Tier I Data Validation Manual

For the

Ohio EPA

Division of Environmental Response and Revitalization

May 2023

Revision Summary

Revision	Date Effective	Summary
Revision 2.0	July 17, 2003	Chapter 11-pH was added. TCLP and Flashpoint chapters were renumbered. The checklist examples were updated to match the most current Tier I Data Validation Checklists.
Revision 2.5	April 1, 2004	Lists of common laboratory contaminants found in Chapter 6 and in Appendix II were modified. Toluene was dropped from the lists and cyclohexane was added. This modification was necessary to be consistent with federal guidance (U.S. EPA National Functional Guidelines for Organic Data Review (OSWER 9240.1-05A-P, EPA540/R-99/008, October 1999).
Revision 3.0	January 9, 2006	The changes increase information about DV responsibilities in Chapter 1; introduce a tracking system (Chapter 1), and introduce a new chapter (Chapter 14) on summarizing data validation information.
Revision 4.0	February 1, 2006	Updating the portions of the Tier I Data Validation Checklist in the manual to match the most current version.
Revision 5.0		Chapter 1 was updated to better define the role of inspectors in the data validation process; a new chapter was added that explain data validation for hexavalent chromium and cyanide analyses (Chapter 14; the former Chapter 14 was re-numbered to Chapter 15).
Revision 6.0	March 21, 2012	The text was revised to reflect that the responsibilities of the former Division of Hazardous Management has been divided to the Division of Environmental Response and Revitalization and Division of Material and Waste Management.

Revision 7.0	May 2023	The text was revised to encompass all Division of Environmental Response and Revitalization programs and comply with current criteria found within SW-846 Test Methods, the U.S. EPA National Functional Guidelines for Organic Data Review, and the U.S. EPA National Functional Guidelines for Inorganic Data Review. All chapters and appendices were updated. Vapor Intrusion and TPH chapters were added, and other chapters were renumbered.

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Chapter 1
Introduction to the Data Validation Manual

1.0 Introduction

The Ohio Environmental Protection Agency (Ohio EPA) uses environmental data from several sources to support its decision-making processes. This manual outlines a Data Validation process that will enable Ohio EPA to review analytical data for consistency, quality, and relevance before using it as a basis for making decisions. In addition, the validity of analytical data is important because it serves as a basis for evaluating compliance with Ohio EPA's rules and for enforcement actions. This chapter discusses the importance of valid analytical data, the concept of data validation, and the role of data validation in quality assurance and quality management, the levels of data validation, and the tools that can aid in the data validation process.

This manual serves as a compendium for data validation methods and examples and a tool to improve the Data Validator's ability to evaluate data reports. It is not intended to be an exhaustive reference, but it provides the fundamental information necessary to evaluate laboratory data commonly received by Ohio EPA. Therefore, the procedures discussed in this manual are confined to common SW-846 analytical methods. The manual focuses on SW-846 methods 1311 (Toxicity Characteristic Leaching Procedure (TCLP)), 8260D (Volatile Organic Compounds (VOCs)), TO-15A (VOCs), 8270E (Semi-Volatile Organic Compounds (SVOCs)), 6010D (metals and trace elements using ICP), 6020A (metals using ICP/MS), 9040C (pH determinations for corrosivity), 1010B (flashpoint determination for ignitability using ASTM Method D93), 8015 (Petroleum Hydrocarbons), 7196A (hexavalent chromium), 7470A (mercury), and 9015 (metal cyanide complexes).

While data validation is a key component of the data evaluation process; this manual does not serve as guidance for data evaluation through establishing data quality objectives (DQOs). Data evaluation is used to determine whether the DQOs for a project are met. Therefore, there are many criteria beyond data validation that may determine the relevance of analytical data. Data evaluation activities may consider, among other topics, the age of the data, the sample collection techniques, or the use of appropriate SW-846 test methods to analyze the samples. Data evaluation is important because the DQOs, or the certainty regulators place in the data, will affect the final management decisions at sites. The manual discusses this subject in Chapter 17, which is titled Data Validation Summary.

Data validation is the process of evaluating the completeness, correctness, consistency, and compliance of a data package against a standard or project-specific criteria. Data validation will identify laboratory and analytical errors that are associated with a data set. In addition, the data validation process may identify potential sampling errors, such as preservation and sample handling methods, which are out of conformance with the sampling plan's DQOs.

In most cases, the standards that will be used in this manual are those described in U.S. EPA's SW-846 Test Methods for Evaluating Solid Waste Physical/Chemical Methods (1993 and later), the National Functional Guidelines for Organic and Inorganic Data Review (2020, 2010, 2009, 2008, 2007, 2002, 2001, 1996, 1994), and the requirements of rules and regulations that DERR is authorized to administer. Data validation criteria may also be consistent with the U.S. Army Corps of Engineers (USACE) data quality management process.

1.1 Data Validation Tiers

Ohio EPA recommends conducting data validation using a tiered approach. Tier I Data Validation includes a general review of sample receipt, analysis, and the ability of the instruments to recover the elements or compounds that were analyzed. The main components of a Tier I Data Validation include assessing the technical holding times, surrogate recoveries, matrix spike/matrix spike duplicates (MD/MSD), laboratory control samples (LCS), and method blanks. The following items are to be evaluated during a Tier I Data Validation review:

Tier I Review Components:

VOCs (8260D and TO-15A), SVOCs (8270E), TPH (8015) and Inorganic Analytes:

- Chain of Custody
- Case narrative
- Field and sample identifications (IDs) cross reference
- Holding times
- Preservation and cooler receipt
- Surrogate recoveries (for organics only)
- Laboratory blank data (method blanks, preparation blanks)
- Spike data (including MS/MSD)
- LCS

Flash Point (ASTM Standard D-93 and SW-846 1010B):

- Chain of Custody
- Case narrative
- Field and sample identifications (IDs) cross reference
- Holding times
- Preservation and cooler receipt
- ASTM test methods
- LCS
- Heating protocols (initial temperature, final temperature, time intervals between flame application)
- Duplicate samples (criteria for duplicates specified in the method)
- Rate of temperature increase information
- Temperature corrected for ambient barometric pressure
- Viscosity information, p-xylene information, stirring rate information

TCLP (1311):

- Chain of Custody
- Case narrative
- Field and sample identifications (IDs) cross reference
- Holding times
- Preservation and cooler receipt
- Percent solids

- TCLP blank
- Extraction fluid information (pre-test information, extraction fluid type, pH, volume)
- Spike recoveries for metals
- Tumbler rate, tumbling time, and room temperature
- Tier II Data Validation includes a more thorough review of parameters that primarily deal with instrument calibration and analysis sensitivity, which will be discussed in a separate guidance document.

1.2 Resources

Resources, such as Tier I Checklists, this manual, and other resources are available to help Data Validators review data generated at their sites. The purpose of these resources is to both aid Data Validators in the validation process and to provide consistency in practice among the various districts. These data validation resources are discussed on the following sections.

1.2.1 Tier I Data Validation Checklists

A series of 14 Tier I Data Validation Checklists have been developed to ensure that all Tier I Data Validations are consistent and address the same QC criteria. It provides a step-by-step guide that begins with helping Data Validators identify the necessary components of a data package. It examines quality control criteria, and judges whether data should be accepted, estimated, or rejected. At each step, the checklists will instruct Data Validators on how to find information in the QC package, contact the lab if it is missing or incomplete, evaluate the information against performance criteria, and gauge the quality of the data.

Checklist #1 focuses on report completeness and technical holding times. Checklist #14 is a Data Validation Summary Checklist that prompts Data Validators to summarize the DQOs and findings, assess bias, and determine whether the quality of the data is sufficient to meet the DQOs. These two checklists should be filled out for each data report that is validated regardless of the media or analytical method. Checklist #2 through Checklist #10 will each guide the Data Validator through validating analytical data for a specific SW-846 method. Checklist #11 through Checklist #13 focus on validating waste characterization data.

1.2.2 Tier I Data Validation Manual

The manual provides an in-depth compilation of decision criteria and examples. It contains basic information about sample extraction, preservation, and analysis criteria as it applies to the quality of data. It also provides several examples to help Data Validators interpret site-specific QC data and apply consistent data qualifiers. This should enhance the usability of the checklist. Appendix A of this manual contains worked Tier I Data Validation Checklist questions that will instruct the Data Validators in the proper way to answer each question.

1.2.3 Additional Data Validation Resources

The purpose of this document is to promote uniformity of data review to help clarify and augment the review guidance of the National Functional Guidelines, to give guidance for areas of data review that require considerable professional judgment, and to specify procedures that are unique to the needs of U.S. EPA Region 5 and Ohio EPA. The references and their short descriptions below are provided as additional tools for Data Validators to utilize during the data validation process.

Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, EPA publication SW-846, Third Edition, Final Updates I (1993), II (1995), IIA (1994), IIB (1995), III (1997), IIIA (1999), IIIB (2005), IV (2008), and V (2015)

The U.S. EPA publication SW-846 is the Office of Solid Waste's official compendium of analytical and sampling methods evaluated and approved for use in complying with the RCRA regulations. SW-846 functions primarily as a guidance document setting forth acceptable, although not required, methods for the regulated and regulatory communities to use in responding to RCRA-related sampling and analysis requirements.

U.S. EPA Guidance for Data Quality Assessment

U.S. EPA has a series of three data quality assessment guidance documents that demonstrate how to use data quality assessment in evaluating environmental data sets and illustrates how to apply some graphical and statistical tools.

U.S. EPA Guidance on Data Verification and Validation

This guidance document describes processes for evaluating the completeness, correctness, and conformance of a specific data set against the method, procedural, or project requirements and determining the analytical quality of a specific data set.

U.S. EPA Requirements for Quality Management Plans

This document outlines U.S. EPA's development and content requirements for quality management plans.

U.S. EPA Guidance for Data Quality Assurance Plans

U.S. EPA's Guidance for Data Quality Assurance Plans (QAPPs) provides guidance on developing Quality Assurance (QA) Project Plans that address EPA specifications and requirements for QAPPs.

U.S. EPA Guidance on Systematic Planning Using the Data Quality Objectives Process

This guidance outlines how to develop DQOs for determining the type, quantity, and quality of data needed to reach defensible decisions or make credible estimates.

U.S. EPA Contract Laboratory Program National Functional Guidelines for Superfund Organic Methods Data Review

This document is designed to offer guidance on Contract Laboratory Program (CLP) organic analytical data evaluation and review. It is intended to assist in the technical review of data generated through the CLP.

U.S. EPA Contract Laboratory Program National Functional Guidelines for Inorganic Data Review

This document is designed to offer guidance on CLP inorganic analytical data evaluation and review. It is intended to assist in the technical review of analytical data generated through the CLP.

U.S. EPA Region I Environmental Data Review Program Guidance

The U.S. EPA Region I Environmental Data Review Program Guidance outlines regional processes to ensure measurement data are adequately reviewed prior to use.

U.S. EPA Region 2 Quality Assurance Guidance and Data Validation Standard Operating Procedures (SOPs)

U.S. EPA Region 2 provides guidance on developing Quality Management Plans and Quality Assurance Plans (QAPPs) as well as SOPs for organic and inorganic data validation.

U.S. EPA Region 4 Data Validation Standard Operating Procedures for Contract Laboratory Program Routine Analytical Services

U.S. EPA Region 4 has SOPs for organic and inorganic data validation.

1.3 Final Data Usability and Satisfaction of Data Quality Objectives

Although the Data Validation tools listed above are helpful in qualifying data, first the data must be qualified in the context in which it was taken - in full consideration of the DQOs under which the analysis was requested. Data that may be deemed acceptable (given a particular set of laboratory QC results, such as spike and surrogate recoveries) in one situation may be unacceptable for another. While some aspects of this evaluation may go beyond what is traditionally thought of as data validation, it is inappropriate to ignore these other factors and validate data in a "vacuum." Such decisions can result in consequences such as ignoring likely exceedances of regulatory levels or risk levels due to contamination remaining at a site. Chapter 17 provides a discussion of sample usability by analytical method that is meant to prompt a thorough analysis of whether data have satisfied the DQOs that triggered the sampling.

Additionally, data usability can also be impacted by bias in the data. An assessment must be made, by method, matrix, and even laboratory batch, of whether there is a directional bias associated with a data set. Typically, we are most concerned about a low bias to results, but a high bias can also be a factor in data usability. While the validator can evaluate the possible presence of bias throughout the process, a summary of any potential bias should be made at the completion of a data validation and included at the end of the checklist.

1.4 Summary

Data validation is an important tool that is not only being used by U.S. EPA and other state agencies but also by entities in the private sector to evaluate the precision and accuracy of data. Accurate data validation will help both Ohio EPA and Ohio EPA-regulated entities make appropriate decisions.

The importance of data validation should be communicated to owners and operators during the planning phase of the clean-up activities. Likewise, when requiring analytical waste evaluations, the importance of requesting laboratory QA/QC documents with all sample results should be communicated. Through communication at the outset of all sampling events, data validation will become a useful quality assurance tool in DERR's cleanup programs.

Chapter 2
Common Analytical Methods

2.0 Introduction

To understand the data validation process, it is helpful to understand how data is generated when a sample is analyzed. The data validation process is complicated by the fact that environmental data is generated from numerous analytical methods and different types of equipment. A discussion of quantitative analytical chemistry is outside of the scope of this manual; however, this chapter will examine the Inductively Coupled Plasma Spectroscopy (ICP), ICP/Mass Spectrometry (ICP-MS), and the Gas Chromatography-Mass Spectroscopy (GC/MS) methods. These analytical methods are the most widely used to analyze samples for metals or for organic compounds. This chapter will focus on the data generation process, and later chapters will discuss data validation issues with the methods that use these types of instruments to analyze data.

No matter what method is being used, or what parameters are being analyzed, the first step in generating analytical data is the preparation of the raw sample into a form that will be introduced to the analytical instrument. The preparatory method can significantly impact the sample results. Therefore, it is critical that the Tier I Data Validator understand which preparatory procedures are being used by a laboratory. It is critical that the Tier I Data Validator verify that the most recent methods outlined in the SW-846 Analytical Methods are implemented by a laboratory. The typical SW-846 preparatory methods used to prepare environmental samples are shown in Table 2.1.

Table 2.1 Common Analytical Methods and Associated Preparatory Procedures
Methods Described in SW-846, Update VII

SW-846 Analytical Method	SW-846 Preparatory Method Description
8260D - Volatile Organics	5021 Head space preparatory method for solid material
	5030B Purge and trap preparatory method for aqueous samples and some solids
	5035 Preparatory method for soil, sediment, and sludge
	TO-15 Determination of VOCs in Air Collected in Specially Prepared Canisters
8270E – Semi-Volatiles	3510C Separatory funnel method for liquids
	3511 Organic Compounds in Water by Microextraction
	3520C Continuous liquid-liquid extraction
	3535A Solid-Phase Extraction (SPE)
	3540C Soxhlet extraction for soils and other solids
	3541 Automated Soxhlet extraction for solids
	3542 Extraction of Semi Volatile Analysis Collected Using Method
	3545A Pressurized Fluid Extraction (PFE)

	3546 Microwave Extraction
	3550C Ultrasonic extraction for solids
	3580A Solvent dilution and extraction for wastes
8015 - Petroleum Hydrocarbons	3560 Supercritical Fluid Extraction of Total Recoverable Petroleum Hydrocarbons
	3561 Supercritical Fluid Extraction of Polynuclear Aromatic Hydrocarbons
6010D or 6020B - Metals	3010A Strong acid digestion for aqueous and solid samples
	3015A Microwave Assisted Acid Digestion of Aqueous Samples and Extracts
	3031 Acid Digestion of Oils for Metals Analysis by Atomic Absorption or ICP Spectrometry
	3050B Acid Digestion of Sediments, Sludges, and Soils
	3051A Microwave Assisted Acid Digestion of Sediments, Sludges, Soils, and Oils
	3052 Microwave assisted digestion for silicates
7471B – Mercury	7471B Mercury in solid waste (Cold Vapor)
9015 - Metal Cyanide Complexes	9010C Total and Amenable Cyanide: Distillation

2.1 Sample Preparation

Many of the procedures described in Table 2.1 are known as either extraction procedures (associated with organic analysis) or digestion procedures (associated with metals analysis). The use of a particular preparatory method will depend upon the type of analysis to be performed, the analytical instrument chosen and the type of sample to be prepared. Common extraction and digestion procedures are discussed in the following sections.

2.1.1 Extraction Procedures for Organic Compounds

Extraction procedures rely chiefly on the chemical affinity of organic pollutants with a solvent. The expression "like dissolves like" describe these chemical phenomena. When a soil or water sample is mixed with organic solvent, chemicals may be released from the sample and dissolve, or be "extracted" into the solvent. For semi-volatile organic compounds (SVOCs), the extraction solvent may preferentially solvate either base/neutral, or acid compounds. Each class of compounds will have a designated set of quality control compounds used in data validation. In certain cases, the sampler may request only the "base-extractable" compounds instead of the entire analyte list of the method. Most preparatory procedures facilitate the extraction process by heating or shaking the samples. After the extraction process is finished, the solvent can then be prepared for analysis.

Volatile organic compounds (VOCs) represent a special set of organic compounds. Many preparatory methods do not call for solvent extraction due to these compounds' natural tendencies to partition from the solid or liquid phase to the air. Preparatory Methods 5021 and 5030B take advantage of this partitioning effect by drawing in a portion of a gaseous sample either from the head space of the sample

or by bubbling an inert gas through the sample and then trapping the volatile compounds. These compounds can then be analyzed. Method 5035 for VOCs in solid samples also requires the addition of a solvent to the sample. However, this solvent is primarily required for preservation, not for extraction. Consult SW-846 for preparatory methods for special matrices or analyses.

2.1.2 Digestion Procedures for Inorganic Compounds

Digestion procedures for solid and aqueous samples primarily use strong acids, such as nitric and hydrochloric acids, to remove metals from solids or to keep metals in a solution. The procedures listed in Table 2.1 also require heating the sample either through applied heat or by a microwave oven technique. It should be emphasized that most of the procedures listed in SW-846 are not total digestion but rather a strong digestion that will dissolve most elements that could become "environmentally available". This means that the entire matrix of a solid sample may not be taken into solution. If a total digestion is required, preparatory Method 3052 is recommended. In addition, there are special preparatory methods for certain metals that are either volatile or are easily oxidized or reduced during the sample preparation step, such as mercury and arsenic. Refer to SW-846 for these methods and any special requirements associated with them.

2.1.3 Distillation Procedures for Cyanide

Distillation procedures for soluble cyanide salts is based on the decomposition of nearly all cyanides by a reflux distillation procedure using a strong acid and a magnesium catalyst. Reflux distillation is a process that cycles the condensate from the distillation process back into the mixture to accelerate the decomposition. Cyanide in the form of hydrocyanic acid (HCN) is released (purged) from the samples and captured into an alkaline scrubber solution, where its concentration is determined by Method 9014 or Method 9213. This method was developed to address the problem of cyanide trace analyses.

2.2 Instrumental Analysis

The samples must be analyzed once they have been properly prepared. Two common quantitative methods used for VOCs/SVOCs, and metals are gas chromatography and emission spectroscopy, respectively. These techniques form the basis for the Gas Chromatography/ Mass spectroscopy (GC/MS, SW-846 Methods 8260D and 8270E) and Inductively Coupled Plasma Optical Emission Spectroscopy (ICP-OES, SW-846 Method 6010D). Please note that for the purposes of this manual, Atomic Emission Spectroscopy or AES, is used in the checklist instead of OES. This section will briefly explain the basics of each analytical system. The Data Validator is invited to gain a more in-depth understanding of these systems by reviewing general college texts on instrumental analysis. Additionally, be aware that the two analytical systems discussed in the following sections are not the only systems of analysis listed in U.S. EPA's SW-846. Many environmental samples for metals are still analyzed by atomic absorption spectroscopy. For example, newer methods utilizing mass spectroscopy and isotope dilution techniques are gaining wide acceptance throughout the environmental community.

2.2.1 Chromatography

Chromatography has been used as a separation technique for organic compounds since early in the twentieth century. The technique usually employs a two-phase system, where compounds in a mobile phase interact with an immobile or stationary phase. In practical terms, the organic chemicals from a prepared environmental sample will be partially trapped by material (solid sorbent) in a column. The sorbent is carefully chosen so that it only retains the compounds but does not fully immobilize them.

The result is that the chemicals moving through the chromatography column will begin to separate (partition) from one another as they move or elute at different rates. The degree of separation is a function of a particular chemical's affinity for the material in the column. The amount of time that a chemical will be retained by the column is known as its retention time. Retention times will vary by the length of the column, the sorbent material chosen, the type of solvent used, and the chemical undergoing separation. A diagram of this process is shown in Figure 2.1.

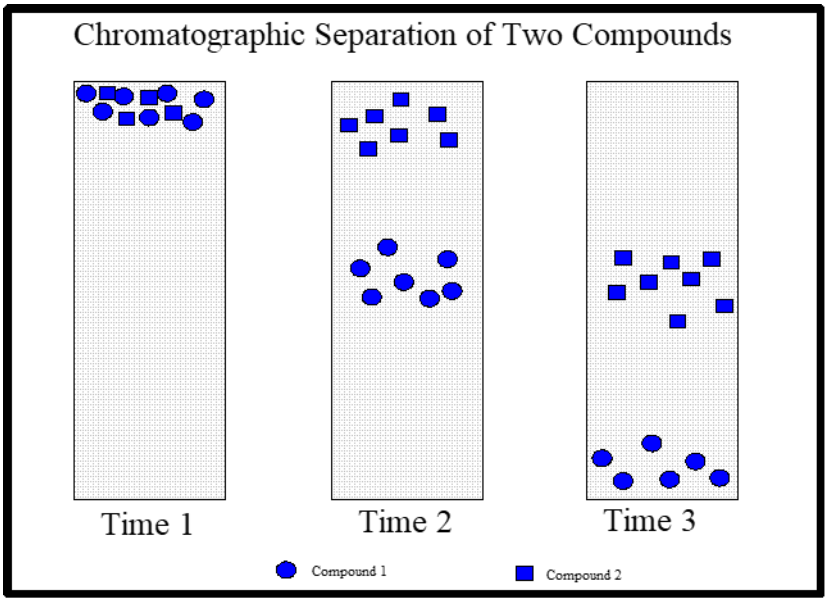
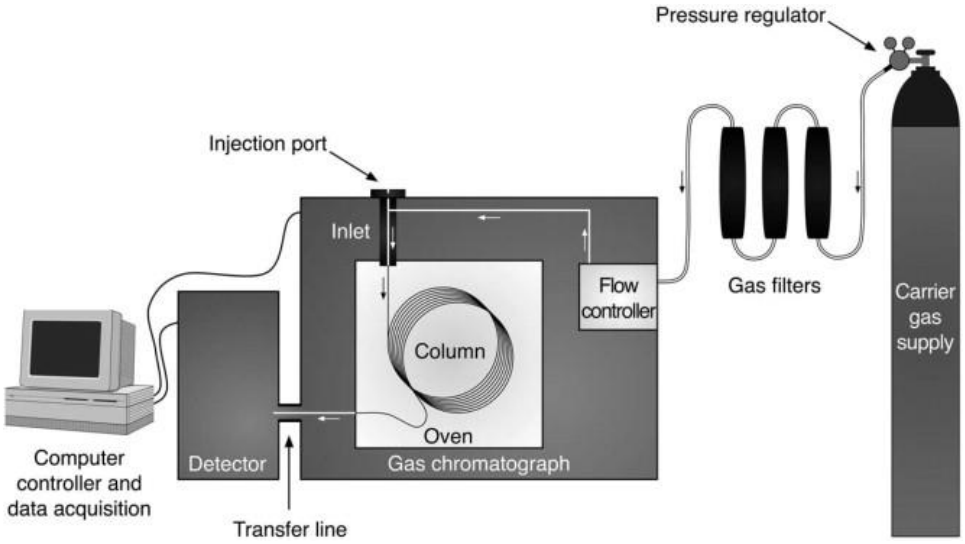


Figure 2.1 The Chromatographic Separation of Two Compounds

Gas chromatography, more correctly called gas-liquid chromatography, is one of the most common analytical techniques used to quantify organic materials in environmental samples. A gas chromatograph is typically constructed as shown in Figure 2.2.



Credit: ScienceDirect.com

Figure 2.2 A Typical Gas Chromatograph Used for Environmental Samples

A gas chromatograph consists of a carrier-gas supply, sample injection port, chromatography column, oven, detector, and some sort of integrator/recording device to manipulate raw data and save the results of the analysis.

The carrier gas is used to transport the organic chemicals from the injection port, through the column, and finally to the detector. Carrier gases are inert and do not chemically interact with the compounds in the samples. Typically, carrier gases are high purity nitrogen or helium. The injector port is where the prepared extract is introduced to the chromatograph. If a liquid sample extract (typically 1 to 10 µL) is directly injected onto the column, the carrier gas will sweep it through the column separating individual compounds along the path. Other methods can also be used to introduce the sample into the chromatograph. For example, VOC analysis from aqueous samples (5 to 25 mL samples, SW-846 Methods 5030B and 8260D) commonly uses a purge and trap technique where carrier gas passes through the liquid sample, liberating the volatile compounds which are then separated on the instrument's column.

The column is housed within an oven where the temperature can be raised or lowered or maintained throughout an analysis. The variable temperature options allow an analyst to program the instrument so that it is very efficient in liberating and separating organic compounds.

The detector is one of the most important devices found on a gas chromatograph. There are many types of detectors, including: flame ionization detectors (FID) and mass spectrographs (MS). These detection systems, described briefly below, are integral to many of the commonly used methods in SW-846.

The FID mixes hydrogen gas and air to produce a very hot (2100°C) flame. FIDs are equipped with a collector electrode, placed above the flame that measures its conductivity. When compounds exiting the chromatograph's column encounter the flame, the organic compounds are ionized (i.e., become charged by losing or gaining electrons). As the compounds are ionized, they create changes in the conductivity of the flame, which can be measured. The relative change in conductivity is associated with a compound's concentration in a sample.

Mass spectroscopy utilizes the mass of organic compounds to identify and quantify the amount of a chemical present in a sample. In GC/MS, effluent from the gas chromatograph is pumped under high vacuum into the mass spectrograph. The organic compounds are bombarded by a high energy electron beam, producing fragments of the original compounds. These fragments are typically charged. These fragments are accelerated through a voltage potential into the center of four parallel rods, called a quadrupole filter. The quadrupole arrangement separates the fragments by their mass to charge ratios. Compounds fragment according to well defined patterns which allows for identification of parent compounds. The quadrupole arrangement separates the fragments by their mass to charge ratios. The number of fragments for a given mass to charge ratio is related to the concentration of the original compound.

2.2.2 Emission Spectroscopy

Emission spectroscopy refers to photons or light emitted and detected from elements as they de-excite from an ionized state. The process usually is described as a solution containing the elements of interest being passed through an energy source. The elements are stripped of one or more of their outer shell electrons and ionized. The ions are in a highly excited state and will de-excite to a more stable state by giving off energy. This energy is a part of the electromagnetic spectrum and may be thought of as light.

The light is given off by each element with a wavelength that is characteristic for that element. The detection of characteristic wavelengths of light (optical emission spectra) allows the analyst to identify each element present in a sample. In addition, the intensity of the light can also be measured. The light intensity is a function of the amount of the element in a sample, which can then be used to determine the element's concentration. The type of optical emission spectroscopy frequently used for environmental samples involves a plasma and is termed Inductively Coupled Plasma Atomic Emission Spectroscopy (ICP-AES), and is the basis for SW-846, Methods 6010D and 6020B. A typical ICP is shown in Figure 2.3.

The sample is introduced to a nebulizer which turns the sample into a fine spray. This spray is then introduced into a plasma. The plasma ionizes the elements initially, then as they cool, they de-excite by emitting light or photons at a characteristic wavelength. The light and its intensity are detected, and the amount of an element is quantified.

In a typical ICP, a plasma is formed by radio-frequency heating of argon (Ar) gas. A plasma is a gaseous mixture of ions. Extremely high temperatures can be reached in the plasma of an ICP, usually on the order of 6,000 to 10,000°K (6,273-10,273°C or 11,323.4-18,523.4°F). The extreme temperature instantly vaporizes the nebulized sample solution. Almost as rapidly, outer shell electrons will be stripped from elements contained in the solution. This produces ions that, in turn, will produce a characteristic spectrum when they de-excite. The detection system used by ICP spectroscopy varies, but many modern instruments utilize detectors built upon the sample principle as in video cameras. These charged-coupled devices (CCDs) record the entire spectrum of light that is generated from an analyzed sample.

In addition, background light generates emissions which can interfere with elemental emission spectral analysis. It can be removed by careful examination of the sample's spectrum. Also, the plasma generates an emission spectrum that may interfere with the emission of another element. Another source of background radiation is the emission of light from molecular species of combined elements, for example, FeO. In addition, elements may ionize into a variety of states, such as Fe(0) and Fe(I). These ions will produce their own characteristic emission spectra. CCDs allow the user to select alternate wavelengths for the detection of elements when interferences are a problem

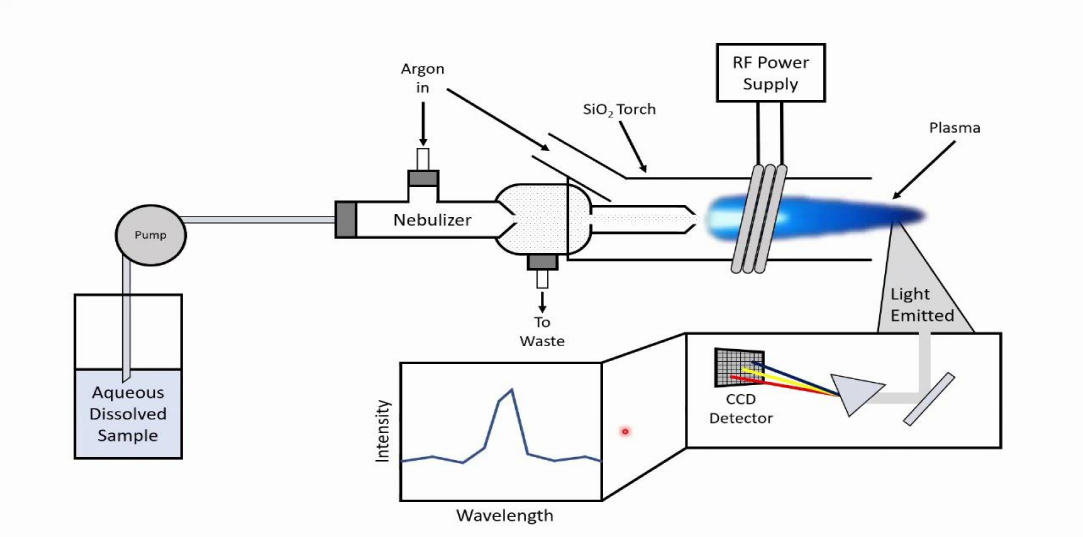


Figure 2.3 A Typical ICP-AES System

Finally, ions commonly will have multiple energy states when they are ionized by the plasma. The de-excitation process usually proceeds through multiple energy states and consequently produces light of varying wavelengths and intensities. Therefore, each element may produce not a single characteristic wavelength, but an entire spectrum of light. Analytical chemists refer to this as "stray light" which may add to the characteristic wavelength of another element. If this stray light is not corrected, a positive bias or interference may result. To further complicate matters, light from emitting ions can produce a negative bias, termed a negative interference, due to sorption by other ions in the spectrum. Both Method 6010D and 6020B contain a procedure to attempt to compensate for these interferences. A set of standards collectively called the Interference Correction Standard (ICS) is used to compensate or identify when interferences are a problem. The ICS consists of two solutions: Solution A and Solution AB. Solution A consists of the interferents, and solution AB consists of the analytes mixed with the interferents. An ICS analysis consists of analyzing both solutions consecutively, starting with solution A, for all wavelengths used for each analyte reported by ICP. The results of these standards are used to determine whether the instrument and its software can overcome potential biases due to sample matrix.

Method 6020B is a newer analytical technique that is being applied to the analysis of metals in soil and aqueous matrices. This method combines the emission spectroscopy techniques of ICP-OES with mass spectroscopy to overcome potential matrix interferences. The method starts by first passing a nebulized sample into the plasma torch. The ions produced are entrained in the plasma gas and introduced, by means of an interface, into a mass spectrometer. The ions produced in the plasma are sorted according to their mass-to-charge ratios and quantified with a channel electron multiplier. Interferences must be assessed, and valid corrections applied, or the data flagged to indicate problems. Interference correction must include compensation for background ions contributed by the plasma gas, reagents, and constituents of the sample matrix.

Chapter 3
Accuracy and Precision

3.0 Introduction

The goals for sampling a site may vary considerably from one project to the next, however, most DQOs will require that measures of accuracy and precision be incorporated into the analysis plan. Accuracy and precision in analytical measurements are prime concerns of data validation. Ideally, analytical systems are both accurate and precise; in reality, however, this is not always the case. Analytical systems may be capable of good accuracy but may not be able to repeat the measurement on a sample through time. Conversely, the analytical system may be able to repeatedly acquire the same result, but the result is inaccurate. Tier I Validators must verify that measures of accuracy and precision fall within acceptable ranges as specified in the sampling and analysis plan or by the lab's quality assurance project plan (QAPP).

3.1 Accuracy

Accuracy can be defined in numerous ways. SW-846 defines analytical accuracy as "the closeness of a measured result to an accepted reference value." This definition implies that analytical measurements are really estimates of the true concentration of a chemical in a sample. Since the goal is to determine the concentration of a compound or element in a sample, how can a determination be made as to whether the estimate is indeed accurate without knowing the true concentration? In addition, what degree of difference is acceptable between the estimated concentration and the true concentration?

The analytical process devised by U.S. EPA and codified in SW-846, Test Methods for Evaluating Solid Waste attempts to provide measures of accuracy within the analytical process. This is accomplished in two ways. First, every testing procedure requires calibration. Calibration is the act of determining the analytical instrument's response to standards which contain compounds at known concentrations. The calibration response curve is then used to establish the concentration of compounds in the samples submitted to the laboratory. Most analytical procedures described in SW-846 and other guidance requires that the lab check the validity of the calibration curve at regular intervals or re-calibrate the instrument each working day. These calibration checks are then used to assure whether the instrument is responding in a proper manner when samples are analyzed over a given period. The review of initial and continuing calibration data, instrument response through time, internal standard response, and retention time of internal standard compounds are important aspects of data validation. However, the review of calibration data is a subject left for the Tier II Data Validation process.

The second approach to determining accuracy is with spikes and system monitoring compounds or surrogate compounds. Surrogate compounds, discussed in detail in Chapter 9, are organic compounds that are not expected to occur in environmental samples, but which behave similarly to target compounds. Surrogate compounds are usually brominated or deuterated (labeled with a "heavy" hydrogen atom in a specific position indicated with a number in the name of the surrogate), making them easy to distinguish from target compounds.

Because surrogate compounds are spiked into each sample extract at known concentrations, a measure of accuracy can be determined based upon a comparison of the measured concentration of the surrogate compound to the actual amount spiked into a sample.

This comparison is usually represented by the Percent Recovery (%R) of a spiked compound. The general formula for the percent recovery is given in the following equation:

Equation 3.1

$$\%Recovery = \frac{Concentration\ Found}{Concentration\ Added} \times 100$$

This equation implies that as the found, or measured, concentration from an analysis approaches the concentration added, or spiked concentration, from a standard, the %R approaches 100 percent.

Surrogate compound analysis gives the Tier I Data Validator important information on what effect the sample material may have on the measurement of a compound in a sample. Therefore, the Validator may also be able to determine whether the accuracy of the measurement may be adversely biased.

Measures of accuracy, such as the %R, are rarely equal to 100%. Usually there is a range of %R values centered around 100 percent. If variability is expected, what %R is acceptable such that the measurements may be considered accurate enough for the goals of the sampling project? The answer to this question is generally predicated on the project's DQOs. In addition, each laboratory specifies its own quality control acceptance level. It is, therefore, important for the Tier I Data Validator to assess the laboratory's quality control acceptance criteria for surrogate recovery ranges prior to analysis in order to determine whether they meet the project specific DQOs. In general, for volatile organic compound analysis, the acceptance criteria %R is 100 +/- 25 %. Recoveries outside of this range are qualified based upon the magnitude of the exceedances.

3.2 Precision

Precision can be defined as the amount of agreement between repeated measurements of a sample or a set of samples. Because of fluctuations in the analytical process, repeated measurements of a sample will commonly differ. If enough measurements are made, the distribution of data points should approximately conform to a standard normal distribution, where data points are distributed about a mean value. In general, the range of scatter in the distribution is a measure of the precision of the analytical process.

The acquisition of sufficient replicates is beyond the scope and budget of most environmental sampling projects. Despite this, a determination must still be made as to whether the analytical process is precise enough to be acceptable.

U.S. EPA has devised a quality control check on analytical precision by requiring the analysis of spiked and spiked duplicate samples (please see Chapter 7 for more information on matrix spike and spike duplicates). Although matrix spikes are primarily discussed in this section, unspiked sample duplicates may also be required to be analyzed alongside an original sample, in which case their precision is evaluated in the same way. The measure of precision is expressed as the relative percent difference (RPD) between the spiked and the spiked duplicate sample results. Most methods in U.S. EPA SW-846 require that a matrix spike (MS) and matrix spike duplicate (MSD) sample be analyzed and evaluated for precision. The formula that is used to calculate the RPD between a spike and its duplicate is given below.

Equation 3.2

$$RPD = \frac{|S - D|}{\left(\frac{S - D}{2}\right)} \times 100$$

Where:
S = Original Sample Result, or MS Result
D = Duplicate Result, or MSD result

It is important to note that the spike and spike duplicate result concentrations be used in Equation 3.2 and not the recoveries for the spike or spike duplicate results. The concentrations are in ug/l, while the recoveries are a percentage.

Equation 3.2 implies that as the results of the spike and spike duplicate begin to deviate from each other, the value of the RPD increases from 0%. Like accuracy, the quality control criteria for precision data must be either required in the work plan, in a contract with a laboratory, or the Tier I Data Validator must know the acceptance level for precision set by the laboratory performing the analyses. In general, the DQO for precision in laboratory analyses is an RPD of 20% or less, however, some analytes and methods may have different criteria.

Chapter 4

Dilution and Detection Limits

4.0 Introduction

Data validation procedures are used to assess the accuracy and precision of a dataset. Most of these procedures evaluate the recovery and reproducibility of spikes. However, detection and quantitation limits are additional important aspects of data assessment that must also be considered in reviewing data. The detection or quantitation limits can have a bearing on successfully meeting a sampling project's DQOs. For example, if the detection limits are above risk-based remediation goals, then few or no decisions may be made concerning whether a site has met its remediation levels or clean-up standards. Interferences from the sample matrix may also act to raise the detection limits of a sample. This chapter will briefly discuss one factor in raised detection limits, namely, dilution. This chapter will also examine the different types of dilution and quantitation limits often associated with environmental data.

4.1 Dilution Factors

Dilution is the act of adding distilled water and/or other preparation reagents to a sample extract or digestate to overcome an interferent or to bring the concentration of a target analyte back into the working calibration range (determined by the concentration range of calibration standards used to develop a response factor ratio for that instrument) of the instrument. Dilution may be thought of as combining a unit volume of a sample with an appropriate volume of a solvent liquid to achieve the desired concentration. The dilution factor is the total number of volumes, including the sample volume, in which the sample will be dissolved. For example, a dilution factor of four (4), or a 1:4 dilution ratio, means combining one volume of diluent (the material to be diluted) + three equal volumes of the solvent medium. The dilution ratio is stated more generally in the following equation:

Equation 4.1

$$Dilution\ Ratio\ = \frac{volume\ of\ sample\ aliquot}{volume\ of\ sample\ aliquot\ + dilution\ volume}$$

For example, a can of soup concentrate is usually diluted with one additional can of water (the dilution solvent) giving a dilution factor of two. The soup concentrate represents one unit volume to which has been added one can (same unit volume) of water. Therefore, the soup concentrate is now distributed through two-unit volumes. This would be called a 1:2 dilution ratio, and the soup is now ½ as concentrated as it was originally. As an exercise, evaluate the dilution factor for the following situation:

Example 4.1

What is the dilution factor, if 500 µl (microliters) of a sample have been added to a volume of 5 ml (milliliters) of distilled water?

Step 1 (Dimensional Analysis):

To complete the exercise, the units of volume must be the same. For this example,

5 ml = 5000 µl and 500 µl = 0.5 ml (on a microliter basis).

Step 2 (Dilution Ratio):

Use Equation 4.1 to determine the dilution ratio.

$$\frac{500\ \mu l}{(500\ \mu l + 5000\ \mu l)} = 1\ to\ 11\ ratio$$

Step 3 (Dilution Factor):

The dilution factor in this case is 11.

Example 4.2

Care should be taken in determining the dilution factors for volumetric data from laboratory bench sheets. For example, if a 500 µl aliquot of a sample is to be part of a <u>total volume</u> of 5 ml then:

Dilution Ratio = $\frac{500\ \mu l}{5000\ \mu l}$ = 1 to 10 ratio and the dilution factor is 10

Another type of dilution that is associated with environmental sample analysis is serial dilution. Certain methods like SW-846, Method 6020B require that serial dilutions be performed if the Quality Control data (matrix spikes) suggest that significant matrix interference exists. As the name implies, a serial dilution is just a series of dilutions. The source of dilution material for each step comes from the diluted material of the previous. In a serial dilution, the total dilution factor at any point is the product of the individual dilution factors in each step up to it (see Figure 4.2). Figure 4.1 shows a set of samples where serial dilution has been performed.

Equation 4.2

$$Total\ Dilution\ Factor\ (DF)\ =\ DF_1 \times DF_2 \times DF_3, etc.$$

Figure 4.1 Serial Dilution

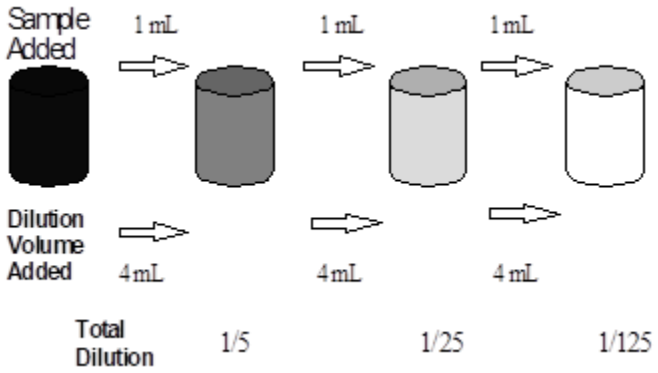


Figure 4.1 shows the process of serial dilution. Each dilution step is made by adding an aliquot from the previous step to a fixed volume of solvent material. The total dilution factor for the serial dilution is determined by multiplying the dilution factor from each step.

4.2 Identifying Dilution

An essential task of data validation is to identify whether a sample or a set of samples have been diluted. This task may be easy, as most laboratories will list the dilution factor used for a sample. However, some data reports may not clearly define the dilution factor. If this is the case, the Tier I Data Validator will have to establish the dilution factor by consulting the laboratory and requesting the information. If this is not possible, the Tier I Data Validator may be able to calculate the dilution factor if sufficient information is present in a data report. If method blank data is present, a comparison of the method detection limits listed with the blank data and the method detection limits listed with the sample results can be used to determine the dilution factor. In this case, the dilution factor is simply the ratio of the two method detection limits. Care must be exercised in using this method. The Tier I Data Validator must not compare method detection limits (MDLs) with reporting or quantitation limits. Comparing detection limits to quantitation limits will greatly exaggerate the dilution factor.

4.3 Consequences of Dilution

As mentioned previously, a laboratory may be forced to dilute a sample for a variety of reasons. Commonly, a sample may contain a constituent of concern at concentrations that are well above the analytical instrument's calibration range. If this is identified, the laboratory will dilute the sample to bring the concentration back into the range of calibration.

Dilution may have several undesirable effects. First, the detection limit will be raised proportionally to the amount of dilution. Secondly, dilution may lessen the signal from other constituents of concern in the sample to the point that they are no longer identified. The consequence is that the sample results may be interpreted as not containing these compounds and the false negative results may bias the sampling effort. Additionally, for organic analyses, surrogate standards that are added to each sample prior to analysis may be diluted to the point that recovery suffers or is non-existent. If this is the case, the Tier I Data Validator will not be able to use the quality control information and the data will be flagged. Consequently, dilution may hinder the validation of a dataset.

Dilution must also be factored into certain data validation calculations. Most notably, the evaluation of blank data requires that the dilution factor be known particularly for applying the 5X and 10X rules. Chapter 6 covers the evaluation of blank data and how to use the dilution factors to accurately assess the significance of blank contamination. Section 6.7 discusses the 5X and 10X rules. If dilution is not accounted for, erroneous conclusions concerning laboratory contamination may result.

4.4 Detection Limits

The majority of data validation activity in the Tier I process is concerned with evaluating the results of quality control samples. However, the Tier I Data Validator is also confronted with issues dealing with the detection limit of analyses. The detection limit of an individual analytical procedure is the lowest amount of analyte in a sample which can be detected but not necessarily quantitated as an exact value. The evaluation of detection limits is important. For example, if dilution of the sample is necessary, the detection limits are raised proportionately to the amount of dilution. If the detection limits are raised above a regulatory or risk level, then the usability of the data is debatable. In addition, there is general confusion concerning the myriad of ways that detection and quantitation limits are reported. This chapter will describe the commonly used detection and quantitation limits and discuss the effect of dilution. This chapter will not present methods of data evaluation concerning raised detection limits and data usability. However, these issues should be discussed in terms of the overall process for a project.

Environmental data may be reported with a variety of detection or quantitation limits. Detection and quantitation limits are not the same. The detection limit is based more upon the sensitivity of an analytical instrument and will only rarely account for the full range of matrix effects that are normally encountered with environmental samples. The most commonly encountered detection limit, the Method Detection Limit, is described below in Section 4.4.1. Quantitation limits will be discussed in Section 4.5.

4.4.1 Method Detection Limit (MDL)

The Method Detection Limit (MDL) is commonly found in environmental data reports. The procedure for determining the MDL is defined in the United States Code of Federal Regulations (40 CFR part 136, Appendix B). The MDL is a statistically defined number based upon the standard deviation of seven replicate analyses of a standard that is analyzed over multiple-day time-period. The MDL is the minimum concentration of an analyte that can be determined with 99 percent confidence that the true value is greater than zero. Most laboratories check their MDLs quarterly and evaluate whether they need to update their MDLs on an annual basis. MDLs do not need to be updated if they are within 50 to 200%. While many data reports still list MDLs, updated SW-846 methods no longer reference them. The U.S. EPA Office of Resource Conservation and Recovery (ORCR), which publishes SW-846 test methods, instead promotes the use of the Lower Limit of Quantitation approach for establishing reporting limits.

4.5 Quantitation Limit (QL)

The quantitation limit (QL) is the lowest amount of an analyte in a sample which can be quantitatively determined with suitable precision and accuracy. It is also referred to as the Lower Limit of Quantitation (LLOQ), and in most cases, it is the lowest concentration in the calibration curve. It is initially verified by spiking a clean control material (e.g., reagent water, method blanks, Ottawa sand, diatomaceous earth etc.) at the QL and processing the material through all preparation and determinative steps of a given method. Once sufficient data points exist, laboratory specific QLs can be established. Ideally, the QL

should be less than the regulatory action levels based on the project-specific requirements. The QL differs from the detection limit in that it accounts for sample matrix effects.

4.5.1 Practical Quantitation Limit (PQL)

A Practical Quantitation Limit (PQL) is defined as the lowest concentration that can be reliably achieved within specified limits of precision and accuracy during routine laboratory operating conditions. However, PQLs do not have method or matrix-specific factors. Although as of 1994, SW-846 no longer uses PQLs, they are still listed in regulatory and guidance documents, and good sampling practices imply that the Tier I Data Validator receive full documentation on the origin of a PQL listed in a data report.

4.5.2 Sample Quantitation Limit (SQL)

The Sample Quantitation Limit (SQL) is similar to the PQL and is commonly found in data reports. Like the PQL, it does not have a specific definition and is not specifically mentioned in SW-846 but is generally 5 to 10 times the MDL. The SQL represents a quantitation limit adjusted to reflect sample-specific actions, such as dilution or use of smaller aliquot sizes, or to report results on a dry-weight basis.

Chapter 5
Sample Report Completeness and Technical Holding Times

5.0 Introduction

The first step in conducting a data validation is ensuring that the sample data report, or laboratory data package, received from a laboratory or regulated facility is complete. Checking for completeness ensures that the sample report has all the components necessary to evaluate the data. The Tier I Data Validator must examine the documents and, if necessary, ask for missing information from the applicable party and/or the laboratory. To determine if sufficient information is present, it is convenient to assume that a typical data report can be divided into three parts: 1) supporting documents, 2) analytical results and 3) quality assurance/quality control (QA/QC) information.

The following information comprises the components of a basic (Tier I) data package:

- Supporting Documents:
  - Chain of custody (COC)
  - Case narrative
  - Statements of quality assurance/data validity
  - Sample receipt form
- Analytical Results:
  - Sample results package
  - Detection limits
- QA/QC Sample Results:
  - Method blanks
  - Duplicates
  - Matrix spike/matrix spike duplicates
  - Laboratory control samples
  - Surrogate recoveries

Obtaining other information that may not be included in a basic data package may also be helpful, but is not required, for the Tier I Data Validator to assess the data package. This type of information may outline project-specific requirements and includes documents such as Sampling and Analysis Plans (SAPs), Quality Assurance Project Plans (QAPPs) and Data Quality Objectives (DQOs). In addition, it may be helpful for the Tier I Data Validator to complete the data validation checklists if they have access to information recorded in the field at the time of sampling, such as field sheets and pre-sampling surveys. If the Tier I Data Validator feels the that these documents are needed to fully assess the data package, then they should reach out to the applicable party to obtain such documents.

5.1 Supporting Documents

Most data reports will include information that can be used in conjunction with other applicable QA/QC information. In some cases, such as with the COC or the statement of quality assurance, documentation is mandatory, because it is needed for litigation purposes. Other important information that can aid the Tier I Data Validator in validating data is found in the case narrative. Case narratives should summarize any quality control problems that were encountered by the laboratory during the analysis of a client's samples, and what steps the laboratory took to rectify these issues. By following the case narrative, the

Tier I Data Validator may be able to focus on significant data problems or areas of concern within a data set. The COC, case narrative, statement of quality assurance, and sample receipt form will be discussed in the following sections.

5.1.1 Chain of Custody

The COC can be strictly defined as a record of all persons who handled the samples prior to relinquishing them to the laboratory for analysis. Figure 5.1 shows an example COC form. For the Tier I Data Validator, the COC also provides a valuable means of checking whether all the sample analyses requested were performed by the laboratory and whether the analyses were performed by the requested SW-846 method (if specified on that particular COC). It can also indicate any special handling procedures that were requested by the samplers. For instance, the COC may specify that only a sub-set of parameters are to be analyzed for specific samples instead of the SW-846 analytical method's full target analyte list. The COC should contain the following information:

- Sample field ID numbers
- Date and time for each sample collected
- List of requested parameters and/or SW-846 test methods
- Preservatives used (if any)
- Sampler name(s)
- Special handling instructions
- Signatures of people with control of the samples, including the person relinquishing the samples to the lab and the person from the laboratory receiving the samples
- Date and time that samples were relinquished to the lab

Note: Anytime control of the sample(s) is being relinquished, the individual relinquishing and accepting control of the sample(s) should mark the COC with the date and time of transfer. However, it is the policy of some carriers to not sign off on the COC for sample transfer.

The Tier I Data Validator will use the COC to determine if there is missing information in the sample data report. The COC stipulates the time and date each sample was collected and can be used as an independent check on technical holding times. The COC also should indicate the preservatives used for each parameter. This information can be cross checked with the sample receipt form to evaluate whether the proper preservatives were used for each sample. Other important information contained on the form includes identification of the sampler, and signatures recording transference of sample custody. If the laboratory has an internal COC, it should also be included with the data package sample receipt form.

Ohio EPA-Division of Environmental Response and Revitalization Lazarus Government Center, 50 W. Town St., Suite 700, Columbus, Ohio 43215					Facility Name:		
Sampler's Name: Split Samples Offered ( ) Accepted ( ) Declined					Facility and Sample Locations:		
Sample ID.	Date	Time	Sample Type (Comp. or Grab)	Analysis Required	Number of Containers	Preservative	Station Description/Remarks
Transferred By:			Time/Date		Received By:		Time/Date
Transferred By:			Time/Date		Received By:		Time/Date
Transferred By:			Time/Date		Received By:		Time/Date

Figure 5.1 Example Chain of Custody Record

5.1.2 Case Narrative

The case narrative is generated by the laboratory and states whether any problems were encountered between sample receipt and analysis. The case narrative must be signed by the laboratory's QA Officer or the Laboratory Manager, include certification that all analyses were performed by SW-846 or other approved methods, and meet any required standards. The case narrative often includes a discussion of general QA/QC procedures and any anomalies, such as QA/QC sample results that did not meet acceptable limits. The client's name associated sample ID numbers, U.S. EPA SW-846 method numbers, an evaluation of technical holding times, and a discussion of potential QA/QC sample concerns should also be included.

5.1.3 Statement of Quality Assurance

A statement of quality assurance should be obtained from the laboratory before an analytical report is accepted for data validation. Ohio EPA's data validation program regards a statement of quality assurance as a legal means of assuring that acceptable and uniform laboratory methods and QA/QC practices were followed by the laboratory. The Tier I Data Validator should review the data package for a statement attesting that all analytical methods were performed using acceptable methods and that the QA/QC procedures stipulated in these methods were followed. Usually, this statement is signed by an officer of the company such as the quality assurance officer or laboratory manager. If this statement is missing from a report, the Tier I Data Validator should contact the applicable party, or the laboratory and new report should be submitted to the Agency with the required statement of quality assurance.

5.1.4 Sample Receipt Form

A sample or cooler receipt form documents the condition of the samples as they are received by the laboratory. Information typically found on a Sample Receipt Form includes the following:

- Client name
- Project name and number
- Lab project manager's name and project number
- Date received
- Turn Around Time (TAT)
- Temperature of the samples within the cooler(s)* and/or internal temperature of the cooler(s) upon receipt
- Type of coolant (wet ice, dry ice, blue ice, none, etc.)
- Sample condition (*i.e.*, are all containers intact?)
- Sample preservation methods utilized
- Presence and condition of custody seals
- Indication that sample labels and COC agree
- Any damaged samples or the presence of air bubbles for volatile samples
- Status of custody seals (if present)
- Courier of samples (drop off, FedEx, UPS, etc.)

* Temperature is an important measurement because many analytical parameters require cooling (see Table 1).

5.2 Analytical Results Package

Each data report should contain a complete set of results for analyses that were requested on the COC form. The Tier I Data Validator should use information, such as the COC and/or the provisions required in the sampling plan, to assure that all the required analyses were performed. In addition, the Tier I Data Validator should review the submittal for obvious clerical mistakes that may affect interpretation of the data, such as use of incorrect units. Finally, if the DQOs for the sample analyses indicate that the data may be used in a risk assessment, it is important to review whether the data is reported using the proper detection or quantitation limits. If inconsistencies in the data set are noted, the Tier I Data Validator should request further information from the applicable party or laboratory.

