP also permits the Sample Coordinator to prepare the appropriate number and type of sampling containers you will need ahead of time. However, be forewarned: there are a limited number of instruments and analysts available to do certain types of testing (e.g. GC/MS-VOC, SVOC-Vols; ICP-metals; etc).

It is prudent to allow for some flexibility in the scheduling of your sampling event and specific turnaround times. If someone else has already booked up that time period the laboratory may ask that you reschedule your sampling event, or extend the turnaround time. There is also the possibility, however remote, that an unscheduled *Legal* case (case with known or high potential for litigation) might preempt your time slot. Depending on the situation, Legal cases may receive priority over **all** other pending work. In any event the lab will do their best to get the work done if it's too late to reschedule, and you will be kept informed of the progress of your project's analyses.

Request rapid turnaround time (1-2 weeks, or less) only when it is absolutely necessary. The laboratory recognizes that this may be appropriate on occasion, but it should not be employed routinely or as a matter of convenience.

6.0 ADVANCED NOTICE OF PLANNED SAMPLING ACTIVITY

Coordination with the laboratory is important to ensure that samples are analyzed properly and timely. The more notice the laboratory has, the better. Advanced notification of a sampling event can be sent to the sample coordinator by fax or email.

DEQ LEAD FAX number: (503) 693-4999

Once notification is received it will be filed in a central location, which everyone involved will check daily. Thus, if the regular liaison person is not available, the information will still reach those involved. This will provide the laboratory with the workload planning information it needs for the work requested. Once this information has been sent, if there are any changes, please send an update.

7.0 QUALITY ASSURANCE

The term quality assurance describes the system of activities intended to provide evidence to the producer or user of a product or service that it meets predefined standards of quality with a stated level of confidence⁷. It consists of related but independent activities: **quality control** and **quality assessment**. Quality control describes those activities and procedures used internally (within the laboratory and in the field) to produce consistent and reliable data. Quality assessment deals with activities to independently evaluate data quality, after it's produced. Data quality is assessed according to the needs of the end data user. The quality needs of the end user should be clearly spelled out in a quality assurance project plan or a sampling and analysis plan.

Scientifically valid test data does not just happen. It results from hard work and considerable cost, in both time and money. Poor quality data can be particularly costly if bad decisions are made based on erroneous data. **It is the DEQ Laboratory's Policy to give higher priority to data quality⁸ over data quantity.** Analytical data can only be as good as the samples collected. Thus, samples that are not representative of the matrix being assessed, those collected in insufficient amounts for the analytical method used, or samples contaminated by sampling or handling procedures cannot be made representative, increased in quantity, or uncontaminated by laboratory efforts. The laboratory must **assume** that everything in the sample container constitutes the sample, that the sample was collected and preserved properly, and that it does not contain extraneous contamination.

Due to the sophistication of today's measurement techniques, and their cost, considerable planning is necessary to insure that test data are meaningful. The goal is to collect an appropriate number of high quality samples which represent the environmental entity being tested. Reduced numbers of samples require even better planning to insure that gaps in the data set, caused by omissions or post-analysis data rejection, don't weaken conclusions or preclude decision-making.

7.1 DEQ Laboratory Sample Acceptance Criteria

7.1.1. Before Sampling

To ensure successful submission of environmental samples for laboratory analysis, the sample collectors must conform to the following sample collection protocols.

The sample collector/project manager must submit a [Quality Assurance Project Plan \(QAPP\)](#) or [Sampling and Analysis Plan \(SAP\)](#) to a QA Officer at least two weeks prior to sampling. SAP templates can be found on [Q-Net](#). The QAPP and SAP are documents detailing the background, scope and goals of the project, the appropriate contact people, the sampling sites, the parameters to be analyzed and their required quantitation limits, types and quantities of QC needed, and other particulars specific to the project. This also provides the laboratory notice so they can ensure proper resources are available to perform the requested analyses within the analytical holding times. **Note:** There may be special circumstances when the QAPP or SAP cannot be completed before the sampling event takes place. In these cases the relevant QAO and/or LEAD section manager should still be notified to discuss some of the details to help ensure project is successful. **Note:** The QAPP or SAP must still be finalized and approved as soon as possible following the sampling event.

7.1.2. Sample Acceptance Criteria

All incoming work will be evaluated against the criteria listed below. Where applicable, data from any samples that do not meet the criteria listed below will be noted on the laboratory report defining the nature and substance of the variation and noting any adjustments to the data quality level (DQL) resulting from the variance. In addition, the sample collector/project manager will be notified after the receipt of the samples.

1. **Samples must be submitted with the proper paperwork (e.g. Chain of Custody (COC) [DEQ06-LAB-0054-FORM](#) including:**

⁷ Taylor, J.K., *Quality Assurance of Chemical Measurements*, (Chelsea, MI: Lewis Publishers, 1987).

⁸ Quality refers to generating data of known and documented quality that meets the needs of the end users.

- Name and address of the project site,
 - Project Q-time number,
 - Name of contact person for the project,
 - Name of sample collector,
 - Latitude and longitude coordinates (in decimal degrees) or corresponding station ID for each sampling location. If station ID is not available, a new one should be created or if sample is not part of the regular monitoring network and will not be repeatedly sampled (e.g. legal, hazardous waste, asbestos), then a station id is not necessary, but Latitude and Longitude are requested
 - Brief description of each sampling location,
 - Sample matrices and QC type (e.g. Matrix: Soil, water, tissue, etc. / QC Type: Equip Blank, (EB) field duplicate (FD), etc.
 - Date and time of sampling,
 - Container ID numbers used at each location, and
 - Requested analyses for each sample.
 - The date and time that each person received or relinquished the sample(s), including their signed name.
 - **Information must be legible**
 - All information must be written on the field form in waterproof blue or black ink.
- 2. Samples must be properly labeled**
- Use durable labels
 - Include a unique identification number traceable back to the COC.
 - Include preservative used (Container codes are acceptable if defined).
 - Use waterproof blue or black ink
 - **Information must be legible**
- 3. Adequate quantity of samples must be collected in appropriate containers, and all relevant preservation measures must be followed.**
- Failure to use the lab-approved container types and/or the proper preservation measures may result in refusal of the submitted samples or flagging of all analytical data.
 - Sufficient sample must be collected to allow for the analysis of matrix quality control samples (e.g. 2 additional 1 liter samples need to be collected per sampling event for extractable organic methods for a matrix spike and matrix spike duplicate e.g. EPA 1664 Oil and Grease, EPA 8270C, etc)
- 4. Samples must be preserved according to the requirements of the requested analytical method.**
- Sample containers must be labeled with the preservation used (e.g. HNO₃, H₂SO₄, etc). The DEQ Laboratory will provide stickers to label sample bottles.
 - Samples must arrive at the laboratory on ice for analyses with temperature preservation requirements.
 - Coolers or other containers used for transporting samples must include a temperature blank⁹. These are available from the laboratory sample receiving section and can be picked up prior to sampling. Most analytical methods require chilling samples to 4° C (other than water samples for metals analysis). For these methods, the criteria are met if the samples are chilled to < 6.0° C and above freezing (0° C). For methods with other temperature criteria (e.g. some bacteriological methods require < 10° C), the samples

⁹ Temperature blanks are water-filled sample bottles that stay in the cooler with the samples for measuring the temperature of the samples during transport to the lab

must arrive within $\pm 2^{\circ}$ C of the required temperature or within the method specified range. Note: Samples that are hand delivered to the laboratory immediately after collection may not have had time to cool sufficiently. In this case the samples will be considered acceptable as long as there is evidence that the chilling process has begun (arrival on ice).

- Chemical preservation (pH) will be verified prior to analysis and the project manager will be notified if there is a discrepancy. ***If analyses will still be performed, all affected results will be flagged to indicate improper preservation.***

5. Special Requirements for Volatile Organic Analyses

- All samples submitted for Volatile Organic analyses must have a Trip Blank submitted at the same time. A trip blank is prepared from organic-free reagent water and carried throughout the sampling, storage, and shipping process.
- Residual chlorine must be neutralized prior to preservation except as noted below. If there is prior knowledge that the samples are not chlorinated, state it on the COC and preserve VOA vials with HCl only. (Note: there are provisions for the analysis of volatiles by 624 and 8260B from unacidified VOAs with shorter holding times.)
- VOA samples to be analyzed for 2-Chloroethyl vinyl ether (2-CVE), Acrolein and Acrylonitrile must NOT be acidified as the acid breaks down these analytes.

6. Parameter/Method Recommended Holding Time must not be exceeded

- Samples must be submitted with sufficient time remaining on the holding time for the laboratory to perform the analyses.

7. Minimum Sampling Plan submitted¹⁰ when more than five (5) samples submitted, including

- Number of samples (by matrix), including QA (duplicates, matrix spikes & duplicates, blanks, etc.),
- Project manager,
- Whom to report data to,
- Analyses (tests) requested, and
- Detection limit needed [qualitative screen, drinking water Maximum Contaminant Limit (MCL), Toxicity Characteristic Leaching Procedure (TCLP), NPDES permit compliance, etc.]

Documentation of any of these elements may be furnished at any time up to that time when the flagged data is released from the laboratory, preferably as soon as possible. If all acceptance criteria are consequently met, qualifying flag will be expunged from the report, on condition that data quality is not affected.

7.1.3. Recommendations for packing samples for shipment.

- Pack samples in ice rather than blue ice packs.
- Soil sample jars should be placed in plastic zip-lock bags. The containers often have dirt around the top and do not seal very well and are prone to intrusion from the water from melted ice.
- Water samples would be best if wrapped with bubble-wrap or paper (newspaper, or paper towels work) and then placed in plastic zip-lock bags.
- Fill extra cooler space with bubble wrap.
- Line bottom of coolers with styrofoam if available in case of cooler being dropped
- When not hand delivering samples to the laboratory, use UPS whenever possible for shipping. If necessary to send by Greyhound or Horizon, please contact the Sample

¹⁰ Failure to submit a sampling plan could result in holding times being exceeded due to prior scheduled work in the laboratory.

Coordinator in advance. For assistance on UPS shipping contact the Technical Services office support specialist at 503-693-5780

7.2 Data Quality Objectives (DQO)

Data quality objectives are quantitative and qualitative statements describing the quality of data that is needed to support a specific environmental decision or action. These descriptors must be considered by a hierarchy of decision makers in order to determine whether data is appropriate for a particular application. Some basic questions that need to be asked are: Why collect these samples? What information is needed? How will data be used? What will data be used for? What resources are available?¹¹ Defining the use of the data to be collected in advance is a key component of any sampling program. If not thought through and considered during sampling design, the resulting data may not be suitable or adequate for project needs, and require expensive re-work or additional sampling.

After the sampling plan is developed, a field inspector or organizational policy controlling data usage should define what quality of data is needed for the specific application intended. Data quality is evaluated using standard Quality Control attributes including Precision, Accuracy, Representativeness, Comparability, Completeness, and Sensitivity (PARCCS). Each of these attributes has a component related to both sampling and analysis activities.

Many data quality definitions already reside in existing analytical methods, statutes, administrative rules, or discharge permits. If not, the data user should evaluate objectives and determine the appropriate method to obtain the needed data, and decide whether that method chosen produces the data quality to fit your needs.

Required detection limits (or reporting limits) are another data quality objective that must be defined during the DQO process. Project required limits should be below regulatory action levels where possible in order to provide some cushion in case there are some problems with the laboratory QC. Regulatory limits for under the drinking water program can be found in [Appendix F](#). Oregon risk based concentrations (RBCs) for soil samples can be found on the Oregon DEQ website at <http://www.deq.state.or.us/lq/pubs/docs/RBDMTable.pdf>.

8.0 SAMPLE COLLECTION

Collecting a **representative sample** can be difficult, but it is the **most crucial in the process of obtaining valid data**. It is now understood that most variability in the sampling and analytical process occurs during sampling. Therefore, success in obtaining a representative analytical result will primarily be determined by the sampling plan design and sample collection process. For more information or assistance with collecting representative samples, please contact the Technical Services Section at LEAD. Collection of duplicate or replicate samples is recommended to measure variability from sample-to-sample and some pre-defined frequency. This QA measure can substantiate your data by demonstrating that your samples are, indeed, representative of the population being measured. For water samples, this process is straightforward, for soil, sediment or other matrices, collection of true replicates can be complex, and should be considered during the sample design. Simple collection of a second nearby sample or spooning sample material from one container to another will not result in a true duplicate or replicates. If a high level of certainty is required to ensure true duplicates in solid matrices, details can be found in guidance from the Interstate Technology and Regulatory Council (ITRC 2102). <http://www.itrcweb.org/ism-1/>

An important factor in collecting a representative sample is the use of **proper sample containers** and **appropriate preservation**. Samples should always be collected in containers supplied by the laboratory; this ensures that the container has been properly cleaned. When the container is filled, the laboratory is ensured they will have enough sample to do the test requested. Samples submitted to the laboratory that are not in a laboratory supplied container (e.g. mayonnaise, pickle, or peanut butter jars) are likely to be

¹¹ Regional QA Management Office, "You and Quality Assurance in Region 10", ES-096, March 1988, pg. 5.

rejected for analysis. Samples must also be properly preserved or they may be rejected. Refer to Appendix "I" for appropriate sample containers, sample preservation and holding times. Apply appropriate preservation sticker to the sample bottles to indicate the preservative used.

It is important to only add sufficient preservative to achieve the required pH. Example: If you normally add 6 drops for a 1 liter bottle, you would only need 3 drops for a 500 mL bottle.

Collecting **representative** samples of liquid matrices requires deciding what it is you want to represent. Are you interested in the effluent itself, the impact of the effluent on its receiving water at the point where they mix (*mixing zone*), or the impact the effluent is having 100 meters downstream from its point of discharge? Should a "background" sample be collected upstream of the discharge point? Collecting a representative sample of a multiphase waste in a steel drum presents different problems than collecting a sample of "pure product." Does a representative soil sample¹² include rocks, vegetation or sticks? Considering these and similar questions before sampling will determine what your sample represents and should be documented in the sampling and analysis plan.

It is up to the sampler to determine what constitutes a "sample," and what it "represents." Unless the laboratory is told otherwise, whatever is in the container is the "sample" and represents the sampled unit in the field. **It is imperative** that the sampler inform the laboratory when interested only in specific fractions of a sample, and it's also necessary to make certain that there is sufficient quantity of the fraction of interest to successfully perform the analyses. If you're not sure, call the laboratory and ask.

For Example: if a sample is ½ liquid and ½ solid:

- do you want the entire sample mixed and analyzed?
- do you want just water fraction analyzed, (Filtered, unfiltered)?
- do you want the water decanted off and have the solid analyzed?

Samples "split" with a source or suspected violator must be as close to identical as it is possible to achieve. If the source or suspected violator intends to have the sample analyzed by their own laboratory it would be prudent to have their laboratory contact DEQ LEAD to agree on what portion each laboratory defines as the "sample." If one laboratory screens out and discards the large rocks, while the other retains them, the concentration of analyte in the two samples will be significantly different.

8.1 WATER SAMPLES

The Water Quality Monitoring (WQM) section collects samples for WQ, HSW, and WMC projects. WQM sampling procedures may be found in their [Mode of Operation Manual \(MOMs\) \(DEQ03-LAB-0036-SOP\)](#), which includes procedures for sampling rivers, streams, estuaries, lakes, groundwater wells, soil, shellfish, fish, and sediment. All sampling performed by DEQ personnel should have an approved SAP and/or Quality Assurance Project Plan (QAPP) or work plan.

8.1.1. Groundwater

Monitoring wells without dedicated pumps may be sampled using bailers. Some bailers are expensive and difficult to decontaminate. Disposable bailers are available from the laboratory Sample Coordinator on request.

Collect drinking water or irrigation well samples by first purging water lines. Fill sample container directly from tap, unless sample is to be split. Insure that split samples are homogeneous: fill large clean container, mix and pour into appropriate containers. Note: Samples collected in purge vials for VOC analyses cannot be split in this manner. They must be filled individually, directly from the tap or bailer ([APPENDIX A SAMPLING FOR VOLATILE ORGANICS](#)).

Groundwater wells must be properly "developed (purged and recharged)" prior to collecting a sample. All sub-samples from a given site should be representative. **Note:** Different programs may have differing standards. In general, unless low-flow techniques are implemented, a specific volume of water should be removed (3 borehole volumes, 3 well casing volumes, or until the well goes dry.) Field parameter testing

¹² DEQ LEAD has received samples containing large rocks, coins, unfired .22 rifle ammunition, etc.

(pH, specific conductivity, eH, temperature, etc.) of each well volume helps to identify when adequate purging has occurred. Groundwater samples should be representative of the aquifer being considered.

8.1.2. Surface water

Samples for many analyses may be collected using a stainless steel bucket. The collection container should first be rinsed with sample, to wash out previous sample. Collect a fresh sample. Avoid dipping bottle into sample, if possible; pour from collection container, with minimal agitation, into sample bottle. Residue from the outside surface of the container, or your hands, could contaminate samples and/or expose you to hazardous materials. If a stainless sampling container is not available, dip bottle directly into sample, install lid, and wipe off outside of container with paper towel.

8.2 AIR

The Air Quality Monitoring (AQM) section collects samples for AQ Program projects. The AQM Procedures Manual contains sampling procedures for pollutant gases (CO, SO₂, O₃, NO_x), PM₁₀ and Suspended Particulate, and canister gas sampling. Air sampling/monitoring equipment is highly specialized, requires considerable logistic support, and is not generally available for use outside of AQM. Anyone interested in proposing a monitoring/sampling project, should contact AQM Supervisor.

DEQ LEAD has sampling capability for PUF and Bubbler samples, and subsequent analyses for PAH (TO-4), volatile toxics (TO-14), and carbonyls (aldehydes & ketones using TO-11).

Air particulate material can be analyzed for specific source-related chemicals, wood fiber, asbestos, etc.. Techniques for particulate sampling include Particle Fallout Samplers and "Sticky Paper." Evacuated Stainless Steel canisters passively sample ambient air which is returned to the laboratory and subjected to gas chromatographic analyses for trace organics (e.g. solvents, gasoline, BTEX, etc).

8.3 SOIL/SEDIMENT

Use a stainless steel spoon or disposable plastic scoop to collect soil/sediment samples. The plastic scoops are useful for soft soils and those contaminated with organics which are difficult to clean off. It is common practice to composite several subsamples of soil to obtain a representative sample of an environmental condition. Composite sampling is achieved by collecting several roughly equal sub-samples and thoroughly mixing to form one sample. Soil samples should contain as few cobbles or stones as possible, unless the sampler wishes them to be included in the analysis.

It is important to note that traditional soil and sediment sampling methods do not address differences in particle sizes and spatial heterogeneity, even over short distances. These differences can result in substantial difference in chemical concentration to difference in spatial distribution of contaminants and differences in surface area on soil or sediment particles, which provide more adsorptive surface. These differences can result in significant differences in concentration, even on sub-samples within the same sample jar. This is important, because analytical results obtained from as little a few grams of extracted material from a sample jar are often used to make decisions on much larger areas in the field.