5.2.1 Sample Results Package

The sample results must contain enough information to determine whether technical holding times were met, the proper analytical methods were used, and all the parameters that were requested were analyzed. In addition to the raw data, the sample results package normally contains the facility or site name, the field sample ID numbers, the laboratory ID numbers, the analytical method numbers, the date of receipt, the date(s) of extraction, and the date(s) of analysis. The analysts' ID number or initials may also be included with the data package.

5.2.2 Detection Limits

The analytical report must contain detection limits or acceptable quantitation limits (which must be presented with dilution factor information). DERR recommends that the Method Detection Limit (MDL),

as defined by 50 FR 46906 and in Chapter 16 of this manual, be reported with the data set. See Chapter 4 for a discussion of detection and quantitation limits.

5.3 Quality Assurance and Quality Control Sample Results

Quality assurance and quality control (QA/QC) data that supports whether the analyses were performed in an acceptable manner, according to the analytical method, and within acceptable criteria for precision and accuracy, must be included in every analytical report. The type and amount of QA/QC information will be dependent upon the analytical method and data quality objectives for which the samples were taken. Most SW-846 methods detail the necessary QA/QC procedures that must be followed.

In general, to complete a Tier I Data Validation for common organic and inorganic analyses, a summary of quality control results for method blanks, matrix spikes/duplicates, laboratory control samples and surrogate recoveries (organic analyses only) should be included with the data package. Each of the quality control data is noted briefly in the following sections and discussed in detail in subsequent chapters.

5.3.1 Method Blanks

Method blanks, or preparation blanks, are used to determine whether laboratory contamination is present and, if so, whether it can significantly bias the analytical results. Method blanks consist of all the reagents that are used in preparing a sample for analysis, including internal standards and surrogate compounds. The data validation procedures for method blanks are given in Chapter 6.

5.3.2 Duplicates

Duplicate samples are separate samples, which are taken from the same source and at as close to the same time as possible, stored separately, and independently analyzed by the same laboratory using the same method. Duplicates are used to demonstrate method precision by the laboratory at the time of analysis.

Field duplicates, which are sometimes called co-located samples, are used to assess improper homogenization of samples in the field, the laboratory's internal sample storage, the reproducibility of preparation and analysis of samples, and matrix heterogeneity. Collection and analysis of field duplicates is often required to assess field and analytical precision. Field split samples are field duplicates where the sample is first homogenized and then divided into two or more aliquots. These subsamples are used to assess variability and may be evaluated by different laboratories and methods. Field split samples may not be recommended for some sample types, such as VOCs in soil, where homogenization may impact sample integrity.

Laboratory duplicates are two sub-samples, created by taking two aliquots of the same sample, usually from the same sample container. These aliquots are taken through the same preparative analytical procedures to evaluate analytical or measurement precision. Laboratory duplicates are used to assess variability associated with sub-sampling and the matrix and are more commonly used for evaluating precision for inorganic and radiological constituents.

5.3.3 Matrix Spike/Matrix Spike Duplicates (MS/MSD)

A matrix spike sample is an aliquot of either soil, water, or other material (i.e., the matrix) that is spiked with known amounts of target analytes. Matrix spikes are analyzed with each analytical batch of samples of a given matrix. Matrix spikes are used to assess the effect or bias of the sample matrix on the analytical results.

Matrix spike duplicates are performed on a second aliquot of the same matrix as the matrix spike. The results of the matrix spike duplicate are compared to the matrix spike results and can give an indication of precision. Criteria for Matrix Spike/Matrix Spike Duplicate data validation are given in Chapter 7.

5.3.4 Laboratory Control Samples (LCS)

Laboratory control samples are analyte-free water or solid clean control matrixes, similar to the sample matrix, that are spiked with target analytes at known concentrations. The performance of an analytical instrument is largely measured with the LCS results. If an analytical instrument does not perform adequately on the LCS sample, the ability of the analytical instrument to accurately analyze non-QC samples is questionable. Immediate corrective action by the laboratory should be performed. The LCS data validation criteria are found in Chapter 8.

5.3.5 Surrogate Compound Analysis

Surrogate compounds, or system monitoring compounds, are spikes of brominated or deuterated compounds incorporated into samples for organic analyses. These analytes have similar characteristics to target analytes but are not commonly found outside the laboratory setting.

Therefore, the recovery of the surrogate compounds is used as a measure of accuracy and to judge the effect of sample matrix on the recovery of target analytes. Surrogate compound data validation procedures are given in Chapter 9.

5.3.6 Regulatory Tests

Regulatory tests including the Toxicity Characteristic Leaching Procedure (TCLP), flashpoint and corrosivity (pH) tests have specified procedures that must be performed by the laboratory. For example, the TCLP requires a minimum of 100 grams for proper extraction of metals and SVOCs in a solid waste sample. Not meeting these specific method requirements could result in data rejection. The tests and the requirements for these tests are outlined in Chapters 11 through 13.

5.4 Data Report Organization

Individual laboratories format their data reports in a variety of different ways. However, most laboratories divide their data packages into sections of inorganic, volatile organic, and semi-volatile organic data. It is recommended that the Tier I Data Validator organize the data report into separate analytical batches (usually identified by a specific batch number) based on the analytical methods, matrices, and laboratory analytical methods or parameters of interest. By doing this, it is possible to associate the pertinent analytical QA/QC data with each batch.

1. Separate the laboratory data into the following report sections:

    - COC form(s)
    - Narrative summary
    - Sample results

- Quality control data

2. Separate sample results by matrix:

    - Water samples (ground water, surface water, etc.)
    - Solid and waste samples (soils, sediments, sludges, solid and liquid wastes leachate, etc.)

3. Separate sample results in water and solid/waste matrices by specific analytical methods:

    - Example: The parameters received include VOCs in ground water, SVOCs in ground water, and (BTEX) compounds in soil. The data package can be arranged in the following way:
        - Place all VOC results by SW-846, Method 8260D together
        - Place all BNA SVOC results by SW-846, Method 8270E together
        - Place all BTEX results by SW-846, Method 8021B together

4. Arrange all sample results for each SW-846 method and matrix in chronological order according to the date of analysis:

    - Based on the number of analyses requested for each sample, there will be one or more groups of sample results placed in chronological order and separated by SW-846 method and sample matrix.

5. Separate the QA/QC data by matrix/method/date (i.e., batch), and combine this information with the appropriate sample results:

    - Laboratories normally state which samples are associated with each QA/QC data sheet. If the data report package is not clear as to which analytical samples are associated with each QA/QC sample/batch, contact the laboratory for clarification.

6. Gather any additional documents that may be needed and proceed to Tier I Checklist.

5.5 Additional Documents

In some cases, additional documents or information may be necessary or helpful when performing data validation. This type of information may outline project-specific requirements and includes documents such as Sampling and Analysis Plans (SAPs), Quality Assurance Project Plans (QAPPs), and Data Quality Objectives (DQOs). It may also be helpful to obtain information recorded in the field at the time of sampling, such as field sheets and pre-sampling surveys. If the Tier I Data Validator feels the that these documents are needed to fully assess the data package, then they should reach out to the applicable party to obtain such documents.

5.6 Technical Holding Times

The technical holding time is the time, usually measured in days, in which a sample must be processed through the steps of collection, preservation, laboratory preparation, and analysis. Technical holding times vary according to the analytical method and matrix. Each party involved with a given sample, whether it is collection, packaging, shipping, receiving, or analytical processing, should perform their duties in a manner that ensures technical holding times are met. This would include the sampler promptly shipping samples with short technical holding times and notifying the laboratory of their time

critical nature, as well as the laboratory promptly contacting sampling representatives if questions exist as to the analytical request. Each party should have standard operating procedures that detail the way respective duties will be carried out.

Table 5.1 (Table 1-2 from the Tier I Checklist #1) Technical Holding Times

Analytes (Method) (Media phase)	Preserved?	From field collection to extraction	From extraction to preparation	From extraction to analysis	Max holding times	Common preservative
VOCs (8260) (aqueous)	Yes	NA	NA	14 days	14 days	Cool to 0-6˚C[2], HCl
VOCs (8260) (aqueous)	No	NA	NA	7 days	7 days	Cool to 0-6˚C
VOCs (8260) Acrolein and Acrylonitrile - only (aqueous)	Yes	NA	NA	7 days	7 days	Cool to 0-6˚C, pH 4-5
VOCs (8260) (liquid/waste)	No	NA	NA	14 days	14 days	Cool to 0-6˚C
VOCs (8260) (soil/waste)	No	NA	NA	NA	14 days	Cool to 0-6˚C or no preservative
VOCs (5035/8260) (soil/waste)	Yes	2 days	NA	12 days	14 days	Encore Sampler or equivalent,

Analytes (Method) (Media phase)	Preserved?	From field collection to extraction	From extraction to preparation	From extraction to analysis	Max holding times	Common preservative
						Cool to approximately 4°C
VOCs (TO-15) (air)	NA	NA	NA	NA	30 days	NA
SVOCs (8270)	Yes	7-14 days	NA	40 days	47 days	Cool to ≤ 6° C
TPH (8015) (GRO) (solid)	No	NA	NA	14 days	14 days	Cool to 4 °C ±2
TPH (8015) (GRO) (aqueous)	Yes	NA	NA	14 days	14 days	Cool to 4 °C ±2; HCl
TPH (8015) (DRO) (solid and aqueous)	No	7-14 days	NA	40 days	47 days	Cool to 4 °C ±2; Keep away from light

Analytes (Method) (Media phase)	Preserved?	From field collection to extraction	From extraction to preparation	From extraction to analysis	Max holding times	Common preservative
Total Metals (6000/7000) (Except Cr $^{6+}$ and Hg)	Yes	NA	NA	180 days	180 days	Nitric Acid (pH<2-aqueous); cool to 4˚C - solid samples
Hexavalent Chromium (7196) (aqueous)	No		NA	24 hours	24 hours	Cool to ≤ 4 °C
Hexavalent Chromium (3060A/7196) (solid)	No	30 days	NA	7 days		≤4±2 °C
Mercury (7470 aqueous and 7471B solid)	Yes	NA	NA	28 days	28 days	Nitric Acid (pH<2-aqueous); cool to ≤ 6ºC
TCLP VOCs (1311/8260)	No	14 days	NA	14 days	28 days	no preservative
TCLP SVOCs (1311/8270)	No	14 days	7 days	40 days	61 days	no preservative

Analytes (Method) (Media phase)	Preserved?	From field collection to extraction	From extraction to preparation	From extraction to analysis	Max holding times	Common preservative
TCLP Metals (except mercury) (1311/6010)	No	180 days	NA	180 days	360 days	no preservative
TCLP Mercury (1311/7470)	No	28 days	NA	28 days	56 days	no preservative
pH (9040)	No	24 hours	NA	NA	1 day	no preservative
Ammonia (Liquid, SM 4500-N)	No	NA	NA	7 days	7 days	Cool to 4˚C
Ammonia (Liquid, SM 4500-N)	Yes	NA	NA	28 days	28 days	Cool to 4˚C; H_2SO_4 to pH <2
Cyanide (Solid, Liquid, Multi-Phase; 9010c)	Yes	NA	NA	14 days	14 days	Cool to 4˚C ±2; NaOH ≥ pH 12

5.7 Specific Information

Evaluation of whether a sample's technical holding time has been met is an essential component of the data validation process. If the technical holding time is not met, it may cause the analytical results to be rejected or qualified as estimated. Technical holding times range from as short as 15 minutes for pH analysis of ground water samples and 48 hours for Method 5035 extraction (EnCoreTM samplers), up to six months for Method 1311, metals extraction. Personnel involved in development of sampling and analysis plans (SAPs) must be aware of these considerations to ensure that their responsibilities for technical holding times are met.

When a technical holding time has been exceeded, it may cause the Tier I Data Validator to qualify the data as "J," estimated, as "UJ," estimated undetected, or as "R," rejected. Qualification does not mean that all the data is unusable. Detected results which are qualified as "J-" should be considered biased low. The reason for, and length of, the technical holding time exceedance in conjunction with the DQOs for that sample will help the sampler or other personnel requesting the analysis to determine whether the data is of value. Additionally, a sample with an exceeded technical holding time may be considered a candidate for re-sampling based on initial results, regulatory or data quality objectives, and sampler and/or program discretion. Furthermore, a sample qualified as "UJ," estimated undetected, may, in fact, contain chemicals of concern above the detection or regulatory limits that remained undetected due only to improper preservation or technical holding time exceedance(s). Such results may be considered unusable, or a candidate for re-sampling, based on the end use of the data and the best professional judgment of the Tier I Data Validator.

Particular attention must be paid to the technical holding time when an extraction or preparation step is performed as part of the analysis. It is not sufficient to evaluate only the time elapsed between sampling and analysis. If a technical holding time is established for the steps of extraction and/or preparation, and these holding times are not met, then the data must be qualified per the Tier I Data Validation Checklist and the sampling DQOs.

5.8 Frequently Asked Questions

Q: What if a particular sample or analyte is repeatedly qualified as "J," estimated, or "UJ," estimated undetected, based on Tier I Data Validation Checklist criteria?

A: If Tier I Data Validation results in an analyte being repeatedly qualified, it may point to greater problems with the procedure or analysis. There is no specific guidance for accepting or rejecting ("R") such data. However, the Tier I Data Validator has the discretion, based on best professional judgment, to accept or reject this data. This decision is best made considering the DQOs for the project (see Chapter 14 for additional discussion of this topic). It is recommended that the Tier II Data Validator be consulted if there is a question regarding how to best qualify such data.

Q: What if a technical holding time exceedance is due to error on behalf of the party requesting the analysis (such as delay in shipment of EnCore or pH samples, or "add on" requests for analysis made to the laboratory after the samples have been received)?

A: If technical holding times are exceeded, regardless of the reason, data should be qualified or rejected using the Checklist #1 and considering DQOs. How this data will be used and other potential measures to be taken, such as re-sampling, will be at the discretion of the sampler and program.

Q: What if a technical holding time is exceeded due to the sampler not field preserving a sample or due to ambiguity as to sample preservation on the COC?

A: The results should still follow Checklist #1 and receive the appropriate qualifiers regarding sample DQOs. However, it should be a standard operating procedure of the laboratory to contact the sampler to clarify any questions or discrepancies that may arise.

Q: What are the technical holding times for pesticides, herbicides, and radiological samples for aqueous matrices?

A: Pesticides and herbicides have holding times of 7 days from sampling until extraction and 40 days from extraction to analysis for a total of 47 days. Most radiological parameters have a holding time of 6 months. However, individual radiological parameter holding times should be checked with the analytical method to verify whether an analysis was performed within holding times.

Chapter 6
Blanks

6.0 Introduction

Blanks are used throughout the analytical process to verify that the analytical equipment, reagents, internal standards, surrogates, and handling procedures do not introduce constituents of concern into the samples at unacceptable levels. For SW-846 methods, blanks are required for both metals and organic compound analysis. The three most common types of blanks found in a Tier I data package are calibration blanks, instrument blanks, and method blanks. Other types of blanks that may be encountered are field blanks, reagent blanks, equipment blanks, and trip blanks. (See Chapter 16 for definitions of the various types of blanks.) These important quality control samples are used to assess whether sampling practices at a field site have imparted an undue bias to the unknown samples. These quality control samples are evaluated with many of the same criteria that are presented in this manual. However, for a Tier I Data Validation, the principal emphasis is on evaluating method blanks.

6.1 Method Blanks

Data from the method blank is used to verify that the reagents and preparation procedures do not impart an unacceptable bias on the sample results. Under optimum conditions, no constituents of concern are measured in the method blank above the Method Detection Limit (MDL). However, it is common to find some target analytes above the detection limits. This is often due to impurities, such as solvents or acids (or impurities found in solvents/acids), which are commonly used in laboratories, contaminating reagents, or cross contamination from other highly contaminated samples. Table 6.1 lists common laboratory contaminants.

Table 6.1: Common Laboratory Contaminants

- Methylene Chloride (8260D)
- Acetone (8260D)
- 2-butanone or methyl ethyl ketone (8260D)
- Cyclohexane (8260D*)
- Phthalate esters (8270D)

***Note:** Cyclohexane is not normally included on the 8260D target analyte list.

Method blanks consist of reagent grade water or other matrix that is treated in the same manner as a sample. Though method blanks are created in the lab, they are extracted and digested in the same manner as a sample collected in the field. At least every one method blank should be analyzed per every batch of twenty samples or less. Batches include both quality control samples and samples of interest. The sequence of method blank analysis is also important. A method blank is analyzed just after each calibration verification sample in each batch.

If samples of interest are divided into different analytical batches, results for more than one method blank should be included with a sample report. In this case, it is important to note which specific sample

results are associated with each method blank. Consequently, a method blank will be analyzed for each matrix type and for each SW-846 method. If no information is given that allows for correlation of sample results with a particular method blank, then either the laboratory or the applicable party must be consulted to ensure the information is provided. Please refer to the boilerplate letter found in Appendix I to simplify requesting more information from a laboratory.

6.2 Data Requirements for Blank Validation

The Data Validator must examine a data package for the following information:

- Batch ID (This information will relate the sample batch QA/QC results to the correct samples.)
- Sample identification
- Instrument identification
- Date and time of analysis
- Results of blanks analysis
- Sample results
- Dilution factors
- Detection limits
- Samples of interest, Laboratory Control Samples (LCS), and Matrix Spikes/Matrix Spike Duplicates (MS/MSD) associated with the blank

Figure 6.1 shows a typical method blank data summary page. The method blank report has a variety of information that may prove useful. This information includes the date the samples were extracted and analyzed, the detection limit, and dilution factor. In this example, analytes, matrix type, and SW-846 method number (8270E) are also listed. A list of samples associated with the method blank is useful information that is not present in this example. This information is especially important when analytes are detected in the method blank. If these same analytes were detected in the samples of interest, then blank evaluation would be necessary. If no analytes were detected in the samples, blank valuation would not be necessary. Laboratories usually summarize most of the required data for their clients.

The QC batch number will enable the Data Validator to associate the sample results, MS/MSD, surrogate, and LCS results with this particular method blank. This can be extremely important if there are numerous samples of different matrices that are spread among different analytical batches. There will be one method blank associated with each batch of samples of a particular matrix. For example, if soil and water samples were analyzed by SW-846, Method 8260D, then at least two method blanks will be associated with the sample results (one for each matrix). Additional SW-846 methods will also have associated method blanks. Finally, if there are sufficient samples that the laboratory has to split them into multiple analytical batches, then each additional batch will have method blank data. The laboratory run log can also be helpful in associating samples with the appropriate method blank (batch QA/QC).

One way to simplify the evaluation of blanks is to separate the sample results and the associated Quality Assurance/Quality Control (QA/QC) data from a data report by matrix. If necessary, the data can be further subdivided by batch. In this manner, large, complicated data sets can be made more manageable.

If any of the required data is missing, the Data Validator must either consult with the laboratory or the applicable party to obtain the necessary information. In addition, the Tier I Data Validator may consult with their district's Tier II Data Validator.

Figure 6.1 Typical Method Blank Results Page for SW-846, Method 8260D

QC Batch: 661077 Analysis Method: EPA 5030/8260
QC Batch Method: EPA 5030/8260 Analysis Description: 8260 MSV TCLP
Laboratory: Analytical Services
Associated Lab Samples: 50307850001, 50307850003, 50307850005, 50307850007

METHOD BLANK: 3044743 Matrix: Water
Associated Lab Samples: 50307850001, 50307850003, 50307850005, 50307850007

Parameter	Units	Blank Result	Reporting Limit	MDL	Analyzed	Qualifiers
1,1-Dichloroethene	ug/L	ND	50.0	25.0	02/07/22 12:44	
1,2-Dichloroethane	ug/L	ND	50.0	25.0	02/07/22 12:44	
2-Butanone (MEK)	ug/L	ND	1000	500	02/07/22 12:44	
Benzene	ug/L	ND	50.0	10.0	02/07/22 12:44	
Carbon tetrachloride	ug/L	ND	50.0	25.0	02/07/22 12:44	
Chlorobenzene	ug/L	ND	50.0	25.0	02/07/22 12:44	
Chloroform	ug/L	ND	50.0	25.0	02/07/22 12:44	
Tetrachloroethene	ug/L	ND	50.0	25.0	02/07/22 12:44	
Trichloroethene	ug/L	ND	50.0	25.0	02/07/22 12:44	
Vinyl chloride	ug/L	ND	20.0	10.0	02/07/22 12:44	
4-Bromofluorobenzene (S)	%.	104	78-117		02/07/22 12:44	
Dibromofluoromethane (S)	%.	99	78-120		02/07/22 12:44	
Toluene-d8 (S)	%.	97	77-118		02/07/22 12:44	

6.3 Data Evaluation

Method blank data is evaluated similarly for both organic compound analysis and inorganic analysis. All data should be reported to the method detection limit, but either the MDL or the QL may be used to evaluate blank and sample results for qualification. Data between the MDL and QL should be evaluated, and data qualified as estimated should be considered as a detection. Ideally, method blank data will not contain any analytes of interest above the detection limit of the instrument. However, when the method blank does contain analytes of interest above the detection limit, the Data Validator must assess whether a positive bias has been imparted to the sample results. This is done by comparing the analytes identified in the method blank with results from the associated samples. Method blanks may be assessed as follows:

- If the method blank does not contain target analytes above the detection limit, no further action or qualification is necessary.

- If the method blank has target analytes above the detection limit, but these same analytes were not identified in the sample results, then no further qualification is necessary.

- If the method blank has target analytes above the detection limit and these same analytes are detected in the sample results, then blank contamination must be assessed to qualify the data, as necessary. In this case, the Data Validator must make sure that correct sample results are associated with the correct method blank (*i.e.*, from the same batch) and there are sufficient data to proceed with the validation.

- The MS/MSD data should also be examined for potential positive bias associated with blank contamination.

- Ohio EPA does not allow subtraction of the method blank from analytical results. If during the Tier I Data Validation process there is reason to suspect that blank subtraction has occurred, a Tier II Data Validation, which includes the review of calibration data, should be performed.

6.4 Blanks for Organic Compound Analysis

The principal criteria used to evaluate blank data are that no target compounds are found in a blank above the Quantitation Limit (QL). For the purposes of data validation, the quantitation limit is defined as the lowest limit of the calibration curve. If contaminants are detected in the blanks, but sample results are below the QL, then generally no action is required. Concern exists when blank contamination is present and sample results are above the quantitation limit, above the blank result, or 2X the blank result for common laboratory contaminants.

Table 6.2 shows blank actions for organic analyses (SW-846, Methods 8260D and 8270E).

Table 6.2 Blank Actions for VOC Analyses*		
Qualification		
Blank Result	Sample Result	Action
Detection	Non-detect	No Action
< QL	< QL	Report at QL and qualify U
< QL	≥ QL but < 2x Blank Result for common laboratory contaminants	Report at QL and qualify U
< QL	≥ QL (≥ 2x Blank Result for common laboratory contaminants)	Report at sample result and qualify J+
≥ QL	< QL	Report at QL and qualify U
≥ QL	≥ QL but < Blank Result	Report at sample result and qualify U
≥QL	≥ QL and ≥ Blank Result or 2x Blank Result for common laboratory contaminants	Report at sample result and qualify J+
Gross contamination**	Detect	Report at sample result and qualify R

*See Table 2-1 in Checklist #2 – VOC Data Validation and Table 4-1 in Checklist #4 – SVOC Data Validation.

** Gross contamination is when blank results are greater than the initial calibration high-point standard concentration.

6.5 Blanks for Inorganic Analysis

Blank evaluation is also important for metals (SW-846, Method 6010D). The procedures for evaluating metals results are similar to organic compounds. The principal criteria used to evaluate blank data are the Method Detection Limit (MDL) and QL. When the blank result is greater than the MDL but the

sample results less than the quantitation limit, the sample result should be considered non-detect. Table 6.3 shows blank actions for metals.

Table 6.3 Blank Actions for Metals Analyses*		
Qualification		
Blank Result	**Sample Result**	**Qualification**
Not analyzed at specified frequency	Non-detect	UJ
	Detect	J
≤QL	Non-detect	UJ
	> MDL but < QL	Report at QL and qualify U
	≥ QL	J+
≥QL	Non-detect	No action
	> MDL but < QL	Report at QL and qualify U
	≥ QL but < 10X the blank result	Report at blank result and qualify J+ or R
	≥ 10X the blank result	No action

*See Table 6-1 in Checklist #6 – Metals Data Validation.

For mercury (SW-846, Method 7470A), the lowest concentration of mercury in the associated samples should be less than 10x the blank concentration if the mercury concentration in the blank is greater than or equal to the QL. If not, all associated samples that have a mercury concentration less than 10x the blank concentration but greater than the QL should be redigested and reanalyzed. Table 6.4 shows the blank actions for mercury.

Table 6.4: Blank Actions for Mercury Analyses*		
Blank Result	**Sample Result**	**Action**
Not analyzed at specified frequency	Non-detect	UJ
	Detect	J
Detect < QL	Non-detect	No qualification
	Detect < QL	Report at QL and qualify U
	Detect > QL	J+ or no qualification
≤ (-MDL) but > (-QL)	Non-detect	UJ
	Detect	J- or no qualification
≥ QL	Non-detect	No qualification
	Detect < QL	Report at QL and qualify U
	≥ QL but < 10x the Preparation Blank Result	Report at Preparation Blank Result and qualify J+ or R
	≥ 10x the Preparation Blank Result	No qualification
≤ (-QL)	Non-detect	UJ
	Detect < QL	J-
	≥ QL but < 10x QL	J-
	≥ 10x QL	No qualification

* See Table 7-1 in Checklist #7 – Mercury Data Validation.

6.6 The 5X and 10X Rules

The 5X and 10X Rules can also be used to evaluate blanks for organic compound analysis (SW-846, Methods 8260D and 8270E). The 5X Rule applies to every organic compound found in a blank except for a select few where the 10X Rule applies. For organic compounds, common laboratory solvents that are often observed contaminating blanks. For inorganic compounds, the 10X Rule applies only to mercury. All blank results for metals are based upon the 5X Rule. Using the 5X and 10X Rules is simple. The rule is designed to gauge if contamination found in a blank could account for apparent contaminant(s) present in a field sample. If a target compound is found in a blank and detected in a sample, but it is not one of the common laboratory contaminants listed above, and if the sample value(s) is less than 5 times the blank concentration (5X Rule), then positive results are qualified "U," undetected. If one of the common laboratory contaminants is detected in both the blank and a sample, and if it is less than 10 times the blank concentration (10X Rule), then the sample result is qualified "U," undetected. If the concentration of a sample is greater than 5 or 10 times the blank concentration, then no qualification is necessary. In other words, the bias imparted by either the contaminated reagents or analytical system is negligible, and the results in the sample can be viewed as representative. The following examples will be useful in illustrating how to apply the 5X and 10X Rules.

Dilution of a sample may be a key factor in evaluating blank contamination. When a sample is diluted, the detection limit is effectively raised by the dilution factor. To evaluate whether blank contamination is significant, the blank and sample results must be compared on the same basis (the amount "seen" by the instrument's detector). In other words, the dilution factor must be considered to correctly apply the 5X or 10X Rules.

Chapter 7
Matrix Spikes and Matrix Spike Duplicates

7.0 Introduction

The Matrix Spike (MS) and Matrix Spike Duplicate (MSD) are quality control samples that are associated with both organic and inorganic analyte analysis. Data for MS/MSD samples are generated to determine long-term precision and accuracy of analytical SW-846 methods for various matrices and to demonstrate acceptable analyte recovery by the laboratory at the time of sample analysis. MS/MSD data alone cannot normally be used to evaluate the precision and accuracy of individual samples (particularly others in the batch that were not subjected to MS spiking). However, when used in conjunction with other available quality control (QC) information, the MS/MSD recoveries provide a strong indication of the laboratory's ability to measure the target analytes in the sample media. A MS/MSD should be included with every batch of samples that is analyzed.

The MS is used to evaluate the effect of the sample matrix on the analysis. MS samples are prepared by spiking known amounts of specific analytes into a sample. The effect of the matrix on the analyte recovery is then evaluated by comparing the recoveries of the added spike with the actual spike value. For example, if 1 mg/kg of chlorobenzene was added as a spike, and the results indicated 1 mg/kg was detected during the analysis, then 100 percent of the spike was recovered. This result would indicate that the matrix had little effect on the ability of the analytical instrument to analyze the analyte.

Matrix spikes are used to provide a measure of accuracy for a batch of samples of the same matrix, such as soil. Due to the inherent heterogeneity of samples from different locations the matrix effects seen in one sample may not be representative of the matrix effects throughout the batch. As a result, the MS/MSD samples provide only an indicator of the potential for matrix interferences. Therefore, the results from one sample cannot be used to flag other samples in the batch without corroboration from other QA/QC data. In addition, if the MS/MSD analysis was not performed on a sample of interest, the Tier I Validator obtains little information regarding accuracy.

The MSD is a spike added to a second aliquot of the same sample used for the matrix spike. The MSD provides a measure of the precision of the analysis. The duplicate is evaluated through the relative percent difference (RPD), or deviation, of the spike recoveries between the two samples. If, after analysis, the matrix spike and the matrix spike duplicate have similar results, then the relative percent difference is low, therefore the assumption is that the effect of the sample matrix on reproducibility is negligible.

7.1 Quality Assurance/Quality Control Specific Information

The MS/MSD are batch specific QA/QC samples. When analyzing by SW-846 methods, the MS/MSD are required for every batch of samples of similar matrix that are analyzed using SW-846, Methods 8260D, 6010D, and 8270E, and 8015B. If the samples in question are spread among different batches, MS/MSD information will be available for each batch. The Tier I Data Validator must be able to relate the correct MS/MSD results to each sample.

The MS/MSD results are evaluated using results from a specific unspiked sample in a batch, the results from the same sample that have been spiked (matrix spike), and the results from a second spiked aliquot of the same sample (matrix spike duplicate).

The matrix spike is evaluated using the percent recovery of the spike. The percent recovery can be determined from the following formula:

Equation 7.1		$\%R = \frac{SSR - SR}{SA} x100$
Where:		
%R	=	percent recovery of the spike analyte
SSR	=	spiking analyte result in the spike sample
SR	=	Result of the same analyte in the original sample
SA	=	actual concentration of the spike added

For example, an analysis determined that 5 mg/kg of TCE (SR) was present in a sample. If 1 mg/kg spike (SA) was added to an aliquot of this sample (matrix spike) and the analysis indicated that 5.9 mg/kg (SSR) of TCE was present in this spike sample, the percent recovery can be determined from equation 7.1 to be:

%R = (5.9 mg/kg - 5.0 mg/kg)/(1 mg/kg) X 100 = 90 % recovery

The matrix spike duplicate is evaluated by the Relative Percent Difference (RPD) between the matrix spike results and the matrix spike duplicate results. The RPD can be evaluated using the following equation:

Equation 7.2		$RPD = \frac{\lvert MSR - MSDR \rvert}{\left(\frac{MSR + MSDR}{2}\right)} x\ 100$
Where:		
RPD	=	Relative Percent Difference
MSR	=	Matrix spike result for the spiking analyte in the MS sample
MSDR	=	Matrix spike result for the spiking analyte in the MSD sample

For example, if the result for a matrix spike is 7 mg/kg (MSR) of TCE and result for the matrix spike duplicate is 6 mg/kg (MSDR), the relative percent difference may be calculated using equation 7.2.

RPD =|7 mg/kg - 6 mg/kg| ÷ [(7 mg/kg + 6 mg/kg)/2] X 100 = 15%

7.2 Information Necessary to Validate MS/MSD Data

The following information is required to complete a review of matrix spike/matrix spike duplicate data:

- Batch ID: This information will relate the sample batch QA/QC results to the correct samples
- Dilution factor of the sample
- Matrix spike recoveries
- Matrix spike duplicate recoveries
- Relative percent differences between the matrix spike and matrix spike duplicate

- Quality control criteria (*i.e.*, control limits)
- Detection limit
- Run log
- Results of blank analysis
- Spike concentrations
- Post-digestion spike information, if applicable (spiked sample result, sample result, spiking solution, %R and control limits)

7.3 Data Validation Criteria

Samples are not normally qualified using MS/MSD results alone. The Tier I Data Validator should first try to determine to what extent the results of the MS/MSD indicate that the associated data is affected by matrix interferences. In instances where it may be determined from other QA/QC sample data that the results of the MS/MSD affect only the spiked sample, then qualification would be limited to that sample alone. However, it may be determined through the MS/MSD results that a laboratory is having a systematic problem in the analysis of one or more analytes which is affecting all associated samples. The Tier I Data Validator must use professional judgment, in conjunction with other QC criteria to determine the need for qualification of positive results of non-spiked analytes. These criteria should be clearly stated in the Tier I Data Validation Checklists #2, #4, and #5 through #10.

The criteria that a specific laboratory uses to evaluate MS/MSD data must be presented in the data report or obtained from the laboratory. Percent recovery criteria usually are 100% +/- 20%. Reproducibility data are usually considered adequate if the RPD is equal to 20% or less.

The Tier I Data Validator must verify that MS and MSD samples were analyzed at the SW-846 required frequency and that results were provided for each sample matrix. If possible, the Tier I Validator must verify that the calculations were performed correctly by using raw data from the laboratory report to verify calculations using equations 7.1 and 7.2.

At least one spiked sample (pre-distillation/pre-digestion) must be prepared and analyzed from each group of samples with a similar matrix type (e.g., solids or water) and concentration (*e.g.*, low, medium) or for each Sample Delivery Group (SDG). An SDG may be either a case of field samples, each set of 20 field samples in a case, or each 14-day calendar period during which a case of field samples are received, beginning with receipt of the first sample.

If two different SW-846 analytical methods are used for the same parameter (*i.e.*, metals analysis) within the same SDG, spiked samples must be run with each SW-846 method. If more than one spiked sample recovery result per matrix and concentration, per analytical SW-846 method, per sample delivery group, is not within control criteria, all the samples of the same matrix, level, and SW-846 method in the sample delivery group would be flagged.

Determination of bias (% recovery) requires a minimum of two matrix spikes. Good sampling practices mandate that a determination of precision be made using a minimum of eight matrix spikes with analyte concentrations within range of the level of interest. These samples are site specific and contain the target analyte at or near the concentration level expected.

The Tier I Data Validator must verify that the field blank samples were not used for the spiked sample analysis. If a lab uses the field blank for spike analysis, then all other data must be carefully checked as

to whether it is acceptable. If the field blank was used, it must be noted in the Tier I Data Validation Checklists.

Good sampling practices for all SW-846 methods, except furnace atomic absorption (AA), mandate a post-digestion/post-distillation spike be run for all parameters not meeting the specified criteria (with the exception of Ag and Hg), if the pre-distillation/pre-digestion metal spike recovery is outside of the control limits, and the sample result does not exceed four (4) times the spike added. The data from post-spikes is NOT to be used to qualify sample results. If this post-digestion data has been used to qualify data, the Tier I Data Validator must note this on the Tier I Checklist. The spike concentration is two times the indigenous level or two times the contract required detection limit, whichever is greater.

Spike %R must be within the established control limits; however, verification must be made that no action was taken to qualify results based on matrix spike alone. If other batch data is outside of specification, spike data can be used to additionally justify qualifying data as estimated, "J," or rejected, "R." If sample concentrations exceed the spike concentration by a factor of four or more, the data would not be qualified even if the %R does not meet the control limits.

If the spike sample analysis was run on the sample chosen for duplicate analysis, good sampling practices mandate that all spike calculations be run on the results from the "original" sample. The average of duplicate results may not be used to determine %R.

7.4 Questions:

Q. What should be done if sample results are greater than 110% of the highest calibration standard or blank?

A. Results must be flagged as "J", estimated.

Q. Should samples be adjusted for bias?

A. Adjustment of sample value for bias is not recommended. However, bias should be evaluated, depending on the bias direction (+ or -), by adding or subtracting the value (% bias x spike concentration) to or from the sample values. Percent bias is the reciprocal value of % recovery (*i.e.*, for 70% recovery there is a negative 30% bias). Use the average recovery from the total number of matrix spikes analyzed. This adjustment approach assumes a spiking concentration equal to the concentration found in the sample.

Q. If one spiked sample recovery is not within control limits, will that affect how all the other samples are treated?

A. If there is more than one spiked sample per matrix and concentration, per analytical SW-846 method, per sample delivery group, and one spiked sample recovery is not within control limit criteria, then qualify all the samples of the same matrix, level and SW-846 method in the sample delivery group.

a. If the spike recovery is >125% and the reported sample results are <QL (quantitation limit), the data is acceptable for use.
b. If the spike recovery is > 125% or < 75% and the sample results are > than the QL, good management practices would qualify the as estimated and it would be flagged with a "J".

c. If the spike recovery results fall within the range of 30% to 74% and the sample results are < QL, the sample results would be qualified as estimated undetected and data flagged with an "UJ".
d. Whenever possible, the potential effects on the data due to spiked sample results outside control limits should be noted in the data review narrative.

Q. For Atomic Adsorption Analysis: Are any furnace results flagged with an (E) by the lab to indicate an interference? If yes, was there a post digestion spike analyzed? If so, was the post digestion spike recovery less than 10% for any of the (E) flagged results?

A. If yes, reject (flag with and "R") all affected data.

7.5 Resources

Department of Energy (DOE):

- Data Quality Objectives
- *Institutionalizing the Data Quality Objectives Process for EM's Environmental Data Collection Activities*
- Steps in the Data Quality Objectives Process–

U.S. EPA:

- How EPA Manages the Quality of its Environmental Information
- Guidance Documents for Data Quality Assurance
- Quality Assurance/Quality Control Guidance for Removal Activities
- Environmental Measurements and Modeling Collection of Methods
- Managing the Quality of Environmental Data at EPA Region 3
- Training Courses on Quality Assurance and Quality Control Activities
- Region 6 QA Training
- Region 7 QA Training
- Data Validation & Laboratory Quality Assurance for Region 9
- Region 9 Superfund Data Evaluation/Validation Guidance

Chapter 8
Laboratory Control Sample

8.0 Introduction

A Laboratory Control Sample (LCS) is a batch specific quality control sample that is used to assess whether the analytical system can perform adequately for a given matrix. An LCS is sometimes referred to as a blank spike. The LCS consists of an aliquot of a clean matrix similar to the sample matrix and of the same weight or volume. The LCS is spiked with the same analytes at the same concentrations as the matrix spike (although, an independently prepared LCS may also be obtained or prepared from a certified reference solution, reagent solid, or alternative lot reagent solid so long as at least one LCS is prepared from the same source as the calibration standard). When the results of the matrix spike and the matrix spike duplicate analysis indicates a potential problem due to the sample matrix itself, the LCS results are used to verify that the laboratory can perform the analysis on a clean matrix.

An LCS is required for the common organic analyses (8270E and 8260D) and for most inorganic analysis methods. The LCS for the volatile (8260D) analysis should at a minimum include 1,1-dichloroethene, trichloroethene, chlorobenzene, toluene, and benzene. The LCS for semi-volatile analysis (8270E) should, at a minimum, include the following compounds:

Base/neutrals	**Acids**
1,2,4-Trichlorobenzene	Pentachlorophenol
Acenaphthene	Phenol
2,4-Dinitrotoluene	2-Chlorophenol
Pyrene	4-Chloro-3-methylphenol
N-Nitroso-di-n-propylamine	4-Nitrophenol
1,4-Dichlorobenzene	

Method 6010D requires the analysis of a matrix spike and a matrix spike duplicate to evaluate matrix interference problems. If a problem is encountered, a post-digestion spike may be analyzed. The data validator should analyze post digestion spike recoveries using the equations and criteria defined in Chapter 7.

The LCS is used in relation to other quality control data such as the matrix spike/matrix spike duplicate recoveries. The matrix spike and its duplicate should contain the same compounds as the LCS and with the same concentrations. The comparisons between the LCS and MS/MSD can be used to verify that a matrix interference problem exists. For example, if a matrix interference is suspected based on matrix spike/matrix spike duplicate data, adequate recovery of compounds in the LCS will assure the validator that the laboratory can analyze samples with accuracy and precision based upon LCS spike recovery. If results show that compounds in the LCS can be recovered within the quality control criteria, a matrix interference can be confirmed. Conversely, if recovery data for compounds in the LCS fail the QC criteria, then the integrity of the analytical system is suspect and corrective measures may be required.

8.1 Quality Assurance/Quality Control Specific Information

The LCS is a batch specific QA/QC sample. When analyzing by SW-846 methods, the LCS is required for every batch of samples of similar matrix that are analyzed using SW-846, Methods 8260D, 6010D, and 8270E. If the samples in question are spread among different batches, LCS information will be available for each batch. The Tier I Data Validator must be able to relate the correct LCS results to each sample The LCS should be spiked such that the final digestate contains each analyte at the level specified in the Quality Assurance Project Plan (QAPP) or at 2x the Quantitation Limit (QL) for the associated matrix.

The LCS is evaluated by the percent recovery of the spike. The percent recovery can be determined from the following formula given in equation 8.1.

Equation 8.1

$$\% \text{ Recovery (R)} = \text{LCS Result}/C_{SA} \times 100$$

Where the LCS result is the analyzed concentration from each of the analytes added to the LCS and CSA is the concentration of the added spike.

8.2 Necessary Information Required to Evaluate LCS data

The following information is required to complete a review of LCS data:

- Batch ID: This information will relate the sample batch QA/QC results to the correct samples
- LCS chemicals and recoveries
- Quality control criteria
- Detection limit
- Spike concentrations
- Post-digestion spike information, if applicable (spiked sample result, sample result, spiking solution, %R and control limits)

Other information that may be useful in an evaluation of LCS data includes the following:

- Sample dilution factor
- Run Log
- Blank analysis results

8.3 Data Validation Criteria

LCS results are evaluated using the percent recovery data calculated using Equation 8.1. If the LCS recovery criteria are not met, then the LCS results should be used to qualify sample data for the specific compounds that are included in the LCS solution.

Professional judgment should be used to qualify data for compounds other than those compounds that are included in the LCS. Professional judgment to qualify non-LCS compounds should consider the compound class, compound recovery efficiency, analytical problems associated with each compound, and comparability in performance of the LCS compound to the non-LCS compound. If the LCS recovery is

greater than the upper acceptance limit, then positive sample results for the affected compound(s) should be qualified with a "J+." If the mass spectral criteria are met but the LCS recovery is less than the lower acceptance limit, then the associated detected target compounds should be qualified "J-" and the associated non-detected target compounds should be qualified "R," If more than half of the compounds in the LCS are not within the recovery criteria, then all of the associated detected target compounds should be qualified "J" and all associated non-detected target compounds should be qualified "R".

Table 8.1: LCS Actions for SVOC Analyses		
Qualification		
LCS Result	Sample Result	Action
> Upper acceptance limit	Detection	J+
< Lower acceptance limit	Detection	J-
< Lower acceptance limit	Non-detect	R
≥ Half target compounds not within recovery criteria	All detections	J
≥ Half target compounds not within recovery criteria	All non-detects	R

Chapter 9
Surrogate Recovery

9.0 Introduction

Surrogates are used in organic SW-846 analytical methods to evaluate what effect the matrix has on accuracy of individual samples. This is accomplished by measuring the percent recovery of the surrogate compounds added to the sample. Surrogates are organic compounds which are similar to the target analytes in chemical composition and behavior, but which are not expected to be detected in environmental media. Most surrogates are target analytes which have been chemically altered through bromination, fluorination, or isotopic labeling. Surrogate compounds are added to every sample, blank, matrix spike (MS), matrix spike duplicate (MSD), matrix spike blank (MSB) and standard prior to any extraction or analysis procedure.

9.1 Quality Assurance/Quality Control Specific Information

Surrogate recovery is used to measure accuracy. The percent recovery is determined using the following equation:

Equation 9.1

$$\%R = \frac{\text{Concentration (or Amount Found) of the Spiked Sample}}{\text{Concentration (or Amount Added) of the Spike}} \times 100$$

Surrogate recovery information must be included within the data report. If this information is not included, the facility or the laboratory should be consulted, and the necessary information supplied to the Tier I Data Validator. A boilerplate letter (to be used for requesting missing information) is available at the end of this document in Appendix C. To assess whether the surrogate recovery is acceptable, the laboratory must also supply surrogate recovery criteria. Good analytical procedures imply that the laboratory provide this information or the individual laboratory's Quality Assurance Program Plan (QAPP) may also be consulted as to its surrogate recovery criteria.

This chapter discusses surrogate recovery procedures for the common organic laboratory SW-846 methods (volatile and semi-volatile analyses).

9.2 Volatile Organic Compound (VOC) Specific Information

The following three surrogate compounds, recommended for SW-846 Method 8260D, are added to all VOC samples and blanks to measure their recovery in environmental samples and blank matrices:

- 1,2-Dichloroethane-d4
- 4-Bromofluorobenzene (BFB)
- Toluene-d8

Other compounds with physicochemical properties better resembling the analyte classes of interest may be used as surrogates provided, they can be unambiguously identified and meet any applicable acceptance criteria for Initial calibration verification (ICV) and continuing calibration verification (CCV).

Surrogate recoveries in volatile organic samples and blanks must be within the limits specified in the SW-846 method. To find the applicable limits for these surrogate compounds, refer to the laboratory's Standard Operating Procedures (SOP) or QAPP. Typical surrogate recovery ranges may be found in Table 9.1. Internal Standards and their associated surrogates for SW-846, Method 8260D may be found in Table 9.2. Most laboratories report surrogate recovery limits on the sample data and blank results sheets.

Table 9.1 Guidelines for Surrogate Recovery for SW-846, Method 8260D

Surrogate Compound	Water	Soil/Sediment
1,2-Dichloroethane-d_4	80-120	80-120
Toluene-d_8	88-110	81-117
4-Bromofluorobenzene	86-115	74-121

Table 9.2 Internal Standards & Their Associated Analytes & Surrogates For SW-846, Method 8260D

Fluorobenzene	Chlorobenzene-d5	1,4-Dichlorobenzene-d4
Acetone	Benzene	p-Bromofluorobenzene (surrogate)
Acrylonitrile	Bromodichloromethane	Bromoform
Bromochloromethane	Carbon tetrachloride	n-Butylbenzene
Bromomethane	Chlorobenzene	sec-Butylbenzene
2-Butanone	Cyclohexane	t-Butylbenzene
Carbon disulfide	Dibromochloromethane	1,2-Dibromo-3-chloropropane
Chloroethane	1,2-Dibromoethane (EDB,	1,2-Dichlorobenzene
Chloroform	Ethylene dibromide)	1,3-Dichlorobenzene
Chloromethane	1,2-Dichloropropane	1,4-Dichlorobenzene
Dichlorodifluoromethane	cis-1,3-Dichloropropene	1,2-Dichlorobenzene-d4 (surrogate)
1,1-Dichloroethane	trans-1,3-Dichloropropene	Hexachlorobutadiene
1,2-Dichloroethane	Ethylbenzene	Isopropylbenzene
1,2-Dichloroethane-d4	2-Hexanone	Isopropyltoluene
(surrogate)	Methyl cyclohexane	Naphthalene
1,1-Dichloroethene	4-Methyl-2-pentanone	n-Propylbenzene
(Vinylidene chloride)	Styrene	1,2,3-Trichloropropane
cis-1,2-Dichloroethene	1,1,1,2-Tetrachloroethane	1,2,4-Trimethylbenzene
trans-1,2-Dichloroethene	1,1,2,2-Tetrachloroethane	1,3,5-Trimethylbenzene
1,4-Difluorobenzene	Tetrachloroethene	1,2,3-Trichlorobenzene
(surrogate)	1,1,1-Trichloroethane	1,2,4-Trichlorobenzene
Freon 113	1,1,2-Trichloroethane	
Methyl acetate	Trichloroethene	
Methylene chloride	(Trichloroethylene)	

Methyl-t-butyl ether (MTBE) Trichlorofluoromethane Vinyl chloride	Toluene Toluene-d8 (surrogate) m-, p-Xylene o-Xylene	

9.2.1 VOC Data Evaluation

The QA/QC information supplied with a data report must be checked to verify that the surrogate recovery information is present and is within the acceptance criteria set by the laboratory or the projects DQOs. If any of the surrogate compounds are outside of these criteria, the sample ID(s) for these compounds should be recorded. According to SW-846, the laboratory should use the method to re-analyze the sample to confirm that the problem is due to sample matrix effects rather than laboratory deficiencies. Often, there is little information presented to indicate that re-analysis was performed. If a surrogate's recovery is outside the acceptance criteria, it is appropriate to confirm that re-analysis was performed with the facility or its laboratory. The data validator may also carefully review the data narrative for an indication that re-analysis was performed. It should be noted that upon successful re-analysis, the laboratory is not required to report the initial, failed analysis, since the second analysis is within the acceptance criteria.

The Tier I Data Validation Checklist does not require that all individual surrogate recoveries be checked mathematically. As part of the Tier I Data Validation Checklist, the Tier I Data Validator should verify that at least one percent recovery calculation was performed correctly. Raw data from the laboratory report should be used to verify calculations using the formula listed in Equation 9.1 or from specific method requirements found in SW-846.

The Tier I Data Validator must check surrogate recoveries associated with the blanks if they are present. If any of this data is out of compliance, it must be reported on the Tier I Data Validation Checklist.

9.2.2 VOC Actions

Based on the findings, good data validation procedures imply that VOC data be qualified using the following criteria:

- If a surrogate compound is above the upper control limit, then all detected results would be qualified as "J+", estimated. Results listed as non-detect would not be qualified.

- If any surrogate recovery is less than the lower criteria, but greater than or equal to 10% recovery, then all detected compounds would be qualified as "J-", estimated, and all non-detect compounds would be qualified as "UJ", estimated undetected.

- If any surrogate recovery is less than 10%, then all detected compounds would be qualified as "J-," estimated, and all non-detect compounds as "R", rejected.

An example showing how to validate surrogate data for a ground water sample analyzed for volatile organic compounds is presented in Appendix A.

9.3 Semi-Volatile Organic Compound (SVOC) Specific Information

Surrogate compounds recommended for SVOC analyses by SW-846, Method 8270E include compounds that can be divided into two fractions: acid compounds and base/neutral compounds. Each class has an assigned set of surrogate compounds. For the base/neutral fraction, the following compounds are recommended as surrogates:

- Nitrobenzene-d5
- 2-Fluorobiphenyl
- p-Terephenyl-d14

For the acid fraction, the following compounds are recommended as surrogates:

- Phenol-d6
- 2-Fluorophenol
- 2,4,6-Tribromophenol

Under certain circumstances, it may be appropriate to use additional surrogates which have similar physiochemical properties such as:

- 1,2-dichlorobenzene-d4
- 1,4-dioxane-d8
- pyridine-d5

Similar to VOC results, surrogate recoveries for SVOC samples and blanks must be within the limits specified by the laboratory. To find the applicable limits for these surrogate compounds, refer to the laboratory's SOP or QAPP. Typical surrogate ranges can be found in Table 9.3. Internal Standards and their associated analytes and surrogates for SW-846, Method 8270D can be found in Table 9.4. Most laboratories report surrogate recovery limits with the sample data and blank results.

Table 9.3 Guidelines for Surrogate Recovery for SW-846, Method 8270E

Surrogate Compound	Water	Soil/Sediment
Nitrobenzene-d5	35-114	23-120
2-Fluorobiphenyl	43-116	30-115
p-Terphenyl-d14	33-141	18-137
Phenol-d6	10-94	24-113
2-Fluorophenol	21-100	25-121
2,4,6-Tribromophenol	10-123	19-122

Note: Sample extracts with high analyte concentrations may not have surrogate recoveries reported due to sample extract dilution. Re-analysis or re-extraction may not be performed since dilution of the extract is due to high analyte concentration and not matrix interferences.

Table 9.4 Internal Standards & Their Associated Analytes &Surrogates For SW-846, Method 8270E

1,4-Dichlorobenzene-d4
Aniline
Benzaldehyde
Bis (2-chloro-1-methyl) ether
2-Fluorophenol (Surr.)
Phenol-d6 (Surr.)
Phenol (CCC)
Pyridine
Bis (2-Chloroethyl) ether
2-Chlorophenol
1,2-Dichlorobenzene
1,3-Dichlorobenzene
1,4-Dichlorobenzene (CCC)
1,4-Dioxane
Ethyl methanesulfonate
Methyl methanesulfonate
Benzyl alcohol
2-Methylphenol
4-Methylphenol
n-Nitroso-di-n-propylamine (SPCC)
Hexachloroethane
2-Picoline

Naphthalene-d8
Nitrobenzene-d8 (Surr.)
Nitrobenzene
n-Nitrosopiperdine
Isophorone
2-Nitrophenol (CCC)
2,4-Dimethylphenol
bis (2-Chloroethoxy)methane
2,4-Dichlorophenol (CCC)
1,2,4-Trichlorobenzene
Benzoic acid
Naphthalene
4-Chloroaniline
Hexachlorobutadiene (CCC)
n-Nitroso-di-n-butylamine
4-Chloro-3-methylphenol
2-Methylnaphthalene
1-Methylnaphthalene
Acetophenone
Caprolactam
2,6-Dichlorophenol
α,α-Dimethylphenethylamine

Acenaphthene-d10
1,2,4,5 - Tetrachlorobenzene
Hexachlorocyclopentadiene
2,4,6-Trichlorophenol (CCC)
2,4,5-Trichlorophenol
2-Fluorobiphenyl (Surr.)
1-Chloronaphthalene
2-Nitroaniline
Dimethylphthalate
Acenaphthylene
2,6-Dinitrotoluene
3-Nitroaniline
Acenaphthene (CCC)
2,4-Dinitrophenol (SPCC)
4-Nitrophenol (SPCC)
Dibenzofuran (CCC)
Pentachlorobenzene
2-Chloronaphthalene
Diethylphthalate
Fluorene
4-Chlorophenyl-phenylether
4-Nitroaniline
1,1'-Biphenyl
1-Naphthylamine
2-Naphthylamine
2,3,4,6-Tetrachlorophenol
2,4,6-Tribromophenol (surr)

Phenanthrene-d10
4,6-Dinitro-2-methylphenol
n-Nitrosodiphenylamine
Diphenylamine (CCC)
4-Bromophenyl-phenylether
Phenacetin
Hexachlorobenzene
Pentachlorophenol (CCC)
Pentachloronitrobenzene
4-Aminobiphenyl
Phenanthrene

Chyrsene-d12
Benzidine
Pyrene
Terphenyl-d14 (Surr.)
Dimethylaminoazobenzene
Butylbenzylphthalate
Benzo [a] anthracene
3,3' - Dichlorobenzidine
Chrysene
Bis (2-ethylhexyl) phthalate
Di-n-octyl phthalate

Perylene-d12
Perylene
Benzo [b] fluoranthene
Benzo [k] fluoranthene
Benzo [a] pyrene (CCC)
3-Methylcholanthracene
Indeno [1,2,3-cd] pyrene
Dibenz [a,h] anthracene
Benzo [g,h,i] perylene
Benzo [e] pyrene (CCC)
Dibenz(a,j)acridine

Anthracene	7,12-
Di-n-butylphthalate	Dimethylbenz(a)anthracene
Fluoranthene (CCC)	
Atrazine	
Carbazole	
4-Nitroquinoline-1-oxide	
Pronamide	

Surrogate = (Surr.)
System Performance Calibration Check = (SPCC)
Continuing Calibration Check = (CCC)

9.3.1 SVOC Data Evaluation

The QA/QC information supplied with a data report must be checked to verify that the recoveries are within the acceptance criteria. The sample ID(s) for any surrogate recovery outside of these criteria should recorded. If any two surrogate compounds in either the acid or base/neutral fraction are out of criteria, then re-analysis should be performed to confirm that the problem is due to sample matrix effects rather than laboratory deficiencies. The report narrative must also contain an indication that re-analysis was performed. As part of the Tier I Data Validation Checklist, the Tier I Data Validator should verify that the percent recovery calculations were performed correctly. This should be done by using raw data from the laboratory report and the formulas available in the specific methods found in SW-846 to verify at least one calculation.

9.3.2 SVOC Actions

If any two base/neutral or acid surrogates are out of the acceptance criteria, or if any one base/neutral or acid extractable surrogate has a recovery of less than 10 percent, then re-analysis should be performed to confirm a matrix effect rather than to identify laboratory deficiencies. The report narrative must also be checked for an indication of re-analysis.

Based on this evaluation, semi-volatile analyses are qualified using the following criteria:

- If any two surrogates in a particular class are above the upper control limit, then all detected results in that class would be qualified as “J+,” estimated. Results listed as non-detect would not be qualified.
- If any two surrogates in a particular class have recoveries less than the lower acceptance criteria, but the recovery is greater than or equal to 10%, then all detected compounds would be qualified as “J-,” estimated, and all non-detect compounds would be qualified as “UJ,” estimated undetected.
- If any surrogate in a particular class has a recovery less than 10%, then all detected compounds would be qualified as “J-,” estimated, and all non-detect compounds as “R,” rejected.

The blank data must be checked for surrogate recoveries out of compliance. If any of this data is out of compliance, this must be reported on the Tier I Data Validation Checklist.

An example showing how to validate surrogate data for a ground water sample analyzed for semi-volatile compounds is presented in Appendix A Section 5.2.

9.4 Target Analytes by Fraction

The Tier I Data Validation guidance and qualification criteria state that target analytes be qualified by either base/neutral or acid fraction. SW-846 does not designate in which fraction each target analyte belongs. In general, acid fraction target analytes will include phenol compounds and other organic acids. The base/neutral fraction will include polynuclear aromatic hydrocarbon (PAH) compounds, such as Pyrene, and chlorinated Benzene compounds. It is important to know to which fraction a target analyte belongs. If, for any compound, it is unknown to which fraction a target analyte belongs, the Tier I Data Validator can consult the Agency's contract laboratory to retrieve that information.

Chapter 10
Batch and Sample QA/QC Summary

10.0 Introduction

The Tier I Data Validation Manual has addressed specific batch and sample quality control (QC) parameters that are used to check the accuracy and precision of environmental data. Batch specific quality control results are applied to all the samples contained in a batch. Results from batch specific QC are generally not used on their own to qualify data. One reason for this is that the QC sample(s) analyzed are included in the batch(es) with sample(s) of concern but may not have been analyzed utilizing sample(s) of concern. Results of this type may indicate problems related to the QC sample's matrix in particular but may not relate to the actual matrix of the sample(s) of concern. Therefore, sample specific quality control results must also be examined when determining whether data should be qualified. Table 10.1 outlines a summary of batch and sample-specific quality control parameters commonly generated with organic and inorganic analyses. This table also indicates the purpose of each QC parameter and what information these samples give the Data Validator concerning the validity of the analytical results.

Table 10.1 Summary of Batch and Sample QA/QC Parameters

QC Parameter Name	Batch, Method, or Sample	Performed on Blank or Sample Itself?	Organics or Inorganics	Purpose
Calibration Standard	Sample	Blank, Sample	Organics	Used to quantify compounds in a sample, to give an indication of matrix interferences, instrumental control and analyst techniques for individual samples.
Internal Standard	Batch	Blank	Both	Monitors the efficiency of the preparation procedures and methods for each sample matrix using the same procedures and analytical methods as the actual samples. Assessed by % Recovery. Used to document overall lab performance of each step during the analysis, using an ideal "sample."
LCS (or Blank Spike)	Batch	Sample	Both	Split sample used to document the precision of a method in a given sample matrix.
Matrix Duplicate	Batch	Sample	Both	Spiking of a sample prior to prep/analysis with a known concentration of target analyte(s). Provides information about the effect of the sample matrix on the digestion and measurement methodology. Used

				to document the bias of a method in a given sample matrix.
Matrix Spike	Batch	Sample	Both	Spike of the same compounds as used in the matrix spike that are added to a second aliquot of the same sample. Intra-lab split samples spiked w/ identical concentration of target analyte(s) prior to prep/analysis. Used to document precision and bias of method in a given sample matrix.
Matrix Spike Duplicate	Batch	Blank	Both	Provides a measure of whether the spiking compounds are inappropriate for a specific batch of samples. For example, organic acids may react with the sample matrix causing unacceptable MS/MSD reproducibility.
Matrix Spike Blank	Batch	Blank	Inorganics	Addition of a known amount of standard after digestion. Also termed analytical spike. Often used to narrow down source(s) of QC problems found in Pre-Digestion Spike.
Post-Digestion Spike	Batch	Blank	Inorganics	See Matrix Spike
Pre-Digestion Spike	Batch	Blank	Inorganics	Spike added at the beginning of a procedure, and therefore subject to preparatory and analytical procedures.
Prep Spike	Sample	Sample	Both	Sample run at specific dilutions to determine whether any significant chemical or physical interferences exist due to sample matrix effects. (ICP only).
Serial Dilution	Method	Sample	Organics	Addition of compounds that are similar to target compounds in physical and chemical properties. Provides indications of matrix interference.
Surrogate	Method	Sample	Organics	Compound known for eluting from the GC column at a particular time. The elution time is then used to confirm consistent performance of the equipment (compared to previous runs).

Chapter 11

Vapor Intrusion Data Validation

11.0 Introduction

Method TO-15A is a method for VOCs in air. It provides basic canister sampling and analysis information, incorporates current technologies and best practices, defines performance criteria, and recommends specific procedures associated with collection and analysis of trace levels of volatile organic compounds (VOCs) in ambient air using specially prepared, evacuated stainless steel canisters. The VOCs targeted method TO-15A may also be measured in soil gas and indoor air during vapor intrusion (VI) investigations. VI is the movement of chemical vapors from contaminated soils and/or ground water into the indoor air of overlying or nearby buildings (Figure 11.1). The chemical vapors can enter buildings through cracks in basements and slab foundations or through other openings such as sump pits, utility conduits and drains.

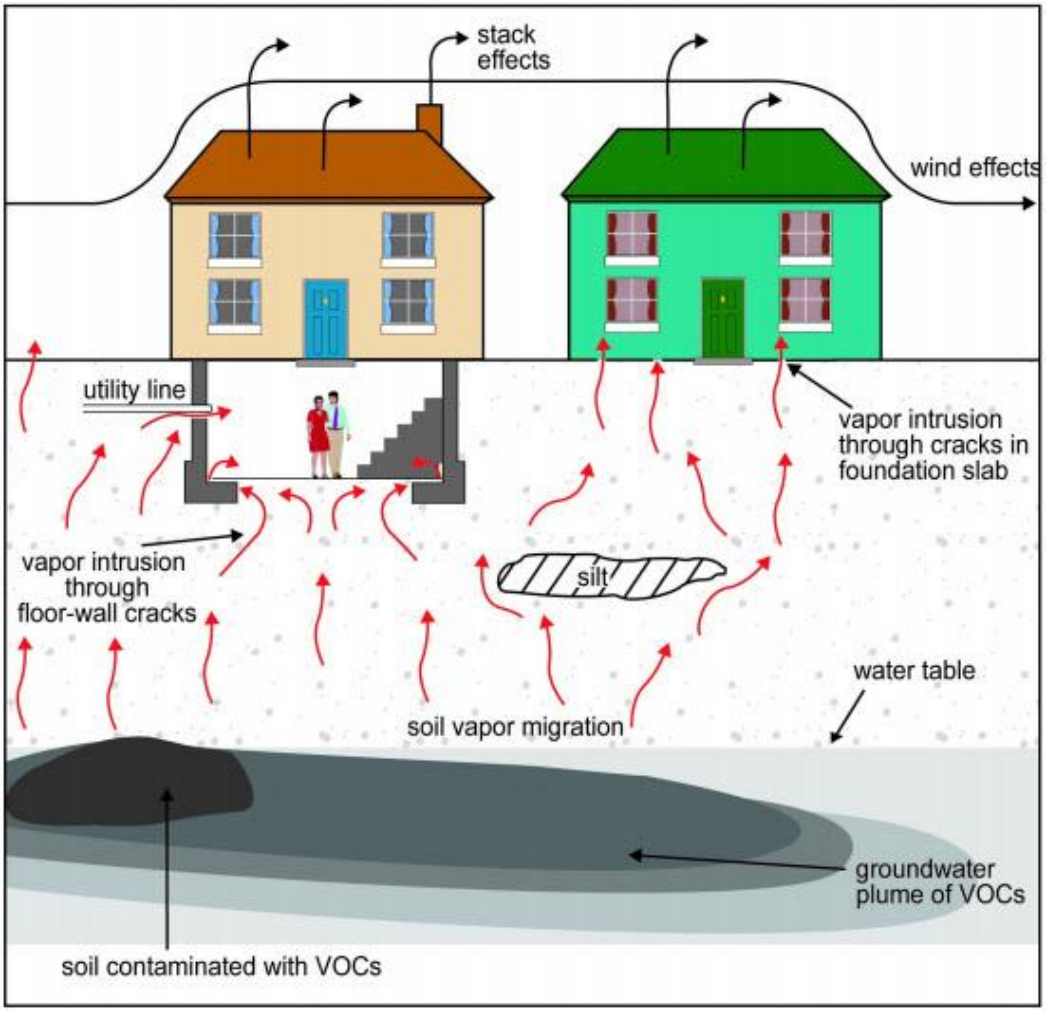


Figure 11.1 Migration of Soil Vapors to Indoor Air

VI sampling is outside the scope of method TO-15A, and modifications to this method may be required to be suitable for this purpose. Modifications may include, but are not limited to, instrument calibration, reduction of the preconcentrated volume, and less aggressive canister cleaning techniques. Different method performance specifications may also be applicable to VI investigation. This chapter focuses on the validation of TO-15A analytical data that has been collected for investigating and evaluating the VI pathway.

Ohio EPA recommends evaluating multiple lines of evidence in a systematic approach to investigate potential VI risk to receptors. Sampling strategy, development of a Conceptual Site Model (CSM), and evaluation of data for VI investigations should follow the guidance provided in "Sample Collection and Evaluation of Vapor Intrusion to Indoor Air" (Ohio EPA, 2020). Most of the lines of evidence should be based on empirical data from environmental media including soil gas, sub-slab vapor, and/or indoor air.

U.S. EPA's June 2015 Office of Solid Waste and Emergency Response (OSWER) Technical Guide for Assessing and Mitigating the Vapor Intrusion Pathway from Subsurface Vapor Sources to Indoor Air (VI Guidance) (U.S. EPA, 2015) states that the chemicals in the subsurface must be both sufficiently volatile and toxic to present a vapor intrusion risk. A chemical is considered "volatile" if it is:

- Vapor pressure is greater than 1 millimeter of mercury (mmHg); or
- Henry's law constant is greater than 10-5 atmosphere-meter cubed per mole (atm m^3 mol-1).

In addition to being sufficiently volatile, a chemical must be potentially toxic to present a vapor intrusion risk. A volatile chemical may be considered toxic regarding vapor intrusion if:

- The vapor concentration of the pure component exceeds the target indoor air concentration when the subsurface vapor source is in soil; or
- The saturated vapor concentration exceeds the target indoor air risk level when the subsurface vapor source is in ground water.

Analytical methods, quantitation limits, qualified data, and blanks should all be evaluated prior to relying on vapor data for decision making. Data are evaluated for several reasons, which should be described in DQOs for the site. Generally, data are evaluated to determine the most logical and efficient next step in the VI investigation or remedial process.

11.1 Sampling and Analytical Methods

The most widely used sample and analytical method for VI investigations is Method TO-15A (U.S. EPA, 2019). Some other common methods for soil gas, sub-slab vapor, and indoor air include:

1) Method TO-11A for Formaldehyde,
2) Method TO-13A for Polycyclic Aromatic Hydrocarbons (PAHs),
3) Method TO-14A for VOCs,
4) TO-15 Selected Ion Monitoring (SIM), and
5) Method TO-17 for VOCs.

Some of these methods use adsorbent cartridges or sorbent tubes for sample collection. However, most soil gas, sub-slab vapor, and indoor air samples are collected in canisters (Figure 11.2). This manual and Checklist #3 focus on Method TO-15A for VI investigation data.

Figure 11.2 Summa Canisters

Method TO-15A provides procedures for measuring a subset of the VOCs included in the hazardous air pollutants (HAPs) listed in Title III of the Clean Air Act Amendments of 1990. In Method TO-15A, VOCs are defined as organic compounds with a vapor pressure ≥ 0.1 mm Hg at 25 °C and standard pressure of 760 mm Hg. This means that Method TO-15A may not include all chemicals that are sufficiently volatile and toxic to present a vapor intrusion risk.

A soil gas, sub-slab vapor, and indoor air sample is collected into an evacuated, specially prepared stainless-steel canister. Air may be collected as a "grab" sample or as a time-integrated sample. A grab sample is taken by opening the canister valve and allowing the canister to fill quickly (within seconds to minutes). A time-integrated sample is collected by filling the canister at a constant rate over a known time period (typically over hours or days) using a regulator. Generally, "grab" samples are appropriate for soil gas or sub-slab vapor sampling, and time-integrated samples are appropriate for indoor air sampling. Refer to Ohio EPA's March 2020 "Sample Collection and Evaluation of Vapor Intrusion to Indoor Air" guidance document for more information about soil gas, sub-slab vapor, and indoor air sampling.

VOCs that are contained in up to 1 L of air are preconcentrated and injected into a gas chromatograph–mass spectrometer (GC-MS) for separation, identification, and quantitation. The preconcentrator captures VOCs from the sample aliquot. Target VOCs are identified through retention times (RTs) and the associated mass spectra by comparing observed fragmentation patterns to reference spectral

patterns established during calibration. The use of both gas chromatographic RTs and mass fragmentation patterns reduces the likelihood of misidentifying compounds.

11.2 Quality Assurance/Quality Control for Vapor Intrusion

Method TO-15A discusses the quality control procedures that should be followed to ensure the data quality data are produced. Checklist #11 guides Data Validators through how to review quality control data for method TO-15A to ensure valid data is used for evaluating and making remedial decisions about the VI pathway. Tier I Data Validation of VI data includes evaluating blanks, laboratory control samples, and duplicates.

A calibration blank should be prepared with each set of standard canisters to be used for an initial calibration. The purpose of the calibration blank is to demonstrate that the diluent gas and dilution apparatus is sufficiently clean so little or no positive bias is imparted during calibration. A calibration blank is analyzed using the same instrument method as standards and field samples when the initial calibration is established and may be included in the calibration curve as a zero-concentration level. An instrument blank should be analyzed at the beginning of each sequence as a preliminary demonstration that the carrier gas and analytical system show acceptably low levels of target VOCs and potential interferences. An instrument blank is a preconcentration analysis cycle performed where all preconcentration steps are taken without introduction of diluent or sample gas into the preconcentrator. A method blank indicates possible laboratory contamination and verifies that target VOCs and potential interferences are acceptably low. A method blank consists of a canister filled with humidified clean diluent gas, and it is analyzed the same as field samples in the analytical sequence.

Precision of the method may be assessed by collecting and analyzing collocated or duplicate samples as well as replicate samples. Precision is evaluated by calculating the absolute RPD of the measurement pair using the following formula:

$$RPD = \left| \frac{X_1 - X_2}{\left(\frac{X_1 + X_2}{2}\right)} \right| \times 100$$

where:

X_1 = target VOC concentration measured in first measurement of the precision pair (pptv)

X_2 = target VOC concentration measured in second measurement of the precision pair (pptv)

Acceptable precision analyses will demonstrate RPD ≤ 25% for each target analyte when both measurements are greater than or equal to 5X the MDL (U.S. EPA 2019). Failure to meet this criterion should prompt the analyst to investigate the reason for the discrepancy, and associated results should be flagged.

The precision of the method and field collection activities can be evaluated through collocated or duplicate samples. Method TO-15A recommends that approximately 5% of the total number of samples should be collected as duplicate or collocated samples.

Replicate analyses are used to demonstrate precision of the instrument and do not provide information on field sampling precision. Each analysis sequence should include a replicate analysis of a sample collected in the field at a rate of either one replicate or replicates of 5% of the field samples, whichever is greater.

Field blanks provide additional verification that the data being collected are reliable. For a field blank, the canister valve is not opened in the field and should not become contaminated. Field blanks that do not meet acceptance criteria should prompt examination of the sample preparation and handling procedures and qualification of the data reported for associated samples. Field blank acceptance criteria should be approximately 20 pptv or less.

A field spike is prepared by filling a canister with humidified standard gas at a concentration in the lower third of the calibration curve. The field spike canister is transported to the field site(s) and treated identically to field samples both in the field and the laboratory. The field spike canister is not opened in the field. Field spike acceptance criteria should be within ±30% of the theoretical spiked concentrations.

Best practices for method TO-15A also include canister cleaning procedures, cleaning of sampling components, leak checks, sampling activities, and procedures for minimizing interreferences.

Collecting samples that contain elevated concentrations of VOCs may result in carryover to subsequent samples, particularly if purging or decontamination is not conducted between samples. All sample equipment should be qualified both initially received and periodically thereafter to demonstrate it is not contributing to measurement bias. The integrity of the canisters used for sampling should always be maintained, including the time of shipment, in the field, while sampling, return shipment, and time of analysis. Canister cleaning verification result should be considered when validating sample results. Canister contamination actions are provided in Table 11.1 below.

Table 11.1: Canister Contamination Actions for TO-15 Analyses

Qualification		
Canister Cleaning Result	Sample Result	Action
Detects	Analytes found in clean canister are non-detect	No Action
< QL	< QL	Report QL as U
	≥ QL and < 2X the QL	Report sample concentration as U
	≥ 2X QL	No Action
> QL	< QL	Report QL as U
	≥ QL and ≤ clean canister value	Report clean canister value as U
	≥ QL and > clean canister value	No Action
= QL	≤ QL	Report QL as U
	> QL	No Action

New canisters should be checked to make sure they are leak-free prior to initial use. This is accomplished by either evacuating or pressurizing the canister. The vacuum of each canister should be

verified prior to deployment and should be measured at the time of setup to minimize contamination, bias, and incomplete sample volumes due to leakage and inadequate starting vacuum.

Performing leak checks on sampling devices allows opportunity to repair sampling equipment prior to field deployment. Leak checks should also be performed at the time of sample collection. Verify the cleanliness of sample collection devices and test the sampling apparatus to ensure the connection is leak free. Preset flow rates and test the operation of sample collection devices.

Interferences that can occur during sample collection include leaks in the sample train, contaminants in the sample train, and contaminants in the canister from previous sample events. Leaks within the sample flow path could result in sample dilution or contaminate field-collected samples. Leaks may also impact time-integrated sampling when unmetered air enters the flow path (See Ohio EPA's March 2020 "Sample Collection and Evaluation of Vapor Intrusion to Indoor Air" guidance document for more information on leak testing).

Particulate matter, insect nests, spider webs, and other materials within the sample flow path may act as sorbents to adsorb VOCs, which could effectively scrub them from the sampled air stream and result in a low bias. The VOCs may desorb later and potentially contaminate subsequent samples.

Interferences in the analytical system can be caused by contamination within the analytical instrument, active sites within the sample flow path, contaminated gases, contaminated water used for humidification, components of the sample matrix such as water or carbon dioxide, or instrument malfunctions.

11.3 Information Necessary to Validate Vapor Intrusion Data

The Data Validator will need the following information to complete the Tier I Data Validation Checklist #3 for validating soil gas, sub-slab vapor, or indoor air data:

- Sample date and start and end times
- Type of sample
- Sample method
- Analysis date
- Sample volume
- Canister pressure at the end of sample collection and when received at the lab
- Blank sample results
- LCS/LCSD sample results
- Field Duplicate and/or Field Spike results, if required per DQOs
- Calibration verification result, if necessary.

11.4 Vapor Intrusion Data Validation Criteria

The criteria that will be used to evaluate VI data are based on the following:

Technical Holding Times: The technical holding time requirement for canister samples is approximately 30 days from field collection to analysis. All results for samples analyzed outside of the technical holding time should be qualified as estimated (*i.e.*, any detected results should be qualified as "J" and non-detect results should be qualified as "UJ").

Blanks: A method blank should be analyzed at least once in each analytical batch to identify possible laboratory contamination and verify that potential interferences are acceptably low in the entire system. Blank actions for TO-15A are shown in Table 11.2.

Table 11.2: Blank Actions for TO-15 Analyses		
Qualification		
Blank Result	Sample Result	Action
Detected	Not detected	No Action
< QL	< QL (2x QL for common laboratory contaminants)	Report QL with a U
	≥ QL (2x QL for common laboratory contaminants) and < 2x QL (4x QL for common laboratory contaminants)	Report Sample Concentration with a U
	≥ 2x QL (4x QL for common laboratory contaminants)	No Action
> QL	< QL (2x QL for common laboratory contaminants)	Report QL with a U
	≥ QL (2x QL for common laboratory contaminants) and ≤ Blank Result	Report blank value for sample concentration with a U
	≥ QL (2x QL for common laboratory contaminants) and > Blank Result	No Action
= QL	≤ QL (2x QL for common laboratory contaminants)	Report QL with a U
	> QL (2x QL for common laboratory contaminants)	No Action
Gross Contamination*	Detects	Report blank value for sample concentration with a U

* Gross contamination is blank contamination > 2x the QL or 4x the QL for common laboratory contaminants.

LCS Recoveries: The LCS demonstrates that the laboratory instrument can produce accurate results. LCS recoveries within the acceptance range should not be qualified. If a sample contains a detectable quantity of compounds, but the LCS recovery is less than the lower acceptance limit, greater than the upper acceptance limit, or less than 50%, then these results should be considered estimated (flagged J). Non-detect results associated with an LCS recovery less than 50% should be rejected (flagged R). LCS actions for detected compounds are shown in Table 11.3.

Table 11.3: LCS/LCSD Actions for TO-15 Analyses		
Criteria	**Action**	
	Detected Associated Compounds	**Non-detected Associated Compounds**
Percent Recovery Criteria		
%R > Upper Acceptance Limit	J	No Action
%R in Acceptance Range	No Action	

%R < Lower Acceptance Limit	J	UJ
%R < 50%	J	R
Relative Percent Difference Criteria		
% RPD ≤ 25%	No Action	
% RPD > 25%	J	UJ

Duplicates: At a minimum, collocated or duplicate samples should be collected at a rate of approximately 5%. The relative percent difference between the parent sample and the collocated or duplicate sample should be greater than or equal to 25% when both results are 5X the MDL.

Chapter 12
Total Petroleum Hydrocarbons

12.0 Introduction

Per U.S. EPA, "*total petroleum hydrocarbons (TPH) is a term used to describe a large family of several hundred chemical compounds that originally come from crude oil. Because there are so many different chemicals in crude oil and in other petroleum products, it is not practical to measure each one separately. However, it is often useful to measure the total amount of TPH at a site*[1]."

TPH data may be used for delineation of bulk oil in the environment, product identification, forensic evaluation of a potential leak source or sources, estimation of risk or hazard to people and the environment, and/or selection of remedial options. In the context of a human health risk assessment, TPH data can be used to determine whether petroleum free product is present in the subsurface.

TPH is a mixture of chemicals which are made mainly from hydrogen and carbon, called hydrocarbons. TPH is often segregated into groups of petroleum hydrocarbons that act alike in soil or water. These groups are called petroleum hydrocarbon fractions. For the purposes of this guidance, we are generally only concerned with the gasoline range organics (GRO) and the diesel range organics (DRO) fractions. Each fraction contains many individual chemicals. The GRO fraction is the lighter fractionation which comprises compounds within the C6-C10 (six to 10 carbon atoms) range. The GRO designation can be a misnomer in the sense that the analytical results do not necessarily measure the presence of gasoline. The results simply indicate that the typical constituents that are found in a gasoline mixture may be present in the sample. The DRO fraction is considered the heavier fraction whereby the typically hydrocarbon compounds range between C10-C28. Similarly, analytical results indicate that the typical constituents that are found in a diesel mixture may be present in the sample.

12.1 Analytical Methods

When a laboratory uses an analytical method to determine TPH, the result is a measure of the general concentration of total petroleum within a sample. An analytical result might not be entirely petroleum, so careful examination of the meaning of the results is important. Some of the more common methods for the analysis of TPH include:

(1) Method 418.1 or Modified 418.1,

(2) Method 413.1 for oil and grease,

(3) Method 8015C for Diesel-Range Organics (DRO), and

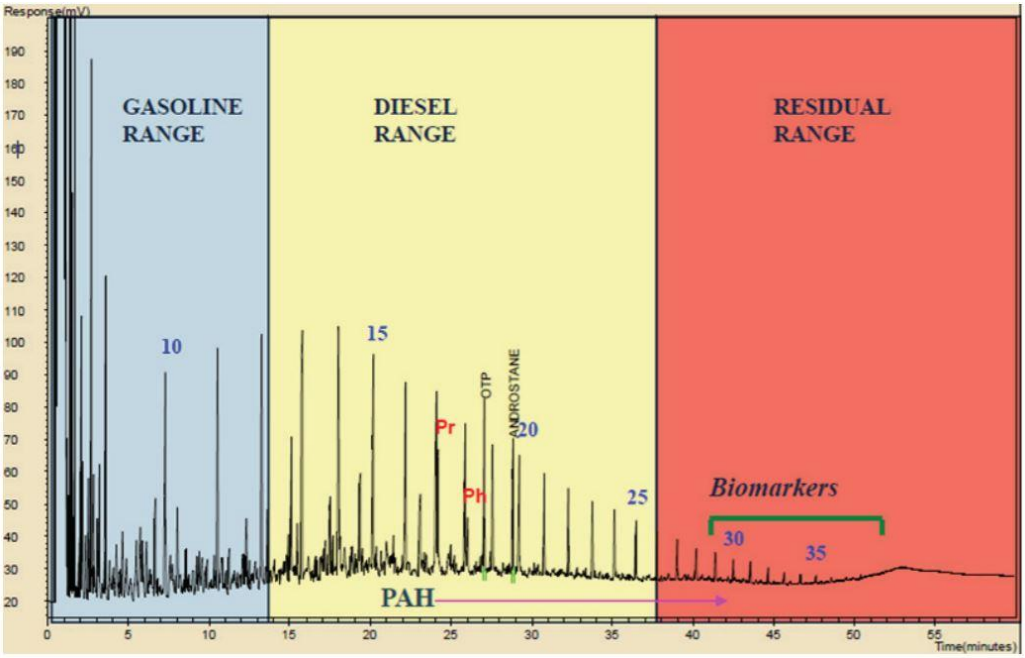
(4) Method 8015C for Gasoline-Range Organics (GRO).

[1]https://www3.epa.gov/region1/eco/uep/tph.html

Method 418.1 consists of solvent extraction followed by treatment in a silica gel column and infrared spectroscopy; Method 8015 for DRO and GRO are solvent extractions followed by gas chromatography. If it is suspected that the sample is predominately a gasoline (*i.e.*, volatile) fraction, purge and trap sample introduction to the gas chromatograph is often used in the determination of GRO. Method 413.1 is a gravimetric method that consists of solvent extraction, evaporation of the solvent, and a weight measurement. For the purposes of this guidance, Method 8015C will be the only TPH analytical method that is discussed going forward.

Method 8015C generates a representative concentration of the total concentration of nonhalogenated volatile (GRO) and semi-volatile (DRO) organic compounds in surface water, ground water, and solid matrices. If one is seeking to determine the concentration of an individual constituent, then the use of Method 8260 (volatiles) or Method 8270 (semi-volatiles) would be more appropriate analytical methods to run as opposed to the use of Method 8015C.

Figure 12.1 below shows a lab-generated chromatogram example which illustrates the generalized carbon ranges for GRO and DRO. Generally, a laboratory will use an algorithm to calculate the area under the response curve to generate a single TPH concentration for each carbon range fraction.



ITRC (Interstate Technology & Regulatory Council). 2018. *TPH Risk Evaluation at Petroleum-Contaminated Sites.* TPHRisk-1. Washington, D.C.: Interstate Technology & Regulatory Council, TPH Risk Evaluation Team. https://tphrisk-1.itrcweb.org.

Figure 12.1 Example Lab-Generated Chromatogram for GRO and DRO

12.2 Complications with Total Petroleum Hydrocarbon Data Validation

Validating TPH data is difficult because analyzing for TPH is a method-defined parameter. This means that depending on the method used, sample preparation and analysis, as well as data interpretation,

results can vary greatly. According to the Interstate Technical Regulatory Council (ITRC) TPH analytical data evaluations are highly variable and can be influenced by the following[2]:

- Potential Effects of Holding-Time Exceedances on TPH Results
- Potential Effects of Blank Detections on TPH Data Interpretation
- Potential Effects of Laboratory Control Sample Results on TPH Data Interpretation
- Potential Effects of Surrogate Recoveries on TPH Data Interpretation
- Potential Effects of Matrix Spike (MS)/MS Duplicates (MSD) on TPH Data Interpretation
- Variability in Evaluating and Interpreting Breakthrough
- Potential Effects of Co-Eluting Contaminants on TPH Results
- Potential Double Counting of Indicator Compounds in Fractionated TPH Data
- Potential Variability Associated with evaluating TPH Chromatograms

Further, depending on the analytical method used, there can be an overlap between the carbon number ranges of different hydrocarbon products when running independent TPH analytical methods. For example, a TPH method designed for gasoline range organics (*i.e.*, C6 to C12) may report some of the hydrocarbons present in diesel fuel (*i.e.*, C10 to C28). The same is also true for TPH analytical tests for diesel range organics which will identify some of the hydrocarbons present in gasoline-contaminated media.

Additional shortcomings identified from American Petroleum Institute (API Publication 4709) include potential validation issues related to:

- Contamination: Sample contamination may occur by diffusion of volatile organics through the septum seal during shipment and storage. The analysis of trip blanks may identify this problem.
- Matrix interferences: Since the FID is non-selective, there is a potential for the interference of non-target compounds.
- Memory Interferences: Carryover may occur whenever high and low concentration samples are analyzed in sequence.

12.3 TPH Data Validation Procedure

Due to the subjective nature of TPH data validation, the Data Validator must understand that the results obtained from the lab are subjective relative to the issues identified above. Therefore, TPH data is often used less for demonstration for compliance with applicable standards, and more so within the context of a screening tool for project site assessment or remediation. Similarly, TPH data validation is often subjective and greater scrutiny of the data may be required which would be beyond the scope of this guidance requiring a Tier II Data Validation.

[2] TPH Risk Evaluation at Contaminated Sites – Chapter 5, Conceptual Site Models (ITRC, November 2008). For a detailed discussion on TPH analytical data usability, interpretation, and implications refer to Sections 5.12.1 through 5.12.10)

The TPH-GRO and TPH-DRO checklists are primarily based on the Method SW 8260 (volatiles) and Method SW 8270 (semi-volatiles) checklists, respectively. The user is instructed to use the appropriate GRO and/or DRO checklist as necessary when validating TPH data. It may be appropriate to request data sheets and quality control information from the laboratory when there is insufficient detail in the data package to validate the data. Questions can be directed to ERAS-central office should the Data Validator need assistance.

Chapter 13
Cyanide and Hexavalent Chromium Analysis

13.0 Introduction

DERR evaluates data from ground water, soil, and waste samples for **cyanide and hexavalent chromium** analyses. These analyses are generally performed in specific instances and are not as common as analyses for other hazardous constituents. Samples for these constituents must be prepared and analyzed in specific ways, and therefore, data validation techniques differ from other data validation activities. For example, cyanide can exist in several forms, and there are specific tests that must be used to characterize each cyanide species. It is a component of the Appendix IX (OAC 3745-54-98) list of ground water monitoring constituents, but it is not listed with the constituents found in Table 1 of OAC 3745-51-24 for the Toxicity Characteristic. DERR evaluates cyanide in soil and ground water samples for human health risk assessment when it could be a chemical of concern or waste constituent at a site undergoing an investigation. Similarly, hexavalent chromium may be evaluated in waste, soil, or ground water analyses for human health risks. Hexavalent chromium analyses may also be used to determine whether a waste is exempt from hazardous waste regulation under OAC 3745-51-04. In this case, wastes which fail the TCLP test because chromium is the sole hazardous constituent can be excluded from hazardous waste management if it can be shown that chromium is primarily trivalent chromium. This chapter will provide an overview of the preparation and analytical methods used to quantify cyanide and hexavalent chromium in solid and liquid matrices. It will also outline those QA/QC requirements that are part of the preparation and analytical methods.

13.1 Cyanide Methods Summary

Cyanide, in its simplest free-form state, consists of a carbon atom and nitrogen atom that act as an anion in aqueous solution. It can form a variety of complexes depending on other constituents in aqueous solution and the solution's pH and oxidation/reduction state. These complexes can significantly affect the transport and toxicity of cyanide. For example, nearly insoluble metal-cyanide complexes, such as Prussian Blue (Fe4(Fe(CN)6)3), can bind cyanide to the soil. In addition, cyanide can sorb into organic matter and be sequestered in the soil column. While cyanide and its complexes can occur naturally, hazardous waste mismanagement, or leachate production from landfills, can notably degrade the environment. Simple, free-form cyanide (CN-) is toxic. Regional Screening Levels (RSLs) for free cyanide can be found in the current RSL tables, and VAP applicable standards for cyanide is located in Appendix A of OAC 3745-300-08.

Since cyanide can take on so many different forms in the environment, different analytical methods exist to quantify different forms of cyanide. Cyanide is usually measured as 1) free cyanide, 2) amenable cyanide, and 3) total cyanide. Free cyanide is a measure of cyanides in the simplest chemical form such as HCN, NaCN, or KCN. These molecular forms are easily soluble and can be readily extracted from aqueous or solid matrices. Amenable cyanides are cyanides amenable to chlorination, and these tests measure common metal cyanide compounds and complexes except for iron cyanides. Total cyanide is a measure of all cyanides, including iron-cyanide complexes.

SW-846 contains a variety of techniques for analyzing cyanide in soil, ground water, and wastes. SW-846 Method 9010C is an acid reflux procedure for water samples that yields total and amenable cyanide

concentrations when the extract is analyzed by SW-846 Method 9012B or 9014. For solid samples and wastes, SW-846 Method 9013A (an amendment to 9010C) extracts soluble cyanide from samples, which are then distilled and extracted with 9010C and analyzed by 9012B, 9014, or 9213. In general, a liquid sample is placed in a refluxing chamber with a strong acid. The acid/sample is continuously refluxed which effectively breaks down complexes liberating the cyanide in the form of HCN gas. This gas is swept into an alkaline scrubbing solution which can be analyzed colorimetrically or with an ion-selective electrode. In the colorimetric measurement, the cyanide is converted to cyanogen chloride (CNCl) by reacting with chloramine-T at a pH less than 8 without hydrolyzing to a cyanate. After the reaction is complete, color is formed on the addition of a pyridine-pyrazolone or pyridine-barbituric acid regent. The absorbance is read at 620 nm when using pyridine-pyrazolone and at 578 nm when using pyridine-barbituric acid. To obtain colors of comparable intensity, it is essential to have the same salt content in both the sample and the standards. Liquid samples effectively generate either total or amenable cyanide concentrations depending on sub-procedures in Method 9010C. Solid samples will yield primarily free cyanides, since the initial alkaline extraction is not strong enough to break down insoluble complexes from the solid matrix.

13.2 Quality Assurance/Quality Control for Cyanide

Since several forms of cyanide may be analyzed, there are a variety of method-specific factors that must be considered when reviewing a data report. These extra considerations are beyond the normal quality assurance and quality control procedures of other methods.

As only a few of these requirements will be discussed here, the reader is referred to the specific SW846 methods: 9010C, 9013A, 9012B, and 9213 when evaluating cyanide for a specific project. It is necessary to strictly adhere to an extraction method for full quantification.

Cyanide concentrations can suffer degradation from improper handling and transport. Aqueous samples must be preserved with a 50% sodium hydroxide solution until a sample pH of 12 is achieved. Samples should be chilled during transport and should not be exposed to light. If properly preserved, samples may be held for 14 days prior to preparation. Sample distillates should be analyzed as soon as possible after preparation.

Cyanide analyses may be subject to chemical interferences that can bias the sample results. Any oxidizing agent, such as chlorine, must be removed prior to distillation of the sample to avoid a negative bias. Methods 9010C and 9013A requires performing an oxidizer test and adding reducing agents to the sample prior to distillation. KI-starch paper is commonly used as a screening procedure for oxidizers. If the oxidizers are present, then reducing reagents, such as ascorbic acid, should be added to the sample until the starch paper indicates that reducing conditions are present. It is necessary to document that the KI-starch paper test was performed and the quantity of reducing reagent added to the sample. Conversely, samples with greater than 10 mg/L of nitrites and nitrates must be treated with sulfamic acid prior to distillation to avoid a positive bias. Once again, any sample treatment must be fully documented and discussed in the case narrative of the data report.

Methods 9010C and 9013A require the following quality control/quality assurance information be provided.

- A reagent blank should be analyzed per analytical batch (every 20 samples). This blank should include all reagents that were used in sample preparation.

- A check standard or Laboratory Control Sample (LCS) should be analyzed per batch and the result should be within 15% of the expected value. If the result is outside of this requirement, the sample should be reanalyzed.
- One sample should be replicated or duplicated per analytical batch. A duplicate is a separate aliquot of a sample that is taken through the preparation and analytical process. Method 9010C states that the Coefficient of Variation of the sample and its replicate should be within 20%. If these criteria are not met, then the samples should be reanalyzed.
- A matrix spike must be analyzed for every batch of 20 samples. This spike should have a concentration of approximately 40 µg/L. It is expected that matrix spike results should be within +/- 30 percent of the expected value (*i.e.*, 70% - 130% recovery).
- A high and a low standard should be distilled per analytical batch and compared to undistilled standard concentrations. The undistilled standards should be within +/- 10% of the distilled standards. If this was not performed or if the standards were not within +/- 10%, then corrective measures by the laboratory should be initiated before proceeding with cyanide analyses.
- The Method of Standard Additions (MSA) may be used when matrix interferences are suspected (i.e., matrix spike performance).

13.3 Information Necessary to Validate Cyanide Data

The Data Validator will need the following information to complete Checklist #10 for validating cyanide data.

- Sampling date
- Extraction/Preparation date
- Weight and/or volume of sample extracted
- pH of sample after necessary adjustments
- Spike sample results, including LCS and matrix spike data
- Calibration verification results
- Blank sample results, including reagent blanks and method blank data
- Method of Standard Additions information, if necessary

13.4 Cyanide Data Validation Criteria

The criteria that will be used to evaluate cyanide data are based on the following:

Preservation: Aqueous samples must be properly preserved using sodium hydroxide to reach a pH of greater than 12. If aqueous samples are received at the lab with a pH less than or equal to 10, then all detected concentrations should be qualified as estimated (J flagged), and all non-detects should be rejected (R). Cyanide samples should also be chilled to 4 °C ± 2, If samples were greater than 6° but less than or equal to 10°, then all detects and non-detects should be qualified as estimates (J flagged of UJ flagged, respectively). If samples were greater than 10°, then all detects should be qualified as estimated with a low bias (J- flagged), and all non-detects should be rejected (R). Table 14.1 shows actions for preservation for cyanide.

Technical Holding Times: The technical holding time requirement for both solid and aqueous samples is 14 days from field preservation to analysis. For detectable quantities of cyanide in samples exceeding 14 days, the results should be considered estimates and data flagged with a

J. All non-detect samples that exceed the technical holding time should be rejected (R). See Table 14.1 for holding time actions.

Interference: Cyanide methods identify interferences that may impact the quality of the results. Oxidizing agents, such as chlorine, decompose most cyanides. Chlorine interferences can be reduced or eliminated by adding a surplus of sodium arsenite before to preserving and storing the sample to reduce chlorine to chloride, which does not interfere. Samples that contain hydrogen sulfide, metal sulfides, or other compounds that may produce hydrogen sulfide may release hydrogen sulfide when the samples are distilled. Sulfide interference can be removed by adding a surplus of bismuth nitrate to the sample before it is distilled to precipitate the sulfide. Results may be biased high for samples that contain nitrate and/or nitrite. Nitrate and nitrite for nitrous acid during distillation, and nitrous acid reacts with some organic compounds to form oximes, which decompose to generate HCN. This interference can be eliminated by pretreating samples with sulfamic acid just before distillation. Table 13.1 shows actions based on interference.

Table 13.1: Preservation and Holding Time Actions

Criteria	Actions	
	Detect	Non-detect
Samples properly preserved and analyzed within specified holding time	No qualification	No qualification
Aqueous/water samples received with pH ≤ 10	J	R
Aqueous/water and soil/sediment/waste samples received or stored at a temperature > 6°C but ≤ 10°C *	J	UJ
Aqueous/water and soil/sediment/waste samples received or stored at a temperature > 10°C*	J-	R
Technical Holding Time: Aqueous/water and SPLP leachates > 14 days	J	R
Technical Holding Time: Soil/sediment/waste samples > 14 days	J	R
Aqueous/water samples received with oxidizing agents present	J	R
Aqueous/water samples received with sulfides present	J	R
Aqueous/water samples received with nitrate/nitrite present and not treated with sulfamic acid	J	R

Blanks: Blanks are required for cyanide analyses. An initial calibration blank should be run just after the calibration sequence but before a verification sample or project samples are analyzed. In addition, a method blank which uses the same reagent and is carried through the distillation process must be analyzed and reported with every batch of samples. Blank actions for cyanide are shown in Table 13.2.

Table 13.2: Blank Actions for Cyanide

Blank Result	Sample Result	Action

Detect ≤ QL	Non-detect	No qualification
	Detect ≤ QL	Report at QL and qualify as U
	> QL	J+ or no qualification
> QL	Non-detect	No qualification
	Detect ≤ QL	Report at QL and qualify as U
	> QL but < 10x the Blank Result	Report at Blank Result and use professional judgment to qualify results as J+ or R
	≥ 10x the Blank Result	No qualification

* Project-specific QAPPs may allow use before assessing any actions for the affected samples for samples that are received with shipping container temperatures greater than 10 °C.

Duplicate Recovery: One field sample should be used as a duplicate and analyzed from each batch (every 20 samples) of a similar matrix aqueous or solid. The coefficient of variation (relative percent difference) for the sample and its replicate should be 20% or less. According to methods 9010C and 9013, if these criteria are not met, then the samples in the batch should be reanalyzed. Duplicate sample actions for cyanide are shown in Table 13.3.

Table 13.3: Duplicate Sample Actions for Cyanide

Criteria	Action	
	Detect	Non-detect
Both original sample and duplicate sample results are ≥ 5x the QL and RPD > 20%*	J	UJ
RPD > 100%	Use professional judgment	Use professional judgment
Both original sample and duplicate sample results are ≥ 5x the QL and RPD ≤ 20%	No qualification	No qualification
Original sample or duplicate sample result < 5x the QL (including non-detects) and absolute difference between sample and duplicate > QL*	J	UJ
Original sample or duplicate sample result < 5x the QL (including non-detects) and absolute difference between sample and duplicate ≤ QL	No qualification	No qualification

* Project DQOs may allow the use of less restrictive criteria (e.g., 35% RPD, 2x the QL) to be assessed against duplicate soil samples due to laboratory variability arising from the sub-sampling of non-homogenous soil samples.

LCS Recoveries: The LCS demonstrates that the laboratory instrument can produce accurate results using a standard clean aqueous or solid matrix. LCS recoveries within 85% to 115% should not be qualified. If a sample contains a detectable quantity of cyanide but has an LCS recovery of 50% to 85% or 115% to 150%, then these results should be considered estimated (flagged J). Non-detect samples in these LCS recovery ranges should also be considered as estimated (flagged UJ). Data associated with LCS recovery below 50% or above 150% should be rejected (flagged R). LCS actions for cyanide are shown in Table 13.4

Table 13.4: Lab Control Sample Actions

Criteria	Action	
	Detect	Non-detect
LCS not prepared with samples	J or R	UJ or R
LCS not prepared at specified concentrations	J	UJ
Aqueous/water and soil/sediment %R < 50%	J-	R
Aqueous/water and soil/sediment %R 50-84%	J-	UJ
Aqueous/water and soil/sediment %R 85-115%	No qualification	No qualification
Aqueous/water and soil/sediment %R 116-140%	J+	No qualification
Aqueous/water and soil/sediment %R > 140%	R	No qualification

Matrix Spikes: Matrix spikes are performed to evaluate the effect of each sample matrix on the sample preparation procedures and the measurement methodology. At least one spiked sample (pre-distillation) should be prepared and analyzed for each batch of samples with a similar matrix type (e.g., aqueous/water or soil/sediment/waste). Table 13.5 shows matrix spike actions for cyanide.

Table 13.5: Matrix Spike Actions for Cyanide

Criteria	Action	
	Detect	Non-detect
Matrix Spike not performed at the specified frequency	J	UJ
Matrix Spike not prepared from a field sample	J	UJ
Matrix Spike %R < 30% Post-distillation spike %R < 75%	J-	R
Matrix Spike %R < 30% Post-distillation spike %R ≥ 75%	J	UJ
Matrix Spike %R 30-74% Post-distillation spike %R < 75%	J-	UJ
Matrix Spike %R 30-74% Post-distillation spike %R ≥ 75%	J	UJ
Matrix Spike %R > 125%	J+	No qualification

Post-distillation spike %R > 125%		
Matrix Spike %R > 125% Post-distillation spike %R ≤ 125%	J	No qualification
Matrix Spike %R < 30% No post-distillation spike performed	J-	R
Matrix Spike %R 30-74% No post-distillation spike performed	J-	UJ
Matrix Spike %R 75-125% No post-distillation is required	No qualification	No qualification
Matrix Spike %R > 125% No post-distillation spike performed	J+	No qualification

NOTE: Project DQOs may allow the use of less restrictive criteria (*e.g.*, 10 %R and 150 %R for the lower and upper limits) to be assessed against spike soil samples due to laboratory variability arising from the sub-sampling of non-homogenous soil samples.

High And Low Calibration Standard Verification: Method 9010C recommends that a high and low standard be distilled and analyzed per batch of samples. This procedure can be used to show the distillation technique is reliable by comparing to similar values on the curve. If distilled standards are not within 10% of undistilled standards, then the cause of the error should be identified before proceeding. Standard verification also establishes the linearity of the curve and can be used to confirm the reporting limit used by the laboratory.

While the evaluation of two standards is considered optional, the laboratory must confirm the calibration curve with an initial calibration verification (ICV) standard and with continuing calibration verification (CCV) standards. Instruments should be calibrated daily or as specified in the project specific QAPP. CCV standards should be analyzed at the frequency specified in the project specific QAPP or at every hour during an analytical sequence. The CCV standard should also be analyzed at the beginning of the analytical sequence and after the last analytical sample. In most cases, the standard should have a concentration near the mid-point of the linear range when a single standard calibration verification test is performed. If the percent recovery (%R) is outside control limits, the laboratory should terminate sample analysis, recalibrate the instrument until acceptable recoveries are verified, and reanalyze all affected samples. If calibration is not performed at the specified frequency, then all results should be rejected. If calibration standards are not distilled, then results should be qualified as estimated (J or UJ). If calibration is incomplete (i.e., there is an insufficient number of standards or required concentrations are missing), then results should be qualified as estimated or rejected based upon professional judgement or project DQOs. If recalibration or reanalysis is not performed, affected samples need to be qualified using the following criteria:

Table 13.6: Calibration Actions for Cyanide

	%R <70%	**%R 70%-84%**	**%R 85%-115%**	**%R 116-130%**	**%R >130%**
Detect	J- or R	J-	Acceptable	J+	J+ or R
Non-detect	UJ or R	UJ	Acceptable	Acceptable	Acceptable

The correlation coefficient of the calibration curve is greater than the value specified in the project specific QAPP for linear fits. Results should be qualified as estimated when correlation coefficients are less than 0.995 or the precent differences are outside 30% or other limits specified in a project specific QAPP.

13.5 Hexavalent Chromium Method Summary

Chromium can exist in nature in a variety of oxidation states including Cr+3 (Cr III), Cr+5 (Cr V), and Cr+6 (Cr VI or hexavalent chromium). The predominant oxidation state of chromium in the environment is Cr III where it occurs as barely soluble oxides and hydroxide species. Cr VI can also occur naturally but is commonly associated with releases to the environment from industrial activities or anthropogenic sources. Cr VI is of special concern as this chromium species is soluble and can be transported under natural conditions into ground water where it may be ingested by human and other ecological receptors. Cr VI is the most toxic form of chromium because it mimics sulfur (sulfur in the plus six oxidation state) and can readily enter into cellular membranes.

This increase in toxicity can be readily seen in Ohio EPA's Generic Cleanup Numbers (GCNs) for direct contact in soil. GCNs for Cr III and Cr VI in soil are 9.54e+4 mg/Kg and 2.02e+2 mg/kg respectively which indicate an approximately 100-fold decrease in the allowable concentration of chromium if the dominate species is Cr VI.

In most cases where chromium can be a constituent of concern, knowledge of the oxidation state is not a primary data quality objective. For example, the Maximum Contaminant Level (MCL) for drinking water is based upon a total chromium concentration, not by the relative concentration level of Cr VI. However, in some situations the determination of chromium species can be important. For example, generators that have wastes that fail the toxicity characteristic for chromium can demonstrate, in part, that the waste should be excluded from hazardous waste management if the chromium in the waste is exclusively trivalent (ORC 3745-51-04(B)(6)(a). In addition, some facilities find it desirable to determine the species of chromium present in various media because it may more accurately represent the human health risk.

There are a variety of methods that are available to determine hexavalent chromium in water, soil, and waste. The methods for hexavalent chromium in SW-846 are listed in Table 13.7.

Table 13.7 Table of SW-846 Methods for the Preparation and Quantification Hexavalent Chromium

SW-846 Method Number	Method Title
3060A	Alkaline Digestion for Hexavalent Chromium
7195	Chromium, Hexavalent (Co precipitation)
7196A	Chromium, Hexavalent (Colorimetric)
7197	Chromium, Hexavalent (Chelation/Extraction)
7198	Chromium, Hexavalent (Differential Pulse Polarography)
7199	Determination of Hexavalent Chromium in Drinking Water, Groundwater and Industrial Wastewater Effluents by Ion Chromatography

While all the methods listed in Table 13.7 are available to Ohio EPA or to a regulated facility, Method 7196A is the most commonly used analytical method for hexavalent chromium. If soil or solid waste is to be analyzed with this method, it must first be extracted with SW-846 method 3060A. Method 3060A must be followed carefully to prevent biasing analytical results due to improper handling of the samples. Method 7196A employs colorimetry to quantify hexavalent chromium in aqueous samples or soil and waste extracts. This method is based upon the reaction of hexavalent chromium with diphenylcarbizide in an acid solution, which produces a red-violet product. The absorbance of 450 nm wavelength light is measured photometrically and compared to a calibration curve. The concentration of the sample can then be determined. A detailed summary of the solid extraction procedure and analytical procedures are presented in the following paragraphs.

13.5.1 Method 3060A, Alkaline Digestion Procedure for Soils and Solid Wastes.

Method 3060A is the preferred extraction procedure for soils and solid wastes that can be used in conjunction with methods 7196A and 7199 (listed in Table 13.7). According to the method, "to quantify total Cr VI in a solid matrix, three criteria must be satisfied: (1) the extracting solution must solubilize all forms of Cr VI, (2) the conditions of the extraction must not induce reduction of native Cr VI to Cr III, and (3) the method must not cause oxidation of native Cr III contained in the sample to Cr VI." The method's procedures reliably perform these tasks. The alkaline solution can solubilize hexavalent chromium from a solid matrix and also minimizes oxidation or reduction of chromium. The method also contains testing procedures to determine whether oxidizer components are present in the matrix of the sample and prescribes the addition of an alkaline buffer containing Mg+2 to prevent sample oxidation.

Method 3060A is unique in that it prescribes that the potential for oxidation/reduction is assessed, in part, by measuring additional soil or waste properties, such as Oxidation Reduction Potential (ORP, ASTM Method D 1498-93), pH (SW-846 Method 9045D), ferrous iron (ASTM Method D3872-86), and sulfide (SW-846 Method 9030B). Other indicators may also be used such as chemical oxygen demand and biological oxygen demand. Because of these additional tests, the necessary soil or waste sample volume must be assessed prior to sampling. For soil and solid waste, the measurement of sample specific parameters such as ORP and pH establishes the tendency of Cr VI to exist or not exist in the unspiked sample(s) and assists in the interpretation of QC data for matrix spike recoveries outside conventionally accepted criteria for total metals. If oxidizing conditions are indicated from the testing procedure in Method 3060A, then the addition of Mg+2 is necessary. Section 3.3 of this method goes on to indicate that special precautions are necessary for soils or wastes that contain soluble chromium. Section 3.3 states, for waste materials or soils containing soluble Cr III concentrations greater than four times the laboratory Cr VI reporting limit, Cr VI results obtained using this method may be biased high due to method-induced oxidation. The addition of Mg+2 in a phosphate buffer to the alkaline extraction solution has been shown to suppress this oxidation. Soluble Cr III can be tested for by performing an extraction using distilled water as the extracting agent.

Maintaining the proper pH through the digestion process is critical. Samples are digested using a sodium carbonate/sodium hydroxide solution that is heated for 60 minutes at 90 degrees centigrade. The efficiency of the procedure to digest both soluble and insoluble chromium is measured through the use of spikes ($K_2Cr_2O_7$ and $PbCrO_4$) that are carried throughout the digestion process.

13.5.2 Method 7196A, Chromium Hexavalent (Colorimetric)

Method 7196A is a colorimetric method that depends upon the reaction of Cr VI with diphenylcarbazide. A calibration curve is developed using stock reagents that are carried through the same digestion procedures as the samples. The calibration curve should be developed daily. Diphenylcarbazide is first added to aqueous samples and soil digestates then acidified to a pH of 2.5 with sulfuric acid. The laboratory should provide proper documentation that this pH was achieved since color development must take place under acidic conditions. Because of some samples' matrices, turbidity may also be a problem. If turbidity is encountered, the laboratory should develop a blank from another portion of the digestate that does not contain diphenylcarbazide.

The absorbance from this blank should be used to correct the reading of the actual sample. Method 7196A is a fairly robust method and not subject to significant interferences. Hexavalent mercury and molybdenum can interfere, but only at significantly high (>200 mg/L) concentrations.

13.6 Hexavalent Chromium Quality Control

Soil and water samples should be collected with non-stainless sampling devices and stored at 4 +/- 2 degrees centigrade until sample extraction (soil or waste) or analysis (aqueous samples). Aqueous samples should be analyzed within 24 hours of collection. Technical holding times for Cr VI are only established for water, but method (3060A) suggests that soil samples can be stored for up to 30 days prior to digestion, when chilled properly, and then must be analyzed within 7 days after digestion. The QA requirements for solid and water samples vary. The following sections illustrate the requirements for these media.