If certainty is needed that the soil or sediment sampled is representative of the chemical concentration in the area sampled in the field, then incremental sampling methods (ISM), which may be considered a type of compositing, is required to obtain this level of confidence. These sampling methods are required to obtain reproducible samples with high confidence that they represent the sampled unit. For detailed information on ISM, see the ITRC (Interstate Technology & Regulatory Council). 2012. Incremental Sampling Methodology. ISM-1 (<http://www.itrcweb.org/ism-1/>).

Note: Composite sampling, achieved by collecting several roughly equal sub-samples and thoroughly mixing in a jar to form one sample, is **not acceptable for the analysis of volatile organics**. Sampling for VOCs in soil is recommended to be completed by USEPA SW-846 Method 5035A, to prevent volatilization prior to analysis. Note that the ITRC incremental sampling (ISM) guidance discussed

situations where ISM can be combined with Method 5035A if representative concentrations of VOCs are necessary for the project.

Subsurface soils can be collected while wells are being drilled, during excavation, or using a hollow-core soil drill. The laboratory has no special equipment to collect subsurface soils, beyond using augers and core samplers.

Hand augers can be used to collect soil samples to depths of approximately 10 feet. The sample is extruded into an aluminum or stainless steel pan followed by immediate placement into appropriate sample containers. It is possible to obtain samples from discrete depths by forcing the soil core from the auger and collecting from the depth of interest. The inspector shall assess whether a lined or stainless steel auger is necessary.

Soil Sampling Guidance documents:

[*A Compendium of Superfund Field Operations Methods \(EPA/540/P-87/001\)*](#)

[*ITRC \(Interstate Technology & Regulatory Council\). 2012. Incremental Sampling Methodology. ISM-1*](#)

8.4 SUSPECT Hazardous Waste CONTAINERS

Use a *disposable* bailer on bulk containers, such as 55 gallon drums, storage tanks, etc. At least one-half foot depth of product is required for the bailer to function. The laboratory also supplies *disposable* glass tubes (approximately 4 feet long) that may be used to "pipette" product out of large containers. Either process is messy. Take an ample supply of clean water and paper towels to clean off sample bottles. Wear nitrile or silver shield gloves to protect hands.

Decontaminate all sampling equipment, except disposable variety, before returning it to the laboratory.

Do not return or transport any contaminated sampling equipment in ice chests with samples.

Spraying with water is generally adequate. Carry a squirt bottle or water-filled pump-type garden sprayer to remote sites. Make your own arrangements to dispose of wastes generated during sampling (*i.e.* disposable equipment, wash water, surgical gloves, and paper towels). **The laboratory has no disposal facilities or resources to accommodate disposal of wastes that accumulate when you decontaminate equipment.** The laboratory will not accept, store or dispose of purge water.

9.0 SUBMITTING SAMPLES, REQUEST FOR ANALYSIS

In order to submit samples to the LEAD, the first step is to contact the Sample Custodian to schedule the analytical work and to clarify what paperwork needs to be submitted with the samples. Having this done in advance will help ensure that your samples will be analyzed in a timely manner.

Typically two forms are submitted with a batch of samples. Data that the sample collector measures in the field (such as temperature or turbidity) are recorded on a Field Data Record form. Sample bottle numbers for laboratory analysis are recorded on a Chain of Custody form.

The Chain of Custody form and the Field Data Sheet [DEQ06-LAB-0054-FORM](#) can be found on Q-Net at <http://deq05/lab/qms/documents.asp>. The forms are divided on separate tabs within the same excel workbook.

9.1 CHAIN OF CUSTODY FORM

The chain of custody form is a legal record documenting the sampling information, custody of the sample and the transfer to the laboratory. ***All sampling events require a chain of custody form.*** The chain of custody form, properly filled out, is also critical for logging samples into the laboratory, and serves as documentation linking analytical data in LIMS with the appropriate project or monitoring event. The information on the form is absolutely necessary.

Include the following information on the **Chain of Custody (COC)** form ([DEQ06-LAB-0054-FORM](#)):

- Sampling Event name.
- Date and time collected
- Sampler's name and organization
- Project Manager or contact to call with questions on the sampling event.
- Program to be charged (QTime number)
- Purpose for sampling (QAPP or SAP number where available)
- Description for each discrete sampling location (Station Name) and LASAR Station ID number (when available). See SOP *Station Naming Conventions* [DEQ06-LAB-0039-SOP](#).
 - **Note:** Submitting specific sample locations to the laboratory is not mandatory for legal samples to protect confidentiality. It is however, **mandatory** that the project manager or field sampler maintains documentation of the specific sample locations. It may be provided to the laboratory at the discretion of the project manager and it will be maintained in the laboratory workorder file. The information however will not be entered into the Laboratory LIMS system to maintain confidentiality.
- Container ID number(s) (in spaces provided).
- **If a QAPP or SAP is not yet available, request specific analysis by name, test group, or method in the Event Comment section of the COC.** For methods with long lists of analytes the laboratory will analyze their standard list of analytes for the test method unless otherwise directed.

To insure that the correct person receives the data report, enter one or more names in the upper right hand space labeled: "Report Recipients". On a COC form without anyone listed, data will be reported to the sample submitter. Further instructions can be found on the back of the COC form

When a sampling event is only one of several within a project, make certain that the same project name is used on each Chain of Custody form for each sampling event. As each case is completed and reported out a database is updated which links the project name with appropriate case numbers and date of reporting. If several sampling events related to the same project are listed using different names, it may be impossible to connect them in the future.

The COC form usually contains the only information the laboratory has about sample origin, possible contaminants and sources, conditions of collection, etc. It would be helpful, when the field sampler has kept a field notebook, if pertinent information was transcribed onto these forms. Information, such as "suspect cyanide in samples," "diesel spill," "samples contain elemental mercury," should be noted as a safety warning to the Sample Coordinator and Analysts that will subsequently be handling the samples.

Note: Please flag on the COC form samples known or suspected to contain sewage, cyanide, toxic solvents or heavy metals, etc. This is safety information for the Sample Coordinator and Analysts who will subsequently handle the container and sample material.

Always keep in mind that the holding time for a sample begins when the sample is collected, not when it arrives at the laboratory. This could lead to problems for samples having extremely short holding times,

such as nitrite, BOD₅ or turbidity (48 hours), and bacteria (e.g. coliforms) samples (8-24 hours). Should a sample be delivered so late as to exceed the holding time the test will be performed, but results will be reported with a qualifier to indicate holding time exceedance.

Each “item” should include a concise **Sampling Point Description (aka Station Name)** of where, or sample identification number, and when (time) sample taken. If this is a new sampling site, Latitudes and Longitudes **MUST** be included for the laboratory staff to create a new Station ID. Be specific, generalized locations (e.g. “in a field”, “across the street”, etc) cannot be used to identify a location. The sampler must also note the matrix (surface water, groundwater, soil, sediment, etc). The LEAD will create Station IDs if there is none available. For more information see *SOP Station Naming Conventions* ([DEQ06-LAB-0039-SOP](#)).

The complete sample description and container/preservation information number must be entered in the appropriate fields on the COC form.

The SAP and/or QAPP should contain a method and analyte list including each analyte, or analyte group, you are interested in having a sample item tested for. Try to be as specific as possible. The laboratory will assign methods appropriate to the analytes you are looking for.

Even though you (or your contractor) have provided a Sampling & Analysis Plan (SAP), it is still necessary for you to fill out the *COC form for all samples submitted*, based on the SAP/QAPP.

Record on “COC form” whether preservatives were added, which preservative, and the amount.

The DEQ LEAD Information Management System (LIMS) assigns a unique “work order” number to each batch of samples that are logged in. A work order number might be assigned to all samples from a specific project on a given sample day, or all samples collected during sampling event spanning more than one day. In no event will a work order number be assigned to include samples from different monitoring projects.

Work order numbers have seven digits. The first two digits represent the year, followed by two digits representing the month, and 3 digits that are assigned sequentially by LIMS and representing the order the cases were logged in during the month (YYMMxxx)

The container sizes, and materials are a result of the various chemical preservations and quantity of sample required to test for different analytes. Tests for several analytes, having identical preservation requirements, are frequently performed on a single bottle. It’s obvious that a bottle preserved with nitric or sulfuric acid cannot be analyzed for nitrate, sulfate, pH, alkalinity, conductivity, etc.; however, those tests can be performed on a bottle (poly) where the only preservation is refrigeration at < 6°C.

Based on the analytes you request, and your Data Quality Objectives (DQOs) (when specified in QAPP/SAP), the laboratory will assign an appropriate analytical method (analysis code). Analytical procedures used by DEQ LEAD comply with requirements of the applicable federal regulations; [e.g. SDWA, NPDES, RCRA, etc.] and are EPA approved¹³ methods. The lab procedures are referenced to current published literature. If DQOs are not specified the laboratory will employ their default method for the test. DEQ LEAD Methods are listed in the most recent laboratory Quality Manual (LQM).

Occasionally non-standard methods may need to be developed when a standard method is not available, or when interfering substances influence the performance of a standard method. Non-standard methods will be appropriately validated, documented, and noted on the laboratory report.

9.2 CHAIN OF CUSTODY PROCESS

The chain of custody process is a means of documenting who has control of the samples and may be different depending on the intended use of the data. First level chain of custody record is for samples unlikely to be used in litigation or enforcement action. The second level is for samples that may be used as evidence in legal processes. Two levels of *Chain-of-Custody* have been developed to eliminate some of the paperwork associated with maintaining a rigorous Chain-of-Custody. **PLEASE NOTE: A CHAIN**

¹³ 40 CFR 136, SW-846, Standard Methods, etc.

OF CUSTODY FORM IS REQUIRED FOR ALL SAMPLING EVENTS REGARDLESS OF THE CUSTODY LEVEL, It should also be noted that the custody level has no bearing whatsoever on analytical data quality; it only documents who had the sample in their possession at any particular time.

9.2.1. FIRST CUSTODY LEVEL

The first custody level is intended for routine, ambient monitoring, and samples unlikely to become involved in litigation. The custody transfer to the LEAD is documented on the COC form at the time samples are relinquished to the DEQ LEAD facility. The sample custodian inspects the shipment and documents any discrepancies from the Sample Acceptance Policy prior to logging the samples into the LIMS. It is always appropriate to make written comments on the Chain-of-Custody form concerning the integrity or condition of samples when received.

Samples are treated routinely in the secure DEQ LEAD facility. This includes: analyses using EPA approved methods, replicate analyses and/or matrix spikes, and QC standards using method and EPA QC criteria at the method required frequency; comparison against historical data, if it exists; data review for consistency, completeness, precision and accuracy; QA review of duplicate sample data; and standardized reporting on the laboratory LIMS. **FIRST CUSTODY LEVEL** samples will be discarded upon completion of analyses and report of results.

9.2.2. SECOND CUSTODY LEVEL

The second level of custody is to be used if the sampling event is involved in litigation or has potential to be involved in litigation. The second level (also see Appendix E) includes everything in **FIRST LEVEL**, plus:

- Custody is thoroughly controlled throughout the sampling process with the use of custody seals and maintaining the samples under lock and key in the vehicle until the delivery to the laboratory.
- Custody is controlled in the laboratory by storing the samples in locked storage area and all transfers of the samples into and out of the storage area are documented in a logbook and witnessed. The samples once checked out for analysis are maintained in the secure laboratory environment. Depending on the situation, type of sample, type of equipment used, duration of the analysis, and other factors, the laboratory analyst may take additional steps to document the security of the samples through use of locks, security tape, or other means. Any extra steps taken should be documented on the COC form.
- Sample remaining after analyses are finished, or empty container when sample is exhausted during analyses, will be retained in secure storage until release is approved in writing by program or sampler. Disposal date will be documented on the custody transfer document.

9.2.3. SAMPLE SHIPMENT

Transport samples to the laboratory immediately after collection. Notify the Sample Coordinator (503 693-5784) **prior to sample collection** to allow adequate time for scheduling analysis, advance preparation, and/or technical assistance. Ship samples¹⁴, packed on ice in a sealed ice chest available from the LEAD, via UPS. For shipment of **legal** samples, refer to "Legal Chain-of-Custody Procedures" [APPENDIX E](#).

Ship samples to:

***DEQ laboratory and Environmental Assessment Division
3150 NW 229th, Suite 150
Hillsboro, OR 97124***

¹⁴ Samples ONLY, via public carrier. Not preservative chemicals, decon solvents, or other sanctioned hazardous materials.

If possible, hand-deliver samples to the facility. We are located off of Evergreen Pkwy just east of 229th in Hillsboro. Building is on the south side of Evergreen Street (Eastbound side). See Map <http://www.deq.state.or.us/about/maps/lab01.pdf>.

VOLATILE ORGANICS: To avoid cross-contamination of samples during shipment, seal samples and "Transport Blank" vials in ziplock bags.

BACTERIAL samples (*fecal coliform*, *E.Coli*) must be received no later than 1200 hours (Noon) on Thursday.

For **Legal** bacterial/microbiological samples, the maximum holding time of 8 hours (30 hours for drinking water) must be adhered to, whereas routine samples can be held for a maximum of 24 hours. Analysis for a "non-routine" organism (*E.coli*, *total coliform*, *fecal strep*) is required, notify the laboratory well in advance so that special media can be prepared for the test. Microbiological testing is currently performed exclusively by the Oregon Public Health laboratory in the same building as DEQ LEAD.

BOD: Samples must be received no later than 1200 hours on Friday.

If it is necessary to sample between 12:00 noon Friday and 12 noon on Monday, the laboratory will need to schedule someone to come in on the weekend. Special notification is required prior to collection and shipment of samples.

9.3 FIELD NOTEBOOKS

A bound field notebook **should** be maintained to provide a daily record of significant events, observations, and measurements during field investigations. (In lieu of a notebook, field data sheets may be used if they contain sufficient documentation and are submitted with the Sampling Event information.) This record should include field measurements, personnel, personal and weather observations, and physical conditions. All entries in field notebooks should be made, signed, and dated in ink. Field notebooks, inspection reports, or other documentation should be kept as a permanent record.

These notebooks are intended to provide sufficient data, observations, and documentation to enable participants to reconstruct events that occurred during an activity, and to refresh their memory when called upon to give testimony during legal proceedings.

9.3.1. CORRECTIONS TO DOCUMENTATION

All original data recorded in field notebooks, chain-of-custody records, and other forms must be written in waterproof (blue or black) INK that does not smear if it gets wet. None of these documents should be destroyed or thrown away, even if they are/become illegible or contain inaccuracies that require a replacement document.

If an error is made on a document assigned to an individual, that individual must make corrections by crossing a single line through the error, enter the correct information, and initial and date the correction.

10.0 GUIDANCE ON CONTRACTING LABORATORIES

This guidance applies to those that contract out laboratory services or to those receiving data from third party labs to support decision-making.

Whenever one uses a laboratory data for any purpose, they must have confidence in the quality of the data. Data of unknown quality should not be accepted without question. It is the responsibility of the user of the data to know where the data is coming from.

Laboratory accreditation is **STRONGLY RECOMMENDED FOR ALL ENVIRONMENTAL TESTING** even though it is only required in Oregon for Safe Drinking Water Act (SDWA) compliance testing and asbestos testing in schools. Keep in mind there are some tests that there is no accreditation available.

Unaccredited laboratories are not subject to oversight unless they have an internal auditing program or are accredited through another agency for another purpose.

Keep in mind that laboratory accreditation does not guarantee that the data will necessarily meet project needs. What accreditation does is to ensure that there has been some external review of the laboratory to help show they have the capabilities to meet project needs. Note; the thoroughness of that external review may vary from auditor to auditor but is significantly better than no review at all.

If the laboratory is not accredited for the tests being reported, it would be advisable to visit the laboratory to ensure they have the equipment and capabilities to perform the tests. Contact a DEQ Quality Assurance Officer or a DEQ LEAD laboratory Section Manager for assistance if you are unfamiliar with laboratories.

Other States require accreditation for various programs. Knowing where the laboratory is located and what type of accreditation they hold can add confidence to the analytical data. Ask the laboratory for a copy of their accreditations. Other agencies (e.g. DOD, DOE) approve labs for their agency use as well. These are good programs and laboratories operating under them have been inspected.

Laboratory participation in proficiency testing (PT) programs is also a good way to help assist in the evaluation of the performance of a laboratory,

Before selecting a laboratory there are many things to consider:

- If DEQ is selecting, make sure the laboratory selected is an approved vendor. DEQ has contracts with 3 preselected laboratories.
- What questions do you need answered?
- Reporting levels / Detection levels / Action levels
- Regulatory Compliance testing or monitoring?
- Methods and QC limits?
- Deliverables
- Are there difficult matrices?
- Does it have to be “Bullet Proof” (Stand up to any possible legal challenge)
- What are your liabilities if there is a problem?
- **Is there a Quality Assurance Project Plan (QAPP) involved?**
 - Get the laboratory in early on the process of creating the QAPP
 - **QAPP Pitfalls**
 - **Not giving the QAPP to the laboratory until the samples arrive.**
 - The laboratory may need to do additional work to meet your needs
 - Slows down the project
 - May cause Turnaround Times to fail?
 - **Not giving the QAPP to the laboratory until after the project is over or not at all.**
 - laboratory may or may not have met QAPP objectives
 - Have to backpedal to try and salvage data

Some recommended things to ask the laboratory.

- **Are they accredited?**
 - For what parameters, media, and test methods?