13.6.1 Quality Control Requirements for Soil and Solid Wastes (Method 3060A)

Method 3060A requires that a preparation blank (method blank) be prepared and analyzed for every batch of samples. The criteria used to evaluate this data are different than for most blanks. The preparation blank must not contain detectable Cr VI (i.e., below the detection limit) or not be greater than 10 percent of the regulatory limit or action limit. If these criteria are not satisfied, then the entire batch must be re-digested.

Soil samples prepared by method 3060A and analyzed by method 7196A should show that the digestate's pH has been adjusted to 7.0 +/- 0.5 units. According to the method (Section 7.7), if this adjustment hasn't been made or if the pH of the digestate is outside of the prescribed range, the digestate should be discarded and a new sample aliquot digested. In addition, soil or waste samples should have one sample in the batch duplicated. This means that a separate aliquot of a sample should be taken, digested and analyzed. The sample and its duplicate should agree within a 20% relative percent difference (RPD). Method 3060A prescribes that both a soluble and insoluble matrix spike be analyzed per batch of samples. The soluble matrix spike should be composed of K2Cr2O7 (at least 40 mg of Cr VI added as a spike) and the insoluble matrix spike composed of PbCrO4 (10 to 20 mg added in the spike). These spikes are added to separate aliquots of a sample in the batch and carried through the digestion process. The criteria used to judge the acceptability of these spikes, and therefore the digestion process, is a percent recovery of 85% to 125%. According to section 8.5 of method 3060A, if the matrix spikes have recoveries that are not within the prescribed acceptance criteria, then the entire batch of samples should be discarded and samples re-digested and re-analyzed. If upon reanalysis, the matrix spike is still outside of criteria, but the LCS is within criteria, method 3060A requires that ancillary parameters be evaluated. These ancillary parameters include the determination of field ORP (Eh) and

pH. If these parameters were not taken in the field, then the time of analysis should be noted. In addition, analyses for COD, BOD and various redox couples (ferric iron and ferrous iron ratio) may also be made. These parameters can help to interpret whether the matrix is oxidizing or reducing. Eh – pH information should be plotted on Table 2 in SW-846 Method 3060A. The position of data plotted on this diagram will give an indication of a sample's oxidizing or reducing state. If the LCS was within acceptance criteria and the pre-digestion matrix spike recoveries for Cr VI were less than the acceptance range minimum (75%), this indicates that the soil samples reduced Cr VI (e.g., anoxic sediments), and no measurable native Cr VI existed in the unspiked sample.

If the data indicate that the sample is not reducing in nature, but the matrix spike is outside of lower criteria (i.e., less than 75%), then additional ancillary parameters data may be used to indicate the cause of the matrix spike failure. Data may be qualified based upon the percent recovery and the LCS data. Alternately, section 8.5 of Method 3060A states "If a low or zero percent pre-digestion matrix spike recovery is obtained, an alternate approach can be used to determine the potential contribution of the sample matrix to Cr VI reduction. This approach consists of performing a mass balance, whereby total chromium is analyzed (Method 3052) for two samples: (1) a separate unspiked aliquot of the sample previously used for spiking, and (2) the digested solids remaining after the alkaline digestion and filtration of the matrix spike (i.e., the filtered solids from the matrix spike in Section 7.6).

The difference between the total chromium measurements should be approximately equal to the amount of the spike added to the matrix spike. If the LCS met the acceptance criteria and the Cr VI spike is accounted for in the filtered solids as total chromium, it is likely that the reduction of the Cr VI to insoluble Cr III resulted from the reducing matrix of the original sample subjected to Cr VI spiking."

A post-digestion spike per batch is required for soil or other solid wastes. The criteria range for acceptance recommended by Method 3060A is a percent recovery between 85% and 115%. If the acceptance criteria are not met, the laboratory should perform the Method of Standard Additions (MSA). If the MSA technique is applied and no spike is observed from the MSA, then these results indicate that the matrix is incompatible with Cr VI.

13.6.2 Quality Control Requirements for Aqueous Matrix

Water or aqueous waste samples require verification that the sample matrix is not unduly biasing the analytical results. The method allows for samples to be blank corrected and also specifies that analytical results can be corrected for turbidity through the analysis of a turbidity blank (sample aliquot that is prepared as usual but does not contain diphenylcarbizide).

Verification is required by the method to ensure that neither a reducing environment nor chemical interference is affecting the analytical results. This evaluation is accomplished by analyzing a second 10-mL aliquot of the pH-adjusted filtrate that has been spiked with Cr VI. The amount of spike added should double the concentration found in the original aliquot. Under no circumstances should the increase be less than 30 µg of Cr VI/liter. To verify the absence of interference, the spike recovery must be between 85% and 115%. Acidic extracts that yield recoveries of less than 85% should be retested to determine if the low spike recovery is due to the presence of residual reducing agent. This determination shall be performed by first making an aliquot of the extract alkaline (pH 8.0 - 8.5) using 1 N sodium hydroxide and then re-spiking and analyzing the aliquot. If a spike recovery of 85-115% is obtained in the alkaline

aliquot of an acidic extract that initially was found to contain less than 5 mg/L Cr(VI), it can concluded that the analytical method has been verified.

If these criteria are not met, upon verification, an alternate method should be chosen to quantify Cr VI in the sample.

13.7 Quality Assurance and Quality Control Samples

Method 7196A requires the following quality control/quality assurance information be acquired.

- A minimum of one blank should be analyzed per batch of samples.
- A continuing calibration standard should be analyzed every 15 samples. The criteria for verification is 80 to 120% recovery of the standard.
- A matrix spike and/or a replicate sample should be analyzed in every batch.
- The Method of Standard Additions should be used for all extracts and for any samples submitted for delisting petitions.

13.8 Information Necessary to Validate Hexavalent Chromium Data

The Data Validator will need the following information to validate hexavalent chromium data:

- Sampling date
- Chain of custody
- Sample receipt log
- Extraction/Preparation date
- Analysis date
- pH of sample after necessary adjustments
- Spiked sample results, including high/low Cr VI spikes for solid material, LCS and matrix spike data
- Interference and oxidizing ancillary data
- Calibration verification results
- Blank sample results, including reagent blanks and method blank data
- Method of Standard Additions information, if necessary

13.9 Data Validation Criteria

The criteria used to validate data are based on whether the sample was solid or aqueous. Data validation criteria are shown below:

1. **Sample Collection and Technical Holding Times**

   Solid material must be collected using non-metallic sampling devices and placed, without head space, in a glass sampling container with a Teflon lid. Samples should be maintained at 4.0 +/- 2 degrees Centigrade and digested within 30 days. Analysis must occur within 7 days after digestion. If technical holding time criteria are not met, then all positive results should be qualified as estimated (J-) and all non-detections should be qualified as estimated. However, if the holding times are greatly exceeded, then the validator may reject all non-detections based upon professional judgment and the project's data quality objectives. If soil was also collected for soil pH and other ancillary parameters (i.e., ORP, other redox couples), these parameters should be analyzed in the field or within 24 hours.

2. Preparation

Solid samples must be pretreated/digested prior to analysis. SW-846 7196A/3060A requires that the pH of alkaline digestates of solid samples must be maintained at 7.5 +/- 0.5, as stated in Section 7.7. If the laboratory failed to maintain the pH, the sample should be re-digested. If pH issues are present with the data, the laboratory must be

contacted to supply supporting information/explanations. If the laboratory cannot provide the information or if data exists to indicate that the proper pH was not maintained, the sample results should be rejected.

3. Blanks

A preparation blank must be prepared and analyzed with each digestion batch. Detected Cr VI concentrations must be less than the method detection limit or one-tenth the regulatory limit or action level, whichever is greater, or the entire batch must be re-digested. If detectable quantities of Cr VI are found in the blank, then the 10X rule can be applied to determine whether the amount is significant enough to bias sample results. If detectable Cr VI is found in the blank and, upon application of the 10X rule, the result is greater than the Cr VI result in the sample, the sample result should be qualified as undetected, and data flagged "UJ". If after application of the 10X rule, the result is below the detected quantity in the sample, the data should be considered valid and not qualified. The Table below can be used to determine Preparation Blank actions.

Table 13.8: Preparation Blank Actions for Hexavalent Chromium

Blank Result	Sample Result	Action
Not analyzed at specified frequency	Non-detect	UJ
	Detect	J
Detect < QL	Non-detect	No qualification
	Detect < QL	Report at QL and qualify U
	Detect > QL	J+ or no qualification
≥ QL	Non-detect	No qualification
	Detect < QL	Report at QL and qualify U
	≥ QL but < 10x the Preparation Blank Result	Report at Preparation Blank Result and qualify J+ or R
	≥ 10x the Preparation Blank Result	No qualification

4. Laboratory Control Sample

One laboratory control sample (LCS) should be analyzed per batch of samples per matrix. The concentration of Cr VI in the LCS should be near the mid-point of the calibration curve. The LCS must utilize the matrix spike solution or the solid matrix spiking agent PbCrO4 (Section 5.6 of method 3060A) to spike into 50 mL of digestion solution (Section 5.7 of method 3060A). Alternatively, the use of a certified solid reference material (if available) is recommended. The criteria for acceptance is a percent recovery between 80 and 135%. If the LCS is outside of the acceptance criteria, the batch of samples should be re-digested and re-analyzed. If the acceptance criteria are not met and the results are reported, the results should be qualified based on the following table:

Table 13.9: LCS Actions for Hexavalent Chromium

	<65%	65%<%R>80%	80%<%R>120%	120%>%R<135%	>135%
Detection	Reject, R	Estimated, J-	Acceptable	Estimated, J+	Reject or estimated*
Non-detection	Reject, R	Estimated, UJ	Acceptable	Acceptable	Acceptable

* Sample results may be rejected based upon professional judgment of the reviewer and the project's data quality objectives.

This table may also be of assistance with LCS actions.

Table 13.10: Additional Criteria and LCS Actions for Hexavalent Chromium		
Criteria	**Action**	
	Detect	**Non-detect**
LCS not prepared with samples	J	UJ
LCS not prepared at specified concentration	J	UJ
Aqueous/water %R < 40%	J-	R
Aqueous/water %R 40-79%	J-	UJ
Aqueous/water %R 80-120%	No qualification	No qualification
Aqueous/water %R 121-150%	J+	No qualification
Aqueous/water %R > 150%	R	No qualification

5. Matrix Spike and Sample Duplicate for Aqueous Samples

A matrix spike (mid-level of the calibration curve) or sample duplicate should be analyzed for every 10 samples (method 7196A, section 8.5). The acceptance criteria for spikes should be within 85-115% recovery. Duplicate sample reproducibility is not discussed in the method. If sample duplication is used, the laboratory should establish criteria for validation. If the matrix spike recovery is outside of the criteria, the laboratory should analyze a post-digestion spike to confirm a matrix interference. Alternately, the Method of Standard Additions can be performed to determine the concentration of Cr VI in the sample. Validation of sample results depends on the results of the LCS. If the LCS recovery is outside of its established criteria, then the reviewer may either qualify results as estimated or reject the results based upon the project's data quality objectives. The table below can aid in determining Duplicate actions.

Table 13.11: Duplicate Analysis Actions for Hexavalent Chromium		
Criteria	**Action**	
	Detect	**Non-Detect**
Duplicate analysis is required by the QAPP, but not performed at the specified frequency	J, or use professional judgement	UJ, or use professional judgement
Both original sample and duplicate sample results are ≥ 5x QL and RPD > 20%	J	UJ
Both original sample and duplicate sample results are ≥ 5x QL and RPD ≤ 20%	No qualification	No qualification

RPD > 100%	Use professional judgement	Use professional judgement
Original sample or duplicate sample results < 5x QL (including non-detects) and absolute difference between sample and duplicate > QL	J	UJ
Original sample or duplicate sample result < 5x QL (including non-detects) and absolute difference between sample and duplicate ≤ QL	No qualification	No qualification

6. Matrix Spikes (soluble and insoluble) for Solid Matrices

According to SW-846 Method 3060A, the analysis of solid matrices requires that both at least one soluble and insoluble pre-digestion matrix spikes be analyzed for every batch of samples. The acceptance range for spike recovery is 75% to 125%. If either spike is outside of control, then re-digestion and re-analysis of the batch should have occurred. If upon re-digestion and re-analysis it is found that the spike recovery(-ies) were still outof control, the LCS results should be reviewed. If the LCS is acceptable, the reviewer should use the following procedure to examine the pre-digestion spike result(s). First, the pH/Eh of the sample should be evaluated using Figure 2 in Method 3060A. Alternatively, the lab can perform a mass balance as described in Section 8.5.2 of SW-846 3060A. If reducing conditions exist, no further action is required. If reducing conditions do not exist, re-analyze the pre-digestion matrix spike(s). If results are acceptable, no further action is required. If matrix spike(s) recovery is between 50 and 74% or >125% and the LCS was in control, no corrective action is required, but samples should be qualified as estimated (J or UJ). If pre-digestion matrix spike(s) recovery is <50% and associated with non-detected results, the non-detected results may be qualified as rejected by the reviewer.

7. Method of Standard Additions (MSA)

The standard addition technique involves adding known amounts of standard to one or more aliquots of the processed sample solution. This technique compensates for a sample constituent that enhances or depresses the analyte signal, thus producing a different slope from that of the calibration standards. The MSA will not correct for additive interferences which cause a baseline shift. The MSA is used for the analysis of all extracts, on all analyses submitted as part of a delisting petition, and whenever a new sample matrix is being analyzed.

Chapter 14
TCLP Extraction

14.0 Introduction

The toxicity characteristic leaching procedure (TCLP) is used to determine the mobility of selected hazardous constituents in wastes. TCLP extraction mimics conditions found in a landfill and attempts to quantify the threat a waste would potentially pose to the environment. Wastes are deemed to be hazardous if they contain extractable levels of constituents at or above certain thresholds, as defined in Ohio Administrative Code (OAC) rule 3745-51-24. TCLP levels for a small number of metals, semi-volatile, and volatile organic compounds are defined in Table 1 of this regulation. The TCLP extraction procedure is defined in SW-846, Method 1311.

TCLP is specifically referred to in the hazardous waste regulations and, therefore, the procedure must be strictly followed. The Tier I Data Validator may have difficulty reviewing TCLP data since most extraction procedure information will be found only in the bench sheets, not in the data report. One of the first steps to completing a data validation of TCLP data may be to request these bench sheets, if they are not provided with the report. Furthermore, the method encompasses not one, but many procedures. The exact procedure used for a sample will depend upon the material extracted, pH of the waste and the analytical parameters.

The Tier I Data Validator must keep in mind that SW-846, Method 1311 is a preparatory procedure, not an analytical procedure. The analytical methods that will accompany TCLP will be the same methods as those used for total constituent analysis, such as SW-846, Method 6010C for metals. Therefore, data validation must include not only the TCLP extraction procedure, but also the QA/QC parameters that are included for each method used to analyze the extract.

14.1 Method Summary

The first step in the extraction process is to characterize the waste as a liquid, solid or semi-solid. If the waste contains less than 0.5 percent solids, it is deemed a liquid and this liquid is defined as the TCLP extract. If the samples contain greater than 99.5 percent solids, the waste is extracted with the appropriate amount and type of extraction fluid and analyzed by the appropriate analytical method.

If a waste contains more than 0.5 but less than 99.5 percent solids, (i.e., semisolid) the liquid portion is retained for analysis, and the solid portion is placed in extraction fluid equaling 20 times the weight of the solid phase. Next, the solid materials must be examined for particle size and filtered. Particles are measured with a ruler and must be less than 1 cm diameter. The sieve is not used to verify particle size for the volatile sample. Both the solid material extract and liquid portions of the waste are analyzed separately and mathematically recombined. Alternately, the multi-phased components may be physically recombined prior to analysis.

The extraction fluid is made of two different strengths of acetic acid depending upon the alkalinity of the solid material. A test must be performed on each waste sample to make this determination. Type 1 Extraction Fluid (fluid #1) is used for samples to be analyzed for VOCs or waste that is acidic to slightly basic. VOC extraction is performed with a special device known as a Zero Head space Extractor Vessel or a ZHE. Type 2 Extraction Fluid (fluid #2) is used if waste is highly alkaline. Both the solid material extract

and liquid portions of the waste are analyzed separately and then mathematically recombined. Alternately, the multi-phased components may be physically recombined prior to analysis. The extraction is performed by placing the extraction vessel in a rotary agitator at 30 +/- 2 rpm for 18 +/- 2 hours. The ambient temperature is maintained at 23 +/- 2°C during agitation. The extracts are defined in more detail below.

14.2 QA/QC Specific Information

The Tier I Validator must pay particular attention to the purpose of TCLP. In addition to waste characterization, TCLP is used to determine if treated wastes meet Land Disposal Restrictions (LDR) (OAC 3745-270). The LDR regulatory levels are very different than the hazardous waste characteristic evaluation. In addition, the Tier I Validator must be aware that there are additional QA/QC requirements for TCLP compared to the normal analytical methods. These tests include:

1. **TCLP Extraction Blanks**: A minimum of one TCLP extraction blank is generated for every 20 extractions processed in a given extraction vessel using the same fluid. Most labs have multiple extraction vessels. The common industry strategy is to generate one TCLP extraction blank for each group of samples processed simultaneously using the same batch of fluid.
2. **Method of Standard Addition**: Four equal-volume, pre-digestion aliquots of sample are measured and known amounts of standards are added to three aliquots. The fourth aliquot is the unknown and no standard is added to it. The concentration of standard added to the first aliquot must be 50% of the expected concentration. The concentration of standard added to the second aliquot must be 100% of the expected concentration, and the concentration of standard added to the third aliquot must be 150% of the expected concentration. The volume of the unspiked and spiked standard must be the same.

   The Method of Standard Addition is to be used for metallic contaminant determinations if both of the following criteria are met:

   - The matrix spike recovery from the TCLP extract is less than 50% and the unpicked sample concentration is less than the regulatory level.
   - The contaminant measured in the sample is within 20% of the regulatory level.

   For the method of standard additions to be correctly applied, the following limitations must be taken into consideration: the plot of sample and standards must be linear over the concentration range of concern, and the effect of the interference must not vary as the ratio of the standard added to the sample matrix changes.

3. **Holding Times**: The holding times outlined in Table 14.1 must be met. Sample results must be evaluated for both time-until-extraction and time- until- analysis. Sample data that exceed holding times are not acceptable for verifying that a waste does not exceed regulatory levels. However, if TCLP extract concentrations exceed regulatory action levels, and holding times are exceeded, the data are considered minimum values, and the data are considered valid.

Table 14.1 Technical Holding Information for TCLP Analysis

Analysis	From field collection until TCLP extraction	From TCLP extraction until	From preparative extraction to analysis	Total elapsed time

		sample preparation		
Volatiles	14 days	NA	14 days	28 days
Semi-Volatiles	14 days	7 days	40 days	61 days
Mercury	28 days	NA	28 days	56 days
Metals	180 days	NA	180 days	360 days

14.3 Information Necessary to Validate TCLP Data

The Data Validator will need the following information to complete the Tier I Data Validation Checklist:

- Sampling date
- TCLP extraction date
- TCLP extract preparation date (for SVOCs only)
- Percent solids
- Weight of sample extracted
- pH of sample after necessary adjustments
- Type and measured pH of extraction fluid used
- Amount of extraction fluid used
- Analyses requested (VOCs, SVOCs, metals, etc.)
- Spike sample results (for metals only)

Contact the facility or the laboratory to request any missing information. A boilerplate letter for requesting additional information is in Appendix I.

14.4 TCLP Data Validation Criteria

If positive results for TCLP constituents above regulatory levels (OAC rule 3745-51-24; Table1) can be qualified, but not rejected, then it is presumed that waste will be managed as hazardous waste. However, if significant data validation criteria are not within limits, then re-sampling and analysis must be considered. If significant data validation criteria are not within limits and results are non-detect or if positive results are found, but are below the regulatory levels, then qualification or invalidation of sample results must be considered. Rejection of results must be considered if insufficient sample weight is used, if an inappropriate extraction solution was used, or if spike results for metals analysis were below the acceptance criteria.

The criteria that Ohio EPA will use to evaluate TCLP data are as follows:

1. If particle size reduction is required, but not performed, then all non-detected results will be qualified as “R,” rejected, and all positive results will be qualified as “J,” estimated. For results near the regulatory limit, use best professional judgment to decide if the results will be flagged “J,” estimated, or “R,” rejected. Positive results above regulatory levels will not be qualified.

2. If an incorrect extraction fluid was used, all non-detected or positive results below the regulatory limit will be qualified as rejected and flagged “R.” Positive results above regulatory levels will be accepted.

3. If an incorrect amount of sample (less than 100 grams for solids analyzed for metals or SVOCs, or 20 grams for VOCs) was used, then all non-detected compounds or elements will be qualified as "R," rejected. Furthermore, if less than 30% of the required sample weight is used, then qualify all positive results below the regulatory threshold as "R," rejected. Positive results above regulatory levels will not be qualified.

4. If the extraction fluid weight is not within +/- 15% of the correct weight (20 times the weight of the sample), then qualify all positive results below the regulatory threshold as "J," estimated. If the extraction fluid weight is more than +/- 30 percent above or below the correct weight, then qualify all positive results and all non-detects as "R," rejected. Positive results above the regulatory limit will be accepted.

5. If a TCLP blank was not analyzed per batch of samples, reject all positive data below the regulatory limits. If a blank was included, use the Tier I Data Validation Checklist Method Blanks section to evaluate blank contamination.

6. If technical holding times were exceeded, then reject all positive results below the regulatory limits. Positive results above the regulatory limits will be accepted.

Chapter 15
pH

15.0 Introduction

Certain wastes must be evaluated for the characteristic of corrosivity to determine whether they are hazardous wastes (OAC 3745-51-22). By definition, a corrosive hazardous waste is either a) aqueous, and has a pH less than or equal to 2 or greater than or equal to 12.5 as defined by SW-846 method 9040, or b) a non-aqueous liquid, and is shown to be corrosive according to SW-846, Method 1110A, "Corrosivity Toward Steel" (steel coupon test) or equivalent. As Ohio EPA does not routinely receive data for Method 1110, this chapter will address the determination of pH by SW-846, Method 9040C. This chapter discusses pH data used to determine corrosivity and the necessary steps for validation of this data.

15.1 QA/QC

The measurement of pH is straightforward and usually can be accomplished without complication. However, there are several important provisions contained within SW-846, Method 9040C that must be observed. This method requires that temperature compensation be made for the final pH determination. Temperature compensation can be either internal, where an instrument uses an automatic temperature compensation (ATC) controller, or external where the temperature is compensated manually. It is important to note that the buffer solution used to calibrate the instrument and the waste pH should be at approximately the same temperature. Method 9040C requires that sample and buffer not differ by more than 2°C without temperature compensation. In addition, the waste temperature should be within the control range of the ATC. For certain wastes, additional information on the instrument's ATC and the temperature of the waste should be obtained in order to evaluate the pH results. For corrosivity characterization, the sample MUST be measured at 25 +/-1°C, if the waste pH is above 12.

The buffer solutions used to calibrate the pH meter must be within their expiration date and must bracket the expected pH of the samples. For a corrosivity determination, at least two pH buffers should be used consisting of a low pH buffer (e.g., 2.0) and/or a high pH buffer (e.g., 12.0), respectively, depending on if the sample is acidic or caustic. Other buffers (e.g., 4, 7 and 10) may also be used to establish a pH meter calibration curve.

Samples should be analyzed as soon as possible after collection. Preferably, the analysis would be performed at the same time as waste generation. If this is not possible, analyses should be performed on the same day as sample receipt by the laboratory.

15.2 Information Necessary to Validate pH Data

Most data reports contain little information that may be used to judge the validity of pH measurements. If it becomes apparent that validation of pH data is necessary, the laboratory should be asked to provide the following information:

- Instrument ID;
- Sample ID and laboratory ID;

- Time and date of sampling;
- Time and date of sample receipt;
- Time and date of analysis;
- Last date of NIST (National Institute of Standards and Technology) instrument certification;
- Calibration procedure (daily calibration log, continuing calibration results and criteria);
- Calibration buffers used;
- Calibration standards results;
- Calibration buffer NIST certification or comparable information from a commercial vendor;
- Expiration date of the buffers;
- Temperature of waste and buffers;
- Temperature compensation (manual or ATC);
- Continuing calibration results (if required by the laboratory QAPP or instrument manufacturer).

Chapter 16
Flashpoint

16.0 Introduction

A liquid, organic waste displays the characteristic of a hazardous waste if its flashpoint is less than 140̊F (Ohio Administrative Code (OAC) 3745-51-21). The flashpoint of wastes may be assessed by several methods specified in this rule. These methods include:

- SW-846 Method 1010B which specifies the use of ASTM D-93-70 or -80 known as Pensky-Martens Closed Cup Tester, or, ASTM D 8175-18 and
- SW-846 Method 1020C which specifies the use of ASTM D-3278-78 or ASTM D 8174-18, Setaflash Closed Cup Tester, or ASTM D8174-18.

The addition of alternate ASTM methods for both Pensky-Martens and Setaflash arose from a rule change in June of 2020. The changes modernized the methods and allowed the use of non-mercury containing thermometers and the ability to use computer control instead of manual controlled equipment. The newer methods also use different flashpoint standards compared to the older methods.

Method 1010B is used most often since this method is appropriate for materials such as paint wastes and parts cleaner solvents. This method is discussed below.

16.1 Information Necessary to Validate Flashpoint Data

Laboratories using Pensky-Martens ASTM D8175-18 to perform flashpoint testing should be able to supply information the necessary information for flashpoint data validation. Typically, the initial data packages often will not have the necessary information to perform data validation if the older D-93 version of the method is used. If necessary, the Tier I Data Validator will need to request bench sheets with this information from the facility or laboratory. A boilerplate letter is available at the end of this document in Appendix I. This letter should help to simplify and standardize Ohio EPA's requests for additional data validation information.

Specific items needed for completing a Tier I Data Validation should be made available by the laboratory. If information is missing or incomplete, use the boilerplate letter to request this information from the facility or laboratory. These items include information for each sample, such as start time and temperature and end time and temperature. Sample results are normally linked by the sample number to information on each sample group. This information may include reference standards evaluated, , the standard flashpoint, results of any duplicates evaluated, the date the sample group was analyzed, and the name of the person who performed the analysis. If Pensky-Martens Method B (ASTM D-93 or ASTM 8175-18) is used, sample viscosities (or a description from the lab on how these were assessed) and the barometric pressure (not adjusted for sea level) at the time of the test are also important information to request. The data should reflect any adjustment made to the flashpoint results for Method B. If these items are not available, the laboratory must submit an explanation as to why that is the case (e.g., the barometric pressure was never recorded; a data sheet was misplaced, etc.).

Flashpoint determinations using D-93 are not computerized or automated. Therefore, the supporting documentation the Tier I Data Validator receives from the laboratory will typically be developed by the

lab and completed by hand. The sheets will likely be non-standard in format and content. Some labs do not even maintain all records required by the method. The Tier I Data Validator may need to qualify or reject data received due to insufficient documentation. Consult with a Tier II Data Validator in this situation.

16.2 Data Validation Criteria

Pensky-Martens contains two methods, A and B. Choosing the correct method depends on sample viscosity, which is information often not recorded by the laboratory. This can make confirmation of the correct method very difficult. Method A, the basic procedure, is used unless the material being tested is a suspension of solids or a highly viscous material. Those materials require the use of Method B.

According to the Pensky-Martens method, "definite rates of temperature increases ... control the precision of the method." Because of this, one of the main criteria to check during data validation is whether the temperature was raised at the proper rate. The method gives a standard rate of temperature increase. The lab should not vary from the rates given in Method A (2.5-3.5˚C per minute) or Method B (1-1.5˚C per minute). The average rate of temperature increase (degrees per minute) needs to be checked based on the starting temperature and time, and the final temperature and time. The rate of increase of temperature is required by the respective method. Raising the temperature too quickly could cause the analyst to miss the flashpoint. Once the flashpoint is exceeded, the atmosphere in the Pensky-Martens cup may become too rich and the sample will not flash. Raising the temperature too slowly could allow more volatile components of a sample to evaporate, artificially elevating the flashpoint of that sample.

A Tier I flashpoint Data Validation may assess the instrument calibration with a certified reference material a (e.g., n-decane, n-undecane), reproducibility of results, corrections made for barometric pressure readings, and proper thermometer choice. Assessment of much of this information may be triggered by a specific problem or inconsistency (e.g., split samples providing markedly different results).

Method B requires the flashpoint result be adjusted for the barometric pressure at the time of the test. Usually, this correction is not large, but it could affect results near the regulatory threshold if it is run on a day with low barometric pressure.

Chapter 17
Data Validation Summary

17.0 Introduction

As illustrated throughout this document, data validation consists of examining quality control information and qualifying sample results based upon pre-defined criteria. By working through the quality control information associated with a method, sample data may be either validated or qualified as estimated or rejected. However, the method of data validation as presented in this manual is limited in its scope. It is meant to acquaint DERR staff that have little or no background in the subject with elementary methods of validating data. Once a Tier I Checklist has been completed, the reviewer may think that this is the end of the process. This is not the case. Data must be summarized, and a final judgment must be made concerning the overall accuracy and precision of the data. Finally, a statement concerning whether the data meets the data quality objectives of the project must be made. This conclusion is not entirely the responsibility of the data validator. Because of the scope of many environmental projects, this final assessment of data usability must be made in consultation with management, risk assessors and field sampling personnel.

The data validation summary does not have a strict format. However, it should contain key elements and a summary of the data validation findings. The elements that may be outlined in the summary include the rational for collecting the data, the statement of the data quality objectives, the summary of findings, an analysis of whether the data quality objectives have been met, or whether additional data validation (higher level) is necessary. Finally, data qualifiers, if any, should be assigned to the data in the report. These elements will be discussed in the following sections.

17.1 Facility and Sampling Information

The summary should begin with a simple statement giving the facility name, facility ID number, date of sampling, the number of samples that were taken, and the media that was sampled. Additional information may include the laboratory name, the sampling location name (*i.e.*, “Former Drum Storage Pad”), and a short description of field or sampling conditions that could affect the sample results. Most of this information may be conveniently summarized on the Tier I Checklist and therefore does not need to be repeated if the summary will be attached to checklist as part of the plan review form. However, if the data validation summary will act as a stand-alone document, then the required information should be provided to serve as a complete record of the sampling event.

17.2 Sampling Rationale and Data Quality Objectives

A statement should be made describing the regulatory basis for collecting the samples. This may be a simple statement such as “the samples were collected to support the closure of the former drum pad storage area.” Other types of sampling activities that DERR oversees includes, but is not limited to, compliance sample data, ground water monitoring data, RCRA Facility Investigations, generator waste analyses, and data derived from complaint investigations.

The statement is important because it relates to the data quality objectives of the sampling event. As a reminder, the DQOs are a process that enhances decision making. The DQO process is a seven-step process that includes the following (U.S. EPA, 2002):

1. State the Problem
2. Identify the Decision
3. Identify Inputs to the Decision
4. Define the Study Boundaries
5. Develop a Decision Rule
6. Specify Limits on the Decision Errors
7. Optimize the Design

For example, samples may be taken to assure the public that a facility permitted to treat hazardous waste is in compliance with its permit and applicable laws and regulations. The decision (step 2) whether the treatment process is functioning properly will be based upon the results of the compliance samples (step 3, sample results are inputs for the decision). The results must be judged against some criteria. In our example, the criteria may be the LDR requirements for the treated waste or whether the waste displays a characteristic of toxicity. The decision whether the data is useful depends on the quality of the data. If sampling or analytical irregularities are such that the data is rejected, then this data would not be able to serve as input into the decision process. Conversely, data that meets all the data quality criteria would meet this aspect of the DQOs.

17.3 Summary of Findings

A summary of the Quality Control data should be included in the assessment. For the most part, if the Tier I Checklist is used as a tool for validating the data, then this summary is complete. In cases where the checklist was not used or when it is necessary to summarize the findings for a judicial action, then the results for each quality control parameter should be briefly discussed. The best approach is to use the Tier I Checklists as an overall outline of QC parameters to present.

As a general outline, the subjects presented include the following:

1. **Sample/Sample Receipt.** Any problems noted with sampling procedures (improper preservation, etc.) should be noted and a list of qualified sample results.
2. **Batch Specific Quality Control**. Batch specific quality control data may include, laboratory control sample results, matrix spike/matrix spike duplicate results and method blank results.
3. **Sample Specific Quality Control**. Sample specific quality control includes surrogate results for organic compound analytical methods and spike (Method of Standard Additions) results for inorganic methods.

For each quality control section, the problems encountered should be briefly discussed and the qualified sample listed.

17.4 Other Information

The validator should also make a note of several other criteria that can have a significant bearing on the usability of the data. For example, any missing data or QA/QC data should be noted. In addition, an evaluation of whether the reported detection limits or quantitation limits meet the regulatory or risk standards must be made. Finally, any deviations of the method must be noted for evaluation. It is also important to assess whether there is a bias in the data, this can be accomplished by reviewing the qualified data. If the quality control data were generally below or above the quality control criteria, then the validator should suspect a bias and use this knowledge when evaluating the data for usability. More

information on bias assessment can be found in Chapter 5 of U. S. EPA's, Data Quality Assessment: A Reviewer's Guide

17.5 Data Assessment

Once all the information has been summarized the reviewer must conclude whether the data is of a sufficient quality to be usable. Unfortunately, this may not be as straight forward as presented in the example in Appendix B. In many instances, professional judgment must be used when assessing the results of the data validation. The reviewer should evaluate all the accumulated data qualifications on a data set and the summary of the findings of the data validation in light of the project's scope and data quality requirements. Thus, if data are qualified as estimated based upon a variety of quality control criteria, it may be deemed unusable for its intended purpose even though the data was not initially rejected. For example, if technical holding times were outside of the acceptance time frame, and batch quality control samples such as the LCS were also below the acceptance criteria, this may indicate that the data does not meet the quality standards necessary to fulfill the project's data quality objectives. This action may also be justified if a bias is found in evaluating the QC data. It must be emphasized that rejection of data or a determination that data is unusable is not an automatic action if data is qualified for multiple reasons. In fact, other actions should also be considered. For example, the reviewer may conclude that additional information may be needed or that a Tier II Data Validation be performed. Another option is to identify if an alternate method can be used to verify the results. This would require that either an additional sample aliquot be analyzed or that the extract be re-analyzed from the original sample. Another option is to consider acquiring additional samples where these extra results can verify the previous sample results. If this action is contemplated, it is crucial to review the necessary changes that must be made by the laboratory to satisfy the project's data quality objects.

14.0 Data Validation Summary	
The results of the Tier I Data Validation must be summarized to be useful in making decisions concerning the use of the analytical data. The final decision on whether the data is usable for its intended purpose must be made in conjunction with the project management team and with the stated DQOs for the project. The following items can be used as a general guideline on preparing a data validation summary. More information can be found in Chapter 17 of the Tier I Data Validation Manual.	
14.1 State the regulatory requirement that prompted the samples to be taken.	**Specify sampling purpose:**
14.2 List the DQOs for the sampling.	**List project specific DQOs:**
14.3 Summarize the findings of each major category of quality assurance data (*e.g.*, blanks, surrogates, spikes, etc.).	**Summarize data validation findings:**
14.4 Assess whether bias is present. **Note: This can be accomplished qualitatively by reviewing the qualified QA/QC data. If most of the QA/QC data are flagged with a "J-then there may be a negative bias present. If most of the QA/QC data is flagged with a "J+"," then there may a positive bias. Additional information on the assessment of bias can be found in U.S.**	**Explain any bias that is present:**

EPA's Guidance for Data Quality Assessment: A Reviewer's Guide (QA/G-9R), EPA/240/B-06/002, February 2006.	
14.5 Is the quality of the data sufficient to meet the DQOs of the project?	**Indicate yes or no, and provide explanation:**

Chapter 18
Definitions

Aliquot: A fraction of a whole, as in aliquots of a sample used for testing or analysis.

Amenable Cyanide: Cyanide in solution that is capable of reacting with chlorine. Amenable cyanide includes both free cyanide and soluble cyanide complexes.

Aqueous: A solution where at least 20 percent of the solution's composition is water.

Batch: A batch is a group of 20 samples that behave similarly, with respect to the analytical procedures being employed, and are prepared and analyzed identically, run consecutively on the same equipment, and associated with the same QA/QC samples.

Bias: The deviation due to matrix effects of the measured value from a known spiked amount. Bias can be assessed by comparing a measured value to an accepted reference value in a sample of known concentration or by determining the recovery of a known amount of contaminant spiked into a sample (matrix spike).

Buffer Solution: A stable solution of known pH, used to calibrate a pH electrode. In addition, buffered solutions or buffers will resist a change in pH when small amounts of acid or base are added. Buffered solutions are used to calibrate analytical instruments.

Control Limits: Established to evaluate lab precision and bias based on the analysis of control samples. Typically, control limits for bias are based on historical mean recovery plus or minus three standard deviation units. Control limits for precision range from zero (no difference between duplicate control samples) to the historical mean relative percent difference plus three standard deviation units.

Dilution: The act of adding distilled water and/or other preparation reagents to a sample extract or digestate to overcome an interferent or to bring the concentration of a target analyte back into the working calibration range of the instrument.

Dilution Factor: The total number of volumes, including the sample volume, in which the sample will be dissolved.

Dilution Ratio: The number of volumes of sample as compared to the dilution factor.

Equipment Blanks: Usually an organic or aqueous solution that is analyte-free and transferred to the site, opened in the field, and poured over or through the decontaminated sample collection device, collected in a sample container, and returned to the laboratory. Generally, one equipment blank is analyzed with each analytical batch or every 20 samples, whichever is more frequent. The results of analysis are used to demonstrate adequate cleanliness and, or decontamination of the sample

equipment. Equipment blanks may not be necessary if dedicated equipment is used for each sample collected (i.e., disposable bailers).

Extraction: The removal of solutes from a material by the application of a solvent. In the case of the TCLP, the extraction process is designed to determine the mobility of specific organic and inorganic analytes present in liquid, solid, and multi-phasic wastes.

Field Blanks: Usually an organic or aqueous solution (as free of analytes as possible) that is transferred from one vessel to another at the sampling site and preserved with the appropriate reagents. This serves as a check on reagent and environmental contamination. Generally, one field blank is analyzed with each analytical batch or every twenty samples, whichever is more frequent.

Free Cyanide: Cyanide that in solution is in the anionic state as CN-.

Hexavalent Chromium: Chromium is commonly found in trace concentrations in aqueous solution in different oxidation states as either chromium III or chromium VI. Hexavalent chromium (Cr VI) is the most oxidized form of chromium that commonly exists in nature. Cr VI is more mobile and toxic in the environment.

Interference: Additions or detractions from a signal generated by analytical instruments. Interferences can either add to the signal received by the instrument producing a positive bias or detract from a signal producing a negative bias. QA samples such as Matrix Spikes, Matrix Spike Duplicates, and Laboratory Control Samples may be used to assess and overcome interferences.

Instrument Blanks: Blanks that are analyzed after any sample that has high concentrations of analytes. The instrument blank assesses whether residual contaminants in the analytical system could be carried over to other samples.

Instrument Detect Limit (IDL): Typically used in metals analysis to evaluate the instrument noise level and response changes over time for analytes of interest. IDLs in µg/L can be estimated as the mean of the blank results plus three times the standard deviation of 10 replicate analyses of the reagent blank solution. (Use zero for the mean if the mean is negative). Each measurement should be performed as though it were a separate calibration standard (i.e., each measurement must be followed by a rinse and/or any other procedure normally performed between the analysis of separate samples). IDLs should be determined at least once using new equipment, after major instrument maintenance such as changing the detector, and/or at a frequency designated by the project. An instrument log book should be kept with the dates and information pertaining to each IDL performed.

Laboratory Control Sample (LCS): A blank spiked with analytes representative of the target analytes used to document laboratory performance prior to the preparation step. An LCS monitors the efficiency of the preparation procedures for analysis which provides the best indication of whether poor analytical results are matrix dependent or a result of an analytical problem. The LCS should be analyzed for each sample matrix (e.g., soil and water) using the same preparation procedures and analytical methods as

the actual samples. Spiked compounds and concentrations are generally the same for LCS and MS/MSD samples.

Matrix Spike: The introduction of a known concentration of analyte(s) into a sample to provide information about the effect of the sample matrix on the digestion and measurement methodology. Matrix spikes are used to provide an indication of bias due to matrix effects and a measure of accuracy of associated results.

Matrix Spike Blank: The introduction of a known concentration of analyte(s) into a blank. It provides a measure of whether the spiking analytes are appropriate for a specific batch of samples.

Matrix Spike Duplicate: Analysis of spiked duplicates is used to provide a measure of the precision in the analytical process. Matrix spikes are evaluated by criteria based upon the relative percent difference of the duplicates.

Method Blanks: Blanks that are prepared using the same techniques and reagents as field samples. Method blanks are used to assess whether a positive bias has been imparted to the results through the analytical procedures or materials used by the laboratory. Method blanks are also referred to as analytical blanks or preparation blanks.

Percent Recovery (%R): Percent recovery of the spike analyte. Used for organics and inorganics. The spike percent recovery and the spike provide information about the effect of each sample matrix on the sample preparation procedures and the measurement methodology. The spike recovery must be within established limits given on the QA/QC sheets provided by the laboratory (i.e., 75-125%). See formula in Equation 7.1 below.

Percent Solids: Liquid samples contain less than 0.5 percent solids and can be used as TCLP extract. Solid samples contain less than 0.5 percent liquids and the entire sample must be extracted. Where samples contain between 0.5 and 99.5 percent solids, the solid and liquid component are analyzed separately, and the results mathematically recombined. Alternately, the multi-phased components may be physically recombined prior to analysis.

pH: The pH is the negative logarithm of the hydronium ion concentration (moles/L) at a specified temperature and pressure. The hydronium ion concentration is small in natural water samples. By defining the pH as the negative log of the concentration, we can conveniently establish the pH scale. The pH scale typically extends from 0 to 14. A pH of zero represents very acidic conditions, 7 indicates neutral conditions, and 14 indicates very basic or alkaline conditions.

Pre-Digestion Spike: (Same as Matrix Spike)

Post-Digestion Spike/Post-Distillation Spike: The addition of a known amount of standard after digestion or distillation (also identified as an analytical spike).

Relative Percent Difference (RPD): Relative percent difference is used for organics and inorganics when comparing the duplicate sample results to the original sample results. Analytical results within 20% of each other indicate that the laboratory followed their Quality Assurance Program Plan (QAPP). See formula in Equation 7.2 below.

Run Log: The log is a chronological record of the instrument history and includes information like sample identifiers of samples analyzed and associated quality control samples.

Serial Dilution: A sample aliquot that is subjected to a multiple or series of dilution steps. Serial dilution is usually performed on new or difficult matrices that may display significant matrix interference.

Spike: A known analyte and volume added to a sample to verify QA/QC results.

Surrogate Recovery (%R): Amount of a specific surrogate compound recovered during analysis, expressed as a percentage. Surrogate recovery is used to measure accuracy.

Target Analyte: The chosen analyte of investigation for which qualitative and/or quantitative data or information is desired

Total Cyanide: All species of cyanide in a sample including free, soluble complexes and insoluble complexes of cyanide.

Total Chromium: Chromium may exist in a number of oxidation states. Total chromium is the combination of all of these chromium oxidation states in solution or in a sample digestate.

Trip Blank: A sample consisting of analyte-free media that is prepared prior to the site visit, transported from the laboratory to the sample site, and then returned to the laboratory without being opened. A trip blank is used to assess contamination attributable to shipping procedures and handling in the field. This type of blank is useful in documenting contamination of samples analyzed for volatile organic compounds (VOCs). The trip blank must accompany sample containers to and from the field when analysis for VOCs is being requested.

Type 1 Extraction Fluid: pH equals 4.93 (+/- 0.05). Created by adding 5.7 ml glacial acetic acid (CH3CH2OOH) to 500 ml reagent water, adding 64.3 ml of 1N NaOH and diluting the volume to one liter. Type 1 Extraction Fluid is always used for extraction of samples to be analyzed for VOCs, as well as acidic to slightly basic wastes.

Type 2 Extraction Fluid: pH equals 2.88 (+/- 0.05). Created by diluting 5.7 ml glacial acetic acid (CH3CH2OOH) with reagent water to a volume of one liter. Type 2 Extraction Fluid is used to extract highly alkaline wastes.

Vapor Intrusion: the movement of chemical vapors from contaminated soils and/or ground water into the indoor air of overlying or nearby buildings

Chapter 19
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Appendix A
Worked Examples and Checklist Sections

1.0 Sample Report Completeness and Technical Holding Times Worked Example

Checklist #1 of the Tier I Data Validation Checklists reviews sample package completeness and deliverables and technical holding times. This section of the manual provides examples of this checklist to illustrate it's use.

The Tier I Data Validator should use the Batch-Specific Data Validation Worksheet included Checklist #1. To facilitate this evaluation, the following documents should be consulted: Chain of Custody (COC), cooler receipt form, sample run log(s), extraction log(s), and bench sheet(s). If the necessary information is not present, the laboratory should be contacted for additional deliverables.

While datasets with limited samples or parameters may not contain extensive technical holding time information, multi-media events typically will. All technical holding time information should be evaluated. While this step of the Tier I Data Validation process is not particularly technical, it may be confusing and present organizational difficulties. Since a review of the completed table may cause analytical data to be qualified or rejected, care and time is required in completing this section. Additional copies of the Batch-Specific Data Validation Worksheet found in Checklist #1 may needed to record all technical holding times.

Checklist section 1.2 below is filled out using example information from Exhibits 1-1 and 1-2 to show an example of a completed review of technical holding times for VOCs and SVOCs for the "Dirty Drum Corporation" site sampling event. The parts of Checklist section 1.2 that did not apply to the Dirty Drum Corporation example were omitted for clarity.

1.1 Example of Filled out Chain of Custody and Checklist #1 Data Validation Worksheet

Exhibit 1-1: Chain of Custody form for Dirty Drum Corporation

Ohio EPA-Division of Environmental Response and Revitalization Lazarus Government Center, 50 W. Town St., Suite 700, Columbus, Ohio 43215					Facility Name: Dirty Drum Corporation		
Sampler's Name: John Doe Split Samples Offered ( ) Accepted (x) Declined					Facility and Sample Locations: 123 Main Street Columbus OH, 43215		
Sample ID.	Date	Time	Sample Type (Comp. or Grab)	Analysis Required	Number of Containers	Preservative	Station Description/Remarks
Waste Pit 1	05/10/01	0915	Grab	Total VOCs 8260D, TCLP VOCs 8260D, TCLP SVOCs 8270E	3	None	N/A
Drum 54	05/10/01	1025	Grab	Total VOCs 8260D, TCLP VOCs 8260D, SVOCs 8270E	3	None	N/A
Transferred By: John Doe Time/Date 1506 / 05/10/01					Received By: Jane Doe Time/Date 1510 / 05/10/01		

Transferred By:	Time/Date	Received By:	Time/Date
Transferred By:	Time/Date	Received By:	Time/Date

Exhibit 1-2: Checklist #1 Data Validation Worksheet for Dirty Drum Corporation

Site Name: Dirty Drum Corporation										
Sample ID	Lab ID	Matrix	Sample Date	Date Received by the Lab	Parameter	Extraction Date	Preparation Date	Analysis Date	QA/QC Data Present (Yes or No)	Batch ID#
Waste Pit 1	51101-9386	solid	5/10/01	5/11/01	Total VOCs 8260D	-	5/22/01	5/22/01	Yes	501522
Waste Pit 1	51101-9386	solid	5/10/01	5/11/01	TCLP VOCs 8260D	5/17/01	5/29/01	5/31/01	Yes	501208
Waste Pit 1	51101-9386	solid	5/10/01	5/11/01	TCLP SVOCs 8270E	5/17/01	5/24/01	5/29/01	Yes	501184
Drum 54	51101-9387	liquid	5/10/01	5/11/01	Total VOCs 8260D	-	5/23/01	5/26/01	Yes	501522
Drum 54	51101-9387	liquid	5/10/01	5/11/01	TCLP VOCs 8260D	5/17/01	5/23/01	5/26/01	Yes	501208

Drum 54	51101-9387	liquid	5/10/01	5/11/01	TCLP SVOCs 8270E	5/17/01	6/1/01	6/6/01	Yes	501184

1.2 Technical Holding Times Checklist #1 (VOCs) - Dirty Drum Corporation

Example 1-1 consists of an excerpt of Checklist #1: Technical Holding Times, completed for the "Dirty Drum Corporation." This example is specific to VOC data.

Example 1-1: Completed Checklist Section for VOC Technical Holdings Times

1.2 Technical Holding Times - Volatile Organic Compounds	
1.2.1 Are samples properly preserved? Check preservation requirements, chain of custody, and sample receipt form for discrepancies. *Action: Note any problems and use the information to qualify results.*	**Indicate yes or no**: No **If no, list any problems**: No preservatives were listed on the COC for either sample. Contacted facility rep., who stated that no preservatives, other than ice, were used. All samples were correctly preserved as they were waste samples.
1.2.2 Were any technical holding times exceeded? *Action: If samples were improperly preserved or unpreserved, if applicable, and the technical holding times were exceeded, qualify all positive results for affected samples as "J-" and all non-detected results as "UJ."*	**Indicate yes or no**: yes **If yes, list sample ID(s) and summarize any actions taken:** Drum 54: Total VOC technical holding time was exceeded from sampling to analysis (16 days instead of the 14 days specified). **Results must be qualified as per the criteria**. Waste Pit: All VOC analyses were within holding times.
1.2.3 Were any technical holding times greater than 2x the time requirement? *Action: If technical holding times are greatly exceeded (> 2x the time requirement) upon analysis or re-analysis then the validator may use professional judgment to qualify all non-detected compounds as "UJ" or "R" based upon professional judgment and on DQOs.*	**Indicate yes or no**: no **If yes, list sample ID(s) and summarize any actions taken:** NA

1.3 Technical Holding Times Checklist #1 (SVOCs) - Dirty Drum Corporation

Example 1-2 consists of an excerpt of Checklist #1: Technical Holding Times, completed for the "Dirty Drum Corporation." This example is specific to SVOC data.

Example 1-2: Completed Checklist Section for SVOC Technical Holdings Times

1.2 Technical Holding Times – Semi-Volatile Organic Compounds	
1.2.5 Were samples properly preserved? Check preservation requirements, chain of custody, and sample receipt form for discrepancies. *Action: Note any problems and use the information to qualify results.*	**Indicate yes or no:** No. **If no, list any problems:** No preservatives were listed on the COC for either sample. Contacted facility rep., who stated that no preservatives, other than ice, were used. All samples were correctly preserved, as they were waste samples.
1.2.6 Were any technical holding times exceeded? *Action: If technical holding times shown in Table 1-2 of Checklist #1 are exceeded, qualify all positive results for affected samples as "J-" and all non-detected results as "UJ."*	**Indicate yes or no:** No **If yes, list sample ID(s) and summarize any actions taken:** Drum 54: TCLP SVOC technical holding times were not exceeded. Waste Pit: TCLP SVOC technical holding times were not exceeded.
1.2.7 Were any technical holding times greater than 2x the time requirement? *Action: If technical holding times are greatly exceeded (> 2x the time requirement), based on the project's DQOs, qualify all positive results as estimated (J-). The validator may use professional judgment to qualify all non-detected compounds as "R" or "UJ".*	**Indicate yes or no:** N/A **If yes, list sample ID(s) and summarize any actions taken:**

In this example, the COC does not indicate the presence of preservatives in the sample containers. The Tier I Data Validator should contact the sampler and/or laboratory to determine if proper sample preservatives were used. In this case, the sampler was contacted and confirmed that no preservatives, other than ice, were used due to the sample matrix being waste. This should have been reported on the COC to alert the laboratory to the need for potentially expedited action(s) based on technical holding times for unpreserved samples.

In this example, technical holding times were not met for one of the two analyses. For Drum 54, VOCs (SW-846, Method 8260D), the analysis was performed in 16 days instead of the method-specified 14 days. For this reason, this analysis must be qualified.

In the "Dirty Drum Corporation" example, the data report did not include vapor intrusion, total petroleum hydrocarbons (TPH), pH, cyanide, inorganic compounds, mercury, or hexavalent chromium, so these sections were not evaluated.

2.0 Blank Worked Examples

The following examples demonstrate how to qualify analytical data using preparation blank results.

2.1 Organic Analysis: Example Blank and Analytical Results

Example 2-1: Qualifying Tetrachloroethylene Data using Blank Results

Sample	Parameter	Results	QL (mg/L)
Soil	Tetrachloroethylene	300	10
Method Blank	Tetrachloroethylene	30	1

Is the Tetrachloroethylene in the sample "real" or a laboratory contaminant?

Steps to evaluate the problem:

1. Is the analyte a common laboratory contaminant? No, tetrachloroethylene is not one of the common laboratory contaminants. Therefore, the sample results should be compared to the blank result.

2. Compare sample result to blank result.

3. Are the results above or below the QL? Both the blank and sample results are above the QL.

Since the sample result is greater than the blank result, the soil sample results should be reported with a J+ qualifier:

Sample	Parameter	Results	QL	Qualifier
Soil Sample	Tetrachloroethylene	300	10	J+

Example 2-2: Qualifying Methylene Chloride Data using Blank Results

Sample	Parameter	Results	QL (mg/L)
Soil	Methylene Chloride	40	5
Method Blank	Methylene Chloride	10	5

Is the Tetrachloroethylene in the sample "real" or a laboratory contaminant?

Steps to evaluate the problem:

1. Is the analyte a common laboratory contaminant? Yes, Methylene Chloride is a common laboratory contaminant.

2. Compare sample result to blank result.

3. Are the results above or below the QL? Both the blank and sample results are above the QL.

4. Are the sample results greater than 2X the blank result? Yes

Since the sample result is greater than 2X the blank result, the soil sample results should be reported with a J+ qualifier:

Sample	Parameter	Results	QL	Qualifier
Soil Sample	Tetrachloroethylene	40	5	J+

2.2 Inorganic Analysis: Example Blank and Analytical Results

Example 2-3: Qualifying Arsenic Data using Blank Results

Sample	Parameter	Sample Result	MDL (mg/L)	QL (mg/L)
Soil	Arsenic	100	5	10
Method Blank	Arsenic	10	0.5	5

Is the Arsenic in the sample "real" or a laboratory contaminant?

Steps to evaluate the problem:

1. Were blank analyzed at the specified frequency? Yes

2. Compare blank result, sample result, MDLs, and QLs.

3. Are the results above or below the QL? Both the blank and sample results are above the QL.

4. Does the sample result greatly exceed the blank result? Yes, the sample result is greater than 10X the blank result.

Since 100 mg/L is greater than 10X the blank result, no qualification is needed. Therefore:

Sample	Parameter	Results	Qualifier
Soil Sample	Arsenic	100	

Example 2-4: Qualifying Mercury Data using Blank Results

Sample	Parameter	Sample Results	MDL (mg/L)	QL (mg/L)
Soil	Mercury	20	1	5
Method Blank	Mercury	2	0.2	1

Is the Mercury in the sample "real" or a laboratory contaminant?

Steps to evaluate the problem:

1. Were blank analyzed at the specified frequency? Yes

2. Compare blank result, sample result, MDLs, and QLs.

3. Are the results above or below the QL? Both the blank and sample results are above the QL.

4. Does the sample result greatly exceed the blank result? No, the sample result is less than 10X the blank result.

Since both results are greater than the QL, but the sample result is less than the QL, the results can be attributed to blank contamination and blank results should be qualified J+ or R. Therefore:

Sample	Parameter	Results	Qualifier

Soil Sample	Mercury	2	J+

2.3 Use of the 5X and 10X Rules

The following examples demonstrate how to qualify analytical data using the 5X and 10X rules.

Example 2-5: Qualifying Benzene Data using the 5X Rule

Sample	Parameter	Results	Method Detection Limits (mg/L)
Soil	Benzene	200	10
Method Blank	Benzene	10	1

Is the Benzene in the sample "real" or a laboratory contaminant?

Steps to evaluate the problem:

1. What is the dilution factor? The MDL is elevated in the sample results as compared to the blank. The dilution factor can be calculated by dividing the MDL of the sample results by the MDL of the blank.

Dilution Factor = 10 mg/L (sample MDL) ÷ 1 mg/L (blank MDL) = 10
The dilution factor must be considered to use the 5X or 10X Rules.

2. Do the 5X or 10X Rules apply? Benzene is not one of the common laboratory contaminants. Therefore, the 5X Rule applies.

3. Dilution Evaluation: The dilution factor must be considered. Either divide the sample concentration by the dilution factor or multiply the blank concentration by the dilution factor before applying the 5X Rule. If dividing the sample result by the dilution factor, then:

200 mg/L ÷ 10 = 20 mg/L (concentration detected by the instrument)

4. Apply the 5X Rule

10 mg/L (blank result) X 5 (5X Rule) = 50 mg/L

Since 50 mg/L is greater than 20 mg/L (sample result corrected for dilution), the results can be attributed to blank contamination. Therefore:

Sample	Parameter	Results	Method Detection Limit	Qualifier
Soil Sample	Benzene	200	10	U

Example 2-6: Qualifying 2-Butanone Data using the 10X Rule

Is the 2-Butanone in the sample "real" or a laboratory contaminant?

Note: This example uses a slightly different method than above. Here the amount detected in the blank is multiplied by 5X or 10X (instead of dividing the sample result) but the result is the same.

Sample	Parameter	Results	Reporting Limit (mg/L)
Soil Sample	2-butanone	300	20
Method Blank	2-butanone	25	20

Steps to evaluate the problem:

1. What is the dilution factor? Apparently, the dilution factor is 1:

Dilution factor = 20 (sample reporting limit) ÷ 20 (blank reporting limit) = 1

However, the report does not list the method detection limit, but rather a reporting limit. Reporting limits are not the same as a method detection limit, but rather a value that the laboratory can reliably achieve for most matrices that it receives. Therefore, if the dilution factor is not listed in the report, it is not possible to determine whether a dilution factor will be accounted for in the blank contamination procedure. At this point either proceed with the calculations or consult the facility or the laboratory for method detection limit information or information on dilution. If proceeding, then:

2. Do the 5X or 10X Rules apply? 2-butanone is a common laboratory contaminant, so the 10X Rule applies.

25 mg/L (method blank concentration) X 10 (10X Rule) = 250 mg/L

3. Dilution Evaluation: The sample result was 300 mg/L of 2-butanone, which is greater than the value of the blank multiplied by 10. Therefore, the amount of 2-butanone observed in the sample is considered "real", and the result is unqualified.

Sample	Parameter	Results	Reporting Limits (mg/L)	Qualifier
Soil Sample	2-butanone	300	20	
Method Blank	2-butanone	25	20	

Remember, the interpretation of the results is predicated on the sample not being diluted. Additional information may change the interpretation entirely. As an exercise, the Data Validator is encouraged to re-evaluate exercise 6.3 with a dilution factor of 2.

Note: When evaluating method blank contamination for solid samples reported in mg/kg, ug/kg, consideration must be given for sample preparation and difference in units (ug/L – ug/kg). As stated above, referral to the raw data from the sample can be of valued assistance.

2.4 Example of Completed Tier I Checklist for Organic Blank Evaluation

The example data report and QC Summary for Any Laboratories, Inc. was used to complete Example 2-5.

Exhibit 2-1: Example VOC Data Report and QC Summary

ANY LABORATORIES, INC. - EPA SW-846, Method 8260D

Project:	Big Site
Project #:	IOQI3
Report Date/Time:	10/11/21; 16:58
Prepared & Analyzed:	09/27/21
Dilution:	1
Batch #:	2802
SAMPLE ID:	**MB**

Analyte(s)	Result	RL	Units	Flag
Dichlorodifluoromethane	ND	10.0	µg/L	
Chloromethane	ND	10.0	µg/L	
Vinyl chloride	5.7	10.0	µg/L	
Bromomethane	ND	10.0	µg/L	

ANY LABORATORIES, INC

Project:	Big Site
Project #:	IOQI3
Report Date/Time:	10/11/21; 16:58
Prepared & Analyzed:	09/27/21
Dilution:	1
Batch #:	2802
SAMPLE ID:	**X102**

EPA SW-846, Method 8260D

Analyte(s)	Result	*RL	Units	Flag
Dichlorodifluoromethane	ND	10.0	µg/L	
Chloromethane	ND	10.0	µg/L	
Vinyl chloride	7.5	10.0	µg/L	U
Bromomethane	ND	10.0	µg/L	
Chloroethane	ND	10.0	µg/L	

Trichlorofluoromethane	ND	5.0	µg/L
Acrolein	ND	10.0	µg/L
Acetone	ND	10.0	µg/L
1,1-Dichloroethene	ND	5.0	µg/L
Methylene chloride	ND	5.0	µg/L

The Tier I Data Validator may question whether the Reporting Limit (RL) is really the quantitation limit. Note that the RL for the blank and the sample results are the same. In addition, the dilution factor is reported as 1, which corresponds with the data presented for the RL in the sample results and in the QA/QC results. For the sake of this example, assume that the RL is the quantitation limit. When there is doubt about the quantitation or detection limit, it is always appropriate to request clarification or additional information from the laboratory.

The results in Exhibit 2-1 indicate that there is a compound detected in the method blank and in the sample; therefore, evaluate whether to qualify the data.

The Tier I Checklist is designed to look at blank data and was developed with U.S. EPA National Functional Guidelines (NFGs) as the general reference. Ohio EPA understands the NFGs were designed to evaluate data from U.S. EPA's Contract Laboratory Program (CLP). The Tier I Checklists were designed to keep the assumptions as generic as possible. The Data Validator will inevitably find a laboratory data package that includes method blank data that does not resemble the forms listed in the NFGs (or in this guidance). However, if sufficient data is given in the package, then validation practices will not be hindered, and the data can be successfully evaluated. The Data Validator is encouraged to thoroughly examine a data package for the required information and pay less attention to the form of the data presentation.

Note: the VOC questions in Checklist #2 and the Semi-Volatile Compounds (SVOC) questions in Checklist #4 are identical.

Example 2-7: Completed Blank Section of VOC Checklist

Laboratory blanks are used to assess whether contamination from the laboratory, reagents, or other samples exists and whether this contamination can bias sample results. The qualification of sample results will depend upon the magnitude of blank contamination.	
2.1.1 Is the method blank data present for each batch (matrix and sample number dependent), including TCLP? *Action: If not present, request information from the facility or laboratory. If the required method blank(s) was not analyzed, sample results may be qualified as estimated ("J," for detected results and "UJ," for non-detected compounds) based upon the validator's judgment. Additional qualification may be warranted based upon other QA/QC information.*	**Indicate yes or no:** Yes, the method blank summary is present. The information necessary to evaluate blank contamination is also present. This data includes a batch ID that can be used to associate sample data with the appropriate blank, detection limit, sample results, blank results, and dilution factor.

The method blank summary is present as are the results. The Data Validator will want to pay particular attention to the detection limits listed for the method blank and the sample results. It is commonly observed that blank analyses are reported with the detection limit, but sample results are reported with a reporting limit. If this is the case, the Data Validator must obtain the detection limit data from the laboratory. This information is necessary to understand whether the reported dilution factor is correct and whether to apply the 5X and 10X Rules.

2.1.3 Is there an indication that the samples associated with the method blank were diluted? **Note: The dilution factor can be found in the data report (a dilution factor of 1 indicates no dilution).**	**Indicate yes or no:** No **If yes, list the sample ID(s) and dilution factor(s):** The dilution factor is 1. Verify this by dividing the detection limit listed for the sample results by the detection limit listed with the method blank.

2.1.4 Do any method/field/trip/equipment blanks have any detected results for any volatile target analytes? Were the same target compounds found in the samples? **Note: A list of samples associated with each of the contaminated blanks should be prepared. Trip blanks are used to qualify samples based on potential contamination during shipment and are not required for non-aqueous matrices.** *Action: Use the criteria in Table 2-1 below to qualify sample results due to blank contamination. Use the largest value from all associated blanks. If any blanks are grossly contaminated, all associated data may be qualified as "R," based upon professional judgment and the project's DQOs.*	**Indicate yes or no:** Yes, VOCs were detected in the method blank. Field, trip, or equipment blanks were not taken. **If yes, list those analytes and results found in both the blanks and samples:** Vinyl chloride was detected in the blank at 5.7 µg/L and in the sample at 7.5 µg/L. **Summarize sample result qualifications based on blank results:** Because the dilution factor is 1, dilution does not need to be considered in the qualification of the data. Vinyl chloride is not a common laboratory contaminant, so the sample result should be compared to the blank result. Since the blank result (5.7 µg/L) is less than the QL, and the sample result of 7.5 µg/L is also below the QL, the sample data should be reported at the QL (10.0 µg/L) and qualified as "U," undetected.

Question 2.1.4 asks the Data Validator to evaluate other blanks that are associated with the sample results. The blanks include field, trip, and equipment blanks. The sample results are compared to these blanks in the same way as with the method blank. If the data set includes these types of blanks, examine the blank results, detection limits, dilution factors, and other required data just as with the method blank examples that have been presented. Note that Table 2-1 is not provided in this appendix but can be found in Checklist #2. The data report, with correct data qualifiers, is shown in Exhibit 2-2

Exhibit 2-2: Example Validated VOC Data Report

ANY LABORATORIES, INC.

Project:	Big Site
Project #:	OQI3
Report Date/Time:	10/11/21; 16:58
Prepared & Analyzed:	09/27/21
Dilution:	1
Batch #:	2802
SAMPLE ID:	**X102**

EPA SW-846, Method 8260D

Analyte(s)	Result	RL	Units	Flag
Dichlorodifluoromethane	ND	10.0	µg/L	
Chloromethane	ND	10.0	µg/L	
Vinyl chloride	**10.0**	10.0	µg/L	U
Bromomethane	ND	10.0	µg/L	
Chloroethane	ND	10.0	µg/L	
Trichlorofluoromethane	ND	5.0	µg/L	
Acrolein	ND	10.0	µg/L	
Acetone	ND	10.0	µg/L	
1,1-Dichloroethene	ND	5.0	µg/L	
Methylene chloride	ND	5.0	µg/L	

2.5 Example of Completed Tier I Checklist for Inorganic Blank Evaluation

The example data report and QC Summary for Any Laboratories, Inc. in Exhibits 2-3 and 2-4 were used to complete Examples 2-6 and 2-7.

Exhibit 2-3: Example Metals and Mercury QC Summary

ANY LABORATORIES, INC. - QC Batch Report

Project:	Big Site
Project #:	IOQI3
Report Date/Time:	10/12/21; 15:54
Prepared & Analyzed:	09/28/21
Dilution:	1
Batch #:	2803
SAMPLE ID:	**MBLK-2803**

Analyte(s)	Result	PQL	Units	Flag
Arsenic	ND	5.0	mg/kg-dry	
Barium	ND	20	mg/kg-dry	
Cadmium	ND	1.0	mg/kg-dry	
Chromium	ND	10	mg/kg-dry	
Lead	ND	20	mg/kg-dry	
Selenium	ND	3.0	mg/kg-dry	
Silver	ND	5.0	mg/kg-dry	
Mercury	ND	0.3	mg/kg-dry	

Exhibit 2-4: Example Metals and Mercury Analytical Data Report

ANY LABORATORIES, INC - EPA SW-846, Method 6010D and Method 7471B

Project:	Big Site
Project #:	IOQI3
Report Date/Time:	10/12/21; 15:54
Prepared & Analyzed:	09/28/21
Dilution:	1
Batch #:	2803
SAMPLE ID:	**SS-103**

EPA SW-846, Method 6010D

Analyte(s)	Result	PQL	Units	Flag
Arsenic	13	6.0	mg/kg-dry	
Barium	150	24	mg/kg-dry	
Cadmium	ND	1.2	mg/kg-dry	
Chromium	ND	12	mg/kg-dry	
Lead	61	24	mg/kg-dry	
Selenium	5.1	3.6	mg/kg-dry	
Silver	ND	6.1	mg/kg-dry	

EPA SW-846, Method 7471B

Analyte(s)	Result	PQL	Units	Flag
Mercury	ND	0.36	mg/kg-dry	

The following sections of Tier I Checklists #6 and #7 were used to evaluate results listed in Exhibit 2-3 and 2-4 for 6010 metals analysis and mercury analysis, respectively.

Example 2-8: Completed Blank Section of 6010 Metals Checklist

6.1.1 Was a method/preparation blank with each batch of samples (for each matrix), including TCLP? *Action: If not present, request information from the laboratory or applicable party. If the required method blanks were not analyzed, sample results may be qualified as "J" for detected results and "UJ" for non-detected compounds. Qualification should consider other QA/QC information and the DQOs.*	**Indicate yes or no:** Yes, MBLK-2803 was prepared with this batch of samples. **Summarize any actions taken:** N/A
6.1.2 Were any samples diluted? *Action: Record the sample ID and dilution factor(s).*	**Indicate yes or no:** No. **Record sample ID(s) and dilution factor(s):** The dilution factor for Sample ID SS-103 is shown to be 1.
6.1.3 Were any metals detected in the blank? Were the same target analytes found in the samples? **Note: Use the information from 6.1.2 to determine whether a dilution factor should be used to determine qualification. When a dilution factor is applied to samples, the contaminant concentration in the samples is divided by the dilution factor. The criteria in Table 6-1 are used to qualify sample results.** *Action: For those metals identified in both the blank and sample, follow the directions in Table 6-1 below for qualifying data based on blank results.*	**Indicate yes or no:** No. No metals were detected in the blank. **If yes, list those analytes and results found in both the blanks and samples and summarize any actions taken:**

Example 2-9: Completed Blank Section of Mercury Checklist

7.1.1 Was a method/preparation blank included with each batch of samples (for each matrix)? *Action: If no method blank was included, consult the laboratory or applicable party and, if possible, have the data submitted. If the data is not available, the data validator may apply best professional judgment to qualify the sample results.*	**Indicate yes or no:** Yes, MBLK-2803 was prepared with this batch of samples.
7.1.2 Were any samples diluted? *Action: Record the sample ID and dilution factor(s).*	**Indicate yes or no:** No. **Record sample ID(s) and dilution factor(s):** The dilution factor for Sample ID SS-103 is shown to be 1.
7.1.3 Did the method blank contain mercury above detectable levels? Was mercury also detected in the sample results? If so, these results are subject to qualification. **Note: If mercury is discovered in the method blank above or equal to the quantitation limit, the lowest concentration of any sample in that batch must be 10X the method blank concentration (after dilution is accounted for). If this is not the case, all samples in that batch should have been re-digested and re-analyzed.** **The Laboratory is not to correct the sample concentration for the blank value.** *Action: Review the blank data. Use Table 7-1 below to qualify results. If the sample results are detected at concentrations greater than or equal*	**Indicate yes or no:** No, Mercury was not detected in the blank. **Summarize any actions taken:** N/A – Mercury was not detected in the blank, so no qualification is needed.

to the QL but less than 10 x the concentration in the blank, the results should have been redigested and reanalyzed.	

No inorganic analytes were detected in the method blank associated with this analytical batch, so results do not need to be qualified for these examples. Because of this, for convenience, Table 6-1 and Table 7-1 were not included in this appendix. These tables can be reviewed in Checklist #6 and Checklist #7.

3.0 Matrix Spikes and Matrix Spike Duplicates Worked Examples

The following examples demonstrate how to qualify analytical data using matrix spike and matrix spike duplicate results.

3.1 Example MS/MSD Analysis for VOCs

The laboratory results page for a soil sample from Boring B12 is listed in Figure 3-1.

Exhibit 3-1: Example VOC Analytical Results

Sample Results from Boring B12			
Report Date:	Oct. 22, 1999		
Sample Delivery Group:	C1986		
Client Name:	Ohio EPA		
Client Address:	122 S. Front St., Columbus, OH 43216		
Batch ID:	C9567	**Lab Sample ID**:	C0009184-23
Method:	8260B	**Extraction Date**:	Sept. 22, 1999
Matrix:	SOLID	**Analysis Date**:	Sept. 22, 1999
Sample ID:	B12		
Sample ID:	B12		
Analytes	**Result: Dry Weight (µg/Kg)**		**RL (µg/Kg)**
1,1-Dichloroethene	<5.0		5.0

Benzene	<5.0	5.0
N-Hexane	8.1	5.0
Toluene	7.4	5.0
Chlorobenzene	<5.0	5.0

QA/QC data that accompanied this report included the information presented in Exhibits 3-2 and 3-3, below. The Tier I Data Validator must note that not all target analytes are analyzed in the MS/MSD samples. In addition, the matrix spike duplicate used a different spiking level compared to the matrix spike (55.6 µg/Kg for the MSD compared to 64.7 µg/Kg for the MS). While different spiking levels are not expressly forbidden, an explanation from the laboratory is warranted. In general, the spike concentrations should be at the same level as the Laboratory Control Sample (LCS).

Figure 3-2: Example VOC Matrix Spike Results

Matrix Spike QA/QC Summary Data for VOCs				
Batch ID:			C9567	
QC Sample ID:			C0009184-23MS	
Sample Affected:			C0009184-23	
Analytes	**Result (µg/Kg)**	**% Recovery**	**QC Limits (%)**	**Spike Level (µg/Kg)**
1,1-dichloroethene	75.6	117	70-130	64.7
Benzene	50.6	74.0	70-130	64.7
N-hexane	36.9	44.5	70-130	64.7
Toluene	55.0	73.6	70-130	64.7
Chlorobenzene	39.3	60.7	70-130	64.7

Figure 3-3: Example VOC Matrix Spike Duplicate Results

Matrix Spike Duplicate QA/QC Summary Data for VOCs						
Batch ID:				C9567		
QC Sample ID:				C0009184-23MS		
Sample Affected:				C0009184-23		
Analytes	**Result (µg/Kg)**	**% Recovery**	**QC Limits (%)**	**Spike Level (µg/Kg)**	**RPD**	**RPD Limits**
1,1-dichloroethene	55.6	100	70-130	55.6	30.5	
Benzene	46.3	78.4	70-130	55.6	8.88	0-30
N-hexane	38.0	53.8	70-130	55.6	2.94	0-30
Toluene	47.7	72.5	70-130	55.6	14.2	0-30
Chlorobenzene	31.5	56.7	70-130	55.6	22.0	0-30

The analytical and QC data provided in the figures above were used to complete Example 3-1, which consists of sections from Tier I Data Validation Checklist #2. The first question from Checklist #2 asks the Tier I Validator to determine whether sufficient information exists to review MS/MSD data. One MS/MSD must be run per batch of 20 or fewer samples for each matrix for each SW-846 analytical method. Verification must also be made that the field blank samples were not used for spiked sample analysis.

Example 3-1: Completed MS/MSD Section of VOC Checklist

2.3.1 Is the matrix spike/matrix spike duplicate recovery data present? **Note: MS/MSD recovery data is more important for aqueous samples, so it is recommended that projects include review of MS/MSD recovery data for water samples in project DQOs.** *Action: If the matrix spike/spike duplicate data are required by the project-specific QAPP or DQOs but missing, the laboratory should be contacted for a resubmittal.*	**Indicate yes or no**: Yes, there is sufficient information to relate the batch QA/QC samples to each specific sample. Spike concentrations, percent recovery and relative percent difference information are also present.

The second question of Example 3-1 asks the Tier I Validator to determine whether any recoveries are outside of the quality control criteria. In this example, the laboratory has conveniently summarized the information in Figures 3-2 and 3-3. The Tier I Validator should note whether %R data is present for both the matrix spike and the matrix spike duplicate. However, RPD is only recorded for the matrix spike duplicate. The question does not specify which spike, the MS, or the MSD, is being referred to. The Tier I validator must note any %R data from either spike sample that is outside of the quality control criteria.

2.3.2 How many VOC spike recoveries are outside the QC limits?	**Record the spike recovery and control limits:** Record spike recovery(ies) and control limits. MS: 2 spike recoveries for N-hexane (44.5%) and chlorobenzene (60.7%) are outside of the 100% + 30% percent recovery criteria for batch C9567 which affects sample C0009184-23 MSD: 2 spike recoveries for N-hexane (58.8%) and chlorobenzene (56.7%) are outside of the 100% ± 30% percent recovery criteria for batch C9567 which affects sample C0009184-23.

If discrepancies from the QA/QC criteria are found, it is appropriate to determine if transcription or calculation errors may be responsible. If the MS/MSD produces low recoveries, it may be due to matrix effects, SW-846 method failure, inadequate background correction or inadequate clean up, improper spiking, degraded spiking solution or a failed spiking device [High MS/MSD recoveries may result from some of the same causes with the addition of possible use of contaminated reagents, gases, or glassware]. Equations 7.1 and 7.2 in Chapter 7 of the Tier I Data Validation Manual can help determine whether recording errors are a possibility. Using the data for N-hexane as an example, the following information was provided in the laboratory report.

Verify the calculations for at least one %R. Using the data for N-hexane as an example, the following information was provided in the laboratory report.

Exhibit 3-4: Example VOC Matrix Spike %R Calculation

Matrix Spike Recovery Calculation for N-Hexane		
	Result (µg/Kg)	
Sample result (Table 7.1)	8.1	Unspiked concentration (SR)
MS concentration (Table 7.2)	64.7	Conc. of Spike added (SA)
MS result (Table 7.1)	36.9	Spiked sample result (SSR)
%R = (SSR-SR)/(SA) X 100 Equation 7.1		
%R = (36.9 µg/Kg – 8.1 µg/Kg)/(64.7 µg/Kg) X 100 = 44.5%		

The result determined through use of equation 7.1, 44.5% is the same as reported in Exhibit 3-4. The Tier I Validator can therefore assume that calculations and transcription errors are minimal.

The next question of Example 3-1 asks the Tier I Data Validator to check the relative percent difference quality control criteria between the MS and MSD. The data to answer this question is found in Figure 3-3. According to quality control criteria listed in the table, RPDs must be below 20%.

2.3.4 How many relative percent differences (RPDs) for matrix spike and matrix spike duplicate recoveries are outside the QC limits for VOCs (≤20%)? **Note: The MS/MSD results may be used in conjunction with other QC criteria to determine the need for data qualification. Outliers should be identified.**	**Record the recovery data out of criteria and control limits. Review surrogate and LCS data to determine if qualification is necessary:** Record the recovery data out of criteria and control limits. Review surrogate recovery and LCS data to determine if qualifiers are necessary. 1,1-dichloroethene is outside of the control limit with and RPD of 30.5. This result affects batch C9567 and sample C0009184-23.

The data can also be used to recalculate a relative percent difference from MS/MSD data. Using the data for N-hexane again as an example, the following information has been provided in the laboratory report.

Exhibit 3-5: Example VOC Matrix Spike RPD Calculation

MS/MSD RPD Calculation for N-Hexane	
	Result (µg/Kg)
MS Result (Table 7.2)	36.9
MSD Result (Table 7.3)	38.0
RPD = (MSR-MSDR)/[(MSR + MSDR)/2] X 100 (Eq. 7.2)	
RPD = (38 µg/Kg – 36.9 µg/Kg)/[36.9 µg/Kg+38 µg/Kg/2] X 100 = **2.94**	

This result is the same as reported in Exhibit 3-3. The Data Validator can assume that transcription or calculation errors are minimal. However, based upon the different spiking levels for the MS/MSD, the relative percent difference results indicate little about reproducibility.

Based upon the matrix spike/matrix spike duplicate analysis, should the data be qualified? The answer is no, unless significant deviations are found in associated quality control data such as LCS results, or surrogate recoveries. However, it may be determined that the matrix of the spiked sample and its duplicate affected the recovery of analytes. The deviations should be noted in the data narrative. In addition, the different spike concentration levels are questionable.

If the spike was performed on a sample of interest, such as one the Validator submitted to the laboratory, the potential negative bias seen in the samples should be noted. To allay any concerns, the Tier I Data Validator may request MS/MSD information on batches with a similar matrix that were analyzed over a period that included the affected MS/MSD. If a trend is apparent, then data quality may be suspect. The results of the MS/MSD may not result in qualification of data, but it may lead the Tier I Data Validator to assess whether the data quality objectives of the sampling event were met.

3.2 Example MS/MSD Analysis for Metals

An example Tier I Checklist #6 Metal Spike Recovery section has been completed based on the following information:

Exhibit 3-6: Example Metals Matrix Spike Results

Matrix Spike QA/QC Summary Data for Metals							
Matrix Spike Analyte	DL (µg/L)	Sample Conc. (µg/L)	Spike Added (µg/L)	MS Conc. (µg/L)	MS % Rec (R)	% Rec. Limits	Data Qualifier
Barium	20	ND	1000	990	99	75-125	J
Cadmium	5	33	1000	896	86.3	75-125	

Chromium	70	519	620	1300	126	75-125	J+
Selenium	20	67	1000	905	89.9	75-125	
Lead - Soil	10	35	5000	5134	102	75-125	J

Exhibit 3-7: Example Metals Matrix Spike Duplicate Results

Matrix Spike Duplicate QA/QC Summary Data for Metals										
Matrix Spike Analyte Duplicate	DL (µg/L)	MSD Conc. (µg/L)	Spike Added (µg/L)	MS % Rec. (µg/L)	% RPD	QC Limits RPD (%)	LCS	LCS Limits (%)	% Rec.	Final Data Qualifier
Barium	20	1350	1000	135	30.7	0 - 20	1208	80-120	121	J+ or R
Cadmium	5	862	1000	82.9	4	0 - 20	789	80-120	79	J
Chromium	70	1100	620	93.7	29.4	0 - 20	616	80-120	99.3	J
Selenium	20	862	1000	79.5	5.3	0 - 20	490	80-120	49	R
Lead - Soil	10	5215	5000	103.6	1.6	0 - 20	5176	80-120	103.5	J+ or R

Example 3-2: Completed MS/MSD Section of 6010 Metals Checklist

6.3.1 Was at least one pre-digestion spiked sample (matrix spike) analyzed per batch, matrix type, or concentration or sample delivery group? *Action: If not present, flag detections "J", non-detections "UJ", and contact the applicable party for re-submittal.*	**Indicate yes or no:** Yes, at least one spiked sample was analyzed per batch. **If no, describe any actions taken**:

At least one spiked sample (pre-distillation/pre-digestion) will be prepared and analyzed from each group of samples with a similar matrix type (e.g., soil and water) and concentration (e.g., low, medium) for each SDG. The SDG may be either a case of field samples (set of 20 field samples in a case) or each 14-day calendar period during which a case of field samples are received, beginning with receipt of the first sample [if there is more than one spiked sample result per matrix, concentration level, sample delivery group, and individual SW-846 analytical method; if one of those spiked sample recovery results is not within control limit criteria, then flag all of the samples of the same matrix, level and SW-846 method in the sample delivery group]. The following worked example does not include all the questions contained in the checklist, but it will serve to illustrate the data validation process for metals data.

6.3.2 Are all spike recoveries within control limits (e.g., 75% to 125%)? **Note: Digestion method 3050B includes optional steps for constituents that are difficult to recover, such as Ag (See Section 7.5). When the spike sample result is less than the instrument detection limit, the percent recovery calculation should use a value of zero (not the detection limit) for the sample result.** *Action: Is the sample concentration ≥ 4 times the spiked concentration? If yes, spike recovery limits do not apply, and data is unqualified. If no, identify those analytes whose concentration is < 4 times the spike added (these would be analytes that should potentially be qualified using professional judgement and other QC results).*	**Indicate yes or no:** No. **If no, list analytes < 4 times the spike added:** Chromium had a spike recovery of 126% in the MS and Barium had 135% in the MSD. The spike concentration for Cr is 620 µg/L. The sample concentration is 519 µg/L. Since the sample concentration is NOT > 4X the spike concentration, this information should be noted in the data narrative and the analyte should be circled. Likewise, barium was not detected and should be circled.
6.3.3 Verify the calculations for at least one %R. Spike Percent Recovery (%R) % Recovery (%R)= $\frac{SSR-SR}{SA}$ X 100 Where: SSR=Spiked sample result SR=Sample result SA=Sample added	**Show results of verified %R calculation:** Cadmium %R= ((896-33)/1000)*100=86.3%
6.3.4 Based on the results of 6.3.2, if the spike recoveries are outside the control limits and the sample results were <4x the spike amount, a post-digestion spike should be analyzed at 2x the indigenous level or QL, whichever is greater. **Note: Post-digestion spikes are not required for Ag or Sb. However, one is typically run if the LCS was out of control. The post-digestion spike confirms a matrix interference and should not be used for qualification.** *Action: Contact the applicable party or laboratory for an explanation if a post-digestion spike was not performed and analyzed in the event that the LCS was out of control. If a satisfactory explanation is not available, use professional judgment to qualify sample results.*	**Summarize results of any post-digestion spikes and actions taken:** There is no evidence that a post-digestion spike was analyzed. The lab should be contacted for an explanation.

6.3.5 Is the %R (pre and post digestion) for any matrix type: 1. Less than 30%? 2. Between 30% and 74%? 3. Greater than 125%? **Note: The criteria in the table below are method requirements for spike samples of any matrix type. However, for technical review purposes only, the QAPP or project-specific DQOs for data review may allow the use of less restrictive criteria (e.g., 10 %R and 150 %R for the lower and upper limits) to be assessed against spike and post-digestion spike soil samples.** *Action: Use the criteria in the table below to determine whether the data needs to be qualified. If qualification is needed, take the necessary actions listed in Table 5-3.*	**Indicate yes or no:** No spike recoveries are less than 30%. No spike recoveries are between 30 and 70%. One spike recovery is 126% and one is 135%. No spike recoveries are >150%. **Summarize any actions taken:** The Cr and Ba results should be qualified as "J+". Both have high %R values in the MS and MSD and the RPDs above 20%. In addition, Cd is "J" flagged and Se is rejected due to low LCS. recoveries. Se also has a low MS %R value.
6.3.6 Was at least one MS and one duplicate unspiked sample, or one matrix spike/matrix spike duplicate (MS/MSD) pair analyzed for each batch of samples processed? **Note: If samples are expected to contain target analytes, laboratories may choose to use an MS and a duplicate analysis of an unspiked field sample. If samples are not expected to contain target analytes, the laboratories should use an MS/MSD pair. Duplicate samples are not required for wipe samples.** *Action: Verify that at least one duplicate sample was prepared and analyzed from each group of samples of a similar matrix type or for each data package.*	**Indicate yes or no:** yes **Summarize any actions taken:**
6.3.7 Are all relative percent difference (RPD) values within control limits? **Note: Acceptance criteria for RPD should be a set of laboratory-derived limits; however, acceptance limits must not exceed 20% for original and duplicate sample values ≥ 5x the QL. For samples analyzed under the Statement of Work (SOW), a control limit of the Quantitation**	**Indicate yes or no:** no **Summarize any actions taken:** - Barium = 30.7% Chromium = 29.4% __ Barium flagged with J+ or R Chromium J- flagged

Limit (QL) should be used if either the sample or duplicate value is < 5x the QL. *Action: Determine whether RPD values exceed laboratory-derived control limits. If control limits have not been developed, use ≤20% as the acceptance criteria.*	
6.3.8 RPD is calculated to evaluate the spike values for precision using the following equation: $\text{RPD} = \frac{\lvert S-D \rvert}{(\frac{S+D}{2})} \times 100$ Where: S = Sample Result (original) D = Duplicate Result When the sample or duplicate result is reported as a non-detect, use a value of zero (0) for calculating the RPD. This will always yield an RPD of 200%. *Action: Verify an RPD calculation for one set of MS/MSD samples. Contact the applicable party or laboratory for an explanation if RPD was not calculated. If a satisfactory explanation is not available, use professional judgment to qualify sample results.*	**Show results of one verified RPD calculation:** Cadmium $\text{RPD}=\frac{\lvert 896-862 \rvert}{(\frac{896+862}{2})} \times 100 = 3.89$

4.0 Laboratory Control Samples Worked Example

The following example will illustrate data validation procedures using laboratory control sample data. Example 4-1 is for semi-volatile organic data, but the validator should find it useful for evaluating volatile analytical data, as the checklist sections are identical.

4.1 Example Analytical Summary and QC Reports for LCS Evaluation

Exhibit 4-1 shows an analytical summary report for a soil sample analyzed for semi-volatile compounds. Exhibit 4-2 shows a summary QC report for the laboratory control sample that was included in the analytical batch.

Exhibit 4-1: Example LCS Report

Laboratory Control Sample					
Login Number: L0108493		Run Date: 08/29/2022		Sample ID: WG103297-03	
Instrument ID: HPMS5		Run Time: 16:50		Method: 8270E	
File ID: 5M18197		Analyst: CLK		Matrix: Solid	
Blank Workgroup: WG103827				Units: ug/kg	
Analyte	**Expected**	**Found**	**%Rec.**	**LCS Limits**	**Qual**
Acenaphthene	1670	1360	81.4	10-123	
Acenaphthylene	1670	1360	81.4	10-109	
Anthracene	1670	1530	91.6	10-149	
Benzo[a]anthracene	1670	1630	97.6	10-159	
Benzo[a]pyrene	1670	1650	98.8	10-152	
Benzo[b]fluoranthene	1670	1640	98.2	10-161	
Benzo[ghi]perylene	1670	1760	105	10-160	
Benzo[k]fluoranthene	1670	1620	97	10-165	
Chrysene	1670	1690	101	10-153	
Dibenz[ah]anthracene	1670	1780	107	10-169	
Fluoranthrene	1670	1610	96.4	10-158	
Fluorene	1670	2510	152	10-122	#
Indeno[1,2,3-cd]pyrene	1670	1740	104	10-162	
Naphthalene	1670	1210	72.5	10-99	
Phenanthrene	1670	1490	89.2	10-144	
Pyrene	1670	1600	95.8	10-161	

Exhibit 4-2: Example SVOC Analytical Results

Results					
Analytica Method: 8270E		Preparatory Method 8270/3550		% Solids: 86	
Matrix: Soil		Initial Calibration ID: HMPS5		Concentration Units: ug/kg	
Date Received: 08/22/2022		Date Extracted: 08/22/2022		Date Analyzed 08/31/2022	
Analyte	**MDL**	**RL**	**Concentration**	**Dilution**	**Qual**
Acenaphthene	959	1900	959	10	ND
Acenaphthylene	959	1900	2700	10	
Anthracene	959	1900	959	10	ND
Benzo[a]anthracene	959	1900	6100	10	
Benzo[a]pyrene	959	1900	7400	10	
Benzo[b]fluoranthene	959	1900	6100	10	
Benzo[ghi]perylene	959	1900	5600	10	
Benzo[k]fluoranthene	959	1900	4600	10	
Chrysene	959	1900	6000	10	
Dibenz[ah]anthracene	959	1900	959	10	ND
Fluoranthrene	959	1900	8900	10	
Fluorene	959	1900	959	10	ND
Indeno[1,2,3-cd]pyrene	959	1900	4400	10	
Naphthalene	959	1900	959	10	ND
Phenanthrene	959	1900	3500	10	
Pyrene	959	1900	11000	10	

4.2 Example of Completed Tier I Checklist for LCS Evaluation

Example 4-1 consists of a section of the SVOC checklist that was completed using the analytical and QC data from Exhibits 4-1 and 4-2.

Example 4-1: Example of Completed LCS Checklist Section for SVOCs

4.2 Semi-Volatile Data Review - Laboratory Control Sample (LCS)	
An LCS should be included with each batch of samples (approx. 20). The LCS consists of an aliquot of a clean (control) matrix similar to the matrix type of the sample and at the same weight or volume. The LCS is spiked with the same analytes at the same concentration as the matrix spike. When the results of the matrix spike indicate a potential problem due to the sample matrix itself, the LCS verifies that the laboratory can perform analyses in a clean matrix.	
4.2.1 Was an LCS prepared, extracted, analyzed, and reported once per batch of 20 samples? *Action: If LCS information is not present, consult the facility or laboratory for re-submission of the data package. If LCS information is not available, qualify all detected results as "J" and all nondetect results as "UJ" or reject all results based on best professional judgment.t*	**Indicate yes or no:** Yes.
4.2.2 Does the LCS contain the following semi-volatile target compounds in addition to the required surrogates? Base/Neutrals Acids 1,2,4-Trichlorobenzene Pentachlorophenol Acenaphthene Phenol 2,4-Dinitrotoluene 2-Chlorophenol Pyrene 4-Chloro-3-methylphenol N-Nitroso-di-n-propylamine 4-Nitrophenol 1,4-Dichlorobenzene **Note: Method 3500C calls for base/neutral compounds to be spiked at 100 mg/L and acid compounds to be spiked at 200 mg/L. However, for waste samples the concentration should be 5 times higher. Other compounds can be spiked into the LCS; however, these compounds should represent the entire range of target analytes. In addition, the compounds in the LCS should be consistent with the compounds included in the matrix spike/matrix spike duplicate.**	**Indicate yes or no:** No. **Identify any target compounds that were present:** The compounds in the LCS only contain PAH compounds of interest and not compounds included within the acid fraction. An explanation was sought from the laboratory. The laboratory responded that only base/neutral fraction analysis was being performed for this batch of samples and therefore acid fraction surrogates were not necessary.
4.2.3 Do the percent recoveries (%R) meet the QC limits provided by the lab? **NOTE: The laboratory should use 70 - 130% as interim acceptance criteria for recoveries of spiked analytes, until in-house LCS limits are developed** *if more than half of the compounds in the LCS are not within the recovery criteria, then all of the associated detected*	**List compounds and sample IDs that do not meet QC limits:** No. The LCS recovery for fluorene (% R = 152) were outside of the acceptance criteria (10-122). Fluorene was not detected in the sample above the reporting limit, but at the MDL. The LCS

target compounds should be qualified as "J," and all associated non-detected compounds should be qualified "R." *Action: Follow the directions in Table 4-2 for qualifying results.*	recovery was above the criteria. Therefore, no qualification is necessary
4.2.4 Verify the calculations for at least one %R. %R = $(\frac{Measured\ Concentration}{Spiked\ amount})$ X 100 *Action: If the %R is not calculated correctly, verify the other %R calculations and/or contact the lab for re-submission. If the recalculated %R values fall within the QC limits, the Data Validator should use professional judgment to determine if the lab should be contacted for re-submission or if the data should be flagged.*	**Show results of verified %R calculation** %R for Fluorene: % R = 2540 /1670 = 1.521 X 100 = 152%

Table 4-2: LCS Actions for SVOC Analyses

Qualification		
LCS Result	Sample Result	Action
> Upper acceptance limit	Detection	J+
< Lower acceptance limit	Detection	J-
< Lower acceptance limit	Non-detect	R
≥ Half target compounds not within recovery criteria	All detections	J
≥ Half target compounds not within recovery criteria	All non-detects	R

Based upon strict conformance with data validation principles, no qualification of the results is necessary based on LCS results. However, the results from the data validation indicate that the laboratory was not in exact conformance with SW-846 Method 8270E for the LCS compounds. If the Data Validator should encounter this problem, a review of the compounds included in the matrix spike/matrix spike duplicate data should be performed. If the compounds are not the same or do not have the same concentrations, the laboratory of the facility should be contacted for an explanation. Without correspondence between these batch QC samples, it will be difficult for the Data Validator to determine whether a matrix interference is present or a system analytical problem exists.

5.0 Surrogate Worked Examples

Surrogates are used to qualify VOC sampling results using Checklist #2, and SVOC sampling results using Checklist #4.

5.1 Example Surrogate Analysis for VOCs

An example showing how to validate soil data is presented using Section 2.4 of the Tier I Data Validation Checklist for VOCs and the following information. Sample results for analytes and surrogate compounds are shown in Exhibit 5-1 below.

Exhibit 5-1: Analytical and Surrogate Results for VOCs

Sample: OH-1-1 Lab ID: 92437606001 Collected: 01/01/2021 12:20 Matrix: solid

Analyte(s)	Result	RDL	Unit	SW-846 Method #	Flag
Chlorobenzene	10	6.1	ug/kg dry	SW-846 8260D	
Ethylbenzene	ND	6.1	ug/kg dry	SW-846 8260D	
o-Xylene	ND	6.1	ug/kg dry	SW-846 8260D	

Surrogate compounds	Surrogate Added	Surrogate Result	Unit	% Recovery	QC Limits
Surrogate: 1,2-Dichloroethane-d4	2,000	2,760	ug/kg	138%	(70-130)
Surrogate: Bromofluorobenzene	2,000	2,200	ug/kg	110%	(70-130)
Surrogate: Toluene-d8	2,000	1,880	ug/kg	94%	(70-130

Example 5-1: Completed Surrogate Section of VOC Checklist

2.4.1 Are the surrogate recovery data present for each batch (method and matrix), including TCLP?	**Indicate yes or no:** Yes, surrogate recovery results are present.

Note: Samples may be included in different batches. When this is the case, separate surrogate recoveries should be provided. *Action: If no, then contact the laboratory for an explanation and report re-submittal.*	
2.4.2 Are any surrogate recoveries are outside the QC limits? **Note: Suggested surrogate recovery limits are 70 to 130% until laboratory or project-specific criteria are developed. QC limits will depend on the surrogates chosen, levels used, and instrument conditions. Acceptance criteria is guidance.** *Action: Identify samples with recoveries outside QC limits.*	**Indicate yes or no:** Yes **If yes, list the sample ID(s), matrix(-ces) and parameter(s):**The surrogate recovery for 1,2-Dichloroethane-d4 under Lab ID 92437606001, was 138% which was above the upper quality Control Criteria of 130%.
2.4.3 Verify the calculations for at least one %R. $Recovery\ \% = \frac{Concentration\ found}{Concentration\ Added}\ x\ 100$	%R = 2760 / 2000 x 100 = 138%
2.4.4 If any surrogate compound was out of compliance was re-analysis performed to confirm a matrix interference? **Note: Check the report narrative for an indication of re-analysis. Additionally, qualification may not be appropriate for TCLP data. Best professional judgment should be used to qualify data.** *Action: Based on the findings, qualify data using the following criteria in Table 2-3 below.*	No reanalysis was performed. Since 1,2-Dichloroethane-d4 was above the upper control limit, the detected result (10) for Chlorobenzene should be qualified as "J+".

Table 2-3: Surrogate Actions for VOC Analyses

	Action	
	Detect	**Non-detect**
Surrogate not present or not at specified concentration	J or R	UJ or R

%R < Expanded Lower Acceptance Limit (10%, excluding surrogates with 10% as a lower acceptance limit, undiluted sample analysis)	J-	R
Expanded Lower Acceptance Limit (10%) ≤ %R (excluding surrogates with 10% as a lower acceptance limit, undiluted sample analysis) < specified Lower Acceptance Limit	J-	UJ
%R < specified Lower Acceptance Limit (diluted sample analysis)	Use professional judgment	Use professional judgment
%R within specified Acceptance Limits	No qualification	No qualification
%R > specified Upper Acceptance Limit	J+	No qualification

In the example, there is no indication that re-analysis was performed. For a real sample report, the Tier I Data Validator must check the data narrative for an indication of re-analysis. If no indication of re-analysis can be found, good data validation practices require that the facility or its laboratory be contacted and confirmation of re-analysis be obtained. If no information is available, the Tier I Data Validator, at his or her discretion, may qualify the affected data using best professional judgment. The Tier I Data Validator is directed to consult with the district Tier II representative for advice.

In example 5-1, 1,2-Dichloroethane-d4, was found to have a percent recovery of 138% which is above the upper bounds of the quality control criteria. In the example, only Chlorobenzene was detected. This value should be flagged with a "J+" to indicate an estimated quantity. The other parameters were not detected and therefore do not require qualification.

The VOC data indicates that Chlorobenzene was detected at 10 micrograms per kilogram (ug/kg) while Ethylbenzene and o-Xylene were non-detect. The surrogate data shows that 1,2-Cichloroethane-d4 is above the upper control limit while Bromofluorobenzene and o-Xylene were within the specified limits.

Since one surrogate compound was above the upper control limit, good data validation practices imply that Chlorobenzene, which is a detected result, be qualified as "J+," estimated. Ethylbenzene and o-Xylene would not be qualified. The qualified laboratory report for Example 5-1 would resemble the following:

Exhibit 5-2: Qualified Laboratory Results for Example 5-1

Analyte(s)	Result	RDL	Unit	SW-846 Method #	Flag
Chlorobenzene	10	6.1	ug/kg dry	SW-846 8260D	J+
Ethylbenzene	ND	6.1	ug/kg dry	SW-846 8260D	
o-Xylene	ND	6.1	ug/kg dry	SW-846 8260D	

5.2 Example Surrogate Analysis for SVOCs

An example showing how to validate soil data is presented using Section 4.4 of the Tier I Data Validation Checklist for SVOCs and the following information. Sample results for analytes and surrogate compounds are shown in Exhibit X below.

Exhibit 5-3: Example Analytical and Surrogate Data for SVOCs

Sample: OH-1-1 Lab ID: 92437606001 Collected: 01/01/2021 12:20 Matrix: water

Analyte(s)	Result	RDL	Unit	SW-846 Method #	Flag
Pyridine	ND	0.05	mg/l	SW-846, 8270E	
Nitrobenzene	50	0.05	mg/l	SW-846, 8270E	
Hexachlorobenzene	ND	0.05	mg/l	SW-846, 8270E	

Surrogate compounds	Surrogate Added	Surrogate Result	Unit	% Recovery	QC Limits
Surrogate: Nitrobenzene-d5	2,000	40	mg/l	2%	(4-140)
Surrogate: 2-Fluorobiphenyl	2,000	180	mg/l	9%	(22-160)
Surrogate: p-Terephenyl-d14	2,000	960	mg/l	48%	(18-137)

Example 5-2: Completed Surrogate Section of SVOC Checklist

4.4.1 Are the surrogate recovery data present for each batch (method and matrix), including TCLP? **Note: Samples may be included in separate sample batches and separate surrogate recoveries should be provided.** *Action: If no, contact the laboratory for explanation and re-submittals.*	**Indicate yes or no**: Yes, surrogate recoveries are present.

4.4.2 Are any surrogate recoveries are outside the QC limits? $\boldsymbol{Recovery}\ \% = \frac{\boldsymbol{Concentration\ found}}{\boldsymbol{Concentration\ added}}\ \boldsymbol{x100}$ **Note: Suggested surrogate recovery limits are 70 to 130% until laboratory or project-specific criteria are developed. QC limits will depend on the surrogates chosen, levels used, and instrument conditions. Acceptance criteria is guidance.** *Action: Identify samples with recoveries outside QC limits*	**Indicate yes or no and then list the sample ID(s), matrix(-ces) and parameter(s) of the outliers:** Yes: Surrogate recoveries for Nitrobenzene-d5 (criteria: 4-140%) and 2-Fluorobiphenyl (criteria 22-160%) for Lab ID: 92437606001, solid matrix, were outside of the lower control limits of the quality control criteria.
4.4.3 Verify the calculations for at least one %R.	**Show results of verified %R calculation:** %R = 180 / 2000 x100 = 9%
4.4.4 Were any TWO surrogate compounds in either the acid or base/neutral fractions out of compliance, was re-analysis performed to confirm a matrix interference? **Note: Check the report narrative for an indication of re-analysis.** *Action: If no information is present, request information from the applicable party or laboratory.*	**List sample ID(s) for surrogate compounds out of compliance and criteria:** Surrogate recoveries for Nitrobenzene-d5 and 2-Fluorobiphenyl for Lab ID: 92437606001are out of compliance (below lower control limit of the quality control criteria). There is no indication of re-analysis in the example. The facility or its laboratory must be contacted and confirmation of re-analysis must be obtained.
4.4.5 Were any surrogate recoveries less than 10% in either the acid or base/neutral fractions? **Note: Check the report narrative for an indication of re-analysis.** *Action: If no information is present, request information from the applicable party or laboratory.*	**List sample ID(s) for surrogate compounds out of compliance and criteria:** Both Nitrobenzene-d5 (base/neutral) and 2-Fluorobiphenyl (base/neutral), under lab ID 92437606001, have less than 10% recovery.
4.4.6 Based on the findings, qualify data in either the acid or base/neutral fractions with the criteria in Table 4-3 below	List the ID(s) of the affected sample(s): Both Nitrobenzene-d5 and 2-Fluorobiphenyl have less than 10% recoveries. All detected compounds under lab ID 92437606001, in base/neutral fraction should be qualified as "J-,"

	estimated, and all non-detect compounds should be rejected and flagged with an "R." For this example, Nitrobenezene was detected, and therefore, this data should be qualified as estimated and flagged with a "J-." Both Pyridine and Hexachlorobenzene were not detected, and therefore, these data should be rejected and the results flagged with an "R."

Table 4-3: Surrogate Actions for SVOC Analyses

	Action	
	Detect	**Non-detect**
Surrogate not present or not at specified concentration	J or R	UJ or R
%R < Expanded Lower Acceptance Limit (10%, excluding surrogates with 10% as a lower acceptance limit, undiluted sample analysis)	J-	R
Expanded Lower Acceptance Limit (10%) ≤ %R (excluding surrogates with 10% as a lower acceptance limit, undiluted sample analysis) < specified Lower Acceptance Limit	J-	UJ
%R < specified Lower Acceptance Limit (diluted sample analysis)	Use professional judgment	Use professional judgment
%R within specified Acceptance Limits	No qualification	No qualification
%R > specified Upper Acceptance Limit	J+	No qualification

The SVOC data indicates that Nitrobenzene was detected at 50 milligrams per liter while Pyridine and Hexachlorobenzene were not detected. The surrogate data show that Nitrobenzene-d5 and 2-Fluorobiphenyl are below the lower QC control limits; and p-Terephenyl is within the specified limits.

Since two surrogate recoveries are below 10% in Example 5-2, Nitrobenzene, which is a detected compound, would be qualified as estimated and the data flagged with a "J." Both Pyridine and Hexachlorobenzene were not detected. They would be qualified as rejected and the data flagged with an "R". The lab results would look like this:

Exhibit 5-4: Qualified Laboratory Results for Example 5-2

Analyte(s)	Result	RDL	Unit	SW-846 Method #	Flag
Pyridine	ND	0.05	mg/l	SW-846, 8270E	R
Nitrobenzene	50	0.05	mg/l	SW-846, 8270E	J-
Hexachlorobenzene	ND	0.05	mg/l	SW-846, 8270E	R

6.0 Vapor Intrusion Worked Example

The following TO-15 analytical and QC data shown in Exhibits 6-1 through 6-4 were used to fill out Checklist #3: Vapor Intrusion, as shown in Example 6-1.

Exhibit 6-1: Example Vapor Intrusion Analytical Data

TO-15 Analysis					
Project: Clean Cleaners		Sample ID: IA-1 03062022		Lab ID: 162392-01	
Collection Date: 3/6/2022		Analysis Date: 3/6/2022		Matrix: Air	
Analyte	**Result**	**Qual**	**Reporting Limit**	**Unit**	**Dilution Factor**
1,2,4-Trimethylbenzene	0.89		0.50	ppbv	1
2-Butanone	1.5		0.50	ppbv	1
Tetrachloroethene	20		0.50	ppbv	1
Trans-1,2-Dichloroethene	ND		0.50	ppbv	1
Trichloroethene	ND		0.20	ppbv	1
Vinyl chloride	ND		0.50	ppbv	1
Surr: Bromofluorobenzene	89.1		60-140	%R	1

Exhibit 6-2: Example VI Blank Data

MBLK						
Lab ID: MBLK-162392		Units: ppbv		Analysis Date: 3/6/2022		
Analyte	**Result**	**PQL**	**SPK Val**	**SPK Ref Val**	**% Rec**	**Control Limit**
1,2,4-Trimethylbenzene	ND	0.50				
2-Butanone	ND	0.50				
Tetrachloroethene	ND	0.50				
Trans-1,2-Dichloroethene	ND	0.50				
Trichloroethene	ND	0.20				
Vinyl chloride	ND	0.50				
Surr: Bromofluorobenzene	8.5	0	10	0	85	60-140

Exhibit 6-3: Example VI Laboratory Control Sample Data

LCS						
Lab ID: LCS-R162392		Units: ppbv		Analysis Date: 3/6/2022		
Analyte	**Result**	**PQL**	**SPK Val**	**SPK Ref Val**	**% Rec**	**Control Limit**
1,2,4-Trimethylbenzene	10.7	0.50	10	0	107	50-162
2-Butanone	9.26	0.50	10	0	92.6	60-140
Tetrachloroethene	10.07	0.50	10	0	101	60-140
Trans-1,2-Dichloroethene	8.97	0.50	10	0	89.7	60-140
Trichloroethene	14.5	0.20	10	0	145	60-140
Vinyl chloride	9.45	0.50	10	0	95	60-140
Surr: Bromofluorobenzene	9.7	0	10	0	97	60-140

Exhibit 6-4: Example VI Field Duplicate Data

TO-15 Duplicate Analysis				
Project: Clean Cleaners Collection Date: 3/6/2022	Analysis Date: 3/6/2022	Sample ID: IA-1 03062022	Lab ID: 162392-01-DUP	Matrix: Air
Analyte	**Native Result**	**Duplicate Result**	**RPD (%)**	**RPD Control Limits (%)**
1,2,4-Trimethylbenzene	0.89	0.97	9	≤25
2-Butanone	1.5	1.8	18	≤25
Tetrachloroethene	20	18	11	≤25
Trans-1,2-Dichloroethene	ND	ND	0	≤25
Trichloroethene	ND	ND	0	≤25
Vinyl chloride	ND	ND	0	≤25
Surr: Bromofluorobenzene	89.1	88.6	0.56	≤25

Example 6-1: Completed Vapor Intrusion Checklist

3.0 Vapor Intrusion Data Validation	
3.1 Vapor Intrusion Data Review – Blank Data	
Method blanks are used to assess whether contamination is from the laboratory and consists of an unused, certified canister that has not left the laboratory. The blank canister is pressurized with humidified, ultra-pure zero air and carried through the same analytical procedure as a field sample. The blank aliquot must contain the same amount of internal standards that are added to each sample. The qualification of sample results will depend upon the magnitude of blank contamination.	
3.1.1 Was a method blank analyzed for each sample batch? *Action: If not present, request information from the applicable party or laboratory. If the required method blank(s) was not analyzed, sample results may be qualified as estimated ("J" for detected results and "UJ" for non-detected compounds) based upon the validator's judgment. Additional*	**Indicate yes or no:** Yes

3.0 Vapor Intrusion Data Validation	
3.1 Vapor Intrusion Data Review – Blank Data	
qualification may be warranted based upon other QA/QC information.	
3.1.2 Is there an indication that the samples associated with the method blank were diluted? **Note: Whenever a high concentration sample is outside the calibration range, a blank analysis should also be performed immediately after the sample to check for carryover effects. The dilution factor can be found in the data report (a dilution factor of 1 indicates no dilution).**	**Indicate yes or no:** No. The dilution factor is shown to be 1. **If yes, list the sample ID(s) and dilution factor(s):** N/A
3.1.3 Do any method blanks have any detected results for any volatile target analytes? Were the same target compounds found in the samples? **Note: A list of samples associated with each of the contaminated blanks should be prepared. Trip blanks are used to qualify samples based on potential contamination during shipment and are not required for non-aqueous matrices.** *Action: Use the criteria in Table 3-1 below to qualify sample results due to blank contamination. Use the largest value from all associated blanks. If any blanks are grossly contaminated, all associated data may be qualified as "R", based upon professional judgment and the project's DQOs.*	**Indicate yes or no:** No. **If yes, list those analytes and results found in both the blanks and samples:** No. **Summarize sample result qualifications based on blank results:** No qualifications are needed based on blank results.
3.1.4 Was a field blank prepared and analyzed?	**Indicate yes, no, N/A:** No.
3.1.5 Were field blank results 20 pptv or less? *Action: If field blank results do not meet acceptance criteria, then examine preparation and sample handling procedures to evaluate reported data for associated field samples based on project DQOs.*	**Indicate yes, no, or N/A:** N/A **Explain any results that do not meet acceptance criteria:**

Table 3-1: Blank Actions for TO-15 Analyses		
Qualification		
Blank Result	Sample Result	Action
Detected	Not detected	No Action
< QL	< QL (2x QL for common laboratory contaminants)	Report QL with a U

Table 3-1: Blank Actions for TO-15 Analyses		
Qualification		
	≥ QL (2x QL for common laboratory contaminants) and < 2x QL (4x QL for common laboratory contaminants)	Report Sample Concentration with a U
	≥ 2x QL (4x QL for common laboratory contaminants)	No Action
> QL	< QL (2x QL for common laboratory contaminants)	Report QL with a U
	≥ QL (2x QL for common laboratory contaminants) and ≤ Blank Result	Report blank value for sample concentration with a U
	≥ QL (2x QL for common laboratory contaminants) and > Blank Result	No Action
= QL	≤ QL (2x QL for common laboratory contaminants)	Report QL with a U
	> QL (2x QL for common laboratory contaminants)	No Action
Gross Contamination*	Detects	Report blank value for sample concentration with a U

* Gross contamination is blank contamination > 2x the QL or 4x the QL for common laboratory contaminants.