- By what agency or accrediting body?
- **Have they had an external audit by a state or federal agency?**
- Can you get a copy of the findings and the labs response? (don't let the number of findings be as much of a concern as the type of findings).
- **Quality Assurance Program**
 - Do they have a Quality Manual that describes their program?
 - Is management involved with the QA program (Quality starts at the top)
 - Separate QA officer (where practical, small labs usually can't afford a separate QA officer)
 - Proficiency Testing Samples
 - Which ones do they participate in and how often?
 - Request results.
 - How wide are the control limits? Do they meet your needs?
 - Are the Reporting Limits acceptable? Can they achieve the project DQO?
 - Do they have an internal auditing and corrective action process
 - Do they qualify the data for QC failures or sampling, preservation, or holding time variances?
- **Control Limits**
 - Do they meet your objectives?
 - How wide / narrow are they.
 - **Note:** Some analytes just don't perform well (examples: Benzidine, ketones, some phenolics. Etc.)
 - Does the laboratory control on all/most compounds or are do they use the minimum subset listed in a method?
 - **Note:** A larger spike list is a good thing.
 - Do they report the QC results in the laboratory report?
- **Reporting Limits and Detection Limits**
 - Within Calibration Range
 - EQL – Estimated Quantitation Limit
 - RL – Reporting Limit
 - CRDL – Contract Required Detection Limit
 - LOD - Limit of Quantitation
 - Below Calibration Range (Must be estimated)
 - MDL – Method Detection Limit
 - IDL – Instrument Detection Limit.
 - Try to avoid reporting to MDLs unless necessary. Need to know all results below the LOQ (reporting limit, etc) must be reported as "*Estimated*"
- **Corrective Action Process**
 - Does the laboratory have a means of tracking non-conformances

- Types
- Frequency
- Do they monitor for trends (Are there recurring problems?)
- **More importantly**, does the laboratory have a mechanism of correcting those problems?
- **Data Qualification**
 - Contrary to popular belief, every analytical run is not perfect.
 - Sample matrix may cause problems with analysis
 - Qualified Data often still supports the questions to be answered.
- **Cautions**
 - Data Qualifiers provide useful information to data users. The laboratory should not be hiding problems.
 - Not always bad. e.g. Tight control limits may lead to more qualifiers
- **Ethical Practices**
 - Do they have an Ethics Policy?
 - Do they have defined manual integration procedures for chromatography methods?
 - Do they perform self auditing and notify clients if there are problems with data? (Do they recall reports when needed?)
 - **Caution:** Watch out for labs that never recall data.
 - Do they perform ethics and manual integration training?
 - Do they use data qualifiers?
- **Customer Service**
 - Are they responsive?
 - Do they have the technical expertise needed for the work being performed?
 - How are they at meeting turnaround times and holding times?
 - How are they about correcting errors?
- **Evaluate laboratory Documentation**
 - At least **once** request a raw data package for all analyses –
 - **Yes**, there is most likely a charge – relative to what is at stake, it is more than worth it.
 - If you don't know what you are looking at... **ask**. You can ask the laboratory that provided the data for clarification or there are 3rd party validators as well that perform the service. **For DEQ staff, ask the DEQ laboratory and Environmental Assessment Division, laboratory Managers or QA staff for assistance.**
 - A raw data package should be requested one time after the analyses have been performed (but not too long after).
- **Verify laboratory Capacity**
 - Does the laboratory have the capacity to deliver what you need?
 - Provide the laboratory with as much information about the number and type of sample as possible.

- If samples are really dirty, it slows down the lab: They need to know.
- Is there a sampling schedule that was agreed upon?
 - Changes in sampling volume can significantly impact the laboratory and TAT.

APPENDIX A SAMPLING FOR VOLATILE ORGANICS

Contamination

Because of the sensitivity of the analytical technique (0.5 parts per billion), and potential for sample contamination, volatile organics sampling is somewhat complex. It is important that all steps be followed. Every effort must be made to avoid contamination, and to document where contamination might originate (e.g. field, transport, laboratory blanks).

I Water Samples

Preparation

Notify the Organic laboratory before collecting any volatile organic samples. The laboratory will prepare the following vials upon proper notification and scheduling:

- A. **LABORATORY BLANK** Two vials filled by Sample Coordinator with purified water and kept in the Organic laboratory for later analysis
- B. **TRANSPORT BLANK** Two vials filled with purified water to accompany samples throughout the sampling run. Do NOT open these vials in the field. Return to the laboratory with samples.
- C. **TRANSFER BLANK** Two vials, filled with purified water by laboratory personnel, carried to the sampling site where the Sampler transfers the contents to two fresh vials.
- D. **SAMPLE VIALS** Three vials: The Sampler is provided with sufficient triplicate numbered vials for TRIPLICATE sampling at each site. Triplicate vials are distinguished by "-A" and "-B" and "-C" to designate first and second and third sample, respectively.

Sampling and Documentation

- 1 The laboratory will provide the Sampler with pre-numbered vials for the Samples, laboratory Blanks, and Transport Blanks. These vial numbers must be listed on the "COC form".
- 2 Transport Blanks are NOT to be opened, and are to accompany the Samples at all times.
- 3 The Sampler is required to prepare two Transfer Blanks at the sampling site, using water supplied by the laboratory.
- 4 All samples are collected in TRIPLICATE, and immediately refrigerated.

Samples should be delivered to the laboratory without delay.

Sample Collection

Collect volatile organic samples in triplicate sets of 40 mL glass purge vials with Teflon-lined (non-reactive) septums in the lids, labeled "A" and "B" and "C". **Fill the vials, in the order "A" followed by "B" and "C"**, from the same source with as little time-lapse as possible between them. **Do not rinse pre-preserved vials prior to filling.** Fill vials all the way to the top and a little above, generating a positive meniscus. No air bubbles should pass through the sample as the bottle is filled, or be trapped in the sample when the bottle is sealed. A positive meniscus at the top of the bottle will help ensure that no air is trapped inside when the cap is screwed on. If bubbles are present empty VOA take a new sample and repeat the check. Place the vials in a plastic bag, close the top, and put the bag in a cooler with ice. Ship the samples to the laboratory as soon as possible.

CAUTION!

Some purge vials from the DEQ LEAD are pre-acidified for preservation. Each vial contains 10 drops of concentrated hydrochloric acid. Acidified containers normally give off fumes, which should not be inhaled. If spilled on clothes or skin, flush immediately with copious amounts of water.

In addition to triplicate sample vials from each site, a sampling event should include vials for laboratory Transport, and Transfer blanks. When sampler desires *Matrix Spike(MS)* or *Matrix Spike Duplicate(MSD)* samples, he must collect one extra set of Triplicate Vials and submit the additional vials for analysis. The sample identified for MS/MSD should be noted on the COC along with vial numbers for the additional sample volume collected.

Note the number of each purge vial on the "COC form" with a site description, date and time sample was taken, as each sample is collected.

- 1 When sampling from a tap, open the tap and allow the system to flush until the water temperature has stabilized (usually 5-10 min.). Adjust flow to about 500 mL/min and collect duplicate samples from the flowing stream. The sampler should ensure that the tap is free of aerator, strainer, hose attachment or water purification device.
- 2 **When sampling a chlorinated water supply for volatiles** it is necessary to preserve the samples with 25 mg Ascorbic acid, then acidify with 2 drops 1:1 hydrochloric acid to destroy any chlorine that may be present. Do not use 40 mL vials that already contain HCl. If this is not done the free chlorine can react with some volatiles, producing chlorinated compounds that did not exist in the original sample.
- 3 When sampling from an open body of water fill a 1 quart bottle or clean beaker with sample, and carefully fill duplicate sample bottles from the container.
- 4 When sampling wells with a bailer the VOC sample should be collected from the first bailer-full after purging well. Precautions should be taken to prevent aeration of sample.
- 5 **Note:** VOA samples to be analyzed for 2-Chloroethyl vinyl ether (2-CVE), Acrolein and Acrylonitrile must NOT be acidified as the acid breaks down these analytes. Take a separate aliquot into an unacidified VOA vial.

Attention: Check VOC vials for Air Bubbles

Invert vial and tap on solid surface. If present, air bubbles will rise

II Soil Samples

Soil samples must be collected in accordance with EPA method 5035A to minimize the loss of volatiles. Bulk samples should not be collected in Jars or brass core sleeves. EPA method 5035A requires the use of special sampling devices (a modified syringe sampler or miniature core sampler, EnCore® or TerraCore®, or similar type device). Which preservation method is chosen will be dependent on the reporting levels that are required for a given Project Plan. Below are some options, check with the laboratory if you have questions:

- a. The sample is extruded into a pre-tared VOA vial containing Methanol (for high level samples) where volatile Organics are solubilized in the Methanol and loss through volatilization is eliminated are at least minimized.
 - Clean VOA vials containing Methanol must be pre-weighed before sampling and weighed again at the laboratory when received.
 - A 5-10 gram portion of soil sample is added to the vial containing Methanol and the vial is quickly sealed.

-
- Use of a modified syringe sampler is recommended for this process as they are easier to use however the Encore® will work as well.
- b. The sample is extruded into a pre-tared VOA vial containing sodium bisulfate preservation (for low level samples). The sodium bisulfate preservation retards microbial growth that can feed off of aromatic compounds, rapidly lowering their concentrations.
- Clean VOA vials containing dilute Sodium bisulfate must be pre-weighed before sampling and weighed again at the laboratory when received.
 - A 5-10 gram portion of soil sample is added to the vial containing Methanol and the vial is quickly sealed.
 - Use of a modified syringe sampler is recommended for this process as they are easier to use however the Encore™ will work as well.
 - **Note:** if 2-CVE, Acrolein, or Acrylonitrile are target analytes, another aliquot would need to be taken and placed in either an empty pre-tared VOA or a pre-tared VOA vial containing reagent water
- c. The sample is extruded into an empty, clean, pre-tared VOA vial (for low level samples).
- Clean VOA vials must be pre-weighed before sampling and weighed again at the laboratory when received.
 - A 5-10 gram portion of soil sample is added to the empty vial and the vial is quickly sealed.
 - Use of a modified syringe sampler is recommended for this process as they are easier to use however the Encore™ will work as well.
 - The Sample must be shipped ASAP as the sample must be frozen within 48 of sampling in order to extend the holding time to 14 days by freezing the samples. It is recommended that Dry Ice be used to freeze samples during transportation to the laboratory.

APPENDIX B SAMPLING PROCEDURE FOR DISSOLVED OXYGEN (Winkler Method Only)

INTRODUCTION

Currently, the Water Quality Monitoring section primarily utilizes a luminescence dissolved oxygen (DO) probe for in situ analysis of DO. However, there are still occasions where the Winkler procedure may be utilized.

Use this procedure for field preservation of water and wastewater samples for dissolved oxygen analysis if using the Winkler method. The preservation method employs dry reagent pillows to "fix" the dissolved oxygen for later analysis. Manganous ion reacts with the dissolved oxygen present in the alkaline solution to form a manganese (IV) oxide hydroxide floc. Azide ion is added to suppress interference by nitrite, which reacts with the free iodine generated during analysis. Following acidification, reduce the manganese (IV) floc with iodide; then titrate the free iodine produced with sodium thiosulfate (or PAO) titrant.

See DEQ03-LABB-0036-SOP, [Water Monitoring and Assessment Mode of Operations Manual \(MOMs\)](#) for assistance on field analysis of Dissolved Oxygen.

SAMPLE COLLECTION

Collect water samples in a clean 300 mL BOD bottle to overflowing with minimal aeration. This ensures that there will be no air bubbles trapped in the bottle. Replace ground glass stopper until you are ready to add reagent pillows.

SAMPLE PRESERVATION

- 1) Remove glass stopper and add contents of one Manganous Sulfate Powder Pillow. Replace cap and invert several times to facilitate mixing.
- 2) Remove glass stopper and add contents of one Alkaline Iodide-Azide Reagent Powder Pillow. Insert stopper and invert bottle several times to mix. A flocculent precipitate will form. It will be brownish-orange if dissolved oxygen is present or white if oxygen is absent.
- 3) Allow the sample to stand until the floc has settled. Again invert the bottle several times to mix, and let stand until the floc settles.
- 4) Transport samples immediately to the laboratory. **Store in the dark** at 10-20°C during transport. Preserved samples can be held for up to 8 hours prior to final titration.

NOTES:

- a) If DO is for **Initial D.O.** measurement used in BOD determinations a second bottle must be collected simultaneously and sent to the laboratory in a cooler with ice (do not add anything to this bottle). The cooler must be carefully packed to protect the sample containers.
- b) When collecting for laboratory BOD measurement, and BOD is expected to be greater than 5 mg/L, collect additional sample in a 1000 mL poly bottle for BOD dilution setups instead of the second BOD bottle. Samples that you suspect will have high BOD should be flagged so laboratory staff will know to prepare dilutions.
- c) Remember to schedule DOs and BOD5s with laboratory so that personnel can be scheduled to perform analyses.

APPENDIX C FIELD FILTRATION PROCEDURE.

APPARATUS

The filtering apparatus consists of a wide-mouth jar, rubber stopper with filter funnel base and filter inserted, and removable magnetic filter funnel. Use a hand-operated vacuum pump to filter the sample. Filters used must be **0.45 micron** cellulose membrane (47 mm diameter).

OPERATION

- 1) Remove cap from clean 250 mL poly bottle labeled as “dissolved” or “filtered” ; place in center of filtering jar.
- 2) Place rubber stopper (with filter base) firmly in jar opening, with filter stem inside poly sample bottle. (Vacuum cannot be produced unless jar is securely sealed).
- 3) Place filter membrane on filter base grid. (Make certain that blue separator disk is not attached to membrane).
- 4) Place funnel over filter.
- 5) Pour approximately 250 mL of sample into the funnel. Evacuate the filtering jar (using hand vacuum pump) until sample begins to pass through filter. If sample is very turbid, filter several small volumes, replacing filter membrane as it becomes plugged, until container is full.
- 6) Remove poly sample bottle from the Mason filtering jar. Rinse filter grid and funnel between samples with distilled water
- 7) Label the sample container to designate that it has been filtered (The DEQ lab will provide stickers for this).

ALTERNATIVE

Alternatively filtration may be performed using a peristaltic pump and clean Tygon™ tubing (cannot use if phthalates are a target analyte) and an in-line cartridge 0.45 micron filter. Use clean tubing for each sample or rinse with at least 1 liter sample before attaching the filter.

Contract DEQ lab staff for more detailed training and use of in-line cartridge filtration.

APPENDIX D EPA QUALITY ASSURANCE PROJECT PLAN OUTLINE¹⁵

A. PROJECT MANAGEMENT

- 1 Title and Approvals
- 2 Table of Contents
- 3 Distribution List
- 4 Introduction
- 5 Purpose
- 6 Project/Task Organization
 - a. Funding Program and Anticipated Resources
- 7 Problem Definition/Background
- 8 Project/Task Description
- 9 Quality Objectives and Criteria
- 10 Special Training/Certification
 - a. Safety
- 11 Documents and Records
- 12 Data Usage
- 13 Monitoring network design and rationale
- 14 Parameters and Frequency
- 15 Project Organization and Responsibilities

B. DATA GENERATION and ACQUISITION

- 2 Sampling Process Design
- 3 Sampling Methods
- 4 Sample Handling and Custody
 - a. Field Notebook
 - b. Corrections to Documentation
 - c. COC
- 5 Analytical Methods
- 6 Quality Control
 - a. Data Quality Objectives
 - Precision
 - Accuracy
 - Limits of Detection (LOD) and/or Quantitation (LOQ)
 - Representativeness
 - Comparability

¹⁵ From *EPA Requirements for Quality Assurance Project Plans* (EPA QA/R-5, March 2001)

- Completeness
 - b. Quality Control Procedures
 - 7 Instrument/Equipment Testing, Inspection, and Maintenance
 - 8 Instrument/Equipment Calibration and Frequency
 - 9 Inspection/Acceptance of Supplies and Consumables
 - 10 Non-direct Measurements
 - 11 Data Management
- C. ASSESSMENT AND OVERSIGHT**
 - 1 Assessments and Response Actions
 - a. Performance and Systems Audits
 - b. Corrective Actions
 - 2 Reports to Management
- D. DATA VALIDATION AND USABILITY**
 - 1 Data Review, Verification, and Validation
 - 2 Verification and Validation Methods
 - 3 Reconciliation with User Requirements

DEQ QAPP template ([DEQ04-LAB-0029-TMPL](#)) can be found on DEQ's intranet site *Q-Net*.

DEQ SAP template ([DEQ11-LAB-0026-TMPL](#)) can also be found on DEQ's intranet site *Q-Net*.

The detail of the contents is dependent on project needs. All components are not necessarily required for all projects.

Note: The QA Plan is to be developed and approved by the Project Manager and QAO prior to the start of the sampling event.

APPENDIX E LEGAL SAMPLE CHAIN OF CUSTODY (Custody Level 2)

Purpose

Sample integrity must be maintained throughout the collection, transport, storage, and analysis process. Consequently all field activities must be fully documented, the samples must be clearly identified, and custody procedures followed in both field and laboratory operations

Evidence for use in a legal action is subject to more stringent rules and criteria than that used for routine monitoring or assessment sampling events.

Written procedures must be available and followed whenever samples destined to become evidence are collected, transferred, stored, analyzed, or discarded. Their primary objective is to create an appropriate and accurate record that can be used to trace the possession of a sample from its collection, through analysis, until its introduction as evidence. A sample is considered to be *in custody* if it is:

- a. In actual physical possession.
- b. In view, after being in physical possession.
- c. Locked up.
- d. Kept in a secured area where access is restricted to authorized personnel.

Pre-sampling

Prior to any "Legal" sample collection, the project manager or field sampler should contact the Inorganic, Organic, and QA Managers at the laboratory to appraise the of the project's regulatory objectives, proposed sampling locations and procedures, matrices, analytical requirements, and Chain-of-Custody procedures to be followed. This is especially true for any project when non-routine sampling procedures are employed.

If legal action is contemplated the potential litigant also must be informed that samples are to be collected. Since analytical results may be used as evidence, the potential litigant is entitled to collect duplicate samples and have them analyzed by someone else, or observe the analysis being done by the DEQ laboratory. Contact your manager or the Environmental Law Specialist assigned to your program, or call Les Carlough in the Office of Compliance and Enforcement at 503-229-5422 for assistance. .

Field Documentation

Field records must be complete, dated, and initialed at the time the sample is collected. Fill out the COC form, providing at least the following information (Additional types of documentation may be relevant and should be identified in the site-specific SAP):

Location of sampling station (include latitude and longitude if there is no station ID)

Date and time sample collected

Case name and reference to QAPP or SAP where available.

Sample container number

Preservatives added and quantity

Number and type of samples shipped;

Number of shipping containers sent;

Analyses required (the COC is the official request for analysis for a laboratory, contact laboratory if unsure of what parameters are needed)

Pertinent field data (e.g., pH, temperature)

Name of sample collector(s)

Name of person performing field tests.

Site observations and photographs (where applicable and with written descriptions);

Equipment numbers and/or calibration information;

Field Sheets or other sample collection forms;

A separate, bound field log book is highly desirable. The necessity, and the specific information to be recorded therein must be determined prior to sampling, by the project manager.

Color photographs of the sampling location are recommended to facilitate identification and subsequent recollection by the sample collector(s). Sign and write date, time, and location on the back of each photo. Handle photographs according to Chain-of-Custody procedures. Keep and protect negatives (if appropriate) as part of the documentation.

Sample Identification

All sample containers must be uniquely identified.

Field Custody Procedures

To ensure proper custody while in the field, the following custody procedures will be followed:

- As few people as possible will handle the samples (each person that handles the samples becomes a potential witness and increases potential for errors);
- Coolers or boxes containing clean sample containers will be sealed with the appropriate custody seals until opened in the field;
- Sample bottles from containers that appear to have been compromised shall not be used;
- The sample collector will assume responsibility for the samples until transferred to another person (or shipping courier) following the appropriate chain-of-custody procedures;
- All sample data will be recorded in ink in a field notebook and on the appropriate field forms;
- A site team leader will assess if additional samples are required;
- All samples requiring thermal preservation will be shipped with an appropriate temperature blank, which will (at a minimum) consist of a 100-mL polyethylene bottle filled with clean water;
- Each cooler (shipping container) in which samples are packed will be sealed and accompanied by one copy of the chain-of-custody record that is sealed in a zip-lock bag and taped to the inside lid of the shipping container; **Note:** If hand delivered, the COC does not need to be inside the cooler.
- A separate chain-of-custody record will accompany each shipment of samples;
- Packaging, marking, labeling, and shipping of samples will comply with all regulations promulgated by the U.S. Dept. of Transportation, 49 CFR 171-177, and International Air Transport Association (IATA); and
- Freight bills and bills of lading will be maintained as part of the permanent project record.
- Written procedures for collection, preservation, and handling, specific for each sample type and analysis must be followed, and any deviations documented.