3.2 Vapor Intrusion Data Review - Laboratory Control Sample (LCS) and Laboratory Control Sample Duplicate (LCSD)	
An LCS and LSCD should be included with each batch of samples (approximately 20). The LCS/LCSD are analyzed using a concentration in the middle of the calibration range and under the same conditions as samples to be analyzed.	
3.2.1 Was an LCS prepared, analyzed, and reported once per 24-hour analytical sequence and concurrently with the samples in each sample delivery group? **Note: This information should be included in the QA package provided by the lab. If not, contact the laboratory and request that the information be submitted.** *Action: If LCS information cannot be found, contact the applicable party or laboratory for re-submittal of the data package. If LCS information is not present, qualify all detected results as "J"*	**Indicate yes or no:** Yes. Samples were collected and analyzed on the same day. **Date(s) LCS analyzed:** 3/6/2022 **Summarize any actions taken:** N/A

3.2 Vapor Intrusion Data Review - Laboratory Control Sample (LCS) and Laboratory Control Sample Duplicate (LCSD)	
and all non-detect results as "UJ" or reject all results based on best professional judgment.	
3.2.2 Do the %R meet the suggested QC limits or limits provided by the lab? **Note: Acceptance criteria of 70 - 130% should be used until appropriate laboratory or project-specific limits are developed.** *Action: Identify samples with recoveries outside QC limits and follow the directions in the table below for qualifying samples results.*	**Indicate yes or no:** No. **If no, list compounds and sample IDs that do not meet QC limits:** %R for all analytes besides trichloroethene are within acceptance criteria. Trichloroethene had a %R of 145, while the limits are listed as 60-140%. **Summarize actions taken:** %R for trichloroethene in the LCS is above the upper acceptance limit. However, trichloroethene was not detected in the sample. Based on Table 3-2 of this checklist, this result does not need to be qualified and no actions need to be taken.
3.2.3 Verify the calculations for at least one %R. $$\%R = \left(\frac{Measured\ Concentration}{Spiked\ Amount}\right) \times 100$$ *Action: If the %R is not calculated correctly, verify the other %R calculations and/or contact the lab for re-submittal. If the re-calculated %R values fall within the QC limits, the validator should use professional judgment to determine if the lab should be contacted for re-submittal or if the data should be flagged.*	**Show results of verified %R calculation:** The result for 1,2,4-Trimethylbenzene in the LCS is 10.7 ppbv. It was originally spiked at 10 ppbv. %R = 10.7 / 10 x 100 = 107%
3.2.4 Do the RPD meet the suggested QC limits or limits provided by the lab? **Note: Acceptable precision analyses should demonstrate RPD ≤ 25% for each target analyte when both measurements are ≥ 5x the method detection limit (MDL).** *Action: follow the instructions in Table 3-2 for qualifying sample results outside QC limits.*	**Indicate yes, no, or N/A:** N/A – RPD was not calculated because an LCSD was not analyzed. **If no, list compounds and sample IDs that do not meet QC limits and explain any discrepancies:** **Summarize any actions taken:** N/A

Table 3-2: LCS/LCSD Actions for TO-15 Analyses		
Criteria	**Action**	
	Detected Associated Compounds	**Non-detected Associated Compounds**
Percent Recovery Criteria		
%R > Upper Acceptance Limit	J	No Action
%R in Acceptance Range	No Action	
%R < Lower Acceptance Limit	J	UJ
%R < 50%	J	R
Relative Percent Difference Criteria		
% RPD ≤ 25%	No Action	
% RPD > 25%	J	UJ

3.3 Vapor Intrusion Data Review – Precision Measurements	
The precision of the method can be assessed by analyzing collocated or duplicate sample and replicate sample analyses. Collocated precision is determined by analyzing samples of the same air mass that were collected simultaneously in two discrete canisters through two separate inlets. Duplicate precision is determined by analyzing samples of the same air mass that were collected simultaneously in two discrete canisters through the same sampling inlet. Replicate precision is determined from repeated analysis of a sample from one canister. Analysis of collocated or duplicate samples determines the precision of both the sampling and analysis processes, however replicate analysis determines only the precision of the analytical process. Field QC samples are not required but provide additional verification that the data being collected are reliable. The canister valve is not opened in the field, so the field QC samples should not become contaminated or otherwise compromised. A field spike is a prepared by filling a cannister with humidified gas at a concentration in the lower third of the calibration curve and should be interspersed among field samples during analysis.	
3.3.1 Did the project SAP, QAPP, or DQOs include collecting collocated or duplicate samples? Note: Method TO-15A suggests collecting collocated or duplicate samples at a rate equal to approximately 5% of the total number of samples. *Action: If yes, then contact the applicable party for an explanation if results are not included in the report provided.*	**Indicate yes, no, or N/A:** Yes. A collocated field duplicate was required, and results for it are provided.
3.3.2 Was the RPD for each pair ≤25% for when both measurements are ≥5x the MDL? $$RPD = \left\| \frac{X_1 - X_2}{\left(\frac{X_1 + X_2}{2}\right)} \right\| \times 100$$ Where:	**Indicate yes, no, or N/A:** Yes. All RPDs are less than 25%. **Summarize any actions taken:** N/A

3.3 Vapor Intrusion Data Review – Precision Measurements	
X_1 = target VOC measured in first measurement X_2 = target VOC measured in the second measurement *Action: If RPD do not meet acceptance criteria, then contact the applicable party or laboratory to investigate the reason for the discrepancy and evaluate results based on project DQOs.*	
3.3.3 Was a field spike prepared and analyzed? If so, were field spike results within ±30% of the spiked concentration? *Action: If field spike results do not meet acceptance criteria, then examine sample preparation and handling procedures and qualify reported data for associated field samples based upon professional judgment and the DQOs for the data.*	**Indicate yes, no, or N/A:** N/A – No field spike results were provided. **Summarize any actions taken:** N/A

7.0 TPH Worked Example

This section provides sample analytical data, QC data, and a completed checklist (Checklist #5) for Total Petroleum Hydrocarbons (TPH).

7.1 TPH Example Analytical and QC Data

This section contains snipped sections of a laboratory report that were used to complete Example 7-1.

Exhibit 7-1: Example Detection Summary

Client Sample ID: HB-06 (4-6')-081016 Lab Sample ID: 240-68175-24

Analyte	Result	Qualifier	RL	MDL	Unit	Dil Fac	D	Method	Prep Type
Gasoline Range Organics [C6 - C10]	420000	B	33000	14000	ug/Kg	5	☼	8015B	Total/NA
Diesel Range Organics [C10 - C28]	1400		200	76	mg/Kg	10	☼	8015B	Total/NA
Arsenic	8.5		0.81	0.33	mg/Kg	1	☼	6010B	Total/NA
Barium	47		16	0.33	mg/Kg	1	☼	6010B	Total/NA
Cadmium	0.11	J	0.16	0.017	mg/Kg	1	☼	6010B	Total/NA
Chromium	12		0.40	0.060	mg/Kg	1	☼	6010B	Total/NA
Lead	8.1		0.24	0.16	mg/Kg	1	☼	6010B	Total/NA
Nickel	21	B	3.2	0.064	mg/Kg	1	☼	6010B	Total/NA
Zinc	53	B	1.6	0.45	mg/Kg	1	☼	6010B	Total/NA

Exhibit 7-2: Analytical Data from HB-06 (4-6') – 081016 Lab Sample ID: 240-68175-24

Client Sample ID: HB-06 (4-6')-081016
Date Collected: 08/10/16 14:30
Date Received: 08/11/16 08:35

Lab Sample ID: 240-68175-24
Matrix: Solid
Percent Solids: 85.7

Method: 8015B - Gasoline Range Organics - (GC)

Analyte	Result	Qualifier	RL	MDL	Unit	D	Prepared	Analyzed	Dil Fac
Gasoline Range Organics [C6 - C10]	420000	B	33000	14000	ug/Kg	☼	08/18/16 05:46	08/23/16 07:51	5

Surrogate	*%Recovery*	*Qualifier*	*Limits*	*Prepared*	*Analyzed*	*Dil Fac*
Trifluorotoluene (Surr)	*91*		*36 - 157*	*08/18/16 05:46*	*08/23/16 07:51*	*5*

Method: 8015B - Diesel Range Organics (DRO) (GC)

Analyte	Result	Qualifier	RL	MDL	Unit	D	Prepared	Analyzed	Dil Fac
Diesel Range Organics [C10 - C28]	1400		200	76	mg/Kg	☼	08/12/16 08:56	08/18/16 19:38	10

Exhibit 7-3: Lab narrative for HB-06 (4-6')- 081016 Lab Sample ID: 240-68175-24

GASOLINE RANGE ORGANICS (GRO)

Samples HB-06 (0-2')-081016 (240-68175-22) and HB-06 (4-6')-081016 (240-68175-24) were analyzed for gasoline range organics (GRO) in accordance with EPA SW-846 Method 8015B - GRO. The samples were analyzed on 08/23/2016.

Samples HB-06 (0-2')-081016 (240-68175-22)[5X] and HB-06 (4-6')-081016 (240-68175-24)[5X] required dilution prior to analysis. The reporting limits have been adjusted accordingly.

Gasoline Range Organics [C6 - C10] was detected in method blank MB 240-243131/1-A at a level that was above the method detection

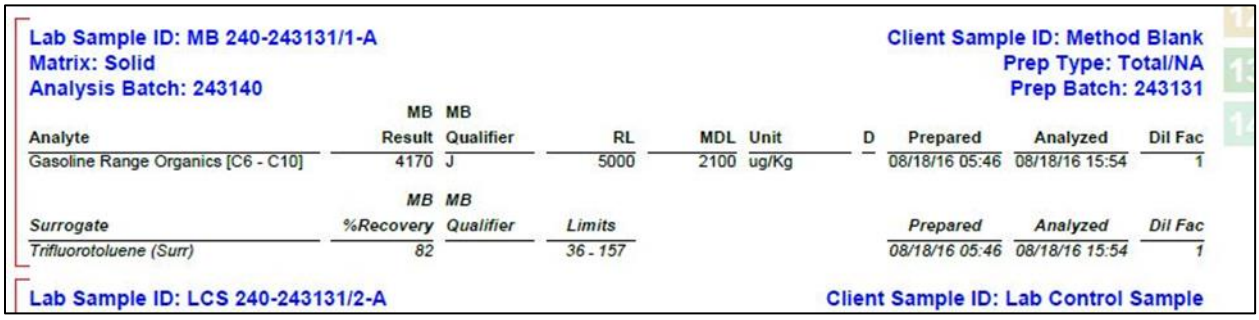
Exhibit 7-4: Surrogate Summary Table for HB-6 (4-6')

Method: 8015B - Gasoline Range Organics - (GC)
Matrix: Solid
Prep Type: Total/NA

		Percent Surrogate Recovery (Acceptance Limits)
Lab Sample ID	Client Sample ID	TFT2 (36-157)
240-68175-22	HB-06 (0-2')-081016	96
240-68175-24	HB-06 (4-6')-081016	91
LCS 240-243131/2-A	Lab Control Sample	101
MB 240-243131/1-A	Method Blank	82

Surrogate Legend
TFT = Trifluorotoluene (Surr)

Exhibit 7-5: Method Blank for HB-06 (4-6') - Method Blank ID MB240-243131/1-A



Lab Sample ID: MB 240-243131/1-A
Matrix: Solid
Analysis Batch: 243140

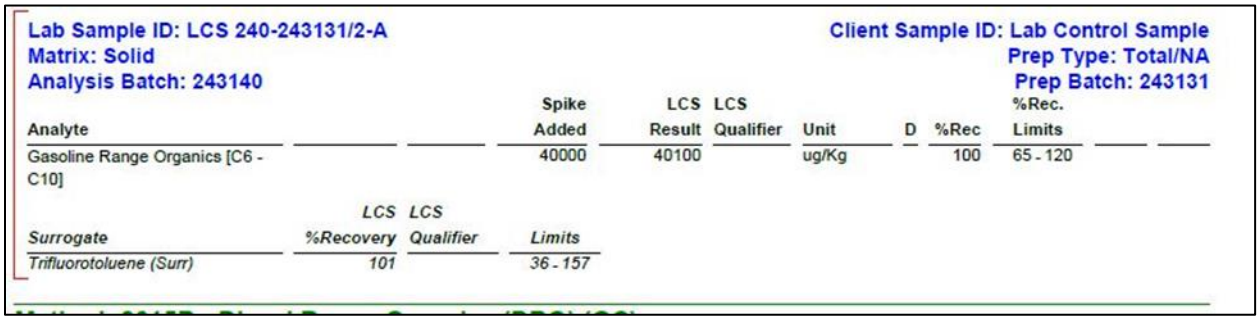
Client Sample ID: Method Blank
Prep Type: Total/NA
Prep Batch: 243131

Analyte	MB Result	MB Qualifier	RL	MDL	Unit	D	Prepared	Analyzed	Dil Fac
Gasoline Range Organics [C6 - C10]	4170	J	5000	2100	ug/Kg		08/18/16 05:46	08/18/16 15:54	1

Surrogate	*MB %Recovery*	*MB Qualifier*	*Limits*	*Prepared*	*Analyzed*	*Dil Fac*
Trifluorotoluene (Surr)	*82*		*36 - 157*	*08/18/16 05:46*	*08/18/16 15:54*	*1*

Lab Sample ID: LCS 240-243131/2-A — Client Sample ID: Lab Control Sample

Exhibit 7-6: LCS Sample for GRO batch 243131



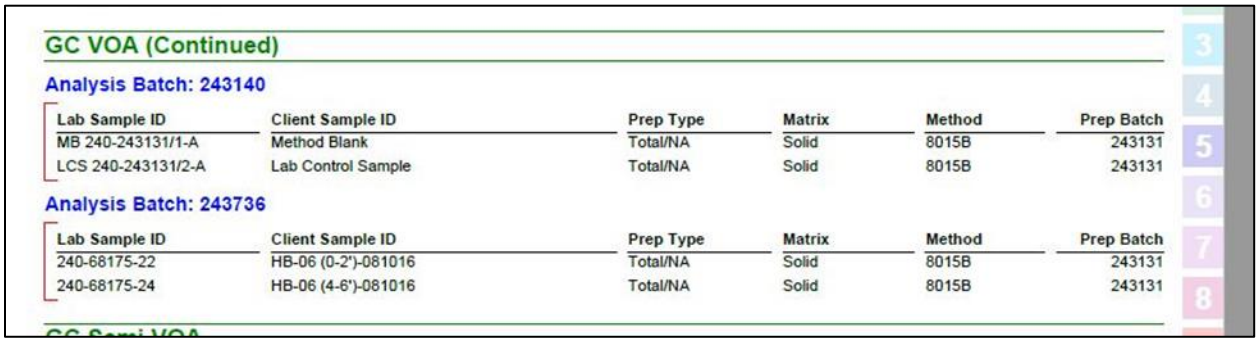
Lab Sample ID: LCS 240-243131/2-A
Matrix: Solid
Analysis Batch: 243140

Client Sample ID: Lab Control Sample
Prep Type: Total/NA
Prep Batch: 243131

Analyte	Spike Added	LCS Result	LCS Qualifier	Unit	D	%Rec	%Rec. Limits
Gasoline Range Organics [C6 - C10]	40000	40100		ug/Kg		100	65 - 120

Surrogate	*LCS %Recovery*	*LCS Qualifier*	*Limits*
Trifluorotoluene (Surr)	*101*		*36 - 157*

Exhibit 7-7: Batch numbers for Method Blank, LCS, and the Sample HB-06 4' to 6'



GC VOA (Continued)

Analysis Batch: 243140

Lab Sample ID	Client Sample ID	Prep Type	Matrix	Method	Prep Batch
MB 240-243131/1-A	Method Blank	Total/NA	Solid	8015B	243131
LCS 240-243131/2-A	Lab Control Sample	Total/NA	Solid	8015B	243131

Analysis Batch: 243736

Lab Sample ID	Client Sample ID	Prep Type	Matrix	Method	Prep Batch
240-68175-22	HB-06 (0-2')-081016	Total/NA	Solid	8015B	243131
240-68175-24	HB-06 (4-6')-081016	Total/NA	Solid	8015B	243131

Exhibit 7-8: Lab Chronicle

Client Sample ID: HB-06 (4-6')-081016 — **Lab Sample ID: 240-68175-24**
Date Collected: 08/10/16 14:30 — **Matrix: Solid**
Date Received: 08/11/16 08:35 — **Percent Solids: 85.7**

Prep Type	Batch Type	Batch Method	Run	Dilution Factor	Batch Number	Prepared or Analyzed	Analyst	Lab
Total/NA	Prep	3540C			242267	08/12/16 08:08	SDE	TAL CAN
Total/NA	Analysis	8270C		1	242912	08/17/16 13:55	TMH	TAL CAN
Total/NA	Prep	5030B			243131	08/18/16 05:46	RTR	TAL CAN
Total/NA	Analysis	8015B		5	243736	08/23/16 07:51	RTR	TAL CAN
Total/NA	Prep	3540C			242285	08/12/16 08:56	SDE	TAL CAN
Total/NA	Analysis	8015B		10	243277	08/18/16 19:38	DEB	TAL CAN
Total/NA	Prep	3540C			242718	08/16/16 08:07	SDE	TAL CAN
Total/NA	Analysis	8082		1	243221	08/18/16 19:51	LSH	TAL CAN

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Exhibit 7-9: Duplicate Sample Results

Lab Sample ID: 240-68269-22 MS — **Client Sample ID: HB-DUP-B (0-2')-081116**
Matrix: Solid — **Prep Type: Total/NA**
Analysis Batch: 243736 — **Prep Batch: 243777**

Analyte	Sample Result	Sample Qualifier	Spike Added	MS Result	MS Qualifier	Unit	D	%Rec	%Rec. Limits
Gasoline Range Organics [C6 - C10]	46	J B	892	792		ug/Kg	☼	84	25 - 120

Surrogate	*MS %Recovery*	*MS Qualifier*	*Limits*
Trifluorotoluene (Surr)	*68*		*43 - 120*

Method: 8015B - Gasoline Range Organics - (GC) (Continued)

Lab Sample ID: 240-68269-22 MSD — **Client Sample ID: HB-DUP-B (0-2')-081116**
Matrix: Solid — **Prep Type: Total/NA**
Analysis Batch: 243736 — **Prep Batch: 243777**

Analyte	Sample Result	Sample Qualifier	Spike Added	MSD Result	MSD Qualifier	Unit	D	%Rec	%Rec. Limits	RPD	RPD Limit
Gasoline Range Organics [C6 - C10]	46	J B	882	789		ug/Kg	☼	84	25 - 120	0	40

Surrogate	*MSD %Recovery*	*MSD Qualifier*	*Limits*
Trifluorotoluene (Surr)	*67*		*43 - 120*

Lab Sample ID: MB 240-243779/1-A — **Client Sample ID: Method Blank**

7.2 Example of Completed TPH Checklist

Example 7-1 consists of Checklist #5, which was completed using the analytical and QC data from Exhibits 7-1 through 7-9.

Example 7-1: Competed TPH Checklist

5.0 Total Petroleum Hydrocarbon Data Validation	
5.1 GRO Data Review – Blank Data	
Laboratory blanks are used to assess whether contamination from the laboratory, reagents, or other samples exists and whether this contamination can bias sample results. The qualification of sample results will depend upon the magnitude of blank contamination.	
5.1.1 Is the method blank data present for each batch? *Action: If not present, request information from the applicable party or laboratory. The data can also be rejected based upon the Data Validator's judgment. If the required method blank(s) was not analyzed, sample results may be qualified as estimated ("J" for positive results and "UJ" for non-detected compounds.*	**Indicate yes or no:** Yes
5.1.2 Is there an indication that the samples associated with the method blank were diluted? **Note: The dilution factor can be found in the data report (a dilution factor of 1 indicates no dilution).**	**Indicate yes or no:** Yes **If yes, list the sample ID(s) and dilution factor(s):** 240-68175-24 Diluted 5 times
5.1.3 Do any method blanks have any detected results? Are blank results below the QL? **Note: A list of samples associated with each of the contaminated blanks should be prepared. The method blanks (MB) must be below the target analyte QL unless all the samples in the batch are below the QL.** *Action: If the MB is not below the QLs then the method blank and all associated samples should be re-extracted and reanalyzed after instrument maintenance and recalibration. Following re-extraction and reanalysis, MB results may be reported and qualified with a "B" for contaminants detected in the method blank with an explanation in the narrative section under the following circumstances:* *1. Insufficient sample volume for re-extraction 2. Expired hold times, 14 days from collection to analysis.* *3. The blank values are below the reporting limit in all the samples.*	**Indicate yes or no:** Yes **If yes, list those analytes and results found in both the blanks and samples:** Method Blank ID MB240-243131/1-A C6-C10 4170 ug/Kg J The method blank is above the quantitation limit. This was mentioned in the laboratory narrative. Sample ID HB-06 (4-6') – 081016 Lab Sample ID: 240-68175-24 420000 ug/Kg B

5.2 GRO Data Review – Laboratory Control Sample (LCS)	
An LCS should be included with each batch of samples (no more than 20). The LCS consists of an aliquot of a clean (control) matrix like the matrix type of the sample and at the same weight or volume. The LCS is spiked with the same analytes at the same concentration as the matrix spike/ matrix spike duplicate and all surrogates. When the results of the matrix spike indicate a potential problem due to the sample matrix itself, the LCS verifies that the laboratory can perform analyses in a clean matrix. The LCS measures the bias in the entire system including the extraction. The LCS is extracted using the identical extraction method. The LCS must be within the laboratory information system statistically derived control limits for each laboratory or piece of equipment.	
5.2.1 Was an LCS prepared, extracted, analyzed, and reported once per batch of 20 samples? **Note: This information should be included in the QA package provided by the lab. If not, contact the laboratory and request that the information be submitted. This information should be found in the injection log.** *Action: If LCS information cannot be found, contact the applicable party or laboratory for re-submittal of the data package. If LCS information is not present, qualify all detected results as "J" and all non-detect results as "UJ" or reject all results based on best professional judgment.*	**Indicate yes or no:** Yes **Summarize any actions taken:** N/A
5.2.2 Does the LCS contain the GRO analytes of interest at the same concentration as the matrix spike/matrix spike duplicate? **Note: When the results of the matrix spike indicate a problem due to sample matrix, the LCS should be checked to determine whether the laboratory can perform the analysis on a clean matrix.**	**Indicate yes or no:** Yes
5.2.3 Do the percent recoveries (%R) meet the statistically derived laboratory control limits? **Note: The laboratory should use statistically derived laboratory control limits calculated by the Laboratory Information Management System (LIMs) to compare recoveries. If any of the target analytes or surrogates in the LCS are outside of the statistically derived control limits, an aliquot of the LCS must be reanalyzed to verify which target analytes or surrogates are out of control. If the exceedance is confirmed**	**Indicate yes or no:** Yes **If no, list compounds and sample IDs that do not meet QC limits and summarize actions taken:** The LCS recovery is 101% well with in the 36% to 157 % control limits.

5.2 GRO Data Review – Laboratory Control Sample (LCS)	
An LCS should be included with each batch of samples (no more than 20). The LCS consists of an aliquot of a clean (control) matrix like the matrix type of the sample and at the same weight or volume. The LCS is spiked with the same analytes at the same concentration as the matrix spike/ matrix spike duplicate and all surrogates. When the results of the matrix spike indicate a potential problem due to the sample matrix itself, the LCS verifies that the laboratory can perform analyses in a clean matrix. The LCS measures the bias in the entire system including the extraction. The LCS is extracted using the identical extraction method. The LCS must be within the laboratory information system statistically derived control limits for each laboratory or piece of equipment.	
upon reanalysis, the batch in question must be re-extracted and reanalyzed along with the LCS. *Action: The data can be qualified using Table 5-1 below under the following limited circumstances:* *1. Insufficient sample volume for re-extraction.* *2. Expired hold times, 14-day hold time from collection to analysis.* *3. The LCS is biased high, and the samples are below the PQL for those target analytes.* *All of these circumstances must be documented in the laboratory report narrative.*	
5.2.4 Verify the calculations for at least one %R. $\%R = \left(\frac{Measured\ Concentration}{Spiked\ Amount}\right) \times 100$ *Action: If the %R is not calculated correctly, verify the other %R calculations and/or contact the lab for re-submittal. If the re-calculated %R values fall within the QC limits, the validator should use professional judgment to determine if the lab should be contacted for re-submittal or if the data should be flagged.*	**Show results of verified %R calculation:** 40100 ug/Kg/40000 ug/Kg x 100 = 101 % well with in the 36% to 157 % control limits.

Table 5-1 LCS Actions for GRO Analyses		
Qualification		
LCS Result	Sample Result	Action
> Upper acceptance limit	Non-detect	J+
< Lower acceptance limit	Detection	R
< Lower acceptance limit	Non-detect	R

5.3 GRO Data Review - Matrix Spike/Matrix Spike Duplicates
Matrix spike and matrix spike duplicates are performed to assess method precision for GRO analyses. One matrix spike and one matrix spike duplicate pair or one matrix spike and one

5.3 GRO Data Review - Matrix Spike/Matrix Spike Duplicates	
duplicate un-spiked sample (if the samples are expected to contain target analytes) is required for every batch of samples (every 20 samples). The Data Validator should be aware that the MS/MSD are batch specific, not sample specific. For example, the MS/MSD information may be analyzed with any sample in the batch, but not necessarily a sample being validated. Because of this, matrix spike and matrix spike duplicate data alone usually are not used to qualify results, but the information is used with the other QA/QC data to qualify data.	
5.3.1 Is matrix spike/matrix spike duplicate recovery data present? *Action: If any matrix spike/spike duplicate data are missing, the laboratory should be contacted for a re-submittal.*	**Indicate yes or no:** Yes
5.3.2 How many VOC spike recoveries are outside the QC limits?	**Record the compound(s) out of compliance, their spike recovery, and the control limits:** None
5.3.3 Verify the calculations for at least one %R. Matrix Spike Recovery $\%R=\frac{SSR-SR}{SA} \times 100$ Where: SSR= spiking analyte result in the spiked sample SR= Result of the same analyte in the original sample SA= spike added in the spiked sample	**Show results of verified %R calculation:** 792 ug/Kg-46ug/Kg / 892 ug/Kg x 100 = 84% well within the control limits of 25% to 120 % 84% recovery is what is reported.
5.3.4 How many relative percent differences (RPDs) for matrix spike and matrix spike duplicate recoveries are outside the QC limits? **Note: The MS/MSD results may be used in conjunction with other QC criteria to determine the need for data qualification. Results outside QC limits should be identified. The laboratory should use statistically derived laboratory control limits calculated by the Laboratory Information System (LIM) to compare relative percent difference.** *Action: Identify RPDs that are outside QC limits.*	**Quantity of RPDs that are outside QC Limits:** N/A **Record the compound(s) that have recovery data out of criteria and control limits. Review surrogate and LCS data to determine if qualification is necessary:** None

5.3 GRO Data Review - Matrix Spike/Matrix Spike Duplicates	
Note: The MS/MSD results may be used in conjunction with other QC criteria to determine the need for data qualification.	
5.3.5 Verify the calculations for at least one RPD. RPD RPD=$\frac{\lvert MSR-MSDR\rvert}{\left(\frac{MSR+MSDR}{2}\right)}$ x 100 Where: MSR= Matrix spike result for the spiking analyte in the MS sample MSDR= Matrix spike result for the spiking analyte in the MSD sample	**Show results of verified RPD calculation:** 84 % - 84 % / (84% + 84 % / 2) = 0 relative percent difference which is well within the control limit of 40% Relative Percent Difference (RPD).

5.4 GRO Data Review - Surrogate Recovery	
Surrogate compounds are spiked compounds of known composition and concentration that are added to samples, blanks, and other QA/QC data. Surrogates are compounds that mimic target analytes but are compounds that are not commonly found in the environment so that they can be identified as QA analytes. The recovery of surrogate compounds allows an assessment of matrix interference. GRO surrogate recoveries are also used with other QA/QC data to qualify sample results and to justify laboratory re-analysis. **<u>Recommended Surrogate Compound</u>** **Trifluorotoluene (TFT)**	
5.4.1 Are the surrogate recovery data present for each sample in each batch? **Note: Samples may be included in different batches. When this is the case, separate surrogate recoveries should be provided.** *Action: If no, then contact the laboratory for an explanation and report re-submittal.*	**Indicate yes or no:** Yes
5.4.2 Are any surrogate recoveries outside the QC limits? **Note: Surrogate recovery limits are statistically derived quality control limits calculated by the laboratory information management system and depend on the surrogates chosen, levels used, and instrument conditions.** *Action: Identify samples with recoveries outside QC limits.*	**Indicate yes or no:** No. **If yes, list the sample ID(s), matrix(-ces) and parameter(s):** Recovery is 91% for Trifluorotoluene in HB-06 4to 6 feet GRO sample. 36% to 157% was the acceptable range.
5.4.3 Verify the calculations for at least one %R.	**Show results of verified %R calculation:**

$Recovery\ \% = \frac{(Concentration\ found)}{(Concentration\ \ added)} \times 100$	91/100 x 100 = 91%
5.4.5 When surrogate recoveries were out of the control limits what corrective action was taken? **Note: When surrogate recoveries are out of limit in samples or quality control samples, reanalyzed, re-extract and reanalyzed, or diluted. Samples do not need to be re-extracted or reanalyzed if there is insufficient sample or surrogates are biased high and the samples are non-detect where no qualification is needed. When there is a cause for the interference and a correction is not possible by the laboratory, the data should be identified by appropriate flagging.** *Action: If surrogate recoveries were out of control, use Table 5-2 below to qualify sample results.*	**Indicate corrective actions taken:** N/A **List sample ID(s) for surrogate compounds out of compliance and criteria:** The sample and blank are within the 36% to 157% control limits.

Table 5-2 Surrogate Actions for GRO Analyses

	Action	
	Detect	**Non-detect**
Surrogate not present or not at specified concentration	J or R	UJ or R
%R within specified Acceptance Limits	No qualification	No qualification
%R > specified Upper Acceptance Limit	J+	No qualification

8.0 Hexavalent Chromium and Cyanide Worked Examples

This section provides sample analytical data, QC data, and completed checklists for hexavalent chromium (Checklist #9) and cyanide (Checklist #10).

8.1 Hexavalent Chromium Worked Example

This section provides sample analytical data and QC data used to fill out Checklist #9. Laboratory reports may provide more or less information than what is shown in this example.

Analytical and QC Results for hexavalent chromium are shown in Exhibits 8-1 through 8-5, below. These exhibits were used to complete Example 8-1.

Exhibit 8-1: Example Analytical Data for Hexavalent Chromium

Sample ID: 1504-02		**Collected**: 07/06/21		**Received**: 07/06/21		**Matrix**: soil		**Digestion Method:** 3060A	
Parameter	Result	Qualifier	Reporting Limit	MDL	Dilution Factor	Units	Prep date	Analysis date	Analytical Method
Chromium, Hexavalent	0.061	J	0.89	0.178	1	Mg/kg	7/8/21	7/9/21	7196A
Sample ID: 1504-03		**Collected**: 07/06/21		**Received**: 07/06/21		**Matrix**: soil		**Digestion Method:** 3060A	
Parameter	Result	Qualifier	Reporting Limit	MDL	Dilution Factor	Units	Prep date	Analysis date	Analytical Method
Chromium, Hexavalent	ND		0.924	0.185	1	Mg/kg	7/8/21	7/9/21	7196A
Sample ID: 1504-04		**Collected**: 07/06/21		**Received**: 07/06/21		**Matrix**: soil		**Digestion Method:** 3060A	
Parameter	Result	Qualifier	Reporting Limit	MDL	Dilution Factor	Units	Prep date	Analysis date	Analytical Method
Chromium, Hexavalent	ND		0.927	0.185	1	Mg/kg	7/8/21	7/9/21	7196A
Sample ID: 1504-05		**Collected**: 07/06/21		**Received**: 07/06/21		**Matrix**: soil		**Digestion Method:** 3060A	
Parameter	Result	Qualifier	Reporting Limit	MDL	Dilution Factor	Units	Prep date	Analysis date	Analytical Method
Chromium, Hexavalent	0.799	J	0.890	0.178	1	Mg/kg	7/8/21	7/9/21	7196A

Exhibit 8-2: Example Preparation Blank Results

Method Blank Analysis for Samples 1504-02 to 1504-05

Parameter	Result	Qualifier	RL	MDL	Units	Prep date	Analysis date	Analytical Method
Chromium, Hexavalent	0.160	J	0.800	0.160	Mg/L	07/08/21	07/08/21	7196A

Exhibit 8-3: Example LCS Results

LCS Analysis for Samples 1504-02 to 1504-05							
Parameter	%LCS Recovery	Qualifier	%LCSD Recovery	%Recovery Limits	Analysis date	RPD	RPD Limits
Chromium, Hexavalent	88			80-120	07/08/21		20

Exhibit 8-4: Example Matrix Spike Results

Matrix Spike Analysis for Samples 1504-02 to 1504-05								
Parameter	Native Sample	MS Added	MS Found	MS %Recovery	MSD found	MSD %Recovery	RPD	Recovery Limits
Chromium, Hexavalent	0.601J	907	850	94				75-125

Exhibit 8-5: Example Duplicate Results

Duplicate Analysis for Samples 1504-02 to 1504-05						
Parameter	Native Sample	Duplicate Sample	MDL	Units	RPD	RPD Limits
Chromium, Hexavalent	0.799J	0.789 J	0.178	Mg/kg	0.1	20

Example 8-1: Completed Hexavalent Chromium Checklist

9.0 Hexavalent Chromium Data Validation

9.1 Hexavalent Chromium Data Review – Blank Data

Chromium in the hexavalent valence state is a concern because of its toxicity and mobility in the environment. SW-846 Method 7196A is used to analyze hexavalent chromium in aqueous matrices and solid extracts. The data validation of hexavalent chromium is dependent on the matrix. Aqueous samples have extraction procedures that differ from solid samples and these differences must be accounted for. Solid samples require ancillary information on the REDOX state of the waste and must use preparation Method 3060A. This preparation method produces an alkaline extract and must be used for valid results. The preparation method requires some review of the waste or the environment from which the samples are derived. Therefore, sampling events for soil and solid wastes must plan for taking measurements of pH, REDOX, ferric-ferrous iron, etc.

9.1.1 For soil and solid waste matrices, was SW-846 preparation method 3060A used to extract the samples? *Action: If not, take no further action and contact the facility. If 3060A was not used to extract solid matrices, then qualify results as rejected, or for TCLP extracted chromium above the regulatory standard, qualify results using professional judgement. The laboratory should be contacted for an explanation and potential re-analysis.*	**Indicate yes or no:** Yes **Summarize any actions taken:**
9.1.2 Was a preparation blank prepared and analyzed for each digestion batch?	**Indicate yes or no:** Yes
9.1.3 Was Cr^{6+} detected in the preparation blank? *Action: Use Table 9-1 below to qualify the data based on the preparation blank results. If the Cr^{6+} results do not meet the criteria, then all samples in the batch should have been re-digested and reanalyzed. Reject all reported results from a batch where the preparation blank does not meet the criteria.*	**Indicate yes or no:** Yes **Summarize any actions taken:** Cr6+ was detected in the blank above the MDL. Therefore, non-detected results do not need to be qualified, but detects greater than the MDL should be qualified. Analytical samples 1504-02 and 1504-05 were had detected Cr6+, with concentrations of 0.061 and 0.799, respectively. These concentrations are greater than the QL, but less than 10x the concentration of the blank result. Therefore, they should have been reported at the blank result and qualified as J+ or R.

Table 9-1: Preparation Blank Actions for Cr^{6+}		
Blank Result	**Sample Result**	**Action**
Not analyzed at specified frequency	Non-detect	UJ
	Detect	J
Detect < QL	Non-detect	No qualification
	Detect < QL	Report at QL and qualify U
	Detect > QL	J+ or no qualification
≥ QL	Non-detect	No qualification
	Detect < QL	Report at QL and qualify U
	≥ QL but < 10x the Preparation Blank Result	Report at Preparation Blank Result and qualify J+ or R
	≥ 10x the Preparation Blank Result	No qualification

9.2 Hexavalent Chromium Data Review – Duplicates	
Duplicate samples, including field duplicates, are used to document precision of the sampling process. Field duplicates are used to assess improper homogenization of the samples in the field, reproducibility of sample preparation and analysis, and heterogeneity of the matrix.	
9.2.1 Was at least one separately prepared duplicate soil sample analyzed at a frequency of one per batch of 20 samples? **Note: At least one duplicate sample should be prepared and analyzed for each data package. Duplicates cannot be averaged for reporting on the Laboratory Report. Additional duplicate sample analyses may be required based on the project's DQOs. A specific sample may be required to be used for the duplicate sample analysis.** *Action: If duplicate analysis is required by the project's DQOs, but not included within the report,*	**Indicate yes or no:** Yes **Summarize any actions taken:** N/A

contact the laboratory or applicable party to determine whether a duplicate sample was prepared and analyzed.	
9.2.2 Are all relative percent difference (RPD) values within control limits? **Note: Acceptance criteria for RPD should have a set of laboratory-derived limits; however, acceptance limits must not exceed 20% for the original sample and its duplicate if both the original and the duplicate are ≥ 5x the QL. A control limit of the QL is used when either the original or the duplicate sample is < 5x the QL.** *Action: Determine whether RPD values exceed control limits by using Table 9-2 below. If duplicate sample results are outside of the criteria, samples with detected results should be qualified as estimated and flagged with "J". Non-detected results should be qualified as estimated and flagged with "UJ".* *For high RPDs (i.e., > 100%), use professional judgment to qualify the data, as this may be indicative of a sampling problem.*	**Indicate yes or no:** Yes. RPD of the duplicates = 1. **Summarize any actions taken:** RPD is within limits, but sample results are less than 5x the QL. The absolute difference between the sample result and duplicate is less than the QL. Therefore, no qualification is needed.
9.2.3 RPD is calculated to evaluate the original and duplicate samples for precision using the following equation: $$RPD = \frac{|S - D|}{(\frac{S - D}{2})} \times 100$$ Where:	**Show the results of one verified RPD calculation:** |0.799-0.789|/ ((0.799+0.789)/2) x100 = 1.26 **Summarize any actions taken:**

S = Original Sample Result D = Duplicate Result *Action: Verify one RPD calculation for one set of original and duplicate samples. Contact the applicable party or laboratory for an explanation if RPD was not calculated. If a satisfactory explanation is not available, use professional judgment to qualify sample results.* **Note: when the Sample or Duplicate Result is reported as a non-detect, use a value of zero (0) only for calculating the RPD. This will always yield an RPD of 200%.**	N/A

Table 9-2: Duplicate Analysis Actions for Cr^{6+}

Criteria	Action	
	Detect	**Non-Detect**
Duplicate analysis is required by the QAPP, but not performed at the specified frequency	J, or use professional judgement	UJ, or use professional judgement
Both original sample and duplicate sample results are ≥ 5x QL and RPD > 20%	J	UJ
Both original sample and duplicate sample results are ≥ 5x QL and RPD ≤ 20%	No qualification	No qualification
RPD > 100%	Use professional judgement	Use professional judgement
Original sample or duplicate sample results < 5x QL (including non-detects) and absolute difference between sample and duplicate > QL	J	UJ
Original sample or duplicate sample result < 5x QL (including non-detects) and absolute difference between sample and duplicate ≤ QL	No qualification	No qualification

9.3 Hexavalent Chromium Data Review – Laboratory Control Sample	
Laboratory Control Samples (LCSs) are analyte-free water or solid, clean control matrixes, similar to the sample matrix, spiked with target analytes at known concentrations. LCSs are analyzed using the same sample preparation, reagents, and analytical methods employed for the samples received. Aqueous/water LCSs should be analyzed for hexavalent chromium utilizing the same sample preparations, analytical methods, and Quality Assurance/Quality Control (QA/QC) procedures as employed for the samples. LCS criteria listed in this section are determined from U.S. EPA's National Functional Guidelines for Inorganic Data Review.	
9.3.1 Was an LCS analyzed per batch of aqueous samples? **Note: The LCS should be spiked such that it contains Cr^{6+} at the levels specified in the Quality Assurance Project Plan (QAPP) or at 2x the QL.** *Action: If LCS information cannot be found, contact the applicable party or laboratory for re-submittal of the data package. If LCS information is not present, qualify all detected results as estimated "J" and all non-detect results as estimated undetected "U" or reject all results based on best professional judgment.*	**Indicate yes or no:** Yes. **Summarize any actions taken:** N/A
9.3.2 Was an LCS analyzed per batch and within suggested QC limits (80% - 120%) or limits provided by the lab? *Action: Use Table 9-3 below to qualify data based on LCS results. If the LCS result is outside the criteria, the batch should have been re-digested and reanalyzed.*	**Indicate yes or no:** Yes. **Summarize any actions taken:** N/A – LCS within QC limits.
9.3.3 Verify the calculations for at least one %R. $$\%R = \left(\frac{Measured\ Concentration}{Spiked\ Amount}\right) \times 100$$	**Show results of verified %R calculation:** The spiked amount and measured concentration of the LCS were not provided alongside the %R.

Action: If the %R is not calculated correctly, verify the other %R calculations and/or contact the lab for re-submittal. If the re-calculated %R values fall within the QC limits, the validator should use professional judgment to determine if the lab should be contacted for re-submittal or if the data should be flagged.	The laboratory should be contacted to retrieve this information.

Table 9-3: LCS Actions for Cr^{6+}

Criteria	Action	
	Detect	**Non-detect**
LCS not prepared with samples	J	UJ
LCS not prepared at specified concentration	J	UJ
Aqueous/water %R < 40%	J-	R
Aqueous/water %R 40-79%	J-	UJ
Aqueous/water %R 80-120%	No qualification	No qualification
Aqueous/water %R 121-150%	J+	No qualification
Aqueous/water %R > 150%	R	No qualification

9.4 Hexavalent Chromium Data Review – Matrix Spike	
Spikes are elements of known composition that are added to blanks and samples to evaluate the effect of each sample matrix on the sample preparation procedures and the measurement methodology. At least one matrix spike (MS) and one matrix spike duplicate (MSD) should be included for each batch of 20 samples or less. Field samples should be used as source samples for matrix spike analyses. Spike recovery criteria listed in this section are determined from U.S. EPA's National Functional Guidelines for Inorganic Data Review. The criteria applied by an individual laboratory may vary. The laboratory should be consulted and have its QA/QC criteria supplied to the validator.	
9.4.1 For aqueous and solid samples, was a matrix spike or a sample replicate (matrix spike duplicate) analyzed at a frequency of once per 20 samples?	**Indicate yes or no:** Yes. **Summarize any actions taken:**

Action: If not present, flag detections "J", non-detections "UJ", and contact the applicable party for re-submittal.	N/A
9.4.2 Are all spike recoveries for aqueous and solid samples within control limits (*e.g.*, 75% to 125%)? **Note: No action is taken on matrix spike data alone. If the LCS is within criteria, the results and potential bias should be noted in the data report's narrative. If other quality control data is outside of criteria, then the matrix spike data may be used to qualify or reject sample results.** *Action: Determine whether spike recoveries are within control limits by using Table 9-4 below. If the matrix spike recoveries are not within these recovery limits, the entire batch should have been rehomogenized, redigested, and reanalyzed.* *If upon reanalysis, the matrix spike is not within the recovery limits, but the LCS is within criteria specified in Section 9.3 of this checklist, ancillary information such pH, Eh, and other REDOX couples must be evaluated (see Figures 1 and 2 and Section 3.1 of Method 3060A). The Cr^{6+} data may be valid for use despite the perceived "QC failure."*	**Indicate yes or no:** Yes. Recovery is 94%. **If no, describe any actions taken:** N/A
9.4.3 Verify the calculations for at least one %R. $$\%Recovery = \frac{SSR - SR}{SA} x100$$ Where:	**Show results of one %R calculation:** %R = (850 – 0.601) / 907 x100 = 94%

SSR = Spike sample Result SR = Sample Result SA = Spike Added **Note: When the sample result is reported as non-detect, the sample result should be set at 0 for calculating the %R.**	
9.4.4 Are all RPD values for the MS/MSD pair within control limits? **Note: Acceptance criteria for RPD should have a set of laboratory-derived limits; however, acceptance limits must not exceed 20% for original and duplicate values ≥ 5x the QL. A control limit of the QL is used when either the original or the duplicate sample is < 5x the QL.** *Action: Determine whether RPD values exceed laboratory-derived control limits. If control limits have not been developed, use ≤20% as the acceptance criteria. For high RPDs (i.e., > 100%), use professional judgment to qualify the data, as this may be indicative of a sampling problem.*	**Indicate yes or no:** N/A – No MSD was analyzed for this batch. **Summarize any actions taken:** Qualification depends on whether the projects DQOs specified an MSD to be performed. If an MSD should have been analyzed, detects should be flagged with "J" and non-detects flagged with "UJ".
9.4.5 RPD is calculated to evaluate the original and duplicate samples for precision using the following equation: $$RPD = \frac{|S - D|}{(\frac{S - D}{2})} \times 100$$ Where: S = Original Sample Result (matrix spike)	**Show the results of one verified RPD calculation:** N/A – No MSD was analyzed. **Summarize any actions taken:**

D = Duplicate Result (matrix spike duplicate) **Note: when the Sample or Duplicate Result is reported as a non-detect, use a value of zero (0) only for calculating the RPD. This will always yield an RPD of 200%.** *Action: Verify one RPD calculation for one MS/MSD pair. Contact the applicable party or laboratory for an explanation if RPD was not calculated. If a satisfactory explanation is not available, use professional judgment to qualify sample results.*	N/A

Table 9-4: Matrix Spike Actions for Cr^{6+}		
Criteria	**Action**	
	Detect	**Non-Detect**
Matrix Spike analysis not performed at the specified frequency	J	UJ
Matrix Spike not prepared from field sample	J	UJ
Matrix Spike %R < 30%	J-	R
Matrix Spike %R 30-74%	J-	UJ
Matrix Spike %R 75-125%	No qualification	No qualification
Matrix Spike %R > 125%	J+	No qualification

9.5 Hexavalent Chromium Data Review – Method of Standard Additions (MSA)	
The standard addition technique involves adding known amounts of standard to one or more aliquots of the processed sample solution. This technique compensates for a sample constituent that enhances or depresses the analyte signal, thus producing a different slope from that of the calibration standards. The MSA will not correct for additive interferences which cause a baseline shift. The MSA is used for the analysis of all extracts, on all analyses submitted as part of a delisting petition, and whenever a new sample matrix is being analyzed.	

9.5.1 Optional: Was the Method of Standard Additions (MSA) used for waste(s) whose generator is seeking a chromium exclusion under OAC 3745-51-04? *Action: If the analysis is performed for a chromium exclusion and the MSA was not performed, the analysis does not have sufficient quality to provide evidence for the exclusion. Additional testing must be performed.*	**Indicate yes or no:** No. **Summarize any actions taken:** N/A
9.5.2 Optional: If the MSA was used, was a spike observed? *Action: If no spike was observed for the MSA results indicate that the matrix is incompatible with Cr^{6+}.*	**Indicate yes or no:** N/A **Summarize any actions taken:** N/A

8.2 Cyanide Worked Example

This section provides sample analytical data and QC data used to fill out Checklist #10, which is shown in Section 8.1.3. Laboratory reports may provide more or less information than what is shown in this example.

Analytical and QC Results for Cyanide are shown in Exhibits 8-6 through 8-10, below. These exhibits were used to complete Example 8-2.

Exhibit 8-6: Example Cyanide Analytical Data

Sample ID: 1504-1		**Date/Time Sampled**: 04/02/2015 / 1150			**Received**: 04/02/2015	
Parameter	CAS #	Result	Reporting Limit	Units	Prep date	Analysis date
Cyanide, Total: SW846-9014	57-12-5	0.071	0.0050	Mg/L	4/13/15	4/13/15
Sample ID: 1504-2		**Date/Time Sampled**: 04/02/2015 / 1152			**Received**: 04/02/2015	
Parameter	CAS #	Result	Reporting Limit	Units	Prep date	Analysis date
Cyanide, Total: SW846-9014	57-12-5	<0.0050	0.0050	Mg/L	4/13/15	4/13/15
Sample ID: 1504-3		**Date/Time Sampled**: 04/02/2015 / 1050			**Received**: 04/02/2015	

Parameter	CAS #	Result	Reporting Limit	Units	Prep date	Analysis date
Cyanide, Total: SW846-9014	57-12-5	1.03	0.0050	Mg/L	4/13/15	4/13/15

Exhibit 8-7: Example Preparation Blank Results

QC Type: Method Blank		**Matrix**: liquid		**Project ID**: 1504
Parameter	Result	Reporting Limit	Units	Analysis date
Cyanide, Total: SW846-9014	<0.0050	0.0050	Mg/L	4/13/2015

Exhibit 8-8: Example LCS Results

QC Type: LCS/LCSD		**Matrix**: liquid			**Project ID**: 1504	
Parameter	Spike Percent Recovery	Spike Percent Recovery	Control Limits	RPD	RPD Control Limits	Analysis date
Cyanide, Total: SW846-9014	95.5	108.0	(74-120)	12.3	(0-20)	4/13/2015

Exhibit 8-9: Example MS/MSD Results

QC Type: MS/MSD		**Matrix**: liquid		**Project ID**: 1504		
Parameter	MS Recovery	MSD Recovery	Control Limits	RPD	RPD Control Limits	Analysis date
Cyanide, Total: SW846-9014	105.9%	161.9%	(74-120)	41.8	(0-18)	4/13/2015

Exhibit 8-10: Example Duplicate Results

QC Type: Sample Duplicate		**Matrix**: liquid		**Project ID**: 1504	
Parameter	Sample Result	Sample Duplicate Result	RPD	RPD Control Limits	Analysis date
Cyanide, Total: SW846-9014	0.071	0.060	16.8	(0-20)	4/13/2015

Example 8-2: Completed Cyanide Checklist

10.0 Cyanide Data Validation
10.1 Cyanide Data Review – Blank Data Analysis
A method blank is used to assess contamination from the laboratory environment, equipment, and/or reagents, so a method blank must be carried throughout the entire sample preparation and analytical process for each batch of samples analyzed. This includes exposure to all glassware, equipment, solvents, filtration, and reagents that are used with field samples. Any free cyanide measured in the method blank that exceeds the quantitation limit indicates that contamination is

present. The source of the contamination should be determined and corrected before performing any sample analysis. Any sample included in an analysis batch that has an unacceptable method blank concentration should be reanalyzed in a subsequent batch after the contamination problem is resolved. One reagent blank per analytical batch or one in every 20 samples should be used to determine if contamination or any memory effects are occurring.	
10.1.1 Is the method blank data present for each batch of approximately 20 samples (matrix and sample number dependent), including TCLP? *Action: If not present, request information from the applicable party or laboratory. If the required method blank(s) was not analyzed, sample results may be qualified as estimated ("J" for positive results and "UJ" for non-detected compounds) based upon the Data Validator's judgment.*	**Indicate yes or no:** Yes. A method blank is present for a batch of 3 analytical samples (1504-1, 1504-2, and 1504-3).
10.1.2 Is there an indication that samples in the batch associated with the blank were diluted? **Note: The dilution factor can be found in the data report (a dilution factor of 1 indicates no dilution).** *Acton: If yes, identify dilution factors.*	**Indicate yes or no:** No. No dilution factor is listed. **If yes, list the dilution factor(s):**
10.1.3 Was cyanide detected in any blanks? Was cyanide found in the samples associated with the blank? **Note: A list of samples associated with each of the contaminated blanks should be prepared.** *Action: If blank contamination is identified, follow the directions in the Table 10-1 below for qualifying data based on blank results.*	**Indicate yes or no:** No. Cyanide was not detected in the blank. **If yes, the sample IDs associated with the blank and summarize any actions taken:** N/A - Cyanide was not detected in the blank, so no qualification is needed.

Table 10-1: Blank Actions for Cyanide

Blank Result	Sample Result	Action
Detect ≤ QL	Non-detect	No qualification
	Detect ≤ QL	Report at QL and qualify as U
	> QL	J+ or no qualification
> QL	Non-detect	No qualification
	Detect ≤ QL	Report at QL and qualify as U
	> QL but < 10x the Blank Result	Report at Blank Result and use professional judgment to qualify results as J+ or R
	≥ 10x the Blank Result	No qualification

10.2 Cyanide Data Review - Duplicates	
Duplicate samples are used to demonstrate acceptable method precision by the laboratory at the time of analysis. Field samples should be used for duplicate sample analysis. At least one duplicate sample should be prepared and analyzed from each batch of samples of a similar matrix type (*e.g.*, aqueous/water or soil/sediment/waste).	
10.2.1 Did the project SAP, QAPP, or DQOs include collecting collocated or duplicate samples? If so, were an appropriate amount duplicates collected?	**Indicate yes, no, or N/A:** This project required the collection and analysis of a duplicate sample.
10.2.2 Was the duplicate analysis performed on a field sample?	**Indicate yes, no, or N/A:** Yes
10.2.3 Were RPDs within the established control limits? *Action: Determine whether RPD values exceed control limits by using Table 10-2 below. If duplicate sample results are outside of the criteria, samples with detected results should be qualified as estimated and flagged with "J". Non-detected results should be qualified as estimated and flagged with "UJ".*	**Record the recovery data out of criteria and control limits.** Yes. The duplicate RPD was less than 20% and both original and duplicate sample results were greater than 5x the QL (0.0050). No qualification is needed.
10.4.4 Verify the calculations for at least one RPD.	**Show results of verified RPD calculation:**

$$RPD = \frac{\lvert S - D \rvert}{\left(\frac{S + D}{2}\right)} \times 100$$ Where: S = Sample result (original) D = Duplicate result	|0.071-0.060| / ((0.071 + 0.060)/2) x 100 = 16.8

Table 10-2: Duplicate Sample Actions for Cyanide

Criteria	Action	
	Detect	Non-detect
Both original sample and duplicate sample results are ≥ 5x the QL and RPD > 20%*	J	UJ
RPD > 100%	Use professional judgment	Use professional judgment
Both original sample and duplicate sample results are ≥ 5x the QL and RPD ≤ 20%	No qualification	No qualification
Original sample or duplicate sample result < 5x the QL (including non-detects) and absolute difference between sample and duplicate > QL*	J	UJ
Original sample or duplicate sample result < 5x the QL (including non-detects) and absolute difference between sample and duplicate ≤ QL	No qualification	No qualification

* Project DQOs may allow the use of less restrictive criteria (*e.g.*, 35% RPD, 2x the QL) to be assessed against duplicate soil samples due to laboratory variability arising from the sub-sampling of non-homogenous soil samples.

10.3 Cyanide Data Review – Laboratory Control Samples

Laboratory Control Samples (LCSs) are analyte-free water or solid, clean control matrixes similar to the sample matrix, spiked with target analytes at known concentrations. LCSs are analyzed using the same sample preparation, reagents, and analytical methods employed for the samples received. The LCS should be spiked at the same levels and using the same spiking materials as the corresponding MS

and MSD. When the results of the MS analysis indicate a potential problem due to the sample matrix itself, the LCS results are used to verify that the laboratory can acceptably perform unbiased analysis in a clean matrix.	
10.3.1 Was an LCS prepared, extracted, analyzed, and reported once per batch of 20 samples? **Note: This information should be included in the QA package provided by the lab. If not, contact the laboratory and request that the information be submitted. This information should be found in the injection log.** *Action: If LCS information cannot be found, contact the applicable party or laboratory for re-submittal of the data package. If LCS information is not present, qualify all detected results as "J" and all non-detect results as "UJ" or reject all results based on best professional judgment. If matrix spikes were not performed either, reject all results.*	**Indicate yes or no:** Yes. **Summarize any actions taken:** N/A
10.3.2 Were LCS results within suggested QC limits (85% - 115%) or limits provided by the lab? **Note: Use 85% - 115% unless appropriate lab-specific LCS limits have been developed. The results for solid and aqueous LCSs should always be within the control limits. If out of limits, the laboratory should terminate the analysis, correct the problem, and the samples should be re-digested and re-analyzed. If still unacceptable, then all samples after the last acceptable method blank must be re-prepared and reanalyzed, along with all other appropriate analysis batch QC samples.** *Action: Refer to Table 10-3 below to determine whether data needs to be qualified. If >115%, qualify all detect data as "J+". If <85%, qualify detect data as "J-" and non-detect data as "UJ".*	**Indicate yes or no:** Yes, both the LCS and LCS duplicate were within limits. **Summarize any actions taken:** N/A
10.3.3 Verify the calculations for at least one %R. $$\%R = \frac{Measured\ Concentration}{Spiked\ Amount} \times 100$$	**Show results of verified %R calculation:**

Action: If the %R is not calculated correctly, verify the other %R calculations and/or contact the lab for re-submittal. If the re-calculated %R values fall within the QC limits, the validator should use professional judgment to determine if the lab should be contacted for re-submittal or if the data should be flagged.	%R values were provided, but spike concentrations and measured amounts were not shown in the report. Therefore, the lab must be contacted to obtain these concentrations.

Table 10-3: Lab Control Sample Actions

Criteria	Action	
	Detect	**Non-detect**
LCS not prepared with samples	J or R	UJ or R
LCS not prepared at specified concentrations	J	UJ
Aqueous/water and soil/sediment %R < 50%	J-	R
Aqueous/water and soil/sediment %R 50-84%	J-	UJ
Aqueous/water and soil/sediment %R 85-115%	No qualification	No qualification
Aqueous/water and soil/sediment %R 116-140%	J+	No qualification
Aqueous/water and soil/sediment %R > 140%	R	No qualification

10.4 Cyanide Data Review – Matrix Spikes	
Matrix spikes are performed to evaluate the effect of each sample matrix on the sample preparation procedures and the measurement methodology. At least one spiked sample (pre-distillation) should be prepared and analyzed for each batch of samples with a similar matrix type (*e.g.*, aqueous/water or soil/sediment/waste). The data user may also require that a specific sample be used for the matrix spike sample analysis.	
10.4.1 Is matrix spike data present for each batch of approximately 20 samples (matrix and sample number dependent)? *Action: If not present, request information from the applicable party or laboratory. If the required method blank(s) was not analyzed, sample results*	**Indicate yes or no:** Yes.

should be qualified as estimated ("J" for detected results and "UJ" for non-detected results).	
10.4.2 Was each matrix spike prepared from a field sample? *Action: If not, sample results should be qualified as estimated ("J" for detected results and "UJ" for non-detected results).*	**Indicate yes or no:** Yes.
10.4.3 Do all pre-distillation matrix spike sample results fall with the established control limits? *Action: If not, verify a post-distillation spike was prepared and analyzed.*	**Indicate yes or no:** No, the MSD has a %R of 161.9%. There is no indication of a post-distillation spike.
10.4.4 If a post-distillation spike was analyzed, were matrix spike sample results within the established control limits? *Action: Use Table 10-4 below to qualify results that are not within the established control limits.*	**Indicate yes or no:** N/A – no post-distillation spike results provided. Because the MSD %R is 161.9%, analytical results with detected concentrations may need to be flagged "J+"
9.4.5 Verify the calculations for at least one %R. $$\%R = \frac{SSR - SR}{SA} \times 100$$ Where: SSR = Spike sample Result SR = Sample Result SA = Spike Added **Note: When the sample result is reported as non-detect, the sample result should be set at 0 for calculating the %R.**	**Show results of verified %R calculation:** %R values were provided, but MS and MSD concentrations were not shown in the report. Therefore, the lab must be contacted to obtain these concentrations.

10.4.6 If a matrix spike duplicate was performed, were any relative percent differences (RPDs) for matrix spike and matrix spike duplicate recoveries are outside the QC limits (RPD > 20%)? **Note: The MS/MSD results may be used to determine the need for data qualification. Outliers should be identified.**	**Record the recovery data out of criteria and control limits.** Yes, the RPD for the MS/MSD was outside QC limits.
10.4.7 Verify the calculations for at least one RPD. $$RPD = \frac{|MSR - MSDR|}{\left(\frac{MSR + MSDR}{2}\right)} \times 100$$ Where: MSR= Matrix spike result for the spiking analyte in the MS sample MSDR= Matrix spike result for the spiking analyte in the MSD sample	**Show results of verified RPD calculation:** MS and MSD results were not shown in the report, so this section is unable to be completed. Therefore, the lab must be contacted to obtain these resulting concentrations.

Table 10-4: Matrix Spike Actions for Cyanide

Criteria	Action	
	Detect	Non-detect
Matrix Spike not performed at the specified frequency	J	UJ
Matrix Spike not prepared from a field sample	J	UJ
Matrix Spike %R < 30% Post-distillation spike %R < 75%	J-	R
Matrix Spike %R < 30% Post-distillation spike %R ≥ 75%	J	UJ
Matrix Spike %R 30-74%	J-	UJ

Post-distillation spike %R < 75%		
Matrix Spike %R 30-74% Post-distillation spike %R ≥ 75%	J	UJ
Matrix Spike %R > 125% Post-distillation spike %R > 125%	J+	No qualification
Matrix Spike %R > 125% Post-distillation spike %R ≤ 125%	J	No qualification
Matrix Spike %R < 30% No post-distillation spike performed	J-	R
Matrix Spike %R 30-74% No post-distillation spike performed	J-	UJ
Matrix Spike %R 75-125% No post-distillation is required	No qualification	No qualification
Matrix Spike %R > 125% No post-distillation spike performed	J+	No qualification

NOTE: Project DQOs may allow the use of less restrictive criteria (*e.g.*, 10 %R and 150 %R for the lower and upper limits) to be assessed against spike soil samples due to laboratory variability arising from the sub-sampling of non-homogenous soil samples.

9.0 TCLP Worked Example

This section provides sample analytical data, QC data, and completed checklists for TCLP data (Checklist #11). Checklist #11 should be used along with Checklists #1, #2, #4, #6, and #7 based on the data received.

9.1 Example TCLP Data Results

Exhibit 9-1 shows a typical bench sheet that a laboratory will use to record method specific information. The Tier I Data Validator will obtain information from the bench sheet to assist in completing Checklist #11 (See section 9.2).

Exhibit 9-1: Example Bench Sheet with TCLP Percent Solids

Date	Sample ID	Filter Weight	Container Weight	Sample +	Container Weight (g)	Total Sample Weight	Res-idue +	Filter Weight (g)	Residue Weight (g)	% Solid Comments
6/22/01	JZ101	1.38	10.65	111.67	101.02	89.65	88.27	12.75	87.38	Multi-Phase Waste
6/22/01	JZ102	1.40	10.65	110.76	100.11	1.89	0.49	99.62	0.49	Filtrate is extraction fluid
6/22/01	JZ103	1.38	10.64	111.17	100.53	101.35	99.97	0.56	99.44	

1. Total Sample Weight = (Sample + Container Weight) - (Container Weight)
2. Residue Weight = (Residue Weight + Filter Weight) - (Filter Weight)
3. Filtrate Weight = (Total Sample Weight) - (Residue Weight)
4. % Solids = [(Residue Weight) ÷ (Total Sample Weight)] X 100

If filtrate is over 0.5%, then the waste is multi-phasic. The filtrate is saved as the extract, and the solid material is extracted with twenty times its weight in the proper extraction fluid.

The results for both the original liquid and the extract are mathematically combined using the equation below.

$$Final\ Analyte\ Concentration\ = \frac{V_1C_1 \times V_1C_2}{V_1 + V_2}$$

Where:

V1 = the volume of the first phase (L),

C1= the concentration of the analyte of concern in the first phase (mg/L),

V2= the volume of the second phase (L), and

C2= the concentration of the analyte of concern in the second phase (mg/L).

Exhibit 9-2 is an example of a typical TCLP Extraction Log that a laboratory will use to record method specific information. The Tier I Data Validator will obtain information from the extraction log to assist in completing Checklist#11 (See section 9.2).

Exhibit 9-2: Example TCLP Extraction Log

Date Extr. Started	Sample ID	Sample Weight (g)	Initial pH	pH After HCl	Ext. Fluid #	Ext. Fluid pH	Vol. of Extraction Fluid (ml)	Time On Tumbler (min.)	Time Off Tumbler (min.)	Final pH Before Filtra-tion	Final pH After Filtra-tion
6/22/01	JZ100	100.45	7.18	1.52	1	4.90	2009	5:15	11:00	5.06	5.00
6/22/01	JZ101	12.62	6.78	1.67	1	4.90	252.4	5:15	11:00	5.87	
6/22/01	JZ102	Extraction fluid is direct filtered									
6/22/01	JZ103	100.61	9.45	5.02	2	2.90	2012.2	5:15	11:00	6.88	6.87

Exhibit 9-3 is an example of a typical TCLP ZHE Extraction Log that a laboratory will use to record method-specific information. The Tier I Data Validator will obtain information from the extraction log to assist in completing Checklist#11 (See section 9.2).

Exhibit 9-3: Example ZHE Extraction for Volatile Compounds

Date Extr. Started	Sample ID	Sample Weight (g)	Initial pH	pH After HCl	Ext. Fluid #	Ext. Fluid pH	Vol. of Ext. Fluid (ml)	Time On Tumbler (min.)	Time Off Tumbler (min.)	Final pH Before Filtration	Final pH After Filtration
6/22/01	JZ100	22	ZHE		1	4.90	440	4:15	12:00	5.06	5.00

9.2 Example of Completed TCLP Checklist

The TCLP analytical data validation example below was completed using data from Exhibits 9-1, 9-2, and 9-3.

Example 9-1: Completed TCLP Checklist

11.0 TCLP Data Validation
The toxicity characteristic leaching procedure (TCLP) is used to determine whether wastes exhibit the toxicity characteristic or whether Land Disposal Restrictions have been met. The TCLP test is specified in OAC Rule 3745-51-24 and defined in SW-846, Method 1311. TCLP data validation requires specific data concerning extraction preparation in addition to the usual data submitted for organic and inorganic analytical methods. In most cases, a laboratory will have to supply bench sheet data to complete the data validation. The Validator may consult the Tier I Data Validation Manual for specific information and examples.

11.1 Did the laboratory calculate TCLP filterable solids? Based on the percent solid calculations, were the correct analytical procedures followed? **Note: TCLP requires that solid waste, semi-solid waste and liquid wastes be prepared based upon the amount of solids in the waste. For waste that has greater than 99.5% solids, the waste is considered solid and 100 grams of material is extracted with 20 times this weight of extraction fluid. For waste that is equal to or less than 0.5% solids, the waste is considered a liquid, and the liquid itself is considered the extract (no additional extraction fluid or tumbling is necessary). If the waste contains both solids and liquids, the solid portion, trapped by filtering, is extracted with 20 times its weight of extraction fluid and then analyzed. In addition, an aliquot of the liquid is analyzed. The results are then mathematically combined. Alternately, the multi-phase components may be physically recombined prior to analysis.** *Action: If percent solids were not calculated, contact the facility for the proper information.* *If, based on the percent solids calculations, the appropriate preparation methods were not used, qualify analytical results using the following criteria: All positive results above the regulatory level should not be qualified.* *All positive results above the detection limits but below the regulatory level should be qualified based on professional judgment and the specific circumstances. The Tier I Data Validator may want to consult the Tier II Validator.* *All non-detected results should be qualified based on professional judgment and the specific circumstances.*	Yes - see Exhibit 9-1, TCLP Percent Solids
11.2 Was the proper amount of material extracted? **Note: For waste samples to be analyzed for metals or SVOCs (in the solid portion), a**	List sample IDs and sample mass(es) used for the extraction. Yes, 100 grams were used for JZ100 and JZ103 which is the correct amount (See Exhibit 9-2,

minimum of 100 grams is required. For waste samples to be analyzed for volatile compounds, approximately 20-25 grams of sample is required. **Liquid samples are directly analyzed as the TCLP extract, no extraction fluid is added to the sample.** *Action: If improper sample mass is used, qualify analytical results using the following criteria:* *All positive results above the regulatory level should not be qualified. All positive results above the detection limits, but below the regulatory level, should initially be qualified as "J" estimated. Based on professional judgment, qualification of data as "R," may be warranted.* *Based on professional judgment, all non-detect results should be qualified as "J" estimated or "R."*	TCLP Extraction Log). Approximately 25 grams of sample were used for JZ100 ZHE extraction (See Exhibit 9-3, ZHE Extraction for Volatile Compounds). For JZ101, 12.62 grams was used for the multiphase sample. Because this is a multiphasic waste, this amount is acceptable (See Exhibit 9-2, TCLP Extraction Log).
11.3 Was the correct extraction fluid used? **Notes: Fluid # 1 is always used for VOC analysis.** **Fluid #1 should be used if the final pH of the pre-test sample is below 5.0.** **If the pH is above 5.0, hydrochloric acid should be added to the pre-test sample (refer to the method for specifics) and re-analyzed for pH. Fluid #2 should be used if the final pH of the pre-test is above 5.0.** *Action: Consult with the facility and have the extraction fluid information submitted. If the improper fluid was used, qualify analytical results using the following criteria:* *All positive results above the regulatory level should not be qualified. All positive results above the detection limits but below the regulatory level, should initially be qualified as "J." Rejection of data may be warranted if other preparatory procedures are outside of criteria.*	List sample IDs and fluid type(s) used for the extraction: Extraction Fluid #1 was used for all samples. Extraction Fluid #2 should have been used for sample JZ-103 because it's pH after HCL is > 5.0 (5.02 for JZ-103). If metals results were just below regulatory levels, consideration of the proper extraction fluid is very important. A more aggressive extraction fluid (i.e., extraction fluid #2) may have extracted more metals.

All non-detected results will be qualified as "R."	
11.4 Did the extraction fluid have the proper pH? Fluid #1 has a pH range of 4.88 to 4.98. Fluid #2 has a pH range of 2.83 to 2.93. *Action: If an improperly prepared extraction fluid is used, qualify analytical results using the following criteria:* *All positive results above the regulatory level should not be qualified.* *All positive results above the detection limits, but below the regulatory level, should initially be qualified as "J." Rejection of data may be warranted if other preparatory procedures are outside of criteria.* *All negative results will be qualified as "R."*	List incorrect fluid pH(s): Only extraction fluid #1 was used and its pH (4.90) was in the proper range. The wrong fluid was used for JZ103. All other sample extraction fluids were acceptable.
11.5 Was the correct weight of extraction fluid used? Laboratory bench sheets may be needed to complete this section. *Action: If the extraction fluid weight is not more than +/- 15% of the correct value (2000 grams for metals; 500 grams for VOCs), qualify all results as estimated "J" or "UJ". These values may be re-qualified if additional problems with TCLP preparation exist.* *If the extraction fluid weight is less than 70% of the proper weight, qualify all results as rejected, "R."* *If the extraction fluid weight is more than 30% greater than the proper weight, qualify all non-detect compounds and positive results below the regulatory level, as rejected "R." All positive results above the regulatory limit will not be qualified.*	The correct weights of extraction fluid were used. Yes, the extraction fluid volumes are within 15% of the correct amount (e.g., 20X the sample weight).

11.6 Was a TCLP blank analyzed with every batch of samples? **Note: TCLP blanks should be prepared using the same extraction fluid as is used for the associated sample's extraction.** *Action: Contact the facility for submittal of missing data. If no blank was analyzed, qualify all positive results as rejected, "R." If data is available, qualify TCLP data as designated in Section 4.0 Blank Data Summary Review.*	List IDs of affected samples: No information is present.
11.7 Was the tumbling time within 18 +/- 2 hours? **Note: Tumbling time (evaluated based on the day and time tumbling begins/is completed) should be noted on the bench sheets. The laboratory should be contacted if this information isn't present.** *Action: If the tumbling time is not within 18 +/- 2 hours, qualify all data as estimated ("J").*	Yes.
11.8 Was the tumbler speed within 30 +/- 2 RPM? **Note: Tumbler speed should be noted on the bench sheets. The laboratory should be contacted if this information isn't present.** *Action: If the tumbler speed is not within 30 +/- 2 RPM, qualify all data as estimated ("J").*	No information is present.
11.9 Was the room temperature during the extraction 23oC +/-2oC? **Note: Data would not be rejected using this criterion except in extreme cases (e.g., very cold temperature with detectable TCLP compounds).** *Action: Mark as estimated ("J" qualify) data for extractions outside this range or when temperature was not recorded.*	No information is present.

VOC, SVOC, Metals, and Mercury results from the TCLP must meet the sample QA/QC criteria outlined in Checklists #1, #2, #4, #6, and #7.

10.0 Corrosivity Checklist Example

The following example will illustrate the appropriate procedures for validation of pH data used to determine the corrosivity characteristic.

A sample of a liquid waste was split between two laboratories for analysis to determine whether the waste met the regulatory criteria for corrosivity. The analysis from a single contract laboratory determined that the waste pH was 12.1. This pH is slightly below the regulatory criteria for corrosivity. Additional information requested from the laboratory is summarized in Exhibit 10-1 and was used to complete Example 10-1 (Checklist #12) below for pH.

Exhibit 10-1: Example pH Calibration and Temperature Information

Sample Collection Date and Time:	09/27/01; 08:35 hours
Lab Sample Receipt Date and Time:	09/27/01; 16:08 hours
Sample Analysis Date and Time:	09/29/01; 13:47 hours
Calibration Buffers:	4, 7, and 10
Buffer Expiration Date:	11/20/01
Calibrated:	Daily
Certified:	Yearly
Continuing Calibration:	No
Temperature Compensation:	Yes, automatic temperature controller
Temperature of sample:	22.3°C

Example 10-1: Example of Completed Corrosivity Checklist

12.0 Corrosivity Data Validation	
pH is an important parameter used in ambient ground water monitoring and for determining if a waste displays the characteristic of corrosivity. For corrosivity determinations, OAC Rule 3745-51-22 specifies that SW-846, Method 9040C be used as the analytical test.	
12.1 Were the pH tests performed as soon as practically possible? **Note: SW-846 Method 9040C does not specify a maximum technical holding time for pH. However, it does state that all tests must be performed as soon as possible. Ohio EPA expects that most laboratories can perform the pH test within 24 hours of sample receipt.** *Action: If analyses were performed within 24 hours, no action is necessary. If analyses were performed after 24 hours, but before the end of 7 days after sample receipt, all sample results between a pH of 2.05 and 12.5 will be flagged as "J." If the results are equal to or less than a pH of 2 or greater than or equal to a pH of 12.5, the results will not be flagged.*	**Note time and date of sampling, sample receipt, and analysis for each sample.** More than 24 hours elapsed between sample receipt and analysis (09/27/01, 16:08 to 09/29/01, 13:47) The sample result of 12.1 should be considered estimated and the results flagged with a "J."

If analyses were performed 7 days or more after sample receipt, all sample results between a pH of 2.05 and 12.45 will be flagged as "R." If the results are equal to or less than a pH of 2 or greater than or equal to a pH of 12.5, the results will not be flagged.	
12.2 Was a yearly NIST certification of the analytical instrument performed? **Note: This information must be part of the Laboratory QAPP. Check the QAPP or request information for the facility or laboratory.** *Action: If a yearly certification was not performed, flag all results between a pH of 2.05 and 12.5 as "J" All results meeting the regulatory criteria for corrosivity will not be flagged.*	**Indicate yes, no, or NA:** According to information from the lab, the instruments are certified once a year. **Summarize any action taken:**
12.3 Were the calibration buffers within their expiration date? **Note: The laboratory can provide a photocopy of the expiration date and the buffer batch ID?** *Action: If the expiration date is exceeded, flag all results between pH 2.05 or 12.45 as "R." Initially, results meeting the regulatory criteria for corrosivity will not be flagged; however, the Validator may qualify results based upon professional judgment and the DQOs for the data.*	**Indicate yes, no, or NA:** Yes, the calibration buffers are within the expiration date. **Summarize any actions taken:**
12.4 Was the instrument calibrated correctly using at least two buffers that bracket the expected pH of the sample? **Note: For corrosivity determinations, the calibration buffers must include a pH 2 buffer and a pH 12 buffer. Review the calibration log for information or request information from the laboratory.** *Action: If an insufficient number of buffers were used (i.e., one) or if the incorrect buffers were used (buffers did not include a pH of 2 or 12 for corrosivity determination), flag all results between a pH of 2.05 and 12.45 as estimated, "J." All results meeting the regulatory criteria for*	**Indicate yes or no:** No. The instrument was calibrated using three buffers, 4, 7, and 10. For most water analyses, this buffer set is adequate. However, SW-846, Method 9040C specifies that for corrosivity determinations, calibration buffers of pH 2 and 12 must be used. In this example, these buffers were not used. **Summarize any actions taken**: Since the pH was determined to 12.1, the result should at least be considered estimated and the result flagged, "J." In addition, the result is within

corrosivity will not be flagged. If the pH of the waste is within 1.5 pH units of the regulatory criteria for corrosivity (3.0 or 11.0) and a pH buffer of 12 was not used, the results may be questionable and additional analyses using the correct buffers standards may be necessary.	1.0 pH units of the regulatory level. The result is questionable, and a second pH determination should be made using the appropriate calibration buffers.
12.5 Was continuing calibration performed? **Note: If continuing calibration was performed, the pH of the continuing calibration buffer must be within 0.5 pH units of the buffer pH. Information on the continuing calibration standard and results must be requested from the laboratory.** *Action: If continuing calibration was performed and the results were within 0.5 pH of the calibration buffer, no action is necessary. If continuing calibration was performed, and the results were greater or less than 0.5 pH units of the correct reading for the calibration buffer, then the analysis must have been terminated and the instrument recalibrated. If recalibration was necessary, but not performed, flag all results between a pH of 2.05 and 12.5 as estimated, "J." Initially, results meeting the regulatory criteria for corrosivity will not be flagged; however, the Validator may qualify results based upon professional judgment and the DQOs for the data.*	**Indicate yes or no:** No, continuing calibration was not performed. **Summarize any actions taken**: N/A
12.6 Were the temperature of the sample and the calibration buffers within 2°C of each other? **Note: Request the information from the laboratory. If the sample and the calibration buffers were not within 2°C, then temperature compensation must have been performed. Request information from the laboratory on manual temperature compensation procedures or whether an automatic temperature compensation was used.** *Action: If temperature compensation was required but not performed, flag all results between pH 2.05 or 12.45 as "J." Initially, results meeting the regulatory criteria for corrosivity will not be flagged; however, the Validator may*	**Indicate yes or no:** The temperature was controlled with automatic temperature compensation. **Summarize any actions taken:** N/A

qualify results based upon professional judgment and the DQOs for the data.	
12.7 If the sample pH was above 12.0, was the temperature of the sample maintained at 25 +/- 1°C? *Action: If the temperature was maintained at 25 +/- 1°C, then no action is necessary. If the temperature was not maintained at 25+/-1°C, but the results meet the regulatory criteria of corrosivity, then the results will not be flagged. If the temperature was not maintained, then reject, "R", all results between 12.0 and 12.5.*	**Indicate yes or no:** No. The temperature of the sample was 22.3°C. Temperature was not maintained at 25+/-1°C. **Summarize any actions taken:** The result should be rejected, and the data should be flagged "R."

This example illustrates that several QA/QC criteria are more specific for corrosivity determinations than for other pH determinations. For example, the calibration buffer solutions must always include pH 2 and pH 12 and the temperature of the sample must be maintained at 25 +/- 1°C. Because of the added specificity of the corrosivity test, it will most likely be necessary to contact the laboratory to receive information about their procedures. It is always appropriate to determine the exact procedures used by a laboratory when making a waste characterization based on corrosivity.

Appendix B
Data Validation Case Study

This appendix presents a Data Validation case study with an example data report and supporting documents that need to be validated. Following the data are completed checklists that illustrate how to review and validate this data using the Tier I checklists.

[redacted] *LABORATORIES OF OHIO, LLC*
WORK ORDER 115782

DELIVERABLES

00001

Checklist for Completeness Review **Work Order:** 115782

Verify that the Certificates of Analyses are signed	✓
Verify customer letter information is correct	✓
Verify appropriate case narratives are submitted	✓
Verify all analyses were completed using appropriate method	✓
Verify all & only the analyses requested are reported	✓
Verify client specific information is submitted	✓
Verify customer COC, sample receipt review sheet, and NCRs (if appropriate) are submitted	✓
Verify sample results for each sample and analyses	✓
Verify QC results for each analysis	✓
Verify table of contents (if appropriate) is provided and accurate	✓

[redacted] **Reviewed by** **Date** 8/13/04

This form has been reviewed by [redacted] Laboratories of Ohio's QA department, 9/15/03.

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[redacted] LABORATORIES OF OHIO, LLC
an Affiliate of THE [redacted] GROUP, INC.
Meeting Today's Needs with a Vision for Tomorrow

July 27, 2004

OHIO ENVIRONMENTAL PROTECTION AGENCY
Division of Hazardous Waste Management
ATTN: Mr.
PO Box 1049
Columbus, OH 43216

Dear Mr.

Please find enclosed the results of analysis and electronic deliverable for the following samples:

Site/Location:

Work Order No.: 115782

Samples No(s): See Sample Case Narrative

Date Received: June 25, 2004

Total No. of Pages: 155

This report shall not be reproduced except in full, without the written approval of [redacted] Laboratories of Ohio, LLC.

The contents of this data package have been reviewed for technical compliance and project completeness. Release of the data contained in this hard copy data package has been authorized by the Laboratory Director or the Director's designee, as verified by the signature below.

We appreciate the opportunity to service your analytical needs. If you have any further questions, please feel free to contact us.

Sincerely,

[redacted]

Project Manager

EC:dmr

Enclosures

[redacted] 00003

Page: 1 of 1
Project #:
GEL Quote #:
COC Number (1):
PO Number:

[redacted] Laboratories of Ohio, LLC
Chain of Custody and Analytical Request

[redacted]

Client Name: Ohio EPA — Phone #: 614/644-2938
Project/Site Name: Blue Knell Industries — Fax #: 614/728-1245
Address: 122 S. Front St., Cols. OH 43215
Collected by: — Send Results To:

Sample ID	Date Collected (mm-dd-yy)	Time Collected (Military) (hhmm)	QC Code (2)	Field Filtered (3)	Sample Matrix (4)	Should this sample be considered: Radioactive	Should this sample be considered: TSCA Regulated	Total number of containers	TCLP VOCs	TCLP Metals	pH	Flashpoint	PCBs	Total VOCs	Tot. RCRA Metals					Comments
DM-028	06-23-04	1545			WW			1	X	X										
RO-02	06-24-04	1110			SS			2	X	X										
002	06-24-04	0930			O			1			X	X	X	X	X					

Sample Analysis Requested (5) (Fill in the number of containers for each test)
<-- Preservative Type (6)

Comments: Note: extra sample is required for sample specific QC

TAT Requested: Normal: ____ Rush: ____ Specify: ____ (Subject to Surcharge) | Fax Results: Yes / (No) | Circle Deliverable: C of A / QC Summary / Level 1 / Level 2 / Level 3 / Level 4

Remarks: *Are there any known hazards applicable to these samples? If so, please list the hazards*

Chain of Custody Signatures

	Relinquished By (Signed)	Date	Time	Received by (signed)	Date	Time
1	[signature]	6/25/04	11:10	[signature]	6/25/04	11:10
2						
3						

Sample Delivery Details / Laboratory Receipt

GEL PM:
Method of Shipment: — Date Shipped:
Airbill #:
Airbill #:

1.) Chain of Custody Number = Client Determined
2.) QC Codes: N = Normal Sample, TB = Trip Blank, FD = Field Duplicate, EB = Equipment Blank, MS = Matrix Spike Sample, MSD = Matrix Spike Duplicate Sample, G = Grab, C = Composite
3.) Field Filtered: For liquid matrices, indicate with a - Y - for yes the sample was field filtered or - N - for sample was not field filtered.
4.) Matrix Codes: DW = Drinking Water, GW = Groundwater, SW = Surface Water, WW = Waste Water, W = Water, SO = Soil, SD = Sediment, SL = Sludge, SS = Solid Waste, O = Oil, F = Filter, P = Wipe, U = Urine, F = Fecal, N = Nasal
5.) Sample Analysis Requested: Analytical method requested (i.e. 8260B, 6010B/7470A) and number of containers provided for each (i.e. 8260B - 3, 6010B/7470A - 1).
6.) Preservative Type: HA = Hydrochloric Acid, NI = Nitric Acid, SH = Sodium Hydroxide, SA = Sulfuric Acid, AA = Ascorbic Acid, HX = Hexane, ST = Sodium Thiosulfate, If no preservative is added = leave field blank

For Lab Receiving Use Only
Custody Seal Intact? YES NO
Cooler Temp: 6 C

SAMPLE RECEIPT REVIEW

Date 6/25/04

Client Ohio EPA

Received by [redacted]

	SAMPLE REVIEW CRITERIA	YES	NO	N/A	COMMENTS
1	Were shipping containers received intact and sealed? If no, notify the Project Manager	X			
2	Were chain of custody documents included?	X			
3	Shipping container temperature(s) checked:	X			
4	Is temperature documented on Chain of Custody		X		Temp: 6°C
5	Was shipping container temperature within specifications (4 +/- 2 C) If no, notify Project Manager	X			
6	Are any of the samples from a client that is known to send radiological samples? If yes, complete radioactive receipt form.		X		
	Container at contact: Samples at contact:				
	Container at 1 meter: Packing material:				
7	Were chain of custody documents completed correctly? (ink, signed, match containers)	X			
8	Were sample containers received intact and sealed? If no, notify the Project Manager.	X			
9	Were all sample containers properly labeled?	X			
10	Were correct sample containers received?	X			
11	Were organic samples checked for residual chlorine?			X	
12	Preserved samples checked for pH?			X	
13	Were samples preserved correctly? If no, notify Project Manager.	X			
14	Were samples received within holding time? If No, notify Project Manager.	X			
15	Were VOA vials free of headspace?			X	

PM(A) Review: [redacted] Date 6/30/04

Samples out of specifications/ PM notification:

Problem upon receipt:

Resolution of problems:

(ALL ACCEPTABLE)
CIRCLE/INITIAL/DATE

[redacted] 6/25/04

[redacted] of Ohio, LLC [redacted]ead, Suite 300, Cincinnati, Ohio, 45242

This form has been reviewed by [redacted] Laboratories of Ohio's QA department, 9/15/03.

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Ohio GenChem Narrative
Ohio Environmental Protection Agency (OEPA)
Work Order 115782
SDG 115782

Method/Analysis Information

Procedure:	**Soil and Waste pH method 9045C**
Analytical Method:	SW846 9045C
Analytical Batch Number:	345466

Sample Analysis

The following samples were analyzed using the analytical protocol as established in SW846 9045C:

Sample ID	Client ID
115782011	002
1200654437	Laboratory Control Sample (LCS)

SOP Reference

Procedure for preparation, analysis and reporting of analytical data are controlled by [redacted] Laboratories of Ohio, LLC as Standard Operating Procedure (SOP). The data discussed in this narrative has been analyzed in accordance with INORG8 REV# 13.

Preparation/Analytical Method Verification

The SOP stated above has been prepared based on technical research and testing conducted by [redacted] Laboratories of Ohio, LLC and with guidance from the regulatory documents listed in this "Method/Analysis Information" section.

Calibration Information

The WetChem: General analysis was performed on a Accumet 25 pH Meter.

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Initial Calibration
The instrument was properly calibrated.

Quality Control (QC) Information

Laboratory Control Sample (LCS) Recovery
The recovery for the laboratory control sample was within the required acceptance limits.

Quality Control
All samples are analyzed in duplicate.

Sample Duplicate Acceptance
The Relative Percent Difference(s) between the sample(s) and duplicate(s) for this batch (was) within the required acceptance limits.

Technical Information

[redacted] assigns holding times based on the date and time of sample collection. Those holding times expressed in hours are calculated in the AlphaLims system by hours. Those holding times expressed as days expire at midnight on the day of expiration.

Holding Times
Samples were analyzed as soon as possible.
The following samples from this sample group were accidentally analyzed outside of the method specified holding time.

Preparation/Analytical Method Verification
All procedures were performed as stated in the SOP.

Sample Reanalysis
No samples in this sample group were reprepped and/or reanalyzed for any reason other than dilutions.

Miscellaneous Information

Additional Comments
No additional comments are needed for this SDG.

Review Validation:

Level 1 Initial [redacted] Date 7/2/04

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[redacted] LABORATORIES OF OHIO, LLC

Certificate of Analysis

Company : Ohio Environmental Protection Agency
Address : P.O. Box 1049
Columbus, Ohio 43216

Report Date: July 2, 2004

Contact:
Project:

Page 1 of 1

Client Sample ID: 002
Sample ID: 115782011
Matrix: Oil
Collect Date: 24-JUN-04 09:30
Receive Date: 25-JUN-04
Collector: Client

Project: OHEP00304
Client ID: OHEPA001

Client Desc.: Oil

Parameter	Qualifier	Result	DL	RL	Units	DF	Analyst	Date	Time	Batch	Method
General Analysis											
SW9045C pH											
pH at Temp 21.9C	H	2.66	1.00	1.00	SU	1	HB1	06/30/04	1649	345466	1

The following Analytical Methods were performed

Method	Description	Analyst Comments
1	SW846 9045C	

Notes:
The Qualifiers in this report are defined as follows :

* Indicates that a quality control analyte recovery is outside of specified acceptance criteria.
< Result is less than amount reported.
> Result is greater than amount reported.
B Target analyte was detected in the sample as well as the associated blank.
E Concentration of the target analyte exceeds the instrument calibration range.
H Analytical holding time exceeded.
J The result was greater than the MDL, but less than the RL and is an estimated value. Values below a CRDL are also flagged.
U Indicates the target analyte was analyzed for but not detected above the MDL.
X Lab-specific qualifier-please see case narrative, data summary package or contact your Project Manager for details.
h Sample preparation or preservation holding time exceeded.

The above sample is reported on an "as received" basis.

Where the analytical method has been performed under NELAP certification, the analysis has met all of the requirements of the NELAC standard unless qualified on the Certificate of Analysis.

This data report has been prepared and reviewed in accordance with [redacted] Laboratories of Ohio, LLC standard operating procedures. Please direct any questions to your Project Manager, [redacted]

7/2/04
Reviewed by

[redacted]

[redacted] LABORATORIES OF OHIO, LLC

QC Summary

Report Date: July 2, 2004
Page 1 of 1

Client : Ohio Environmental Protection Agency
P.O. Box 1049
Columbus, Ohio

Contact:

Workorder: 115782

Parmname	NOM	Sample	Qual	QC	Units	RPD%	REC%	Range	Anlst	Date	Time
WetChem: General											
Batch 345466											
QC1200654437 LCS											
pH	6.87			6.75	SU		98	(96%-104%)	HB1	06/30/04	15:26

Notes:
The Qualifiers in this report are defined as follows:

- * Indicates that a quality control analyte recovery is outside of specified acceptance criteria.
- < Result is less than amount reported.
- > Result is greater than amount reported.
- B Target analyte was detected in the sample as well as the associated blank.
- E Concentration of the target analyte exceeds the instrument calibration range.
- H Analytical holding time exceeded.
- J The result was greater than the MDL, but less than the RL and is an estimated value. Values below a CRDL are also flagged.
- U Indicates the target analyte was analyzed for but not detected above the MDL.
- X Lab-specific qualifier-please see case narrative, data summary package or contact your Project Manager for details.
- h Sample preparation or preservation holding time exceeded.

N/A indicates that spike recovery limits do not apply when sample concentration exceeds spike conc. by a factor of 4 or more.
^ The Relative Percent Difference (RPD) obtained from the sample duplicate (DUP) is evaluated against the acceptance criteria when the sample is greater than five times (5X) the contract required detection limit (RL). In cases where either the sample or duplicate value is less than 5X the RL, a control limit of +/- the RL is used to evaluate the DUP result.
For PS, PSD, and SDILT results, the values listed are the measured amounts, not final concentrations.

Where the analytical method has been performed under NELAP certification, the analysis has met all of the requirements of the NELAC standard unless qualified on the QC Summary.

7/2/04

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[redacted] OD Ohio Notebook No. 1559

PROJECT Batch: 3454616

Continued From Page

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pH 9045C
date 6/30/04

LCS: SWC.OH0629-01

Sample #	pH	Temp°C	Time	pH	Temp°C	Time
~~BEGCV~~	6.996	20.2	1517	6.986	20.3	1518
LCS 1200054434	6.754	23.4	1526	6.749	25.1	1528
1153200001	5.922	21.6	1541	5.917	22.7	1542
1153200002	5.800	21.3	1549	5.790	21.4	1550
1153210009	5.758	21.4	1556	5.763	21.4	1557
1153210011	5.751	21.3	1559	5.740	21.2	1602
1157820611 2.66	~~8.655~~	21.9	1649	~~8.627~~ 2.66	22.0	1651
CCV	6.951	20.7	1630	6.951	21.0	1634
CCV	6.971	21.0	1654	6.979	21.1	1655

HB 6/30/04

MM 7/1/04

Continued on Page—

Read and Understood By

[redacted] Signed — 6/30/04 Date — Matt Mann Signed — 7-1-04 Date

00010

Ohio GenChem Narrative
Ohio Environmental Protection Agency (OEPA)
Work Order 115782
SDG 115782

Method/Analysis Information

Procedure:	**Ignitability (Flash Point Determination) EPA Method 1010**
Analytical Method:	SW846 1010
Analytical Batch Number:	350989

Sample Analysis

The following samples were analyzed using the analytical protocol as established in SW846 1010:

Sample ID	Client ID
115782005	014
115782006	016
115782007	018
115782011	002
1200667723	Laboratory Control Sample (LCS)

SOP Reference

Procedure for preparation, analysis and reporting of analytical data are controlled by [redacted] Laboratories of Ohio, LLC as Standard Operating Procedure (SOP). The data discussed in this narrative has been analyzed in accordance with INORG30 REV# 11.

Preparation/Analytical Method Verification

The SOP stated above has been prepared based on technical research and testing conducted by [redacted] Laboratories of Ohio, LLC and with guidance from the regulatory documents listed in this

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"Method/Analysis Information" section.

Calibration Information

The WetChem: General analysis was performed on a Koehler Flashpoint.

Initial Calibration
The instrument was properly calibrated.

Calibration Verification Information (CCV)
Not required.

Quality Control (QC) Information

Laboratory Control Sample (LCS) Recovery
The recovery for the laboratory control sample was within the required acceptance limits.

Quality Control
All samples are analyzed in duplicate.

Sample Duplicate Acceptance
The Relative Percent Difference(s) between the sample(s) and duplicate(s) for this batch (was) within the required acceptance limits.

Technical Information

[redacted]assigns holding times based on the date and time of sample collection. Those holding times expressed in hours are calculated in the AlphaLims system by hours. Those holding times expressed as days expire at midnight on the day of expiration.

Holding Times
All samples from this sample group were analyzed within the required holding time for this method.

Preparation/Analytical Method Verification
All procedures were performed as stated in the SOP.

Sample Reanalysis
No samples in this sample group were reprepped and/or reanalyzed for any reason other than duplicates.

Miscellaneous Information

Nonconformance Reports
Nonconformance reports are generated to document any procedural anomalies that may deviate from referenced SOP or contractual documents. An NCR was not generated for this SDG.

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Additional Comments
No additional comments are needed for this SDG.

Review Validation:

Level 1 Initial CMA Date 7-26-04

00013

[redacted] LABORATORIES OF OHIO, LLC

Certificate of Analysis

Company : Ohio Environmental Protection Agency
Address : P.O. Box 1049
Columbus, Ohio 43216

Report Date: July 26, 2004

Contact:
Project:

Page 1 of 1

Client Sample ID: 002
Sample ID: 115782011
Matrix: Oil
Collect Date: 24-JUN-04 09:30
Receive Date: 25-JUN-04
Collector: Client

Project: OHEP00304
Client ID: OHEPA001

Client Desc.: Oil

Parameter	Qualifier	Result	DL	RL	Units	DF	Analyst	Date	Time	Batch	Method
General Analysis											
SW1010 Closedcup Flash Pt 200											
Flashpoint-200		138	68.0	68.0	Fahrenheit	1	HB1	07/21/04	0915	350989	1

The following Analytical Methods were performed

Method	Description	Analyst Comments
1	SW846 1010	
2	SW846 9045C	

Notes:
The Qualifiers in this report are defined as follows :

* Indicates that a quality control analyte recovery is outside of specified acceptance criteria.
< Result is less than amount reported.
> Result is greater than amount reported.
B Target analyte was detected in the sample as well as the associated blank.
E Concentration of the target analyte exceeds the instrument calibration range.
H Analytical holding time exceeded.
J The result was greater than the MDL, but less than the RL and is an estimated value. Values below a CRDL are also flagged.
P The concentration between the confirmation column and the primary column is >40 RPD for EPA methods 8081A, 8082 & 608.
U Indicates the target analyte was analyzed for but not detected above the MDL.
X Lab-specific qualifier-please see case narrative, data summary package or contact your Project Manager for details.
h Sample preparation or preservation holding time exceeded.

The above sample is reported on an "as received" basis.

Where the analytical method has been performed under NELAP certification, the analysis has met all of the requirements of the NELAC standard unless qualified on the Certificate of Analysis.

This data report has been prepared and reviewed in accordance with [redacted] Laboratories of Ohio, LLC standard operating procedures. Please direct any questions to your Project Manager, [redacted]

[redacted] 7-26-04
Reviewed by

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LABORATORIES OF OHIO, LLC

QC Summary

Report Date: July 26, 2004
Page 1 of 1

Client : Ohio Environmental Protection Agency
P.O. Box 1049
Columbus, Ohio

Contact:

Workorder: 115782

Parmname	NOM	Sample	Qual	QC	Units	RPD%	REC%	Range	Anlst	Date	Time
WetChem: General											
Batch 350989											
QC1200667723 LCS Flashpoint-200	77.0			77.7	Fahrenheit		101	(95%-105%)	HB1	07/21/04	09:15

Notes:
The Qualifiers in this report are defined as follows:

- * Indicates that a quality control analyte recovery is outside of specified acceptance criteria.
- < Result is less than amount reported.
- > Result is greater than amount reported.
- B Target analyte was detected in the sample as well as the associated blank.
- E Concentration of the target analyte exceeds the instrument calibration range.
- H Analytical holding time exceeded.
- J The result was greater than the MDL, but less than the RL and is an estimated value. Values below a CRDL are also flagged.
- P The concentration between the confirmation column and the primary column is >40 RPD for EPA methods 8081A, 8082 & 608.
- U Indicates the target analyte was analyzed for but not detected above the MDL.
- X Lab-specific qualifier-please see case narrative, data summary package or contact your Project Manager for details.
- h Sample preparation or preservation holding time exceeded.

N/A indicates that spike recovery limits do not apply when sample concentration exceeds spike conc. by a factor of 4 or more.
^ The Relative Percent Difference (RPD) obtained from the sample duplicate (DUP) is evaluated against the acceptance criteria when the sample is greater than five times (5X) the contract required detection limit (RL). In cases where either the sample or duplicate value is less than 5X the RL, a control limit of +/- the RL is used to evaluate the DUP result.
For PS, PSD, and SDILT results, the values listed are the measured amounts, not final concentrations.

Where the analytical method has been performed under NELAP certification, the analysis has met all of the requirements of the NELAC standard unless qualified on the QC Summary.

7-26-04

00015

Method 1010 Sample Run Log
Analyst: Holly Bricker
Date: 7/21/04

Sample ID#	Client ID#	Result	Start Time	End Time
1200667713	LCS	77	10:15 am	
1200667714	LCS	78		
115781886	Form. X1	142		
115781887	Form. X2	145		
115781888	Form. X3	143		
115781888D	Dup.	144		12:20 pm
1200667723	LCS	77	1:40 pm	
1200667724	LCS	77		
115782005	BR549	>200		
115782006	R2D2	148		
115782007	Dy4U	177		
115782011	002	138		
115782011D	Dup.	138		4:05 pm

Ohio Metals
Ohio Environmental Protection Agency (OEPA)
Work Order 115782
SDG 115782

Method/Analysis Information

Procedure:	**ICP Analysis according to EPA Method 6010B**
Analytical Method:	SW846 6010B
Prep Method:	SW846 3010A
Ohio SW846 1311 Metals TCLP Leaching Solids Method:	SW846 1311
Analytical Batch Number:	346202
Prep Batch Number:	346201
Ohio SW846 1311 Metals TCLP Batch Number:	345991

Sample Analysis

The following samples were analyzed using the analytical protocol as established in SW846 6010B:

Sample ID	Client ID
115782001	028
115782002	RO-2
115782003	001
115782006	016
1200655630	TCLP Blank (TB)
1200656091	Method Blank (MB)

00017

1200656095	Laboratory Control Sample (LCS)
1200656092	115782006(016) Sample Duplicate (DUP)
1200656093	115782006(016) Matrix Spike (MS)
1200656094	115782006(016) Matrix Spike Duplicate (MSD)

SOP Reference

Procedure for preparation, analysis and reporting of analytical data are controlled by [redacted] Laboratories of Ohio, LLC as Standard Operating Procedure (SOP). The data discussed in this narrative has been analyzed in accordance with INORG1 REV# 13.

Preparation/Analytical Method Verification

The SOP stated above has been prepared based on technical research and testing conducted by [redacted] Laboratories of Ohio, LLC and with guidance from the regulatory documents listed in this "Method/Analysis Information" section.

Calibration Information

The Metals: ICP analysis was performed on a Thermo Jarrell-Ash Enviro I ICAP 61E.

Instrument Calibration
The instrument calibrations are conducted using the method and instrument manufacturer s specifications. All initial calibration requirements have been met for this SDG.

Initial Calibration (ICV) Requirements
All initial calibration verification requirements have been met for this SDG.

ICSA/ICSAB Statement
All interference check samples (ICSA and ICSAB) associated with this SDG met the established acceptance criteria.

Continuing Calibration Blank (CCB) Requirements
All continuing calibration blanks (CCB) bracketing this batch met the established acceptance criteria.

Continuing Calibration Verification (CCV) Requirements
All continuing calibration verifications (CCV) bracketing this SDG met the acceptance criteria.

Quality Control (QC) Information

Blank Acceptance

00018

The tumble blank contained Barium RL but <5% of the TCLP regulatory limit. The method blank showed no contamination above the MDLs for parameters of interest.

LCS/LCSD Recovery Statement
The laboratory control sample (LCS) met the acceptance criteria for percent recovery (%R) for all applicable analytes.

Quality Control (QC) Sample Statement
The following sample was selected as the quality control (QC) sample for this batch: 115782006 (016).

Matrix Spike Recovery Statement
The percent recovery (%R) obtained from the MS analyses are evaluated when the sample concentration is less than four times (4X) the spike concentration added. All applicable elements met the acceptance criteria.

Matrix Spike Duplicate Recovery Statement
The percent recovery (%R) obtained from the MSD analyses are evaluated when the sample concentration is less than four time (4X) the spike concentration added. All applicable elements met the acceptance criteria.

MSD RPD Statement
The relative percent difference (RPD) obtained from the designated matrix spike duplicate (MSD) is evaluated based on acceptance criteria of 20%. The RPD between qualifying elements results in the MS and MSD were within the acceptance limits of 20%.

Duplicate RPD Statement
The relative percent difference (RPD) obtained from the designated sample duplicate (DUP) is evaluated based on acceptance criteria of 20% when the sample is 5X the contract required detection limit(RL). In cases where either the sample or duplicate value is less than 5X the RL, a control of +/-RL is used to evaluate the DUP results. All applicable analytes met these requirements.

Technical Information

[redacted] assigns holding times based on the date and time of sample collection. Those holding times expressed in hours are calculated in the AlphaLims system by hours. Those holding times expressed as days expire at midnight on the day of expiration.

Holding Time Specifications
All the samples were prepared and/or analyzed within the required holding time period.

Preparation/Analytical Method Verification
All procedures performed in association with this SDG followed the Standard Operating Procedure (SOP) guidelines. All samples in this SDG were prepared in accordance with the referenced SW-846 procedures.

00019

Sample Dilutions
Dilutions are performed to minimize matrix interferences resulting from elevated mineral element concentrations present in soil samples and/or to bring over range target analyte concentrations into the linear calibration range of the instrument. No sample dilutions were needed in this SDG.

Re-prep/Re-analysis
No samples in this SDG required redigestion and/or reanalysis.

Miscellaneous Information

Additional Comments
No additional comments are needed for this sample group.

Method/Analysis Information

Procedure:	**ICP Analysis according to EPA Method 6010B**
Analytical Method:	SW846 6010B
Prep Method:	SW846 3050B
Analytical Batch Number:	346537
Prep Batch Number:	346534

Sample Analysis

The following samples were analyzed using the analytical protocol as established in SW846 6010B:

Sample ID	Client ID
115782011	002
1200656824	Method Blank (MB)
1200656828	Laboratory Control Sample (LCS)
1200656825	115782011(002) Sample Duplicate (DUP)
1200656826	115782011(002) Matrix Spike (MS)
1200656827	115782011(002) Matrix Spike Duplicate (MSD)

00020

SOP Reference

Procedure for preparation, analysis and reporting of analytical data are controlled by [redacted] Laboratories of Ohio, LLC as Standard Operating Procedure (SOP). The data discussed in this narrative has been analyzed in accordance with INORG1 REV# 13.

Preparation/Analytical Method Verification

The SOP stated above has been prepared based on technical research and testing conducted by [redacted] Laboratories of Ohio, LLC and with guidance from the regulatory documents listed in this "Method/Analysis Information" section.

Calibration Information

The Metals: ICP analysis was performed on a Thermo Jarrell-Ash Enviro I ICAP 61E.

Instrument Calibration
The instrument calibrations are conducted using the method and instrument manufacturer s specifications. All initial calibration requirements have been met for this SDG.

Initial Calibration (ICV) Requirements
All initial calibration verification requirements have been met for this SDG.

ICSA/ICSAB Statement
All interference check samples (ICSA and ICSAB) associated with this SDG met the established acceptance criteria.

Continuing Calibration Blank (CCB) Requirements
All continuing calibration blanks (CCB) bracketing this batch met the established acceptance criteria.

Continuing Calibration Verification (CCV) Requirements
All continuing calibration verifications (CCV) bracketing this SDG met the acceptance criteria.

Quality Control (QC) Information

Blank Acceptance
The method blank showed no contamination above the MDL for any parameters of interest except for Arsenic which was detected MDL but <1/2RL.

LCS/LCSD Recovery Statement
The laboratory control sample (LCS) met the acceptance criteria for percent recovery (%R) for all applicable analytes.

00021

Quality Control (QC) Sample Statement
The following sample was selected as the quality control (QC) sample for this batch: 115782011 (002).

Matrix Spike Recovery Statement
The percent recovery (%R) obtained from the MS analyses are evaluated when the sample concentration is less than four times (4X) the spike concentration added. All applicable elements met the acceptance criteria.

Matrix Spike Duplicate Recovery Statement
The percent recovery (%R) obtained from the MSD analyses are evaluated when the sample concentration is less than four time (4X) the spike concentration added. All applicable elements met the acceptance criteria.

MSD RPD Statement
The relative percent difference (RPD) obtained from the designated matrix spike duplicate (MSD) is evaluated based on acceptance criteria of 20%. The RPD between qualifying elements results in the MS and MSD were within the acceptance limits of 20%.

Duplicate RPD Statement
The relative percent difference (RPD) obtained from the designated sample duplicate (DUP) is evaluated based on acceptance criteria of 20% when the sample is 5X the contract required detection limit(RL). In cases where either the sample or duplicate value is less than 5X the RL, a control of +/-RL is used to evaluate the DUP results. All applicable analytes met these requirements.

Technical Information

[redacted] assigns holding times based on the date and time of sample collection. Those holding times expressed in hours are calculated in the AlphaLims system by hours. Those holding times expressed as days expire at midnight on the day of expiration.

Holding Time Specifications
All the samples were prepared and/or analyzed within the required holding time period.