Custody Seals

Custody seals/tape must be present on all shipping containers. These seals are designed to show evidence of tampering or disturbance and must be present on the shipping container in as many places as necessary to ensure security. The seals must be dated and signed before application to the shipping containers. The seals may be covered in clear tape to prevent accidental damage during the shipping process. Custody seals on individual containers are not required in most cases as long as the samples are shipped the same day of sampling and are not left unattended. If samples are collected over multiple days and then shipped/delivered in a single shipment, then custody seals on individual sample containers would be recommended. On a case by case basis, DEQ's Office of Compliance and Enforcement (OCE) may also request that seals are placed in individual containers. If custody seals **are** to be placed on

individual sample containers, the containers must be protected from excessive moisture (ice water) that may cause the custody seals to be damaged or come off the container. Sealing the containers in zip-lock plastic bags is highly recommended.

Once sealed, a cutter is required to remove the tape, assuring the integrity of the samples to their delivery destination. A copy of the COC form should be retained by the sample collector. Copies of receipts from post offices, or bills of lading, will be retained by the laboratory as part of the permanent chain-of-custody documentation.

An effective **security tape/seal** is any material (e.g. tape) that cannot be removed without detection. Filament packing tape wrapped several times completely around the cooler makes for good sealing material. The tape should be signed or initialed and dated using a permanent marker pen across the tail-end of the tape.

The DEQ security seals available from LEAD, are generally effective as seals on individual glass and plastic sample containers (too much water may cause damage to the seals).

If samples are relinquished for transport to the laboratory, the transfer must be properly documented, i.e. signatures of both parties, date, and time of transfer recorded on the COC form. Note: Shipment of a secure cooler/ice chest via third party transport does not require signature by the company agent.

The sample collector must complete a Chain-of-Custody form for all samples that are delivered directly to the laboratory. The sample collector will receive a copy to acknowledge receipt of the samples by the Sample Coordinator.

Transfer of Custody

When transferring possession/custody of samples the date and time of transfer must be recorded on the chain of custody form, and all persons involved must sign the record. The sample collector is responsible for proper packaging, security, and transport of samples to the laboratory for analysis, as well as providing all necessary sample documentation.

Laboratory and Environmental Assessment Division (LEAD) Custody

This section includes all of the routine practices stated in first level custody and the field.

The Sample Coordinator and Sample Custodian are the principal custodians of samples in LEAD.

Samples should be handled by a minimum number of persons.

If custody or evidence seals/tape are present, they are inspected for integrity on the sample coolers and/or individual sample containers. The presence/absence of seals on both coolers and sample containers and their condition are noted on the sample receipt checklist.

➤ Locked Storage

Sampling event numbers and container ID numbers for each sample are entered into the legal custody logbook binder and the all sample containers are placed into the locked storage refrigerator. The process is witnessed and time, date, and the initials of receiver and witness are also entered into the logbook.

All samples in the locked storage must be signed out and back in with the same information for each transfer of custody.

All empty containers are returned to the locked storage in the same fashion.

➤ Laboratory Custody

Once signed out to an analyst, they are responsible for the integrity of the sample until signed back in to the locked storage. The laboratory is considered a secure area for samples during processing as there is limited key card access. The original samples is processed and returned to secure storage.

Original samples and sample containers are retained until Office of Enforcement informs the sample coordinator that the samples are no longer needed and may be disposed.

Records Management

The Chain of Custody and Field Data Sheets are scanned into the electronic report and kept into the physical sampling event (case) file.

The sample receipt checklist is stored with the physical case file.

The custody log for the case is maintained in the logbook binder until the samples have been disposed. Once disposed, a copy of the log page(s) from the binder is placed in the physical file folder of the sampling event.

Sample Control maintains a locked refrigerator, with limited key card access, exclusively for storing **SECOND CUSTODY LEVEL** samples. The refrigerator is accompanied by a log book in which to record sample transfers. The Sample Coordinator, sample Custodian, QA Chemist, Inorganic and Organic laboratory Managers, and laboratory Division Administrator are the only staff that has key card access to the locked refrigerator.

It is always appropriate to make written comments on the Chain-of-Custody form concerning the integrity or condition of samples.

If the samples have been mailed or shipped to the laboratory, the Security Seal number must be recorded on the Chain-of-Custody form (if there is no number on the seal, document the presence/absence of an intact seal. Transportation receipts are attached to the copy for filing, and a second copy is given to the sample collector to acknowledge receipt.

SECOND CUSTODY LEVEL samples are distributed for analysis or secured in a locked refrigerator, as appropriate. A copy of the Chain-of-Custody form must be kept with samples. Samples are considered to be in a secure area upon custody transfer from the Sample Custodian to the laboratory staff.

All laboratory documentation and logs are filled out as usual, but the samples are identified as "LEGAL" to alert the analyst of the need to follow custody procedures.

The laboratory area is designated a secure area, and access is restricted to authorized personnel only.

Laboratory personnel are responsible for the care and custody of the sample, once it is in their possession. They should be prepared to testify that the sample was in their view, or secured in the laboratory area, at all times while in their custody. The samples may be stored in Key card-access refrigerators for short term storage until the samples are returned to sample control and signed back in.

Approved methods for laboratory analysis will be used and documented. All analytical results are recorded in bound notebooks or on bench sheets and are retained as a permanent record in the laboratory, as well as on LIMS.

When sample analyses are complete the unused portion of sample must be returned to secure storage until data is reported out of the laboratory.

Unused portion of samples logged into the laboratory under **FIRST CUSTODY LEVEL** will be discarded as soon as the analytical data is reported.

Unused portion of samples logged into the laboratory under **SECOND CUSTODY LEVEL** will be discarded/destroyed only by order of Legal Council or the Division Administrator after consultation with enforcement officials, when it is verified that samples are no longer required. The disposal of the samples is also documented (date, time, DEQ staff) in the custody logbook form. A copy of the internal custody logbook forms will be maintained in the case/event file.

References

NPDES Compliance Inspection Manual, U.S. EPA, 305-X-04-001, Office of Enforcement and Compliance Assurance Office of Compliance, July 2004 (Appendix J and H updated 2006)

Handbook for Analytical Quality Control in Water and Wastewater laboratories, U.S. EPA 600/4-79-019,
EMSL - Cincinnati, Office of Research and Development, March, 1979.

APPENDIX F OSHD and EPA DRINKING WATER STANDARDS¹⁶

EPA PRIMARY STANDARDS mg/L

Goal: to protect human health in public drinking water supplies. MCLs are enforceable, MCLGs are not.

ORGANICS MCL¹⁷ MCLG¹⁸

Disinfection By-Products (DBPs)

a) Trihalomethanes

Sum of 4 THM's below:

Chloroform		0.07
Bromoform		zero
Chlorodibromomethane		0.06
Bromodichloromethane		zero
Total THMs	0.080	

b) Haloacetic Acids (HAAs)

Sum of 4 THM's below:

Dichloroacetic acid		zero
Monochloroacetic acid		0.07
Trichloroacetic acid		0.02
Bromoacetic acid		--
Dibromoacetic acid		--
Total HAA 5	0.060	

c) Bromate 0.010 zero

d) Chlorite 1.0 0.8

Pesticides & PCBs

Alachlor	0.002	zero
Aldicarb	0.003	0.001
Aldicarb sulfoxide	0.004	0.003
Aldicarb sulfone	0.002	0.002

¹⁶ From 40 CFR parts 141 and 142, July 2008

¹⁷ Maximum Contaminant Level, mg/L

¹⁸ Maximum Contaminant Level Goal, mg/L

Atrazine	0.003	0.003
Carbofuran	0.04	0.04
Chlordane	0.002	zero
Dibromochloropropane (DBCP)	0.0002	zero
Endrin	0.002	0.002
Ethylene dibromide (EDB)	0.00005	zero
Heptachlor	0.0004	zero
Heptachlor epoxide	0.0002	zero
Lindane	0.0002	0.0002
PCBs	0.0005	zero
Pentachlorophenol	0.001	zero
Toxaphene	0.003	zero
Methoxychlor	0.04	0.04

Chlorophenoxy Herbicides

2,4-D	0.07	0.07
2,4,5-TP (Silvex)	0.05	0.05

VOC's - regulated

	MCL	MCLG
Benzene	0.005	zero
Carbon tetrachloride	0.005	zero
1,2-Dichlorobenzene (ortho)	0.6	0.6
1,4-Dichlorobenzene (para)	0.075	0.075
1,2-Dichloroethane	0.005	zero
1,1-Dichloroethylene	0.007	0.007
cis-1,2-Dichloroethylene	0.07	0.07
trans-1,2-Dichloroethylene	0.1	0.1
Dichloromethane	0.005	zero
1,2-Dichloropropane	0.005	zero
Ethylbenzene	0.7	0.7
Chlorobenzene (mono)	0.1	0.1
Styrene	0.1	0.1
Tetrachloroethylene	0.005	zero
Toluene	1	1
1,1,1-Trichloroethane	0.200	0.20
1,1,2-Trichloroethane	0.005	0.003

Trichloroethylene	0.005	zero
Vinyl Chloride	0.002	zero
Xylenes	10	10

SOCs

	MCL	MCLG
Di(ethylhexyl)adipate	0.4	0.4
Di(ethylhexyl)phthalate	0.006	zero
Hexachlorobenzene	0.001	zero
Hexachlorocyclopentadiene	0.05	0.05
1,2,4-Trichlorobenzene	0.07	0.07
2,3,7,8-TCDD (Dioxin)	0.00000003	zero
Dalapon	0.2	0.2
Dinoseb	0.007	0.007
Diquat	0.02	0.02
Endothall	0.1	0.1
Glyphosate	0.7	0.7
Picloram	0.5	0.5
Simazine	0.004	0.004
Oxamyl (Vydate)	0.2	0.2

e) PAHs

Benzo(a)pyrene	0.0002	zero
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INORGANICS

Asbestos	7 MFL ¹⁹	7 MFL
Cyanide (CN)	0.2	0.2 ²⁰
Fluoride (F)	4.0	4.0
Nitrate as N	10	10
Nitrite as N	1	1
Nitrate + Nitrite as N	10	10
Antimony (Sb)	0.006	0.006
Arsenic (As)	0.010	zero
Barium (Ba)	2	2

¹⁹ Million fibers per Liter > 10 microns

²⁰ As Free Cyanide, Amenable Cyanide or Weak Acid Dissociable Cyanide

Beryllium (Be)	0.004	0.004
Cadmium (Cd)	0.005	0.005
Chromium (Cr)	0.1	0.1
Copper (Cu)	1.3 ²¹	1.3
Lead (Pb)	0.015 ²¹	zero
Mercury (Hg)	0.002	0.002
Selenium (Se)	0.05	0.05
Thallium (Tl)	0.002	0.0005

BACTERIOLOGICAL

Total Coliform Bacteria 2.2/100 mLs

OSHD & EPA SECONDARY Standards (quality) SMCL²³'s (mg/L)

Goal: protection of aesthetics (taste & appearance) of Public Drinking Water supplies. **NOT Primary MCLs.**

Silver (Ag)	0.1 mg/L
Aluminum (Al)	0.05 to 0.02 mg/L
Beryllium (Be)	0.004 mg/L
Chloride (Cl)	250 mg/L
Copper (Cu)	1.0 mg/L
Fluoride (F)	2.0 mg/L
Iron (Fe)	0.3 mg/L
Manganese (Mn)	0.05 mg/L
Sulfate (SO ₄)	250 mg/L
Zinc (Zn)	5.0 mg/L
Total Dissolved Solids (TDS)	500 mg/L
Total Hardness	250 mg/L
Surfactants (MBAS)	0.5 mg/L
pH	6.5 – 8.5 pH Units
Odor	3 threshold odor number
Color	15 color units
Corrosivity	non-corrosive

Calcium also monitored as a parameter for corrosivity.

²¹ "Action Level", not MCL.
²³ Secondary Maximum Contaminant Limit

APPENDIX G DETECTION/QUANTITATION LIMITS

DL?RL ?MDL?PQL?LOD?LOQ?

Method Detection Limit and *Practical Quantitation Limit* are common terms used to describe detection levels and though there are technical differences from "Limit of Detection" and "Limit of Quantitation", they are generally used interchangeably (MDL/LOD and PQL/LOQ). Note many laboratories use the term Reporting Limit (RL) synonymously with PQL and LOQ.

Prior to development of sophisticated analytical instrumentation common today (GC, GC/MS, ICAP, ICP/MS, HPLC, etc), chemical analysis was "a kinder and gentler" field of expertise. Whereas chemists formerly separated, purified, and weighed an analyte of concern, analysts now measure an optical, electrical, or electromagnetic property of an analyte, or a discreet fragment thereof, assisted by a highly refined computer that controls the measurement system and statistically evaluates the instrument signal. At some concentration it becomes impossible to distinguish between signal from analyte, and that arising from electronic "noise."

METHOD DETECTION LIMIT (MDL) The method detection limit (MDL) is defined as the minimum concentration of a substance 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 (USEPA 40CFR136 Appendix B).

The MDL considers all operations performed on a sample (digestion, extraction, etc). A MDL is measured by analyzing a minimum of seven samples in a given matrix spiked at very low concentration levels (between 1X and 10X estimated MDL) using procedures in 40 CFR 136 Appendix B. The MDL is the standard deviation (s) of these replicate analyses multiplied by the Student's T value for 99% confidence with n-1 degrees of freedom.

A measured value is credible and meaningful only when it is larger than the uncertainty associated with it (e.g. an observation of 50 mg/L \pm 100% is not as believable as 50 mg/L \pm 20%).

As the measurement concentration increases the relative uncertainty decreases.. The **LIMIT OF QUANTITATION (LOQ)** or **PRACTICAL QUANTITATION LIMIT (PQL)** are defined as the concentration above which quantitative results may be obtained with a specified degree of confidence. For practical purposes, this is represented by laboratory control limits for the analyte of concern. The levels of the LOQ or PQL should be such that they are incorporated in the calibration range of the method.

METHOD DETECTION and PRACTICAL QUANTITATION LIMITS are NOT method constants; they depend on analyst expertise, quality control employed, and the matrix being measured. Although it is customary for labs to adopt MDLs and PQLs published in EPA methods, they should periodically be measured by each laboratory for each matrix they analyze. The PQL of an analyte in various matrices can range from 50 to 500 times higher than in groundwater.

Use of data reported as below MDL (< MDL) can be a problem. ACS and ASTM take the position that observations below MDL should be reported as "Not Detected" (ND), with the numerical MDL reported in parentheses; however EPA's position is that this data should be reported (only some programs). DEQ uses the convention of reporting values below a detection level, be it the LOD or LOQ as <LOD or <LOQ with a numeric value of the LOD/LOQ following the "<". One problem with reporting <MDL as "ND" is that the data may be treated as "zero" in subsequent calculations. This type of censoring biases the summary statistics, and ignores important information about data variability at these low levels.

Two additional practices commonly used for treating values below the LOD data in calculations, besides treating "ND" measurement as zero, are: consistently treat the measurement as if it were AT the MDL (EPA preferred), rather than below it, or treat the measurement as if it were one-half the numerical value of the MDL. Thus <0.0005 becomes either 0.0005 or 0.00025 for calculation purposes. Whichever convention is chosen, some bias is inevitable. For Relative Percent Difference (RPD) calculation on QA Reports DEQ LEAD uses 0 for results < MDL. In these cases an absolute difference is used to evaluate precision. All of these conventions have their merits but whatever convention is chosen, it must be declared and consistently applied.

ON DEQ LABORATORY REPORTS

When diluting samples at the time of preparation or at the time of analysis the LOQ and LOD will be adjusted based on the dilution and the sample result will also be adjusted accordingly

APPENDIX H MATRIX QUALITY CONTROL (SPIKES AND DUPLICATES)

What is meant by **Matrix Spike**? **Matrix Spike Duplicate**? The concept can be very confusing.

Matrix Spike (MS) a matrix spike is the addition of a known amount of target analyte to a sample prior to preparation and analysis. A matrix spike is useful for assessing the **performance of a method in a specific sample matrix**. *i.e.* systematic errors arising from, sample extraction efficiency sample matrix effects and other interferences. Matrix spikes are generally not used as an indication of laboratory batch QC performance. It tells us how well suited a method is for a particular matrix.

Matrix Spike Duplicate (MSD) are just as the name describes: a duplicate of the Matrix Spike. MSDs are useful for assessing **analytical precision and sample homogeneity**, *i.e.* random errors arising from instrument instability, variations in sample composition, faulty technique, tolerances, etc.. Analytical precision sample homogeneity can also be determined by analysis of two portions of the same sample, *e.g.* duplicate samples, without spiking. Analysis of field duplicate samples can be used to assess field sampling precision (representativeness of sample collection). Various methods developed under different programs (CWA, SDWA, & RCRA) require MSs and MSDs at a frequency from 5 to 10% of the samples collected.

Most analytical methods are developed using relatively clean samples under well controlled conditions. When the method is applied by a *real-world* analyst to a *real-world* sample matrix containing chemical compounds that interfere with the method, by either reducing or enhancing the recovery of the analyte of interest, we are said to have a bias. Although it may not appear logical, it is not uncommon to add an accurately known quantity of analyte to a sample, analyze it, and get back considerably less or more than 100% of what was added. In most cases bias is characterized by occurring in the same direction, *e.g.* for a given analyst-matrix-method combination the results are nearly always biased in the same direction, either high or low.

The degree of bias and precision in test data is a result of both systematic and random error in both the sampling and analytical phases of the project. Assessing which uncertainty is attributable to which activity is not a precise science, given the limited amount of data associated with each individual project and unique matrix.

Theoretically the sample, MS, and MSD analyses are performed on identical portions of homogeneous sample material originating in a single sample container. This is doable for some types of sample, *e.g.* aqueous, or very finely divided, dry solid (*e.g.* powdered sand), but it is not possible for every sample matrix or method. The key to this admonition is that the sampled material must be homogeneous, and there must be enough sample quantity to prepare and analyze several portions of the same sample.

The basis for this technique is that by analyzing a portion of the original sample, as it comes from the field, we determine the concentration of those analytes in all portions of that sample, including those portions we subsequently spike (MS and MSD) with a known concentration of analyte.

It is necessary to find the analyte concentration in the unspiked, original sample material before it is possible to calculate the recovery of the added (spiked) concentration. Matrix spike recovery is calculated as follows:

$$\text{Percent Spike Recovery} = 100 \times \frac{\text{Observed Concentration}}{(\text{Spike Concentration}) + (\text{Initial Concentration})}$$

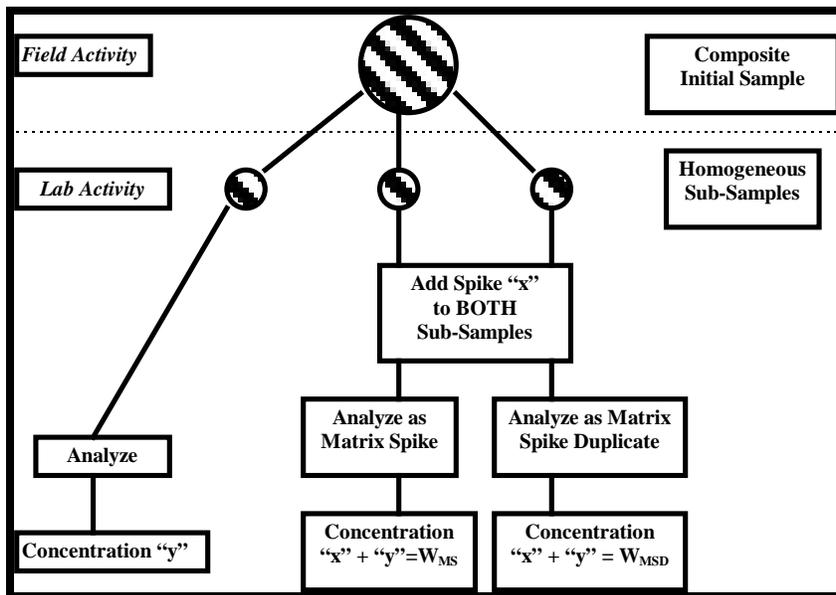
Where: **Observed Concentration** = concentration of analyte found in spiked sample

Spike Concentration = concentration of analyte spike added

Initial Concentration = concentration of analyte in unspiked sample

Uncertainty may be created when sample, MS, and MSD are collected as separate, discreet samples; a question of homogeneity arises, particularly for soils or solids, raising concerns about the relevance of MS & MSD analyses. The probability of collecting two or more soil grab samples, even out of the same hole, and have them be identical is practically non-existent. For valid samples for spiking multiple grab samples, mix them together to produce a homogeneous composite, and then sub-sample the composite. Although it would be best to send a single, bulk sample to the lab, if the composite is *thoroughly* mixed it could be packaged in multiple containers. This approach should work for semi-volatiles, pesticides, TPH, herbicides, PAH, and PCBs in soil as well as for all inorganic parameters.

Ideally the diagram below is the way MS & MSD samples should originate, not by collecting separate grab samples for each entity.



Mixing a composite of soil grab samples is generally not a valid approach for Volatile Organic samples; samples for VOCs should be disturbed as little as possible to preclude loss of volatiles. Instead it is better to collect several smaller grab samples and place them in the container undisturbed.

Where a MS/MSD (or Duplicate sample analysis) is needed for a project, please note the information on the Chain of Custody. The sampler may designate a specific sample on the COC or the lab may select one if a specific sample is not designated.

The quantity of sample submitted can hamper doing MS and MSD analyses. With soils it is less of a concern, because relatively small quantities of sample are necessary for analyses. However for aqueous samples most organic methods require a 1 liter sample for each analysis. This does not allow performing multiple analyses on a single container for semi-volatiles, pesticides, herbicides, etc., because it takes one full container for each analysis. In these cases additional sample must be collected and submitted in order for the laboratory to perform the MS/MSD. For most Inorganic analyses there is enough sample in a container to run the QC out of sample submitted without having to collect additional volume though if there are a lot of tests that come out of the same container, an additional one may still be needed. If you are coordinating with the laboratory ahead of time, they can help ensure you have sufficient containers for any additional QC samples.

In many organic analytical methods, **Surrogate Spikes**²⁴ are added to each sample analyzed, as a quality control measure. A *Surrogate* is a stable compound that is chemically similar to the analytes

²⁴ Anywhere from one to six surrogate compounds may be used depending on the method.

being analyzed for, but either isotopically labeled or else does not occur as a natural or manmade substance normally observed in environmental samples. The surrogate spike performs a similar function as a matrix spike but is added to every sample. Percent recovery of these surrogates must fall within a specific range for the analysis to be considered "acceptable." Examples include D6-Benzene (benzene with all six hydrogen atoms replaced by deuterium), bromofluorobenzene, various di- or tri-substituted fluorobenzenes, etc.

Problems can occur with surrogate recoveries on samples containing elevated concentrations of one or more analytes. Surrogate spikes are prepared at a concentration to function within the normal calibration range of the analytical instrument. Observation of very high concentrations of one or more analytes requires the sample be diluted to bring these elevated concentrations within the valid concentration range. Substantial sample dilution often results in dilution of the surrogate concentration to a level that is below their detection limit. Although surrogate recovery may appear bad, it may not be when all the facts are known.

APPENDIX I LABORATORY SAMPLE AND PRESERVATION REQUIREMENTS

ANALYTE	CONTAINER ²⁵	MIN. QUANTITY	PRESERVATION	HOLDING TIME ²⁷
Acidity	1L Poly	100 mL	Cool ≤ 6°C	14 days
Alcohols	1L Amber	50 mL	Cool ≤ 6°C	14 days
Alkalinity	1L Poly	100 mL	Cool ≤ 6°C	14 days
Asbestos (bulk)	Zip Lock Bag	200 g	None	N/A
BOD	1L Poly, BOD Bottle	300 mL	Cool ≤ 6°C	48 hours
BOD ₅	1L Poly, BOD Bottle	300 mL	Cool ≤ 6°C	48 hours
CBOD	1L Poly, BOD Bottle	300 mL	Cool ≤ 6°C	48 hours
NBOD ²⁸	1L Poly, 2x BOD Bottle	600 mL	Cool ≤ 6°C	48 hours
Chloride	250mL, 500mL, 1L Poly	50 mL	None	28 days
Chlorine	1L Poly	50 mL	Cool ≤ 6°C	Immediate
Chlorophyll	Petri dish	1 filter	Field Filter, acetone w/ MgCl, dry ice Avoid light	28 days
COD	500mL Poly	50 mL	H2SO4 pH <2, Cool < 6°C	28 days
Color	1L Poly	50 mL	Cool ≤ 6°C	48 hours
Conductivity	1L Poly	50 mL	Cool ≤ 6°C	28 days

²⁵ Wide Mouth Jars (4, 6, or 8 oz) can be used for all soil analyses with the exception of Volatile Organics. Preservation for soil samples is Cool < 6°C in almost all cases, freezing may extend the HT for some parameters.

²⁷ Holding Time for water and soil samples may be different.

²⁸ Nitrogenous BOD determined by difference between BOD & CBOD.

ANALYTE	CONTAINER ²⁵	MIN. QUANTITY	PRESERVATION	HOLDING TIME ²⁷
Cyanide	1L Poly	500 mL	NaOH pH > 10 (dechloro if needed) Cool ≤ 6°C	14 days
Dissolved Oxygen (DO)	BOD bottle, P	300 mL	Cool ≤ 6°C	0.5 h, 8h/8h ²⁹
Ethylene Dibromide (EDB)	40 mL VOA x 2	2 x 40 mL	No Headspace Cool ≤ 6°C	14 days
Flash Point	500 mL glass jar	200 g	Cool ≤ 6°C	30 days
Fluorescein	1L Amber	1000 mL	Cool ≤ 6°C	30 days
Fluoride	DP	50 mL	None Required	28 days
Formaldehyde	1L Amber	1000 mL	Cool ≤ 6°C	30 days
Glycol	1L Amber	1000 mL	Cool ≤ 6°C	30 days
Herbicides	1L Amber	1000 mL	Cool ≤ 6°C	7d/40d
Hexane Extractable Material (formerly Oil and Grease)	1L Amber	1000 mL	H2SO4 pH <2, Cool < 6°C	28 days
Hydrocarbon ID	1L Amber	1000 mL	Cool ≤ 6°C	7d/40d
Ketones-Aldehydes (Air)	Cartridge DNPH	1	Cool ≤ 6°C	14d/30d
Lignin-Tannin	1L Poly	50 mL	Cool ≤ 6°C	N/A
MBAS	1L Poly	250 mL	Cool ≤ 6°C	48 hours
METALS				
Chromium +6	250 or 500mL Poly	100 mL	Cool ≤ 6°C	24 hours
Mercury	500mL Poly	100 mL	HNO ₃ pH <2. Cool ≤ 6°C	28 days
Hardness (calc. Ca/Mg)	250 or 500mL Poly	100 mL	Field Filter, HNO ₃ pH <2	6 months
Metals, Total.Recoverable	250 or 500mL Poly	100 mL	HNO ₃ pH <2	6 months

²⁹ Analyze immediately. Winkler allows stabilization & holding for 8 hours until titration.

ANALYTE	CONTAINER ²⁵	MIN. QUANTITY	PRESERVATION	HOLDING TIME ²⁷
Metals, Dissolved	250 or 500mL Poly	100 mL	Field Filter, HNO ₃ pH <2	6 months
Enterococci	Sterile Plastic Bac-T	100 mL	Cool ≤ 6°C	8 hours Legal, 30 hours routine
Fecal coliform, non-chlorinated	Sterile Plastic Bac-T	100 mL	Cool ≤ 6°C	8 hours NPDES compliance 24 hours non-compliance
Total coliform, chlorinated	Sterile Plastic Bac-T	100 mL	Sodium thiosulfate Cool < 6°C	8 hours NPDES compliance 24 hours non-compliance
Ammonia nitrogen	500mL Poly	50 mL	H ₂ SO ₄ pH <2, Cool < 6°C	28 days
Nitrite nitrogen	DP, 1L Poly	50 mL	Cool ≤ 6°C	48 hours
Nitrate+Nitrite nitrogen	500mL Poly	50 mL	H ₂ SO ₄ pH <2, Cool < 6°C	28 days
Total Kjeldahl Nitrogen	500mL Poly	50 mL	H ₂ SO ₄ pH <2, Cool ≤ 6°C	28 days
Organotins (TBT, etc)	Polycarbonate 4-8 oz jar (soils)	1000 mL	Cool ≤ 6°C	7d / 40d (water), 14d / 40d (soil) 8wk / 40d (soil-frozen)
Ortho Phosphate	250 or 500mL Poly	100 mL	Field Filter, Cool ≤ 6°C	48 hours
Pesticides, Chlorinated or Nitro-Phos	1L Amber	1000 mL	Cool ≤ 6°C	7 days to extract, 40 days after
Pharmaceutical and Personal Care products	1L Amber (two bottles ea.)	2000 mL	Cool ≤ 6°C	7 days to extract, 40 days after

ANALYTE	CONTAINER ²⁵	MIN. QUANTITY	PRESERVATION	HOLDING TIME ²⁷
Polynuclear Aromatic Hydrocarbons (PAH)	1L Amber	1000 mL	Cool $\leq 6^{\circ}\text{C}$	7 days to extract, 40-days after
Polynuclear Aromatic Hydrocarbons (PAH) (Air)	Cartridge PUF	1	Cool $\leq 6^{\circ}\text{C}$	14d/30d
PCB	1L Amber, L	1000 mL	Cool $\leq 6^{\circ}\text{C}$	1 year to extract, 1 year after
Pentachlorophenol	1L Amber	1000 mL	Cool $\leq 6^{\circ}\text{C}$	7 days to extract, 40-days after
Percent Fat	Foil	1 fish	Freeze $< 10^{\circ}\text{C}$	6 months
pH	1L Poly	100 mL	Cool $\leq 6^{\circ}\text{C}$	Immediate (24 hrs)
Phenolics	1L Poly ,1L Amber,	500 mL	H ₂ SO ₄ pH <2, Cool < 6oC	28 days
Pheophytin	Chlorophyll tube	8 mL	Field Filter, acetone w/ MgCl, dry ice Avoid light	7 days
Phthalates	1L Amber	1000 mL	Cool $\leq 6^{\circ}\text{C}$	7 days to extract, 40-days after
Sediment moisture	glass jar	200 g	Cool $< 6^{\circ}\text{C}$	28 days
Semivolatile Organics (PPOE, BNA)	1L Amber	1000 mL, 10 g	Cool $\leq 6^{\circ}\text{C}$	7 days to extract, 40-days after
Steroids and Hormones	1L Amber	1000 mL, 10 g	Cool $\leq 6^{\circ}\text{C}$	7 days to extract, 40-days after
Sulfate	250mL, 500mL, 1L Poly	50 mL	Cool $\leq 6^{\circ}\text{C}$	28 days
Sulfides	250 mL Poly	100 mL	10 drops 2N Zn Acetate NaOH pH >9 Cool $\leq 6^{\circ}\text{C}$	7 days
Percent Solids (soil/solids)	glass jar	200 g	Cool $< 6^{\circ}\text{C}$	28 days

ANALYTE	CONTAINER ²⁵	MIN. QUANTITY	PRESERVATION	HOLDING TIME ²⁷
Solvents	40 mL VOA	40 mL	No Headspace, Cool \leq 6°C	14 days
Solids(Dissolved, Total, or Suspended)	1L Poly	200 mL ea	Cool \leq 6°C	7 days
TCLP	250mL, 500mL, 1L Poly, 1L Glass or Amber, glass jar	200 mL, 500g	Cool \leq 6°C	7 days to extract 28 days to extract if metals only (6 mos. if Hg not target analyte).
Total Organic Carbon	500mL Poly	10 mL	H2SO4 pH <2, Cool < 6oC	28 days
Total Phosphate	500mL Poly	100 mL	H2SO4 pH <2, Cool < 6oC	28 days
Total Volatile Solids	1L Poly	200 mL	Cool \leq 6°C	7 days
Total Organic Halides	250mL, 500mL Amber	100 mL	H2SO4 pH <2, Cool < 6oC	28 days
Turbidity	1L Poly	50 mL	Cool \leq 6°C	48 hours
Volatile Aromatics	40 mL VOA x 2 L	2 x 40 mL, 5 g	HCL, No Headspace, Cool \leq 6°C	14 days
Volatile Organics (Water)	40 mL VOA x 2	2 x 40 mL	HCL, No Headspace, Cool \leq 6°C ³⁰ No Headspace, Cool \leq 6°C	14 days 3 days - Acrolein 7 days – Aromatics 14 days – Other VOCs
Volatile Organics (Soil)	40 mL VOA w/ Methanol 40 mL VOA	5 g	Cool \leq 6°C Cool \leq 6°C / Freeze - 10°C	14 days 48 hrs / 14 days

³⁰ If 2-CVE, Acrolein, or Acrylonitrile are target analytes a 2nd ‘ DO NOT USE HCL, a separate unacidified sample is required.

APPENDIX J DEQ LABORATORY ANALYTICAL TECHNIQUES and LOQ
INORGANIC ANALYSES

	ANALYTICAL	
ANALYTE	TECHNIQUE	LOQ ³¹ (waters)
Aluminum	ICP	0.050 mg/L
Aluminum	ICP Mass Spectrometry	20.0 µg/L
Antimony	ICP	0.015 mg/L
Antimony	ICP Mass Spectrometry	0.30 µg/L
Arsenic	ICP	0.010 mg/L
Arsenic	ICP Mass Spectrometry	0.25 µg/L
Barium	ICP	0.0020 mg/L
Barium	ICP Mass Spectrometry	2.0 µg/L
Beryllium	ICP	0.00050 mg/L
Beryllium	ICP Mass Spectrometry	0.10 µg/L
Boron	ICP	0.020 mg/L
Boron	ICP Mass Spectrometry	20 µg/L
Cadmium	ICP	0.0050 mg/L
Cadmium	ICP Mass Spectrometry	0.10 µg/L
Calcium	ICP	0.10 mg/L
Calcium	ICP Mass Spectrometry	200 µg/L
Chromium	ICP	0.0020 mg/L
Chromium	ICP Mass Spectrometry	1.0 µg/L
Cobalt	ICP	0.0030 mg/L
Cobalt	ICP Mass Spectrometry	0.20 µg/L
Copper	ICP	0.010 mg/L
Copper	ICP Mass Spectrometry	1.5 µg/L
Lead	ICP	0.010 mg/L
Lead	ICP Mass Spectrometry	0.20 µg/L
Lithium	ICP	0.015 mg/L
Magnesium	ICP	0.10 mg/L
Magnesium	ICP Mass Spectrometry	50.0 µg/L
Manganese	ICP	0.0050 mg/L

³¹ LOQ = Limit of Quantitation

	ANALYTICAL	
ANALYTE	TECHNIQUE	LOQ ³¹ (waters)
Manganese	ICP Mass Spectrometry	2.0 µg/L
Mercury	Manual Cold Vapor	0.000005 mg/L
Molybdenum	ICP	0.004 mg/L
Molybdenum	ICP Mass Spectrometry	3.0 µg/L
Nickel	ICP	0.004 mg/L
Nickel	ICP Mass Spectrometry	1.0 µg/L
Potassium	ICP	0.50 mg/L
Potassium	ICP Mass Spectrometry	200 µg/L
Selenium	ICP	0.010 mg/L
Selenium	ICP Mass Spectrometry	2.0 µg/L
Silver	ICP	0.0020 mg/L
Silver	ICP Mass Spectrometry	0.10 µg/L
Sodium	ICP	0.30 mg/L
Sodium	ICP Mass Spectrometry	100 µg/L
Silica (SiO ₂)	ICP	0.50 mg/L
Silica (SiO ₂)	ICP Mass Spectrometry	0.32 mg/L
Strontium	ICP	0.020 mg/L
Strontium	ICP Mass Spectrometry	1.0 µg/L
Tin	ICP Mass Spectrometry	2.0 µg/L
Titanium	ICP Mass Spectrometry	15 µg/L
Thallium	ICP	0.015 mg/L
Thallium	ICP Mass Spectrometry	0.10 µg/L
Vanadium	ICP	0.0020 mg/L
Vanadium	ICP Mass Spectrometry	4.0 µg/L
Zinc	ICP	0.030 mg/L
Zinc	ICP Mass Spectrometry	5.0µg/L
Uranium	ICP Mass Spectrometry	0.10 µg/L
Alkalinity	Titration	1 mg/L
BOD ₅	Winkler-Azide Modification/ LDO	2 mg/L
Bromide	Ion Chromatography	0.03 mg/L
CBOD ₅	Winkler-Azide Mod./Inhib / LDO	2 mg/L

	ANALYTICAL	
ANALYTE	TECHNIQUE	LOQ ³¹ (waters)
Chloride	Ion Chromatography	0.5 mg/L
Chlorine	Amperometric Titration	0.02 mg/L
Fluoride	Ion Chromatography	0.1 mg/L
Ammonia-Nitrogen	Ion Chromatography	0.01 mg/L
Tot.Kjeldahl Nitrogen	S-Auto Block Digestion	0.2 mg/L
Nitrate-Nitrogen	Auto Cadmium Reduction	0.005 mg/L
Nitrate-Nitrogen	Ion Chromatography	0.05 mg/L
Nitrite-Nitrogen	Auto Colorimetric	0.005 mg/L
Nitrate+Nitrite-Nitrogen	Auto Cadmium Reduction	0.005 mg/L
Ortho Phosphate-P	Colorimetric Ascorbic Acid	0.005 mg/L
Total Phosphate-P	Colorimetric Ascorbic Acid	0.01 mg/L
Sulfate	Ion Chromatography	0.2 mg/L
DISINFECTION BY-PRODUCTS (DBP) (Drinking Water Samples)		
Bromate	Ion Chromatography	5 µg/L
Bromide	Ion Chromatography	30 µg/L
Chlorate	Ion Chromatography	30 µg/L
Chlorite	Ion Chromatography	12 µg/L
INORGANIC PHYSICAL PARAMETERS		
Color	Colorimetric Pd/Co	5 CU ³²
Conductivity	Wheatstone Bridge	1 µmho/cm ²
pH	Electrometric	0.01 SU ³³
RESIDUES (SOLIDS)		
Filterable (TDS)	Gravimetric, 180°C	10 mg/L
Nonfilterable (TSS)	Gravimetric, 103-105°C	1 mg/L
Total (TS)	Gravimetric, 103-105°C	10 mg/L
Volatile (TVS)	Gravimetric, Ignition @ 550°C	10 mg/L
Turbidity	Nephelometric	1 NTU ³⁴

³² CU = Color Units

³³ SU = Standard pH Units

³⁴ NTU = Nephelometric Turbidity Units

	ANALYTICAL	
ANALYTE	TECHNIQUE	LOQ ³¹ (waters)

ORGANIC ANALYSES

	ANALYTICAL	
ANALYTE	TECHNIQUE	LOQ ³⁵ (waters)
Adipates	Solvent Ext, GC/MS	0.001 mg/L
BTEX	GC/MS	0.0005 mg/L
Chlorinated Pesticides	Solvent Ext., GCMS	0.00002 mg /L (20 ng/L)
Chlorinated Herbicides	micro solvent extraction with derivitization, GC	0.0001 – 0.0006 mg/L
Cyanide Amenable to Chlorination (See 4.1.3)	Colorimetric Barbiturate	0.01 mg/L
EDB, DBCP	GC	0.05 µg/L
Flash Point		
Formaldehyde	HPLC/UV	
Hydrocarbon ID (HCID)	GC/FID	0.6 mg/L
Ketones & Aldehydes	HPLC/UV	
Lignin-Tannin		1 mg/L
PCBs	Solvent Ext., GCMS	< 0.001 µg/L
N-P Pesticides	LCMS	< 0.040 µg/L
Hexane Extractable Material (Oil and Grease)	Hexane Extraction, Gravimetric	5 mg/L
Organotins	Non-DEQ analysis	Non-DEQ analysis
PAHs	Solvent Extract, GC/MS / GCMS SIM	0.001 mg/L / 0.1 µg/L
Pentachlorophenol	Semi-Volatile GC/MS	0.0001 mg/L
Phenolics	Colorimetric, Automated 4-AAP with Distillation Not performed by DEQ Lab	0.005 mg/L
Phthalates	Extraction, GC/MS	0.001 mg/L
SemiVols (PPOE, BNA)	Extraction, GC/MS	0.001 mg/L

³⁵ LOQs vary by compound. The limits shown in the table are only a general expectation and do not necessarily reflect all compounds in a group. Contact the laboratory for compound specific reporting limits

	ANALYTICAL	
ANALYTE	TECHNIQUE	LOQ ³⁵ (waters)
Trihalomethanes	Purge & Trap, GC/MS	0.0005 mg/L
Volatile Organic Compunds (VOCs)	Purge & Trap, GC/MS	0.0005 mg/L
COD	Spectrophotometric, Cr	5 mg/L
TOC	Wet Oxidation, IR Not currently performed by DEQ Lab	1 mg/L
TOX	Pyrolysis, μ coulometric titration Not performed by DEQ Lab	0.005 mg/L
UST/LUST SOIL CLEANUP [OAR 340-122- (0205-0360)		
TPH HCID	Capillary GC/FID	
TPH-Gx	Purge & Trap GC/FID	0.05 mg/Kg Gasoline
TPH-Dx	Sonic Extr., GC/FID	20 mg/Kg Diesel 50 mg/Kg Oil

APPENDIX K METHOD REFERENCES BY ANALYTE AND ANALYTE GROUP

Parameter	Method Reference <u>Non-Potable</u> Water	Method Reference <u>Solids and Tissue</u>	Method Reference <u>Potable</u> Water (SDWA)
Inorganics-Metals			
Aluminum	EPA 200.7 / 6010 C	EPA 6010 C	EPA 200.7
Aluminum	EPA 200.8 / 6020 A	EPA 6020 A	EPA 200.8
Antimony	EPA 200.7 / 6010 C	EPA 6010 C	
Antimony	EPA 200.8 / 6020 A	EPA 6020 A	EPA 200.8
Antimony	EPA 200.9	EPA 7010	EPA 200.9
Arsenic	EPA 200.7 / 6010 C	EPA 6010 C	EPA 200.7
Arsenic	EPA 200.8 / 6020 A	EPA 6020 A	EPA 200.8
Barium	EPA 200.7 / 6010 C	EPA 6010 C	EPA 200.7
Barium	EPA 200.8 / 6020 A	EPA 6020 A	EPA 200.8
Beryllium	EPA 200.7 / 6010 C	EPA 6010 C	EPA 200.7
Beryllium	EPA 200.8 / 6020 A	EPA 6020 A	EPA 200.8
Boron	EPA 200.7 / 6010 C	EPA 6010 C	EPA 200.7
Boron	EPA 200.8 / 6020 A	EPA 6020 A	EPA 200.8
Cadmium	EPA 200.7 / 6010 C	EPA 6010 C	EPA 200.7
Cadmium	EPA 200.8 / 6020 A	EPA 6020 A	EPA 200.8
Calcium	EPA 200.7 / 6010 C	EPA 6010 C	EPA 200.7
Calcium	EPA 200.8 / 6020 A	EPA 6020 A	
Chromium (total)	EPA 200.7 / 6010 C	EPA 6010 C	EPA 200.7
Chromium (total)	EPA 200.8 / 6020 A	EPA 6020 A	EPA 200.8
Cobalt	EPA 200.7 / 6010 C	EPA 6010 C	EPA 200.7
Cobalt	EPA 200.8 / 6020 A	EPA 6020 A	EPA 200.8
Copper	EPA 200.7 / 6010 C	EPA 6010 C	EPA 200.7
Copper	EPA 200.8 / 6020 A	EPA 6020 A	EPA 200.8
Iron	EPA 200.7 / 6010 C	EPA 6010 C	EPA 200.7
Iron	EPA 200.8 / 6020 A	EPA 6020 A	EPA 200.8
Lead	EPA 200.7 / 6010 C	EPA 6010 C	
Lead	EPA 200.8 / 6020 A	EPA 6020 A	EPA 200.8
Lead	EPA 200.9	EPA 7010	EPA 200.9
Magnesium	EPA 200.7 / 6010 C	EPA 6010 C	EPA 200.7

Parameter	Method Reference Non-Potable Water	Method Reference Solids and Tissue	Method Reference Potable Water (SDWA)
Magnesium	EPA 200.8 / 6020 A	EPA 6020 A	
Manganese	EPA 200.7 / 6010 C	EPA 6010 C	EPA 200.7
Manganese	EPA 200.8 / 6020 A	EPA 6020 A	EPA 200.8
Mercury (inorganic)	EPA 245.1 / 7470A	EPA 7473	EPA 245.1
Molybdenum	EPA 200.7 / 6010 C	EPA 6010 C	EPA 200.7
Molybdenum	EPA 200.8 / 6020 A	EPA 6020 A	EPA 200.8
Nickel	EPA 200.7 / 6010 C	EPA 6010 C	EPA 200.7
Nickel	EPA 200.8 / 6020 A	EPA 6020 A	EPA 200.8
Potassium	EPA 200.7 / 6010 C	EPA 6010 C	EPA 200.7
Selenium	EPA 200.7 / 6010 C	EPA 6010 C	
Selenium	EPA 200.8 / 6020 A	EPA 6020 A	EPA 200.8
Selenium	EPA 200.9	EPA 7010	EPA 200.9
Silica	EPA 200.7 / 6010 C	EPA 6010 C	EPA 200.7
Silica	EPA 200.8 / 6020 A	EPA 6020 A	
Silver	EPA 200.7 / 6010 C	EPA 6010 C	EPA 200.7
Silver	EPA 200.8 / 6020 A	EPA 6020 A	EPA 200.8
Sodium	EPA 200.7 / 6010 C	EPA 6010 C	EPA 200.7
Strontium	EPA 200.7 / 6010 C	EPA 6010 C	EPA 200.7
Strontium	EPA 200.8 / 6020 A	EPA 6020 A	EPA 200.8
Thallium	EPA 200.7 / 6010 C	EPA 6010 C	
Thallium	EPA 200.8 / 6020 A	EPA 6020 A	EPA 200.8
Thallium	EPA 200.9	EPA 7010	EPA 200.9
Tin	EPA 200.7 / 6010 C	EPA 6010 C	EPA 200.7
Tin	EPA 200.8 / 6020 A	EPA 6020 A	EPA 200.8
Titanium	EPA 200.7 / 6010 C	EPA 6010 C	EPA 200.7
Titanium	EPA 200.8 / 6020 A	EPA 6020 A	EPA 200.8
Vanadium	EPA 200.7 / 6010 C	EPA 6010 C	EPA 200.7
Vanadium	EPA 200.8 / 6020 A	EPA 6020 A	EPA 200.8
Zinc	EPA 200.7 / 6010 C	EPA 6010 C	EPA 200.7
Zinc	EPA 200.8 / 6020 A	EPA 6020 A	EPA 200.8
Uranium	EPA 200.8 / 6020 A	EPA 6020 A	EPA 200.8

Inorganics-General Chemistry

Parameter	Method Reference Non-Potable Water	Method Reference Solids and Tissue	Method Reference Potable Water (SDWA)
Alkalinity	SM 2320 B		SM 2320 B
Ammonia	SM 4500-NH3 H		
Ammonia	SM 4500-NH3 G		
BOD	SM 5210 B/4500-O C		
Bromide	EPA 300.0/300.1	EPA 9056	EPA 300.0/300.1
Calcium Hardness	SM 2340 B		
CBOD	SM 5210 B		
Chloride	EPA 300.0/300.1	EPA 9056	EPA 300.0/300.1
Chlorophyll / Pheophyton	10200-H		
COD	SM 5220 D		
Color	SM 2120 B		SM 2120 B
Conductivity / Specific Conductance	SM 2510 B		SM 2510 B
Cyanide, total (see 4.1.3)	SM 4500-CN E / EPA 9014	EPA 9010 B/ 9014	SM 4500-CN E
Cyanide, Amenable to Chlorination (see 4.1.3)	SM 4500-CN G, E	EPA 9010 B/ 9014	SM 4500-CN G, E
Fluoride	EPA 300.0/300.1	EPA 9056	EPA 300.0/300.1
Fluoride	SM 4500-F E		SM 4500-F E
Nitrate (measured as Nitrogen)	EPA 300.0/300.1	EPA 9056	EPA 300.0/300.1
Nitrate (measured as Nitrogen)	SM 4500-NO3 F/EPA 353.2		SM 4500-NO3 F/EPA 353.2
Nitrate+Nitrite (measured as Nitrogen)	SM 4500-NO3 F/EPA 353.2		SM 4500-NO3 F/EPA 353.2
Nitrite (measured as Nitrogen)	SM 4500-NO2 B/EPA 353.2		SM 4500-NO2 B/EPA 353.2
Oil & Grease	EPA 1664	EPA 9071	
Orthophosphate as P	SM 4500-P E	EPA 9056	SM 4500-P E
pH	EPA 4500H+B	EPA 9040C / 9045D	EPA 4500H+B
Phenolics, total	EPA 420.1	EPA 9065	
Settleable Solids	SM 2540 F		
Sulfate	EPA 300.0	EPA 9056	EPA 300.0/300.1

Parameter	Method Reference Non-Potable Water	Method Reference Solids and Tissue	Method Reference Potable Water (SDWA)
Total Dissolved Solids	SM 2540 C		SM 2540 C
Total Hardness	SM 2340 B		SM 2340 B
Total Kjeldahl Nitrogen	SM 4500 Norg D/EPA 351.2		
Total or Dissolved Organic Carbon	SM 5310 B / EPA 9060		SM 5310 B
Total Phosphorus as P	SM 4500-P E		
Total Residual Chlorine	SM 4500-CL D		SM 4500-CL E
Total Solids	SM 2540 B	SM 2540 G	SM 2540 B
Total Suspended Solids	SM 2540 D		SM 2540 D
Turbidity	SM 2130 B		SM 2130 B
Chlorine (as Cl ₂)			SM 4500-CL D
Residual Free Chlorine			SM 4500-CL G
Inorganic Disinfection Byproducts			
Bromate			EPA 300.1
Bromide			EPA 300.1
Chlorate			EPA 300.1
Chlorite			EPA 300.1
Herbicides			
2,4,5-TP (Silvex)	SM 6640B	SM 6640B	EPA 515.4
2,4,5-Trichlorophenoxyacetic acid	SM 6640B	SM 6640B	EPA 515.4
2,4-D	SM 6640B	SM 6640B	EPA 515.4
Dicamba	SM 6640B	SM 6640B	10.1.1. EPA 515.4
Dacthal (DCPA)	SM 6640B	SM 6640B	EPA 515.4
2,4-DP (Dichlorprop)	SM 6640B	SM 6640B	EPA 515.4
Dinoseb	SM 6640B	SM 6640B	EPA 515.4
MCPA	SM 6640B	SM 6640B	
MCPP	SM 6640B	SM 6640B	

Parameter	Method Reference Non-Potable Water	Method Reference Solids and Tissue	Method Reference Potable Water (SDWA)
Pentachlorophenol	SM 6640B	SM 6640B	EPA 515.4
Picloram	SM 6640B	SM 6640B	EPA 515.4
2,4-DB			EPA 515.4
3,5-Dichloro benzoic acid			EPA 515.4
Acifluorfen			EPA 515.4
Polychlorinated biphenyls (PCBs)			
Aroclor 1016/1242	EPA 8082/608	EPA 8082	EPA 508 / 508A
Aroclor 1016	EPA 8082/608	EPA 8082	EPA 508 / 508A
Aroclor 1221	EPA 8082/608	EPA 8082	EPA 508 / 508A
Aroclor 1232	EPA 8082/608	EPA 8082	EPA 508 / 508A
Aroclor 1242	EPA 8082/608	EPA 8082	EPA 508 / 508A
Aroclor 1248	EPA 8082/608	EPA 8082	EPA 508 / 508A
Aroclor 1254	EPA 8082/608	EPA 8082	EPA 508 / 508A
Aroclor 1260	EPA 8082/608	EPA 8082	EPA 508 / 508A
Volatile Organics			
1,1,1,2-Tetrachlorethane	EPA 8260C/624	EPA 8260C	EPA 524.2
1,1,1-Trichloroethane	EPA 8260C/624	EPA 8260C	EPA 524.2
1,1,2,2-Tetrachloroethane	EPA 8260C/624	EPA 8260C	EPA 524.2
1,1,2-Trichloroethane	EPA 8260C/624	EPA 8260C	EPA 524.2
1,1-Dichloroethane	EPA 8260C/624	EPA 8260C	EPA 524.2
1,1-Dichloroethylene	EPA 8260C/624	EPA 8260C	EPA 524.2
1,1-Dichloropropene	EPA 8260C/624	EPA 8260C	EPA 524.2
1,2,3-Trichlorobezene	EPA 8260C/624	EPA 8260C	EPA 524.2
1,2,3-Trichloropropane (TCP)	EPA 8260C/624	EPA 8260C	EPA 504.1
1,2,4-Trichlorobenzene	EPA 8260C/624	EPA 8260C	EPA 524.2
1,2,4-Trimethylbenzene	EPA 8260C/624	EPA 8260C	EPA 524.2
1,2-Dibromo-3-chloropropane (DBCP)	EPA 8260C/624	EPA 8260C	EPA 504.1
1,2-Dibromomethane (Ethylene dibromide)	EPA 8260C/624	EPA 8260C	EPA 504.1

Parameter	Method Reference Non-Potable Water	Method Reference Solids and Tissue	Method Reference Potable Water (SDWA)
1,2-Dichlorobenzene	EPA 8260C/624	EPA 8260C	EPA 524.2
1,2-Dichloroethane	EPA 8260C/624	EPA 8260C	EPA 524.2
1,2-Dichloropropane	EPA 8260C/624	EPA 8260C	EPA 524.2
1,2-Dimethylbenzene	EPA 8260C/624	EPA 8260C	
1,3-Dichlorobenzene	EPA 8260C/624	EPA 8260C	EPA 524.2
1,3-Dichloropropane	EPA 8260C/624	EPA 8260C	EPA 524.2
1,3,5-Trimethylbenzene	EPA 8260C/624	EPA 8260C	EPA 524.2
1,4-Dichlorobenzene	EPA 8260C/624	EPA 8260C	EPA 524.2
1,4/1,3-Dimethylbenzene	EPA 8260C/624	EPA 8260C	
2,2-Dichloropropane	EPA 8260C/624	EPA 8260C	EPA 524.2
2-Butanone (MEK)	EPA 8260C/624	EPA 8260C	
2-Chloroethylvinyl ether	Special Request	Special Request	
2-Chlorotoluene	EPA 8260C/624	EPA 8260C	EPA 524.2
2-Hexanone	EPA 8260C/624	EPA 8260C	
4-Chlorotoluene	EPA 8260C/624	EPA 8260C	EPA 524.2
4-Isopropyltoluene	EPA 8260C/624	EPA 8260C	EPA 524.2
Acetone	EPA 8260C/624	EPA 8260C	
Acrolein	Special Request	Special Request	
Benzene	EPA 8260C/624	EPA 8260C	EPA 524.2
Bromobenzene	EPA 8260C/624	EPA 8260C	EPA 524.2
Bromochloromethane	EPA 8260C/624	EPA 8260C	EPA 524.2
Bromodichloromethane	EPA 8260C/624	EPA 8260C	EPA 524.2
Bromoform	EPA 8260C/624	EPA 8260C	EPA 524.2
Bromomethane	EPA 8260C/624	EPA 8260C	EPA 524.2
n-Butylbenzene	EPA 8260C/624	EPA 8260C	EPA 524.2
sec-Butylbenzene	EPA 8260C/624	EPA 8260C	EPA 524.2
tert-Butylbenzene	EPA 8260C/624	EPA 8260C	EPA 524.2
Carbon disulfide	EPA 8260C/624	EPA 8260C	
Carbon tetrachloride	EPA 8260C/624	EPA 8260C	EPA 524.2
Chlorobenzene	EPA 8260C/624	EPA 8260C	EPA 524.2
Chlorodibromomethane	EPA 8260C/624	EPA 8260C	EPA 524.2
Chloroethane	EPA 8260C/624	EPA 8260C	EPA 524.2

Parameter	Method Reference Non-Potable Water	Method Reference Solids and Tissue	Method Reference Potable Water (SDWA)
Chloroform	EPA 8260C/624	EPA 8260C	EPA 524.2
Chloromethane	EPA 8260C/624	EPA 8260C	EPA 524.2
cis-1,2-Dichloroethylene	EPA 8260C/624	EPA 8260C	EPA 524.2
cis-1,3-Dichloropropene	EPA 8260C/624	EPA 8260C	EPA 524.2
Dibromochloromethane	EPA 8260C/624	EPA 8260C	EPA 524.2
Dibromomethane	EPA 8260C/624	EPA 8260C	EPA 524.2
Dichlorodifluoromethane	EPA 8260C/624	EPA 8260C	EPA 524.2
Dichloromethane (Methylene chloride)	EPA 8260C/624	EPA 8260C	EPA 524.2
Ethylbenzene	EPA 8260C/624	EPA 8260C	EPA 524.2
Hexachlorobutadiene	EPA 8260C/624	EPA 8260C	EPA 524.2
Isopropylbenzene (Cumene)	EPA 8260C/624	EPA 8260C	EPA 524.2
Methyl-isobutyl ketone (MIBK)	EPA 8260C/624	EPA 8260C	
Methy tert-butyl ether (MTBE)	EPA 8260C/624	EPA 8260C	EPA 524.2
Naphthalene	EPA 8260C/624	EPA 8260C	EPA 524.2
n-Propylbenzene	EPA 8260C/624	EPA 8260C	EPA 524.2
Styrene	EPA 8260C/624	EPA 8260C	EPA 524.2
Tetrachloroethylene	EPA 8260C/624	EPA 8260C	EPA 524.2
Toluene	EPA 8260C/624	EPA 8260C	EPA 524.2
trans-1,2-Dichloroethylene	EPA 8260C/624	EPA 8260C	EPA 524.2
trans-1,3-Dichloropropene	EPA 8260C/624	EPA 8260C	EPA 524.2
Trichloroethylene	EPA 8260C/624	EPA 8260C	EPA 524.2
Trichlorofluoromethane	EPA 8260C/624	EPA 8260C	EPA 524.2
Vinyl chloride	EPA 8260C/624	EPA 8260C	EPA 524.2
Xylenes (total) m&p-Xylenes + o-xylene	EPA 8260C/624	EPA 8260C	EPA 524.2
o-Xylene		EPA 8260C	EPA 524.2
m&p-Xylenes		EPA 8260C	EPA 524.2
Semivolatile Organics - Base Neutrals			

Parameter	Method Reference Non-Potable Water	Method Reference Solids and Tissue	Method Reference Potable Water (SDWA)
1,2,4-Trichlorobenzene	EPA 8270D/625	EPA 8270D	
1,2-Dichlorobenzene	EPA 8270D/625	EPA 8270D	
1,3-Dichlorobenzene	EPA 8270D/625	EPA 8270D	
1,4-Dichlorobenzene	EPA 8270D/625	EPA 8270D	
2,4-Dinitrotoluene	EPA 8270D/625	EPA 8270D	
2,6-Dinitrotoluene	EPA 8270D/625	EPA 8270D	
2-Chloronaphthalene	EPA 8270D/625	EPA 8270D	
3,3'-Dichlorobenzidine	EPA 8270D/625	EPA 8270D	
4-Bromophenyl-phenylether	EPA 8270D/625	EPA 8270D	
4-Chlorophenyl-phenylether	EPA 8270D/625	EPA 8270D	
Acenaphthene	EPA 8270D/625	EPA 8270D	
Acenaphthylene	EPA 8270D/625	EPA 8270D	EPA 525.2
Anthracene	EPA 8270D/625	EPA 8270D	EPA 525.2
Benzo(a)anthracene	EPA 8270D/625	EPA 8270D	EPA 525.2
Benzo(a)pyrene	EPA 8270D/625	EPA 8270D	EPA 525.2
Benzo(b)fluoranthene	EPA 8270D/625	EPA 8270D	EPA 525.2
Benzo(g,h,i)perylene	EPA 8270D/625	EPA 8270D	EPA 525.2
Benzo(k)fluoranthene	EPA 8270D/625	EPA 8270D	EPA 525.2
Bis(2-chloroethoxy)methane	EPA 8270D/625	EPA 8270D	
Bis(2-chloroethyl)ether	EPA 8270D/625	EPA 8270D	
Bis(2-chloroisopropyl)ether	EPA 8270D/625	EPA 8270D	
Bis(2-ethylhexyl) phthalate	EPA 8270D/625	EPA 8270D	EPA 525.2
Bis(2-ethylhexyl)adipate			EPA 525.2
Butylbenzyl phthalate	EPA 8270D/625	EPA 8270D	EPA 525.2
Chrysene	EPA 8270D/625	EPA 8270D	EPA 525.2
Dibenz(a,h)anthracene	EPA 8270D/625	EPA 8270D	EPA 525.2
Dibenzofuran	EPA 8270D/625	EPA 8270D	
Diethyl phthalate	EPA 8270D/625	EPA 8270D	EPA 525.2
Dimethyl phthalate	EPA 8270D/625	EPA 8270D	EPA 525.2
Di-n-butyl phthalate	EPA 8270D/625	EPA 8270D	EPA 525.2
Di-n-octyl phthalate	EPA 8270D/625	EPA 8270D	
Fluorene	EPA 8270D/625	EPA 8270D	EPA 525.2

Parameter	Method Reference Non-Potable Water	Method Reference Solids and Tissue	Method Reference Potable Water (SDWA)
Fluoranthene	EPA 8270D/625	EPA 8270D	
Hexachlorobenzene	EPA 8270D/625	EPA 8270D	EPA 525.2 / EPA 508
Hexachlorobutadiene	EPA 8270D/625	EPA 8270D	
Hexachlorocyclopentadiene	EPA 8270D/625	EPA 8270D	
Hexachloroethane	EPA 8270D/625	EPA 8270D	
Indeno(1,2,3-c,d)pyrene	EPA 8270D/625	EPA 8270D	EPA 525.2
Isophorone	EPA 8270D/625	EPA 8270D	
Naphthalene	EPA 8270D/625	EPA 8270D	
Nitrobenzene	EPA 8270D/625	EPA 8270D	
N-Nitrosodiethylamine	EPA 8270D/625	EPA 8270D	
N-Nitrosodimethylamine	EPA 8270D/625	EPA 8270D	
N-Nitroso-di-n-propylamine	EPA 8270D/625	EPA 8270D	
N-Nitrosodiphenylamine	EPA 8270D/625	EPA 8270D	
Phenanthrene	EPA 8270D/625	EPA 8270D	EPA 525.2
Pyrene	EPA 8270D/625	EPA 8270D	EPA 525.2
Semivolatile Organics- Acids			
2,4,5-Trichlorophenol	EPA 8270D/625	EPA 8270D	
2,4,6-Trichlorophenol	EPA 8270D/625	EPA 8270D	
2,4-Dichlorophenol	EPA 8270D/625	EPA 8270D	
2,4-Dimethylphenol	EPA 8270D/625	EPA 8270D	
2,4-Dinitrophenol	EPA 8270D/625	EPA 8270D	
2,6-Dichlorophenol	EPA 8270D/625	EPA 8270D	
2-Chlorophenol	EPA 8270D/625	EPA 8270D	
2-Methylphenol	EPA 8270D/625	EPA 8270D	
2-Nitrophenol	EPA 8270D/625	EPA 8270D	
4,6-Dinitro-2-methylphenol	EPA 8270D/625	EPA 8270D	
4-Chloro-3-methylphenol	EPA 8270D/625	EPA 8270D	
4-Methylphenol	EPA 8270D/625	EPA 8270D	
Pentachlorophenol	EPA 8270D/625	EPA 8270D	
Phenol	EPA 8270D/625	EPA 8270D	

Parameter	Method Reference Non-Potable Water	Method Reference Solids and Tissue	Method Reference Potable Water (SDWA)
Chlorinated Pesticides			
4,4'-DDD	EPA 8081B/608	EPA 8081B	
	EPA 8270D/625	EPA 8270D	
4,4'-DDE	EPA 8081B/608	EPA 8081B	
	EPA 8270D/625	EPA 8270D	
4,4'-DDT	EPA 8081B/608	EPA 8081B	
	EPA 8270D/625	EPA 8270D	
Aldrin	EPA 8081B/608	EPA 8081B	EPA 508
	EPA 8270D/625	EPA 8270D	EPA 525.2
alpha-BHC	EPA 8081B/608	EPA 8081B	
	EPA 8270D/625	EPA 8270D	
alpha-Chlordane	EPA 8081B/608	EPA 8081B	
beta-BHC	EPA 8081B/608	EPA 8081B	
	EPA 8270D/625	EPA 8270D	
Chlordane, technical	EPA 8081B/608	EPA 8081B	EPA 508
	EPA 8270D/625	EPA 8270D	
delta-BHC	EPA 8081B/608	EPA 8081B	
	EPA 8270D/625	EPA 8270D	
Dieldrin	EPA 8081B/608	EPA 8081B	EPA 508
	EPA 8270D/625	EPA 8270D	EPA 525.2
Endosulfan I	EPA 8081B/608	EPA 8081B	
	EPA 8270D/625	EPA 8270D	
Endosulfan II	EPA 8081B/608	EPA 8081B	
	EPA 8270D/625	EPA 8270D	
Endosulfan sulfate	EPA 8081B/608	EPA 8081B	
	EPA 8270D/625	EPA 8270D	
Endrin	EPA 8081B/608	EPA 8081B	EPA 508
	EPA 8270D/625	EPA 8270D	EPA 525.2
Endrin Aldehyde	EPA 8081B/608	EPA 8081B	
Endrin ketone	EPA 8081B/608	EPA 8081B	
gamma-BHC (Lindane)	EPA 8081B/608	EPA 8081B	EPA 508

Parameter	Method Reference Non-Potable Water	Method Reference Solids and Tissue	Method Reference Potable Water (SDWA)
	EPA 8270D/625	EPA 8270D	EPA 525.2
gamma-Chlordane	EPA 8081B/608	EPA 8081B	
Heptachlor	EPA 8081B/608	EPA 8081B	EPA 508
	EPA 8270D/625	EPA 8270D	EPA 525.2
Heptachlor epoxide (beta)	EPA 8081B/608	EPA 8081B	EPA 508
	EPA 8270D/625	EPA 8270D	
Methoxychlor	EPA 8081B/608	EPA 8081B	EPA 508
			EPA 525.2
Toxaphene	EPA 8081B/608	EPA 8081B	EPA 508
PAHs			
Acenaphthene	EPA 8270 SIM	EPA 8270 SIM	
Acenaphthylene	EPA 8270 SIM	EPA 8270 SIM	
Anthracene	EPA 8270 SIM	EPA 8270 SIM	
Benzo(a)anthracene	EPA 8270 SIM	EPA 8270 SIM	
Benzo(a)pyrene	EPA 8270 SIM	EPA 8270 SIM	
Benzo(b)fluoranthene	EPA 8270 SIM	EPA 8270 SIM	
Benzo(g,h,i)perylene	EPA 8270 SIM	EPA 8270 SIM	
Benzo(k)fluoranthene	EPA 8270 SIM	EPA 8270 SIM	
Chrysene	EPA 8270 SIM	EPA 8270 SIM	
Dibenz(a,h)anthracene	EPA 8270 SIM	EPA 8270 SIM	
Fluorene	EPA 8270 SIM	EPA 8270 SIM	
Fluoroanthene	EPA 8270 SIM	EPA 8270 SIM	
Indeno(1,2,3-c,d)pyrene	EPA 8270 SIM	EPA 8270 SIM	
Naphthalene	EPA 8270 SIM	EPA 8270 SIM	
Phenanthrene	EPA 8270 SIM	EPA 8270 SIM	
Pyrene	EPA 8270 SIM	EPA 8270 SIM	
Organophosphorus Pesticides (OPP)			
Azinphos-methyl (Guthion)	EPA 8270D	EPA 8270D	
Chlorpyrifos	EPA 8270D	EPA 8270D	
Diazinon	EPA 8270D	EPA 8270D	EPA 525.2

Parameter	Method Reference Non-Potable Water	Method Reference Solids and Tissue	Method Reference Potable Water (SDWA)
Dichlorvos (DDVP)	EPA 8270D	EPA 8270D	
Dimethoate	EPA 8270D	EPA 8270D	
Disulfoton	EPA 8270D	EPA 8270D	
Ethoprop	EPA 8270D	EPA 8270D	
Ethyl Parathion	EPA 8270D	EPA 8270D	
Malathion	EPA 8270D	EPA 8270D	
Methyl Parathion	EPA 8270D	EPA 8270D	
Phosmet	EPA 8270D	EPA 8270D	
Stirophos	EPA 8270D	EPA 8270D	
Terbufos	EPA 8270D	EPA 8270D	

Carbamate Pesticides (EPA 8321M / DEQ11-LAB-0031-SOP)			
Baygon	EPA 8321M	EPA 8321M	
Bromacil	EPA 8321M	EPA 8321M	EPA 525.2
Carbaryl	EPA 8321M	EPA 8321M	
Carbofuran	EPA 8321M	EPA 8321M	
Diuron	EPA 8321M	EPA 8321M	
Methiocarb	EPA 8321M	EPA 8321M	
Methomyl	EPA 8321M	EPA 8321M	
Molinate	EPA 8321M	EPA 8321M	EPA 525.2
Oxamyl (vydate)	EPA 8321M	EPA 8321M	

Nitrogen Pesticides (EPA 8321M or EPA 8270D)			
Alachlor	EPA 8321M / EPA 8270D	EPA 8321M / EPA 8270D	EPA 525.2
Ametryn	EPA 8321	EPA 8321	
Atrazine	EPA 8321M / EPA 8270D	EPA 8321M / EPA 8270D	EPA 525.2
Butachlor	EPA 8270D	EPA 8270D	EPA 525.2 / EPA 508.1
Butylate	EPA 8270D	EPA 8270D	
Cyanazine	EPA 8270D	EPA 8270D	

Parameter	Method Reference Non-Potable Water	Method Reference Solids and Tissue	Method Reference Potable Water (SDWA)
EPTC (Eptam)	EPA 8270D	EPA 8270D	
Hexazinone	EPA 8270D	EPA 8270D	
Metolachlor	EPA 8321M / EPA 8270D	EPA 8321M / EPA 8270D	EPA 525.2 / EPA 508.1
Metribuzin	EPA 8270D	EPA 8270D	EPA 508.1
Napropamide	EPA 8321M / EPA 8270D	EPA 8321M / EPA 8270D	
Prometon	EPA 8321M	EPA 8321M	EPA 525.2
Prometryn	EPA 8321M	EPA 8321M	
Pronamide	EPA 8270D	EPA 8270D	
Propachlor	EPA 8270D	EPA 8270D	EPA 525.2 / EPA 508
Propazine	EPA 8321M / EPA 8270D	EPA 8321 / EPA 8270D	
Simazine	EPA 8321M / EPA 8270D	EPA 8321M / EPA 8270D	EPA 525.2
Terbacil	EPA 8270D	EPA 8270D	
Trifluralin	EPA 8270D	EPA 8270D	EPA 525.2 / EPA 508
Organic Disinfection By Products Haloacetic Acids (HAA)			
Bromochloroacetic acid			EPA 552.2
Dibromoacetic acid			EPA 552.2
Dichloroacetic acid			EPA 552.2
Monobromoacetic-acid			EPA 552.2
Monochloroacetic acid			EPA 552.2
Trichloroacetic acid			EPA 552.2

Steroids and Hormones			
17a-Estradiol	EPA 1698	EPA 1698	
17a-Ethynyl Estradiol	EPA 1698	EPA 1698	
17β-Estradiol	EPA 1698	EPA 1698	
Cholesterol	EPA 1698	EPA 1698	
Coprostanol	EPA 1698	EPA 1698	
Estriol	EPA 1698	EPA 1698	

Parameter	Method Reference Non-Potable Water	Method Reference Solids and Tissue	Method Reference Potable Water (SDWA)
Estrone	EPA 1698	EPA 1698	

Pharmaceuticals and Personal Care Products (PCPs)

Acetaminophen	EPA 1694	EPA 1694	
Codeine	EPA 1694	EPA 1694	
Caffeine	EPA 1694	EPA 1694	
Sulfamethoxazole	EPA 1694	EPA 1694	
Venlafaxine	EPA 1694	EPA 1694	
Diphenhydramine	EPA 1694	EPA 1694	
Carbamazepine	EPA 1694	EPA 1694	

Polybrominated Diphenyl Ethers (PBDE) - Flame Retardants

PBDE-17	EPA 8270D	EPA 8270D	
PBDE 28	EPA 8270D	EPA 8270D	
PBDE 47	EPA 8270D	EPA 8270D	
PBDE 66	EPA 8270D	EPA 8270D	
PBDE-85	EPA 8270D	EPA 8270D	
PBDE-99	EPA 8270D	EPA 8270D	
PBDE 100	EPA 8270D	EPA 8270D	
PBDE 138	EPA 8270D	EPA 8270D	
PBDE 153	EPA 8270D	EPA 8270D	
PBDE 154	EPA 8270D	EPA 8270D	
PBDE 183	EPA 8270D	EPA 8270D	

PCB Congeners

BZ-8	EPA 8270D	EPA 8270D	
BZ-18	EPA 8270D	EPA 8270D	
BZ-28	EPA 8270D	EPA 8270D	
BZ-44	EPA 8270D	EPA 8270D	
BZ-52	EPA 8270D	EPA 8270D	
BZ-66	EPA 8270D	EPA 8270D	
BZ-77	EPA 8270D	EPA 8270D	

Parameter	Method Reference Non-Potable Water	Method Reference Solids and Tissue	Method Reference Potable Water (SDWA)
BZ-101	EPA 8270D	EPA 8270D	
BZ-105	EPA 8270D	EPA 8270D	
BZ-110	EPA 8270D	EPA 8270D	
BZ-118	EPA 8270D	EPA 8270D	
BZ-126	EPA 8270D	EPA 8270D	
BZ-128	EPA 8270D	EPA 8270D	
BZ-138	EPA 8270D	EPA 8270D	
BZ-153	EPA 8270D	EPA 8270D	
BZ-170	EPA 8270D	EPA 8270D	
BZ-180	EPA 8270D	EPA 8270D	
BZ-187	EPA 8270D	EPA 8270D	
BZ-195	EPA 8270D	EPA 8270D	
BZ-206	EPA 8270D	EPA 8270D	
BZ-209	EPA 8270D	EPA 8270D	

Explosives by HPLC			
1,3,5-Trinitrobenzene	EPA 8330A	EPA 8330A	
1,3-Dinitrobenzene	EPA 8330A	EPA 8330A	
2,4,6-Trinitrotoluene	EPA 8330A	EPA 8330A	
2,4-Dinitrotoluene	EPA 8330A	EPA 8330A	
2,6-Dinitrotoluene	EPA 8330A	EPA 8330A	
2-Amino-4,6-dinitrotoluene	EPA 8330A	EPA 8330A	
2-Nitrotoluene	EPA 8330A	EPA 8330A	
3-Nitrotoluene	EPA 8330A	EPA 8330A	
4-Amino-2,6-dinitrotoluene	EPA 8330A	EPA 8330A	
4-Nitrotoluene	EPA 8330A	EPA 8330A	
Hexahydro-1,3,5-trinitro- 1,3,5-triazine	EPA 8330A	EPA 8330A	
Methyl-,2,4,6- trinitrophenylnitramine (Tetryl)	EPA 8330A	EPA 8330A	
Nitrobenzene	EPA 8330A	EPA 8330A	

Parameter	Method Reference Non-Potable Water	Method Reference Solids and Tissue	Method Reference Potable Water (SDWA)
Octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine	EPA 8330A	EPA 8330A	

Formaldehyde			
Formaldehyde	EPA 8315	EPA 8315	
Formaldehyde	Hantzch Method	Titration (Product only)	

Fuels			
Gasoline Range Organics	NWTPH-Gx	NWTPH-Gx	
Diesel Range Organics	NWTPH-DX	NWTPH-DX	
Oil Range Organics	NWTPH-DX	NWTPH-DX	
Hydrocarbon ID	NWTPH-HCID	NWTPH-HCID	
Hexane Extractable Material	EPA 1664	EPA 1664	

Aldehydes and Ketones in Air by EPA TO-11			
2,5-Dimethylbenzaldehyde	NA	NA	NA
2-Butanone (MEK)	NA	NA	NA
Acetaldehyde	NA	NA	NA
Acetone	NA	NA	NA
Benzaldehyde	NA	NA	NA
Butyraldehyde	NA	NA	NA
Crotonaldehyde (2-Butenal, (E))	NA	NA	NA
Formaldehyde	NA	NA	NA
Hexaldehyde	NA	NA	NA
Isovaleraldehyde	NA	NA	NA
m-Tolualdehyde	NA	NA	NA
o-Tolualdehyde	NA	NA	NA
Propionaldehyde	NA	NA	NA
p-Tolualdehyde	NA	NA	NA

Parameter	Method Reference Non-Potable Water	Method Reference Solids and Tissue	Method Reference Potable Water (SDWA)
Valeraldehyde	NA	NA	NA

Semivolatile Organics in Air by EPA TO-13			
Acenaphthene	NA	NA	NA
Acenaphthylene	NA	NA	NA
Anthracene	NA	NA	NA
Benzo[a]anthracene	NA	NA	NA
Benzo[a]pyrene	NA	NA	NA
Benzo[b]fluoranthene	NA	NA	NA
Benzo[e]pyrene	NA	NA	NA
Benzo[g,h,i]perylene	NA	NA	NA
Benzo[k]fluoranthene	NA	NA	NA
Chrysene	NA	NA	NA
Coronene	NA	NA	NA
Dibenz[a,h]anthracene	NA	NA	NA
Dibenzofuran	NA	NA	NA
Dibenzothiophene	NA	NA	NA
Fluoranthene	NA	NA	NA
Fluorene	NA	NA	NA
Indeno[1,2,3-cd]pyrene	NA	NA	NA
Naphthalene	NA	NA	NA
Perylene	NA	NA	NA
Phenanthrene	NA	NA	NA
Pyrene	NA	NA	NA

Volatile Organics in Air by EPA TO-15			
1,1,1-Trichloroethane	NA	NA	NA
1,1,2,2-Tetrachloroethane	NA	NA	NA
1,1,2-Trichloroethane	NA	NA	NA
1,1-Dichloroethane	NA	NA	NA
1,1-Dichloroethylene	NA	NA	NA
1,2,4-Trichlorobenzene	NA	NA	NA

Parameter	Method Reference Non-Potable Water	Method Reference Solids and Tissue	Method Reference Potable Water (SDWA)
1,2,4-Trimethylbenzene	NA	NA	NA
1,2-Dibromoethane (EDB)	NA	NA	NA
1,2-Dichlorobenzene	NA	NA	NA
1,2-Dichloroethane	NA	NA	NA
1,2-Dichloropropane	NA	NA	NA
1,2-Dimethylbenzene	NA	NA	NA
1,3,5-Trimethylbenzene	NA	NA	NA
1,3-Butadiene	NA	NA	NA
1,3-Dichlorobenzene	NA	NA	NA
1,4/1,3-Dimethylbenzene	NA	NA	NA
1,4-Dichlorobenzene	NA	NA	NA
2,2,4-Trimethylpentane	NA	NA	NA
2-Butanone (MEK)	NA	NA	NA
2-Hexanone	NA	NA	NA
3-Chloropropene	NA	NA	NA
4-Ethyltoluene	NA	NA	NA
4-Methyl-2-Pentanone (MIBK)	NA	NA	NA
Acetone	NA	NA	NA
Acrylonitrile	NA	NA	NA
Benzene	NA	NA	NA
Bromodichloromethane	NA	NA	NA
Bromoform	NA	NA	NA
Bromomethane	NA	NA	NA
Carbon Disulfide	NA	NA	NA
Carbon Tetrachloride	NA	NA	NA
Chlorobenzene	NA	NA	NA
Chloroethane	NA	NA	NA
Chloroform	NA	NA	NA
Chloromethane	NA	NA	NA
cis-1,2-Dichloroethylene	NA	NA	NA
cis-1,3-Dichloropropene	NA	NA	NA

Parameter	Method Reference <u>Non-Potable</u> Water	Method Reference <u>Solids and Tissue</u>	Method Reference <u>Potable</u> Water (SDWA)
Cyclohexane	NA	NA	NA
Dibromochloromethane	NA	NA	NA
Dichlorodifluoromethane	NA	NA	NA
Dichlorotetrafluoroethane	NA	NA	NA
Ethylbenzene	NA	NA	NA
Hexachloro-1,3-Butadiene	NA	NA	NA
Isopropanol	NA	NA	NA
Methylene Chloride	NA	NA	NA
MtBE	NA	NA	NA
n-Heptane	NA	NA	NA
n-Hexane	NA	NA	NA
Styrene	NA	NA	NA
Tetrachloroethylene	NA	NA	NA
Tetrahydrofuran	NA	NA	NA
Toluene	NA	NA	NA
trans-1,2-Dichloroethene	NA	NA	NA
trans-1,3-Dichloropropene	NA	NA	NA
Trichloroethylene	NA	NA	NA
Trichlorofluoromethane	NA	NA	NA
Trichlorotrifluoroethane	NA	NA	NA
Vinyl bromide	NA	NA	NA
Vinyl Chloride	NA	NA	NA

Method References

- *Standard Methods for the Analysis of Water and Wastewater*, APHA, AWWA and WEF: 21st Edition, 2005. (Methods with prefix "SM")
- *Methods for the Determination of Metals in Environmental Samples*, EPA/600R-94/111, Supplement I May, 1994 (EPA 200 Series Methods).
- *Methods for the Determination of Inorganic Substances in Environmental Samples*, EPA-600/R-93-100, Revised August 1993. (Note: EPA Method 300.1 Update 1997). (EPA 300 Series methods)
- *Methods for the Determination of Organic Compounds in Drinking Water*, USEPA December 1988, revised July 1991; Supplement I, July 1990; Supplement II, August 1992 (EPA 500 series Methods)

- *Test Methods for Evaluating Solid Wastes, SW 846* USEPA Office of Solid Waste, 3rd Edition (1986) including Update I (1992) Update II, IIA, IIB (1993-1995) Update III, IIIA, IIIB (1996-2004) and Update IV (2007) (EPA 1000, 6000, 7000, 8000, 9000 series Methods).
- Compendium of Methods for the Determination of Toxic Organic Compounds in Ambient Air, USEPA, EPA/625/R-96/010b, January 1999. ("TO" Methods)
- Some other methods not otherwise referenced can be found on EPA's Office of Water website <http://www.epa.gov/waterscience/methods/method/>

APPENDIX L ICP and ICPMS TEST GROUPS

The Inductively Coupled Argon Plasma (ICP) and Inductively Coupled Plasma Mass Spectrometer (ICPMS) instruments are capable of simultaneously scanning a single sample for multiple trace metals. To simplify logging in samples to LIMS for multiple trace metals a number of *Test Groups* have been designated to meet the needs of different program objectives (e.g. landfill monitoring wells, groundwater, surface water, drinking water, etc.).

The major difference between the ICP and ICPMS is their sensitivity. The ICPMS more sensitive and is designed to analyze clean matrix samples for ultra-trace level constituents. In almost all cases, the ICPMS has replaced the graphite furnace Atomic Absorption technology that was used for extremely low level analyses. The ICP is better suited for trace metals analysis of dirtier water matrices or soil matrices where sub- $\mu\text{g/L}$ reporting is not a necessity.

It is frequently more cost-effective to analyze for a Test Group, than to analyze for a single metal. Some common "test Groups" are listed below. Contact the laboratory to develop a list for your project. The Quantitation limits for the analytes below and additional analytes are listed in [APPENDIX J](#):

PPM=Primary Pollutant Metals #1 DW = Drinking Water TAL = EPA Target Analyte List

	Cations	PPM ^a	DW ^a	TAL ^a	RCRA/TCLP ^a	Other
Aluminum	X		S ^b	X		
Antimony		X	X	X		
Arsenic		X	X	X	X	
Barium		X	X	X	X	
Beryllium		X	X	X		
Boron	X					
Calcium	X			X		
Cadmium		X	X	X	X	
Chromium		X	X	X	X	
Cobalt		X		X		
Copper		X	X	X		
Hardness as CaCO ₃						X
Iron	X			X		
Lead		X	X	X	X	
Lithium	X					
Magnesium	X			X		
Manganese	X		S ^b	X		
Molybdenum		X				
Nickel		X	X	X		
Potassium	X			X		
Selenium		X	X	X	X	

	Cations	PPM^a	DW^a	TAL^a	RCRA/TCLP^a	Other
Silica (SiO ₂)	X					
Silver		X	X	X	X	
Sodium	X			X		
Strontium						X
Thallium		X	X	X		
Titanium						X
Uranium		X				
Vanadium		X				
Zinc		X	X	X		
Zirconium						X

^a Mercury is also in the PPM, TAL, DW and RCRA/TCLP List must be requested separately.

^b S = Supplemental Drinking Water parameter

APPENDIX M UNITS & CONVERSIONS

Metric System

The basic units of measurement in the metric system are the **meter, gram, and liter**, all of the rest are multiples of these quantities using an exponent of 10.

Prefix	Symbol	Multiple	Decimal	Name
tera	T	10^{12}	1,000,000,000,000	trillion
giga	G	10^9	1,000,000,000	billion
mega	M	10^6	1,000,000	million
kilo	k	10^3	1,000	thousand
hecto	h	10^2	100	hundred
Basic unit:			Meter (m), gram (g), liter (L)	
centi	c	10^2	0.01	hundredth
milli	m	10^{-3}	0.001	thousandth
micro	μ	10^{-6}	0.000,001	millionth
nano	n	10^{-9}	0.000,000,001	billionth
pico	p	10^{-12}	0.000,000,000,001	trillionth

1 liter = 1000 milliliters = 1,000,000 microliters = 1000 cm³

1 kilogram = 1000 grams = 1,000,000 milligrams = 1,000,000,000 micrograms

Conversions

Unit	Metric Equivalent	English Equivalent
Acre	0.40468564 hectares	43560 square feet
Acre	40468564 meters	4840 yards
Acre	0.0040468564 sq.kilometers	0.0015625 sq.miles
Barrel (petroleum)	158.98729 liters	42 gallons
Bushel	35.23907 liters	4 pecks
Chain (surveyor's)	20.1168 meters	66 feet
Cord (wood)	3.624556 cubic meters	128 cubic feet
Cup	0.2365882 liters	8 ounces liquid (US)
Degree (temperature)	$^{\circ}\text{C}=(5/9)(^{\circ}\text{F}-32)$	$^{\circ}\text{F}={}^{\circ}\text{C}(9/5)+32$
Fathom	1.8288 meters	6 feet
Foot	30.48 centimeters	12 inches
Foot	0.3048 meters	0.333333 yards
Foot ²	929.0304 cm ²	144 in ²

Unit	Metric Equivalent	English Equivalent
Foot ³	28.316846 liters	7.480519 gallons (US)
Foot ³	0.028316846 meter ³	1728 in ³
Gallon, liq.(US)	3.785411784 liters	4 quarts, liq.(US)
Grain	64.79891 milligrams	0.00228571 ounces (advp)
Gram	1000 milligrams	0.03527396 ounces (advp)
hectare	10000 meters ²	2.4710538 acres
inch	2.54 centimeters	0.08333333 feet
inch ²	6.4516 centimeters ²	0.00694444 feet ²
inch ³	16.387064 cm ³ (or milliliters)	0.0346320 pints, liq.
Kilogram	0.001 metric ton	2.204623 pounds
Kilometer	1000 meters	0.62137119 miles
Kilometer ²	100 hectares	247.10538 acres
knot (nautical)	1.852 kilometers/hr	1.151 miles/hr
league (nautical)	5.559552 kilometers	3 nautical miles
liter	1000 milliliters (or cm ³)	1.056688 quarts, liq.
Meter	100 centimeters	1.093613 yards
Meter ²	10000 cm ²	1.195990 yards ²
Meter ³	1000 liters	1.307951 yards ³
micron	0.000001 meter	0.0000394 inch
mil	0.0254 millimeters	0.001 inch
mile, nautical	1.852 kilometers	1.1507794 miles
mile, statute	1.609344 kilometers	5280 feet or 8 furlongs
ounce, advp	28.349523125 grams	437.5 grains
ounce, liquid	29.57353 milliliters	0.0625 pint, liquid
pace	76.2 centimeters	30 inches
pint, liquid	0.473176473 liter	0.5 quart, liquid
point (typographical)	0.3514598 millimeter	0.013837 inch
pound, advp	453.59237 grams	16 ounces, advp
quart, liquid	0.946352946 liter	2 pints, liquid
rod	5.0292 meter	5.5 yards
section (US)	2.5899881 kilometer ²	1 mile ²
tablespoon	14.78676 milliliters	3 teaspoons
teaspoon	4.928922 milliliters	0.33333333 tablespoons

Unit	Metric Equivalent	English Equivalent
ton, metric	1000 kilograms	2204.623 pounds
ton, short	907.18474 kilograms	2000 pounds
yard	0.9144 meter	3 feet
yard ²	0.83612736 meters ²	9 feet ²
yard ³	764.554857984 liters	27 ft ³ or 201.974 gallons

Perspective

part-per-million	ppm	1 mg/kg,	1 mg/L
part-per-billion	ppb	1 µg/kg,	1 µg/L
part-per-trillion	ppt	1 pg/kg,	1 pg/L

1,000,000 ppt = 1,000 ppb = 1 ppm

One **ppm**: 1 inch in 15.782 miles or 1 second in 11.57 days

One **ppb**: 1 inch in 15782 miles or 1 second in 31.71 years

One **ppt**: 1 inch in 15,782,828 miles or 1 second in 31,710 years

Percentage

Percent means parts-per-hundred. Seldom used as a unit of environmental analytical measurement, because it is too large.

Percent, %	ppm
1	10,000
0.1	1000
0.01	100
0.001	10
0.0001	1
0.00001	0.1

APPENDIX N LIST OF ACRONYMS

AQ	Air Quality
AQM	Air Quality Monitoring
BOD	Biochemical Oxygen Demand
CN	Cyanide
COD	Chemical Oxygen Demand
CWA	Clean Water Act
DEQ	Department of Environmental Quality
DQO	Data Quality Objective
EPA	Environmental Protection Agency
ER	Eastern Region
FID	Flame Ionization Detector
GC	Gas Chromatograph (or Chromatography)
GCMS	Gas Chromatograph Mass Spectrometer (or Spectrometry)
HEM	Hexane Extractable Material
HPLC	High Pressure Liquid Chromatography
HQ	Headquarters
HR	Human Resources
HRGC	High Resolution Gas Chromatograph
HRMS	High Resolution Mass Spectrometer
IC	Ion Chromatography
ICAP	Inductively Couple Argon Plasma
ICP	Inductively Couple Plasma
ISE	Ion Selective Electrode
LEAD	Laboratory and Environmental Assessment Division
LIMS	Laboratory Information Management System
LOD	Limit of Detection
LQ	Land Quality
MCL	Maximum Contaminant Level
MDL	Method Detection Limit
MOMs	Mode of Operation Manual
MSDS	Material Safety Data Sheet
NPDES	National Pollution Discharge Elimination System
NWR	Northwest Region
NWTPH	Northwest Total Petroleum Hydrocarbons
OCE	Office of Compliance and Enforcement

OSHD	Oregon State Health Department
OVA	Organic Vapor Analysis
PBDE	Polybrominated Diphenyl Ethers
PCB	Polychlorinated Biphenyls
PCDD	Polychlorinated Dibenzo-Dioxins
PCDF	Polychlorinated Dibenzo-Furans
PID	Photoionization Detector
PPM	Priority Pollutant Metals
PQL	Practical Quantitation Limit
QA	Quality Assurance
QAO	Quality Assurance Officer
QAPP	Quality Assurance Project Plan
QC	Quality Control
RBC	Risk Based Concentration
RCRA	Resource Conservation Recovery Act
SAP	Sampling and Analysis Plan
SDWA	Safe Drinking Water Act
SMCL	Secondary Maximum Contaminant Level
STP	Sewage Treatment Plant
SVOC	Semi-volatile Organic Compounds
SW	Solid Waste
TAL	Target Analyte List
TCLP	Toxicity Characteristic Leaching Procedure
TDS	Total Dissolved Solids
TOC	Total Organic Carbon
TOX	Total Organic Halogen
TS	Total Solids
TSS	Total Suspended Solids
TVS	Total Volatile Solids
VIP	Vehicle Inspection Program
UPS	United Parcel Service
VOC	Volatile Organic Compounds
WAD	Weak Acid Dissociable (cyanide)
WPCF	Water Pollution Control Facility
WR	Western Region

APPENDIX O REVISION HISTORY.

Revision	Date	Changes	Editor
7.0	November 2009	Updated method references, clarification of Cyanides, Analyte groups updated to reflect current practices, added section on third party labs, updated custody procedures and contact information. Simplified detection limit discussion. Added VOC soil sampling guidance and additional info for soil sampling. Added acronym definitions. Reformatted entire document.	SCH
8.0	July 2013	Removed references to LASAR throughout (it still refers to an external database, we just don't know what it will be called in the future). Updated information based on Element LIMS system, including container and preservation table. Updated method references to reflect changes in 40 CFR 136 (e.g. addition of Free Cyanide, 8 hr HT for micro methods). Additional discussion on sampling and incremental sampling (section 8)	SCH