Preparation/Analytical Method Verification
All procedures performed in association with this SDG followed the Standard Operating Procedure (SOP) guidelines. All samples in this SDG were prepared in accordance with the referenced SW-846 procedures.

Sample Dilutions
Dilutions are performed to minimize matrix interferences resulting from elevated mineral element concentrations present in soil samples and/or to bring over range target analyte concentrations into the linear calibration range of the instrument. No sample dilutions were needed in this SDG.

00022

expressed as days expire at midnight on the day of expiration.

Holding Time Specifications
All the samples were prepared and/or analyzed within the required holding time period.

Preparation/Analytical Method Verification
All procedures performed in association with this SDG followed the Standard Operating Procedure (SOP) guidelines. All samples in this SDG were prepared in accordance with the referenced SW-846 procedures.

Sample Dilutions
Dilutions are performed to minimize matrix interferences resulting from elevated mineral element concentrations present in soil samples and/or to bring over range target analyte concentrations into the linear calibration range of the instrument. No sample dilutions were needed in this SDG.

Re-prep/Re-analysis
No samples in this SDG required redigestion and/or reanalysis.

Miscellaneous Information

Additional Comments
No additional comments are needed for this sample group.

Method/Analysis Information

Procedure:	**Mercury by Cold Vapor Method 7470A**
Analytical Method:	SW846 7470A
Prep Method:	SW846 7470A Prep
Ohio SW846 1311 Metals TCLP Leaching Solids Method:	SW846 1311
Analytical Batch Number:	346229
Prep Batch Number:	346228
Ohio SW846 1311 Metals TCLP Leaching Solids Batch Number:	345991

Sample Analysis

The following samples were analyzed using the analytical protocol as established in SW846

00023

7470A:

Sample ID	Client ID
115782001	028
115782002	RO-2
115782003	001
115782006	016
1200655630	TCLP Blank (TB)
1200656155	Method Blank (MB)
1200656161	Laboratory Control Sample (LCS)
1200656156	115782001(028) Sample Duplicate (DUP)
1200656158	115782001(028) Matrix Spike (MS)
1200656159	115782001(028) Matrix Spike Duplicate (MSD)

SOP Reference

Procedure for preparation, analysis and reporting of analytical data are controlled by [redacted] Laboratories of Ohio, LLC as Standard Operating Procedure (SOP). The data discussed in this narrative has been analyzed in accordance with INORG3 REV# 12.

Preparation/Analytical Method Verification

The SOP stated above has been prepared based on technical research and testing conducted by [redacted] Laboratories of Ohio, LLC and with guidance from the regulatory documents listed in this "Method/Analysis Information" section.

Calibration Information

The Metals: Mercury analysis was performed on a PSA Mercury Analyzer.

Instrument Calibration
The instrument calibrations are conducted using the method and instrument manufacturer s specifications. All initial calibration requirements have been met for this SDG.

Initial Calibration (ICV) Requirements
All initial calibration verification requirements have been met for this SDG.

00024

ICSA/ICSAB Statement
ICSA and ICSAB analysis does not apply to this method.

Continuing Calibration Blank (CCB) Requirements
All continuing calibration blanks (CCB) bracketing this batch met the established acceptance criteria.

Continuing Calibration Verification (CCV) Requirements
All continuing calibration verifications (CCV) bracketing this SDG met the acceptance criteria.

Quality Control (QC) Information

Blank Acceptance
The method blank associated with this SDG showed no contamination.

LCS/LCSD Recovery Statement
The laboratory control sample (LCS) met the acceptance criteria for percent recovery (%R) for all applicable analytes.

Quality Control (QC) Sample Statement
A DUP, MS and MSD were run on sample 115782001.
The following sample was selected as the quality control (QC) sample for this batch: 115782001 (Trailer 028).

Matrix Spike Recovery Statement
The MS recovery associated with this SDG passed.

Matrix Spike Duplicate Recovery Statement
The MSD recovery associated with this SDG passed.

MSD RPD Statement
The RPD between the MS and the MSD associated with this SDG passed.

Duplicate RPD Statement
The RPD between the sample and the duplicate associated with this SDG passed.

Technical Information

[redacted] assigns holding times based on the date and time of sample collection. Those holding times expressed in hours are calculated in the AlphaLims system by hours. Those holding times expressed as days expire at midnight on the day of expiration.

Holding Time Specifications
All the samples were prepared and/or analyzed within the required holding time period.

Preparation/Analytical Method Verification

(CCD25

Sample Dilutions
Dilutions are performed to minimize matrix interferences resulting from elevated mineral element concentrations present in soil samples and/or to bring over range target analyte concentrations into the linear calibration range of the instrument. No sample dilutions were needed in this SDG.

Re-prep/Re-analysis
No samples in this SDG required redigestion and/or reanalysis.

Miscellaneous Information

Nonconformance Documentation
Nonconformance reports (NCRs) are generated to document procedural anomalies that may deviate from referenced SOP or contractual documents. An NCR was not generated for this SDG.

Additional Comments
No additional comments are needed for this sample group.

Method/Analysis Information

Procedure:	**Mercury by Cold Vapor 7471A**
Analytical Method:	SW846 7471A
Prep Method:	SW846 7471A Prep
Analytical Batch Number:	344889
Prep Batch Number:	344887

Sample Analysis

The following samples were analyzed using the analytical protocol as established in SW846 7471A:

Sample ID	Client ID
115782011	002
1200653094	Method Blank (MB)
1200653100	Laboratory Control Sample (LCS)

00026

1200653095	115320001(02) Sample Duplicate (DUP)
1200653097	115320001(02) Matrix Spike (MS)
1200653098	115320001(02) Matrix Spike Duplicate (MSD)

SOP Reference

Procedure for preparation, analysis and reporting of analytical data are controlled by [redacted] Laboratories of Ohio, LLC as Standard Operating Procedure (SOP). The data discussed in this narrative has been analyzed in accordance with INORG60 REV# 3.

Preparation/Analytical Method Verification

The SOP stated above has been prepared based on technical research and testing conducted by [redacted] Laboratories of Ohio, LLC and with guidance from the regulatory documents listed in this "Method/Analysis Information" section.

Calibration Information

The Metals: Mercury analysis was performed on a PSA Mercury Analyzer.

Instrument Calibration
The instrument calibrations are conducted using the method and instrument manufacturer s specifications. All initial calibration requirements have been met for this SDG.

Initial Calibration (ICV) Requirements
All initial calibration verification requirements have been met for this SDG.

ICSA/ICSAB Statement
ICSA and ICSAB analysis does not apply to this method.

Continuing Calibration Blank (CCB) Requirements
All continuing calibration blanks (CCB) bracketing this batch met the established acceptance criteria.

Continuing Calibration Verification (CCV) Requirements
All continuing calibration verifications (CCV) bracketing this SDG met the acceptance criteria.

Quality Control (QC) Information

Blank Acceptance
The method blank associated with this SDG showed no contamination.

LCS/LCSD Recovery Statement

00027

The laboratory control sample (LCS) met the acceptance criteria for percent recovery (%R) for all applicable analytes.

Quality Control (QC) Sample Statement
A DUP,MS and MSD were run on sample 115320001.
The following sample was selected as the quality control (QC) sample for this batch: 115320001 (02).

Matrix Spike Recovery Statement
The MS recovery associated with this SDG passed.

Matrix Spike Duplicate Recovery Statement
The MSD recovery associated with this SDG passed.

MSD RPD Statement
The RPD between the MS and the MSD associated with this SDG passed.

Duplicate RPD Statement
The RPD between the sample and the duplicate associated with this SDG passed.

Technical Information

[redacted] assigns holding times based on the date and time of sample collection. Those holding times expressed in hours are calculated in the AlphaLims system by hours. Those holding times expressed as days expire at midnight on the day of expiration.

Holding Time Specifications
All the samples were prepared and/or analyzed within the required holding time period.

Preparation/Analytical Method Verification
All procedures performed in association with this SDG followed the Standard Operating Procedure (SOP) guidelines. All samples in this SDG were prepared in accordance with the referenced SW-846 procedures.

Sample Dilutions
Dilutions are performed to minimize matrix interferences resulting from elevated mineral element concentrations present in soil samples and/or to bring over range target analyte concentrations into the linear calibration range of the instrument. No sample dilutions were needed in this SDG.

Re-prep/Re-analysis
No samples in this SDG required redigestion and/or reanalysis.

Miscellaneous Information

Nonconformance Documentation

00028

Nonconformance reports (NCRs) are generated to document procedural anomalies that may deviate from referenced SOP or contractual documents. The following NCR was generated for this SDG:
NCR 123629 was generated due to Failed Recovery for LCS/MS/PS (rounds to 80%).

Additional Comments
No additional comments are needed for this sample group.

Review Validation:

Level 1 Initial MA Date 7/21/09

00029

[redacted] LABORATORIES OF OHIO, LLC

Certificate of Analysis

Company : Ohio Environmental Protection Agency
Address : P.O. Box 1049
Columbus, Ohio 43216

Report Date: July 21, 2004

Contact:
Project:

Page 1 of 2

Client Sample ID: 028
Sample ID: 115782001
Matrix: Aqueous
Collect Date: 23-JUN-04 15:45
Receive Date: 25-JUN-04
Collector: Client

Project: OHEP00304
Client ID: OHEPA001

Client Desc.:

Parameter	Qualifier	Result	DL	RL	Units	DF	AnalystDate	Time	Batch	Method
ICP Analysis										
TCLP Metals by 6010 Solids										
Arsenic	U	ND	0.0204	0.100	mg/L	1	JML 07/06/04	1634	346202	1
Barium		0.169	0.0006	0.010	mg/L	1				
Cadmium		0.787	0.0026	0.010	mg/L	1				
Chromium	J	0.0165	0.005	0.025	mg/L	1				
Lead	U	ND	0.0161	0.100	mg/L	1				
Selenium	U	ND	0.0189	0.080	mg/L	1				
Silver	U	ND	0.0032	0.025	mg/L	1				
Mercury Analysis										
TCLP Hg Solids										
Mercury	U	ND	0.000079	0.0002	mg/L	1	MAK 07/06/04	1228	346229	2

The following Prep Methods were performed

Method	Description	Analyst	Date	Time	Prep Batch
SW846 1311	SW846 1311 Metals TCLP Leaching Solids	TU1	07/01/04	1530	345991
SW846 3010A	Metals Leachate Digestion SW846 3010A	TU1	07/02/04	1400	346201
SW846 7470A Prep	EPA 7470A Mercury Prep TCLP Liquids	PS1	07/06/04	0730	346228

The following Analytical Methods were performed

Method	Description	Analyst Comments
1	SW846 6010B	
2	SW846 7470A	

Notes:
The Qualifiers in this report are defined as follows :

* Indicates that a quality control analyte recovery is outside of specified acceptance criteria.
< Result is less than amount reported.
> Result is greater than amount reported.
B Target analyte was detected in the sample as well as the associated blank.
E Concentration of the target analyte exceeds the instrument calibration range.
H Analytical holding time exceeded.
J The result was greater than the MDL, but less than the RL and is an estimated value. Values below a CRDL are also flagged.
U Indicates the target analyte was analyzed for but not detected above the MDL.
X Lab-specific qualifier-please see case narrative, data summary package or contact your Project Manager for details.

00030

[redacted] LABORATORIES OF OHIO, LLC

Certificate of Analysis

Company: Ohio Environmental Protection Agency
Address: P.O. Box 1049
Columbus, Ohio 43216

Report Date: July 21, 2004

Contact:
Project:

Page 2 of 2

Client Sample ID: 028
Sample ID: 115782001

Project: OHEP00304
Client ID: OHEPA001

Parameter	Qualifier	Result	DL	RL	Units	DF	AnalystDate	Time	Batch	Method

h Sample preparation or preservation holding time exceeded.

The above sample is reported on an "as received" basis.

Where the analytical method has been performed under NELAP certification, the analysis has met all of the requirements of the NELAC standard unless qualified on the Certificate of Analysis.

This data report has been prepared and reviewed in accordance with [redacted] Laboratories of Ohio, LLC standard operating procedures. Please direct any questions to your Project Manager, [redacted].

Reviewed by 7/21/04

00031

[redacted] LABORATORIES OF OHIO, LLC

Certificate of Analysis

Company : Ohio Environmental Protection Agency
Address : P.O. Box 1049
Columbus, Ohio 43216

Report Date: July 21, 2004

Contact:
Project:

Page 1 of 2

Client Sample ID: RO-2
Sample ID: 115782002
Matrix: Misc Solid
Collect Date: 24-JUN-04 11:10
Receive Date: 25-JUN-04
Collector: Client

Project: OHEP00304
Client ID: OHEPA001

Client Desc.:

Parameter	Qualifier	Result	DL	RL	Units	DF	AnalystDate	Time	Batch	Method
ICP Analysis										
TCLP Metals by 6010 Solids										
Arsenic	U	ND	0.0204	0.100	mg/L	1	JML 07/06/04	1650	346202	1
Barium		2.65	0.0006	0.010	mg/L	1				
Cadmium		0.387	0.0026	0.010	mg/L	1				
Chromium		0.486	0.005	0.025	mg/L	1				
Lead		0.296	0.0161	0.100	mg/L	1				
Selenium	J	0.0356	0.0189	0.080	mg/L	1				
Silver	J	0.00358	0.0032	0.025	mg/L	1				
Mercury Analysis										
TCLP Hg Solids										
Mercury		0.00073	0.000079	0.0002	mg/L	1	MAK 07/06/04	1240	346229	2

The following Prep Methods were performed

Method	Description	Analyst	Date	Time	Prep Batch
SW846 1311	SW846 1311 Metals TCLP Leaching Solids	TU1	07/01/04	1530	345991
SW846 3010A	Metals Leachate Digestion SW846 3010A	TU1	07/02/04	1400	346201
SW846 7470A Prep	EPA 7470A Mercury Prep TCLP Liquids	PS1	07/06/04	0730	346228

The following Analytical Methods were performed

Method	Description	Analyst Comments
1	SW846 6010B	
2	SW846 7470A	

Notes:
The Qualifiers in this report are defined as follows :

* Indicates that a quality control analyte recovery is outside of specified acceptance criteria.
< Result is less than amount reported.
> Result is greater than amount reported.
B Target analyte was detected in the sample as well as the associated blank.
E Concentration of the target analyte exceeds the instrument calibration range.
H Analytical holding time exceeded.
J The result was greater than the MDL, but less than the RL and is an estimated value. *Values below a CRDL are also flagged.*
U Indicates the target analyte was analyzed for but not detected above the MDL.
X Lab-specific qualifier-please see case narrative, data summary package or contact your Project Manager for details.
h Sample preparation or preservation holding time exceeded.

00032

[redacted] LABORATORIES OF OHIO, LLC

Certificate of Analysis

Company : Ohio Environmental Protection Agency
Address : P.O. Box 1049
Columbus, Ohio 43216

Report Date: July 21, 2004

Contact:
Project:

Page 2 of 2

Client Sample ID: RO-2
Sample ID: 115782002

Project: OHEP00304
Client ID: OHEPA001

Parameter	Qualifier	Result	DL	RL	Units	DF	AnalystDate	Time	Batch	Method

The above sample is reported on an "as received" basis.

Where the analytical method has been performed under NELAP certification, the analysis has met all of the requirements of the NELAC standard unless qualified on the Certificate of Analysis.

This data report has been prepared and reviewed in accordance with [redacted] Laboratories of Ohio, LLC standard operating procedures. Please direct any questions to your Project Manager, [redacted].

7/21/04

Reviewed by

00033

[redacted] LABORATORIES OF OHIO, LLC

Certificate of Analysis

Company : Ohio Environmental Protection Agency
Address : P.O. Box 1049
Columbus, Ohio 43216

Report Date: July 21, 2004

Contact:
Project:

Page 1 of 2

Client Sample ID: 002
Sample ID: 115782011
Matrix: Oil
Collect Date: 24-JUN-04 09:30
Receive Date: 25-JUN-04
Collector: Client

Project: OHEP00304
Client ID: OHEPA001

Client Desc.: Oil

Parameter	Qualifier	Result	DL	RL	Units	DF	AnalystDate	Time	Batch	Method
ICP Analysis										
6010/3050 ICP SCAN Metals Soil										
Arsenic	U	ND	1.69	9.23	mg/kg	1	JML 07/15/04	1031	346537	1
Barium	U	ND	0.175	1.38	mg/kg	1				
Cadmium	U	ND	0.129	0.923	mg/kg	1				
Chromium	U	ND	1.10	3.69	mg/kg	1				
Lead	U	ND	3.84	9.23	mg/kg	1				
Selenium	U	ND	1.93	9.23	mg/kg	1				
Silver	U	ND	0.563	2.31	mg/kg	1				
Mercury Analysis										
7471 Cold Vapor Hg in Solid										
Mercury	U	ND	0.00309	0.00995	mg/kg	1	MAK 06/30/04	1228	344889	2

The following Prep Methods were performed

Method	Description	Analyst	Date	Time	Prep Batch
SW846 3050B	SW846 3050BS Prep Solids	TU1	07/07/04	1425	346534
SW846 7471A Prep	EPA 7471A Mercury Prep Solids	PS1	06/30/04	0805	344887

The following Analytical Methods were performed

Method	Description	Analyst Comments
1	SW846 6010B	
2	SW846 7471A	

Notes:
The Qualifiers in this report are defined as follows :

* Indicates that a quality control analyte recovery is outside of specified acceptance criteria.
< Result is less than amount reported.
> Result is greater than amount reported.
B Target analyte was detected in the sample as well as the associated blank.
E Concentration of the target analyte exceeds the instrument calibration range.
H Analytical holding time exceeded.
J The result was greater than the MDL, but less than the RL and is an estimated value. Values below a CRDL are also flagged.
P The concentration between the confirmation column and the primary column is >40 RPD for EPA methods 8081A, 8082 & 608.
U Indicates the target analyte was analyzed for but not detected above the MDL.
X Lab-specific qualifier-please see case narrative, data summary package or contact your Project Manager for details.
h Sample preparation or preservation holding time exceeded.

00034

[redacted] LABORATORIES OF OHIO, LLC

Certificate of Analysis

Company : Ohio Environmental Protection Agency
Address : P.O. Box 1049
Columbus, Ohio 43216

Report Date: July 21, 2004

Contact:
Project:

Page 2 of 2

Client Sample ID: 002
Sample ID: 115782011

Project: OHEP00304
Client ID: OHEPA001

Parameter	Qualifier	Result	DL	RL	Units	DF	AnalystDate	Time	Batch	Method

The above sample is reported on an "as received" basis.

Where the analytical method has been performed under NELAP certification, the analysis has met all of the requirements of the NELAC standard unless qualified on the Certificate of Analysis.

This data report has been prepared and reviewed in accordance with [redacted] Laboratories of Ohio, LLC standard operating procedures. Please direct any questions to your Project Manager, [redacted]

7/21/04

Reviewed by

00035

[redacted] LABORATORIES OF OHIO, LLC

QC Summary

Report Date: August 13, 2004
Page 1 of 7

Client : Ohio Environmental Protection Agency
P.O. Box 1049
Columbus, Ohio

Contact:

Workorder: 115782

Parmname	NOM	Sample	Qual	QC	Units	RPD%	REC%	Range	Anlst	Date Time
Metals: ICP										
Batch 346202										
QC1200656092 115782006 DUP										
Arsenic		U ND	U	ND	mg/L	N/A		(+/-0.100)	JML	07/06/04 16:37
Barium		0.0621		0.0612	mg/L	2		(0%-20%)		
Cadmium		U ND	U	ND	mg/L	N/A		(+/-0.010)		
Chromium		U ND	U	ND	mg/L	N/A		(+/-0.025)		
Lead		U ND	U	ND	mg/L	N/A		(+/-0.100)		
Selenium		U ND	U	ND	mg/L	N/A		(+/-0.080)		
Silver		U ND	U	ND	mg/L	N/A		(+/-0.025)		
QC1200656095 LCS										
Arsenic	1.00			0.984	mg/L		98	(80%-120%)		07/06/04 16:30
Barium	1.00			1.01	mg/L		101	(80%-120%)		
Cadmium	0.500			0.488	mg/L		98	(80%-120%)		
Chromium	0.500			0.492	mg/L		98	(80%-120%)		
Lead	2.00			1.92	mg/L		96	(80%-120%)		
Selenium	0.500			0.462	mg/L		92	(80%-120%)		
Silver	0.500			0.447	mg/L		89	(80%-120%)		
QC1200656091 MB										
Arsenic			U	ND	mg/L					07/06/04 16:26
Barium			U	ND	mg/L					
Cadmium			U	ND	mg/L					
Chromium			U	ND	mg/L					
Lead			U	ND	mg/L					
Selenium			U	ND	mg/L					
Silver			U	ND	mg/L					
QC1200656093 115782006 MS										
Arsenic	1.00	U ND		0.971	mg/L		97	(75%-125%)		07/06/04 16:41
Barium	1.00	0.0621		0.981	mg/L		92	(75%-125%)		
Cadmium	0.500	U ND		0.477	mg/L		95	(75%-125%)		
Chromium	0.500	U ND		0.483	mg/L		97	(75%-125%)		
Lead	2.00	U ND		1.90	mg/L		94	(75%-125%)		
Selenium	0.500	U ND		0.502	mg/L		100	(75%-125%)		
Silver	0.500	U ND		0.440	mg/L		88	(75%-125%)		
QC1200656094 115782006 MSD										
Arsenic	1.00	U ND		0.988	mg/L	2	99	(0%-20%)		07/06/04 16:45
Barium	1.00	0.0621		0.990	mg/L	1	93	(0%-20%)		
Cadmium	0.500	U ND		0.484	mg/L	1	97	(0%-20%)		
Chromium	0.500	U ND		0.479	mg/L	1	96	(0%-20%)		
Lead	2.00	U ND		1.93	mg/L	2	96	(0%-20%)		
Selenium	0.500	U ND		0.498	mg/L	1	100	(0%-20%)		
Silver	0.500	U ND		0.447	mg/L	2	89	(0%-20%)		
QC1200655630 TB										
Arsenic			U	ND	mg/L					07/06/04 16:23
Barium				0.0233	mg/L					

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[redacted] LABORATORIES OF OHIO, LLC

QC Summary

Workorder: 115782

Parmname	NOM	Sample	Qual	QC	Units	RPD%	REC%	Range	Anlst	Date Time
Metals: ICP										
Batch 346202										
Cadmium			U	ND	mg/L					
Chromium			U	ND	mg/L				JML	07/06/04 16:23
Lead			U	ND	mg/L					
Selenium			U	ND	mg/L					
Silver			U	ND	mg/L					
Batch 346537										
QC1200656825 115782011 DUP										
Arsenic		U	ND	U	ND	mg/kg	N/A	(+/-9.86)	JML	07/15/04 10:35
Barium		U	ND	U	ND	mg/kg	N/A	(+/-1.48)		
Cadmium		U	ND	U	ND	mg/kg	N/A	(+/-0.986)		
Chromium		U	ND	U	ND	mg/kg	N/A	(+/-3.94)		
Lead		U	ND	U	ND	mg/kg	N/A	(+/-9.86)		
Selenium		U	ND	U	ND	mg/kg	N/A	(+/-9.86)		
Silver		U	ND	U	ND	mg/kg	N/A	(+/-2.46)		
QC1200656828 LCS										
Arsenic	100				83.0	mg/kg		83	(80%-120%)	07/15/04 10:28
Barium	100				93.7	mg/kg		94	(80%-120%)	
Cadmium	50.0				40.5	mg/kg		81	(80%-120%)	
Chromium	50.0				42.3	mg/kg		85	(80%-120%)	
Lead	200				161	mg/kg		81	(80%-120%)	
Selenium	50.0				40.0	mg/kg		80	(80%-120%)	
Silver	50.0				39.4	mg/kg		79	(75%-120%)	
QC1200656824 MB										
Arsenic				J	1.90	mg/kg				07/15/04 10:24
Barium				U	ND	mg/kg				
Cadmium				U	ND	mg/kg				
Chromium				U	ND	mg/kg				
Lead				U	ND	mg/kg				
Selenium				U	ND	mg/kg				
Silver				U	ND	mg/kg				
QC1200656826 115782011 MS										
Arsenic	95.0	U	ND		79.8	mg/kg		84	(75%-125%)	07/15/04 10:39
Barium	95.0	U	ND		89.4	mg/kg		94	(75%-125%)	
Cadmium	47.5	U	ND		39.7	mg/kg		84	(75%-125%)	
Chromium	47.5	U	ND		40.2	mg/kg		85	(75%-125%)	
Lead	190	U	ND		159	mg/kg		83	(75%-125%)	
Selenium	47.5	U	ND		40.5	mg/kg		85	(75%-125%)	
Silver	47.5	U	ND		38.8	mg/kg		82	(75%-125%)	
QC1200656827 115782011 MSD										
Arsenic	97.9	U	ND		83.5	mg/kg	5	85	(0%-20%)	07/15/04 10:42
Barium	97.9	U	ND		93.4	mg/kg	4	95	(0%-20%)	
Cadmium	49.0	U	ND		40.8	mg/kg	3	83	(0%-20%)	
Chromium	49.0	U	ND		41.2	mg/kg	3	84	(0%-20%)	
Lead	196	U	ND		160	mg/kg	1	81	(0%-20%)	
Selenium	49.0	U	ND		41.6	mg/kg	3	85	(0%-20%)	
Silver	49.0	U	ND		38.8	mg/kg	0	79	(0%-20%)	
Batch 346941										
QC1200657897 115782004 DUP										

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LABORATORIES OF OHIO, LLC

QC Summary

Workorder: 115782

Parmname	NOM	Sample	Qual	QC	Units	RPD%	REC%	Range	Anlst	Date Time
Metals: Mercury										
Batch 344889										
QC1200653095 115320001 DUP										
Mercury		U	ND	J	0.00347	mg/kg	N/A		(+/-0.00978)	MAK 06/30/04 11:58
QC1200653100 LCS										
Mercury	0.250				0.221	mg/kg		88	(88%-108%)	06/30/04 11:52
QC1200653094 MB										
Mercury				U	ND	mg/kg				06/30/04 11:48
QC1200653097 115320001 MS										
Mercury	0.248	U	ND		0.198	mg/kg		80	(80%-120%)	06/30/04 12:00
QC1200653098 115320001 MSD										
Mercury	0.249	U	ND		0.204	mg/kg	3	81	(0%-20%)	06/30/04 12:07
Batch 346229										
QC1200656156 115782001 DUP										
Mercury		U	ND	U	ND	mg/L	N/A		(+/-0.0002)	MAK 07/06/04 12:31
QC1200656161 LCS										
Mercury	0.005				0.00541	mg/L		108	(80%-120%)	07/06/04 12:06
QC1200656155 MB										
Mercury				U	ND	mg/L				07/06/04 12:04
QC1200656158 115782001 MS										
Mercury	0.005	U	ND		0.0049	mg/L		98	(75%-125%)	07/06/04 12:33
QC1200656159 115782001 MSD										
Mercury	0.005	U	ND		0.0052	mg/L	6	104	(0%-20%)	07/06/04 12:36
QC1200655625 TB										
Mercury				U	ND	mg/L				07/06/04 11:56
QC1200655630 TB										
Mercury				U	ND	mg/L				07/06/04 11:58
QC1200656758 TB										
Mercury				U	ND	mg/L				07/06/04 12:00
Batch 346944										
QC1200657908 115782005 DUP										
Mercury		U	ND	J	0.000111	mg/L	N/A		(+/-0.0002)	MAK 07/08/04 17:00
QC1200657914 LCS										
Mercury	0.005				0.0051	mg/L		102	(80%-120%)	07/08/04 16:37
QC1200657907 MB										
Mercury				U	ND	mg/L				07/08/04 16:35
QC1200657911 115782005 MS										
Mercury	0.005	U	ND		0.00541	mg/L		107	(75%-125%)	07/08/04 17:02
QC1200657912 115782005 MSD										
Mercury	0.005	U	ND		0.00529	mg/L	2	104	(0%-20%)	07/08/04 17:07
QC1200657776 TB										
Mercury				J	0.000111	mg/L				07/08/04 16:31
QC1200657894 TB										
Mercury				J	0.000111	mg/L				07/08/04 16:33
Batch 348686										
QC1200662206 LCS										
Mercury	0.005				0.00511	mg/L		102	(80%-120%)	MAK 07/14/04 11:19
QC1200662201 MB										
Mercury				U	ND	mg/L				07/14/04 11:17
QC1200661438 TB										
Mercury				U	ND	mg/L				07/14/04 11:13
QC1200661439 TB										

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Prep LogBook

Analyst: TU1 Verified by:_____
Batch: 346201
Lab SOP: INORG6 REV# 9

Type	Sample Id	Lot. Id	Spike Amount	Spike Units
LCS	1200656095	SMT040609-01	.25	mL
LCS	1200656095	SMT040518-02	.25	mL
MS	1200656093	SMT040609-01	.25	mL
MS	1200656093	SMT040518-02	.25	mL
MSD	1200656094	SMT040609-01	.25	mL
MSD	1200656094	SMT040518-02	.25	mL

+4. 7/6/04 12:45

Sample Type	Sample ID	Parent Sample ID	Method	Prep Date	Ph	Initial Wt.	Final Volume	Prep Factor	Matrix
TB	1200655630		SW846 3010A	02-JUL-2004 14:00	5	50 mL	50 mL	1	MISC SOLID
MB	1200656091		SW846 3010A	02-JUL-2004 14:00	7	50 mL	50 mL	1	MISC SOLID
LCS	1200656095		SW846 3010A	02-JUL-2004 14:00	7	50 mL	50 mL	1	MISC SOLID
SAMPLE	115782001		SW846 3010A	02-JUL-2004 14:00	6	50 mL	50 mL	1	AQUEOUS
SAMPLE	115782002		SW846 3010A	02-JUL-2004 14:00	6	50 mL	50 mL	1	MISC SOLID
SAMPLE	115782003		SW846 3010A	02-JUL-2004 14:00	5	50 mL	50 mL	1	AQUEOUS
SAMPLE	115782006		SW846 3010A	02-JUL-2004 14:00	5	50 mL	50 mL	1	MISC SOLID
DUP	1200656092	115782006	SW846 3010A	02-JUL-2004 14:00	5	50 mL	50 mL	1	MISC SOLID
MS	1200656093	115782006	SW846 3010A	02-JUL-2004 14:00	5	50 mL	50 mL	1	MISC SOLID
MSD	1200656094	115782006	SW846 3010A	02-JUL-2004 14:00	5	50 mL	50 mL	1	MISC SOLID

Reagent/Solvent Lot ID	Amount	Description
217179-C	5 mL	1:1 hydrchloric acid
202473-A	3 mL	NITRIC ACID

Comments: Hotplate/Hotblock Temperature: 90 C

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Prep Data Logbook Version 1:1

[redacted]

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Prep LogBook

BS, 7/8/04

Analyst: TU1 Verified by:_____
Batch: 346534
Lab SOP: INORG5 REV# 10

Type	Sample Id	Lot. Id	Spike Amount	Spike Units
LCS	1200656828	SMT040609-01	.5	mL
LCS	1200656828	SMT040518-02	.5	mL
MS	1200656826	SMT040609-01	.5	mL
MS	1200656826	SMT040518-02	.5	mL
MSD	1200656827	SMT040609-01	.5	mL
MSD	1200656827	SMT040518-02	.5	mL

Sample Type	Sample ID	Parent Sample ID	Method	Prep Date	Initial Wt.	Final Volume	Prep Factor	Matrix
MB	1200656824		SW846 3050B	07-JUL-2004 14:25	1 g	100 mL	100	MISC SOLID
LCS	1200656828		SW846 3050B	07-JUL-2004 14:25	1 g	100 mL	100	MISC SOLID
DUP	1200656825	115782010	SW846 3050B	07-JUL-2004 14:25	1.0144 g	100 mL	98.58044	MISC SOLID
MS	1200656826	115782010	SW846 3050B	07-JUL-2004 14:25	1.0522 g	100 mL	95.03897	MISC SOLID
MSD	1200656827	115782010	SW846 3050B	07-JUL-2004 14:25	1.021 g	100 mL	97.94319	MISC SOLID
SAMPLE	115782011		SW846 3050B	07-JUL-2004 14:25	1.0835 g	100 mL	92.29349	MISC SOLID

Reagent/Solvent Lot ID	Amount	Description
217177-C	10 mL	1:1 NITRIC ACID
040615-A	10 mL	30% HYDROGEN PEROXIDE
213555-A	10 mL	Hydrochloric Acid
202473-A	10 mL	NITRIC ACID

Comments: Hotplate/Hotblock Temperature: 92 C

CJM 7-20-04

00040

Prep Data Logbook Version 1:1

[redacted]

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TCLP LOGBOOK

Report run on: July 2, 2004 2:42 PM

Batch Id: 345991

Employee ID: [redacted]

Sop ID: INORG10

Extraction Start Date: 01-JUL-04 15:30:00 @ 23 C

Extraction Stop Date: 02-JUL-04 07:30:00 @ 22 C

Filtration Complete Date: 02-JUL-04 08:30:00 @ 23 C

T.U. 7/2/04

Sample Id	Matrix	Inst. ID	Inst. Speed	Sample Properties	Aliquot	Weight of Container	Weight of Filter Cake	Initial Ph	pH Temp C	2nd pH	20 X % wet solid X wt.sample	Volume of Filtrate	pH of Filtrate	Positive Pressure (Y/N)	Extraction Vessel number
115782001	m	TCLP01	30	99.2% Liquid (Liquid)	100.08g	N/A	N/A	8	50	NA	NA	125	6	N/A	N/A
115782002	m	TCLP01	30	Solid without Particle Size Reduction	100.1g	N/A	N/A	8	50	3.567	2002	125	6	N/A	N/A
115782003	m	TCLP01	30	99.8% Liquid (Liquid)	100.3g	N/A	N/A	7	50	NA	NA	125	5	N/A	N/A
115782006	m	TCLP01	30	Solid without Particle Size Reduction	100.12g	N/A	N/A	8	50	1.595	2003	125	5	N/A	N/A
1200655630 (TB)	m	TCLP01	30	Solid without Particle Size Reduction	100g	N/A	N/A	4.912	N/A	N/A	N/A	125	5	N/A	N/A

Bottle Lot Number: 111

Filter Lot Number: 92041201w

Tumbled with Buffer #1- M108 pH = 5.09

Note: The aliquot for metals matrix QC is taken from the parent sample filtrate.

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TCLP Logbook version 3 Modified 01-29-2002

[redacted]

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PS. 7/6/04

Prep LogBook

Analyst: PSI Verified by:_____
Batch: 346228
Lab SOP: INORG3 REV# 12

Type	Sample Id	Lot. Id	Spike Amount	Spike Units
LCS	1200656161	MMT222088-11	.1	mL
MS	1200656158	MMT222088-11	.1	mL
MSD	1200656159	MMT222088-11	.1	mL

Sample Type	Sample ID	Parent Sample ID	Method	Prep Date	Ph	Initial Wt.	Final Volume	Prep Factor	Matrix
TB	1200655625		SW846 7470A Prep	06-JUL-2004 07:30	5	30 mL	30 mL	1	SOIL
TB	1200655630		SW846 7470A Prep	06-JUL-2004 07:30	5	30 mL	30 mL	1	MISC SOLID
MB	1200656155		SW846 7470A Prep	06-JUL-2004 07:30	7	30 mL	30 mL	1	MISC SOLID
TB	1200656758		SW846 7470A Prep	06-JUL-2004 07:30	7	30 mL	30 mL	1	MISC LIQUID
LCS	1200656161		SW846 7470A Prep	06-JUL-2004 07:30	7	30 mL	30 mL	1	MISC SOLID
SAMPLE	115741001		SW846 7470A Prep	06-JUL-2004 07:30	6	30 mL	30 mL	1	SOIL
SAMPLE	115782001		SW846 7470A Prep	06-JUL-2004 07:30	6	30 mL	30 mL	1	MISC SOLID
DUP	1200656156	115782001	SW846 7470A Prep	06-JUL-2004 07:30	6	30 mL	30 mL	1	AQUEOUS
MS	1200656158	115782001	SW846 7470A Prep	06-JUL-2004 07:30	6	30 mL	30 mL	1	AQUEOUS
MSD	1200656159	115782001	SW846 7470A Prep	06-JUL-2004 07:30	6	30 mL	30 mL	1	AQUEOUS
SAMPLE	115782002		SW846 7470A Prep	06-JUL-2004 07:30	6	30 mL	30 mL	1	MISC SOLID
SAMPLE	115782003		SW846 7470A Prep	06-JUL-2004 07:30	5	30 mL	30 mL	1	MISC SOLID
SAMPLE	115782006		SW846 7470A Prep	06-JUL-2004 07:30	5	30 mL	30 mL	1	MISC SOLID
SAMPLE	115783001		SW846 7470A Prep	06-JUL-2004 07:30	7	5 mL	30 mL	6	MISC LIQUID
SAMPLE	115783002		SW846 7470A Prep	06-JUL-2004 07:30	9	5 mL	30 mL	6	MISC LIQUID
SAMPLE	115783003		SW846 7470A Prep	06-JUL-2004 07:30	6	5 mL	30 mL	6	MISC LIQUID
SAMPLE	115783004		SW846 7470A Prep	06-JUL-2004 07:30	7	5 mL	30 mL	6	MISC LIQUID

Reagent/Solvent Lot ID	Amount	Description
202473-A	.75 mL	NITRIC ACID
150564-A	1.5 mL	SULFURIC ACID FOR CV OHIO
221744-C	1.8 mL	HYDROXYLAMINE SULFATE SOLUTION FOR CV O
221741-C	2.5 mL	POTASSIUM PERSULFATE SOLUTION
221746-C	4.5 mL	POTASSIUM PERMANGANATE SOLUTION

Comments: Digestion Start Date: 06-JUL-04 08:10
Digestion End Date: 06-JUL-04 10:10
Hotplate/Hotblock Temperature: 90 C
Samples 115783001,115783002,115783003 and 115783004 were diluted 1-6 due to high organics in the sample.

[redacted]

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C0042

PS 6/30/04

Prep LogBook

Analyst: PS1 Verified by:_____
Batch: 344887
Lab SOP: INORG60 REV# 3

Type	Sample Id	Lot. Id	Spike Amount	Spike Units
LCS	1200653100	MMT222088-11	.1	mL
MS	1200653097	MMT222088-11	.1	mL
MSD	1200653098	MMT222088-11	.1	mL

Sample Type	Sample ID	Parent Sample ID	Method	Prep Date	Initial Wt.	Final Volume	Prep Factor	Matrix
MB	1200653094		SW846 7471A Prep	30-JUN-2004 08:05	0.6 g	30 mL	50	MISC SOLID
LCS	1200653100		SW846 7471A Prep	30-JUN-2004 08:05	0.6 g	30 mL	50	MISC SOLID
SAMPLE	115320001		SW846 7471A Prep	30-JUN-2004 08:05	0.6104 g	30 mL	49.1481	MISC SOLID
DUP	1200653095	115320001	SW846 7471A Prep	30-JUN-2004 08:05	0.6135 g	30 mL	48.89976	AQUEOUS
MS	1200653097	115320001	SW846 7471A Prep	30-JUN-2004 08:05	0.6056 g	30 mL	49.53765	AQUEOUS
MSD	1200653098	115320001	SW846 7471A Prep	30-JUN-2004 08:05	0.6015 g	30 mL	49.87531	AQUEOUS
SAMPLE	115320002		SW846 7471A Prep	30-JUN-2004 08:05	0.6014 g	30 mL	49.8836	MISC SOLID
SAMPLE	115320003		SW846 7471A Prep	30-JUN-2004 08:05	0.6063 g	30 mL	49.48046	MISC SOLID
SAMPLE	115321008		SW846 7471A Prep	30-JUN-2004 08:05	0.6034 g	30 mL	49.71826	MISC SOLID
SAMPLE	115321009		SW846 7471A Prep	30-JUN-2004 08:05	0.6097 g	30 mL	49.20453	MISC SOLID
SAMPLE	115321011		SW846 7471A Prep	30-JUN-2004 08:05	0.6047 g	30 mL	49.61138	MISC SOLID
SAMPLE	115764001		SW846 7471A Prep	30-JUN-2004 08:05	0.6016 g	30 mL	49.86702	SLUDGE
SAMPLE	115782010		SW846 7471A Prep	30-JUN-2004 08:05	0.6054 g	30 mL	49.55401	MISC SOLID
SAMPLE	115782011		SW846 7471A Prep	30-JUN-2004 08:05	0.6033 g	30 mL	49.7265	MISC SOLID

Reagent/Solvent Lot ID	Amount	Description
223099-C	2.5 mL	AQUA REGIA FOR CV OHIO
221744-C	1.8 mL	HYDROXYLAMINE SULFATE SOLUTION FOR CV O
221746-C	4.5 mL	POTASSIUM PERMANGANATE SOLUTION

Comments: Date: 6/30/04 09:15
Date: 6/30/04 09:45
Temperature: 90 C
Temperature 2: 90 C

Prep Data Logbook Version 1:1

[redacted]

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MR 6/30/04

00043

Volatiles Case Narrative
Ohio Environmental Protection Agency (OEPA)
Work Order 115782
SDG 115782

Method/Analysis Information

Procedure:	**GC-MS Analysis of Volatiles 8260B**
Analytical Method:	SW846 8260B
Prep Method:	SW846 1311
Analytical Batch Number:	347359
Prep Batch Number:	345667

Sample Analysis

The following samples were analyzed using the analytical protocol as established in SW846 8260B:

Sample ID	Client ID
115782001	028
115782002	RO-2
115782003	001
115782004	013
115782005	014
115782006	016
115782007	018
115782008	018
115782009	025
1200654868	TCLP Blank (TB)
1200658905	Laboratory Control Sample (LCS)
1200658906	115782001(028) Post Spike (PS)
1200658907	115782001(028) Post Spike Duplicate (PSD)

SOP Reference

Procedure for preparation, analysis and reporting of analytical data are controlled by [redacted] Laboratories of Ohio, LLC as Standard Operating Procedure (SOP). The data discussed in this narrative has been analyzed in accordance with ORG11 REV# 13.

Preparation/Analytical Method Verification

C0054

The SOP stated above has been prepared based on technical research and testing conducted by [redacted] Laboratories of Ohio, LLC and with guidance from the regulatory documents listed in this "Method/Analysis Information" section.

Calibration Information

The Volatiles: GC-MS analysis was performed on a HP 5973 Mass Spectrometer.

Initial Calibration
All the initial calibration requirements were met.

Continuing Calibration Verification Requirements
All the calibration verification standard (CCV) requirements were met.

Quality Control (QC) Information

Method Blank Acceptance
Target analytes were not detected above the reporting limit in the blank.

Surrogate Recovery
Sample 115782006 was analyzed twice and both time it had low surrogate recoveries. 115782006 (016).

Laboratory Control Sample Recovery Statement (LCS)
All the required analyte recoveries in the LCS were within the acceptance limits.

QC Sample Designation
Spike analyses were performed on the following sample: 115782001 (028).

Spike Recovery Statement
All the required spike recoveries were within the acceptance limits.

Spike Duplicate Recovery Statement
All the required spike recoveries were within the acceptance limits.

Relative Percent Difference Statement (RPD)
The RPD between spike recoveries were within the acceptance limits.

Internal Standard (ISTD) Acceptance
The internal standard responses, in all samples and quality control samples, met the required acceptance criteria.

Technical Information

[redacted] assigns holding times based on the date and time of sample collection. Those holding times expressed in hours are calculated in the AlphaLims system by hours. Those

0055

holding times expressed as days expire at midnight on the day of expiration.

Holding Time Specifications
All samples were originally analyzed with in hold time. Some of the samples had to be re-analyzed more dilute and due to internal standard and surrogate failures. The re-analysis took place out of hold time. 115782003 (001), 115782006 (016), 115782007 (018), 115782008 (018) and 115782009 (025).

Sample Preservation and Integrity
All samples met the sample preservation and integrity requirements.

Preparation/Analytical Method Verification
All procedures were performed as stated in the SOP.

Sample Dilutions
TCLP samples are routinely analyzed at a 50X dilution to minimize the potential for system contamination. TCLP reporting limits are still met at a 50X dilution. Some of these samples had to be analyzed at a higher dilution. Sample were however analyzed at the lowest dilution possible with out contaminating the instrument.

Sample Re-prep/Re-analysis
Sample 115782006, 007, 008 and 009 had to be re-analyzed due to low internal standard and surrogate recoveries. 115782006 (016), 115782007 (018), 115782008 (018) and 115782009 (025).

Miscellaneous Information

Nonconformance (NCR) Documentation
NCR ID 129468 The following NCR was generated for this SDG: NCR 129468 was generated due to Sample Analyzed out of Holding.

Manual Integrations
Manual integrations were performed as per SOP GEN 43, details are documented in the raw data.

TIC Comment
Tentatively identified compounds (TIC) were not required for this sample delivery group/work order.

Additional Comments
There were no additional comments.

Review Validation:
Initial VF Date 7/26/14

00056

Volatiles Case Narrative
Ohio Environmental Protection Agency (OEPA)
Work Order 115782
SDG 115782

Method/Analysis Information

Procedure:	**GC-MS Analysis of Volatiles 8260B**
Analytical Method:	SW846 8260B
Prep Method:	SW846 5035
Analytical Batch Number:	347358
Prep Batch Number:	347357

Sample Analysis

The following samples were analyzed using the analytical protocol as established in SW846 8260B:

Sample ID	Client ID
115782011	002
1200658896	Method Blank (MB)
1200658897	Laboratory Control Sample (LCS)
1200658898	115782011(002) Post Spike (PS)
1200658899	115782011(002) Post Spike Duplicate (PSD)

SOP Reference

Procedure for preparation, analysis and reporting of analytical data are controlled by [redacted] Laboratories of Ohio, LLC as Standard Operating Procedure (SOP). The data discussed in this narrative has been analyzed in accordance with ORG11 REV# 13.

Preparation/Analytical Method Verification

The SOP stated above has been prepared based on technical research and testing conducted by [redacted] Laboratories of Ohio, LLC and with guidance from the regulatory documents listed in this "Method/Analysis Information" section.

Calibration Information

The Volatiles: GC-MS analysis was performed on a HP 5973 Mass Spectrometer.

Initial Calibration
All the initial calibration requirements were met.

Continuing Calibration Verification Requirements
All the calibration verification standard (CCV) requirements were met.

00057

Quality Control (QC) Information

Method Blank Acceptance
Target analytes were not detected above the reporting limit in the blank.

Surrogate Recovery
Surrogate recoveries in all samples and quality control samples were within the established acceptance limits.

Laboratory Control Sample Recovery Statement (LCS)
The LCS had low recovery for Bromomethane and Trichlorofluoromethane. These compounds are often low on Methanol prep samples. 1200658897 (LCS).

QC Sample Designation
Spike analyses were performed on the following sample: 115782011 (002).

Spike Recovery Statement
The MS and MSD had low recoveries for Bromomethane, Trichlorofluoromethane, Acetone and 2-Butanone. 1200658898 (002) and 1200658899 (002).

Spike Duplicate Recovery Statement
The MS and MSD had low recoveries for Bromomethane, Trichlorofluoromethane, Acetone and 2-Butanone. 1200658898 (002) and 1200658899 (002).

Relative Percent Difference Statement (RPD)
The %RPD failed for 2-Butanone and Acetone. 1200658898 (002) and 1200658899 (002).

Internal Standard (ISTD) Acceptance
The internal standard responses, in all samples and quality control samples, met the required acceptance criteria.

Technical Information

[redacted] assigns holding times based on the date and time of sample collection. Those holding times expressed in hours are calculated in the AlphaLims system by hours. Those holding times expressed as days expire at midnight on the day of expiration.

Holding Time Specifications
All the samples were prepared and/or analyzed within the required holding time period.

Sample Preservation and Integrity
All samples met the sample preservation and integrity requirements.

Preparation/Analytical Method Verification

00058

All procedures were performed as stated in the SOP.

Sample Dilutions
This sample and QC samples were prepped by Methanol dilution and analyzed at a 50X dilution.

Sample Re-prep/Re-analysis
Reanalyses were not required for samples in this sample group/work order.

Miscellaneous Information

Nonconformance (NCR) Documentation
NCR 129035 was generated due to Failed Recovery for LCS/MS/PS, Failed RPD for LCS/LCSD, MS/MSD and or PS/PSD.

Manual Integrations
Manual integrations were performed as per SOP GEN 43, details are documented in the raw data.

TIC Comment
Tentatively identified compounds (TIC) were not required for this sample delivery group/work order.

Additional Comments
There were no additional comments.

Review Validation:
Initial VF Date 7/24/11

00059

[redacted] LABORATORIES OF OHIO, LLC

Certificate of Analysis

Company : Ohio Environmental Protection Agency
Address : P.O. Box 1049
Columbus, Ohio 43216

Report Date: July 26, 2004

Contact:
Project:

Page 1 of 2

Client Sample ID: 028
Sample ID: 115782001
Matrix: Misc Solid
Collect Date: 23-JUN-04 15:45
Receive Date: 25-JUN-04
Collector: Client

Project: OHEP00304
Client ID: OHEPA001

Client Desc.:

Parameter	Qualifier	Result	DL	RL	Units	DF	AnalystDate	Time	Batch	Method
Volatiles Analysis										
TCLP Volatiles in Solid										
1,1-Dichloroethylene	U	ND	0.0112	0.050	mg/L	50	MW1 07/16/04	0102	347359	1
1,2-Dichloroethane	U	ND	0.0221	0.050	mg/L	50				
1,4-Dichlorobenzene	U	ND	0.0103	0.050	mg/L	50				
2-Butanone	U	ND	0.117	0.500	mg/L	50				
Benzene	U	ND	0.0171	0.050	mg/L	50				
Carbon tetrachloride	U	ND	0.0075	0.050	mg/L	50				
Chlorobenzene	U	ND	0.0178	0.050	mg/L	50				
Chloroform	U	ND	0.0166	0.050	mg/L	50				
Tetrachloroethylene	J	0.0113	0.0102	0.050	mg/L	50				
Trichloroethylene	U	ND	0.0151	0.050	mg/L	50				
Vinyl chloride	U	ND	0.00925	0.050	mg/L	50				

The following Prep Methods were performed

Method	Description	Analyst	Date	Time	Prep Batch
SW846 1311	SW846 1311 TCLP Volatiles Prep Solids	MW1	07/06/04	1524	345667

The following Analytical Methods were performed

Method	Description	Analyst Comments
1	SW846 8260B	

Surrogate/Tracer recovery	Test	Recovery%	Acceptable Limits
1,2-Dichloroethane-d4	TCLP Volatiles in Solid	99	(78%-110%)
Bromofluorobenzene	TCLP Volatiles in Solid	106	(95%-108%)
Dibromofluoromethane	TCLP Volatiles in Solid	97	(79%-115%)
Toluene-d8	TCLP Volatiles in Solid	100	(94%-107%)

Notes:
The Qualifiers in this report are defined as follows :

* Indicates that a quality control analyte recovery is outside of specified acceptance criteria.
< Result is less than amount reported.
> Result is greater than amount reported.
B Target analyte was detected in the sample as well as the associated blank.
E Concentration of the target analyte exceeds the instrument calibration range.

00060--

[redacted] LABORATORIES OF OHIO, LLC

Certificate of Analysis

Company : Ohio Environmental Protection Agency
Address : P.O. Box 1049
Columbus, Ohio 43216

Report Date: July 26, 2004

Contact:
Project:

Page 2 of 2

Client Sample ID: 028
Sample ID: 115782001

Project: OHEP00304
Client ID: OHEPA001

Parameter	Qualifier	Result	DL	RL	Units	DF	AnalystDate	Time	Batch	Method

H Analytical holding time exceeded.
J The result was greater than the MDL, but less than the RL and is an estimated value. Values below a CRDL are also flagged.
U Indicates the target analyte was analyzed for but not detected above the MDL.
X Lab-specific qualifier-please see case narrative, data summary package or contact your Project Manager for details.
h Sample preparation or preservation holding time exceeded.

The above sample is reported on an "as received" basis.

Where the analytical method has been performed under NELAP certification, the analysis has met all of the requirements of the NELAC standard unless qualified on the Certificate of Analysis.

This data report has been prepared and reviewed in accordance with [redacted] Laboratories of Ohio, LLC standard operating procedures. Please direct any questions to your Project Manager, [redacted].

Reviewed by

00061

[redacted] LABORATORIES OF OHIO, LLC

Certificate of Analysis

Company : Ohio Environmental Protection Agency
Address : P.O. Box 1049
Columbus, Ohio 43216

Report Date: July 26, 2004

Contact:
Project:

Client Sample ID: RO-2
Sample ID: 115782002
Matrix: Misc Solid
Collect Date: 24-JUN-04 11:10
Receive Date: 25-JUN-04
Collector: Client

Project: OHEP00304
Client ID: OHEPA001

Client Desc.:

Parameter	Qualifier	Result	DL	RL	Units	DF	Analyst	Date	Time	Batch	Method
Volatiles Analysis											
TCLP Volatiles In Solid											
1,1-Dichloroethylene	U	ND	0.0223	0.100	mg/L	100	MW1	07/16/04	0141	347359	1
1,2-Dichloroethane	U	ND	0.0442	0.100	mg/L	100					
1,4-Dichlorobenzene	U	ND	0.0205	0.100	mg/L	100					
2-Butanone		1.25	0.235	1.00	mg/L	100					
Benzene	U	ND	0.0342	0.100	mg/L	100					
Carbon tetrachloride	U	ND	0.015	0.100	mg/L	100					
Chlorobenzene	U	ND	0.0355	0.100	mg/L	100					
Chloroform	U	ND	0.0331	0.100	mg/L	100					
Tetrachloroethylene	J	0.0361	0.0203	0.100	mg/L	100					
Trichloroethylene	U	ND	0.0301	0.100	mg/L	100					
Vinyl chloride	J	0.201	0.0185	0.100	mg/L	100					

The following Prep Methods were performed

Method	Description	Analyst	Date	Time	Prep Batch
SW846 1311	SW846 1311 TCLP Volatiles Prep Solids	MW1	07/06/04	1524	345667

The following Analytical Methods were performed

Method	Description	Analyst Comments
1	SW846 8260B	

Surrogate/Tracer recovery	Test	Recovery%	Acceptable Limits
1,2-Dichloroethane-d4	TCLP Volatiles in Solid	109	(78%-110%)
Bromofluorobenzene	TCLP Volatiles in Solid	118*	(95%-108%)
Dibromofluoromethane	TCLP Volatiles in Solid	115	(79%-115%)
Toluene-d8	TCLP Volatiles in Solid	116*	(94%-107%)

Notes:
The Qualifiers in this report are defined as follows :

* Indicates that a quality control analyte recovery is outside of specified acceptance criteria.
< Result is less than amount reported.
> Result is greater than amount reported.
B Target analyte was detected in the sample as well as the associated blank.
E Concentration of the target analyte exceeds the instrument calibration range.
H Analytical holding time exceeded.

00062

[redacted] LABORATORIES OF OHIO, LLC

Certificate of Analysis

Company : Ohio Environmental Protection Agency
Address : P.O. Box 1049
Columbus, Ohio 43216

Report Date: July 26, 2004

Contact:

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Project:

Client Sample ID: -RO-2
Sample ID: 115782002

Project: OHEP00304
Client ID: OHEPA001

Parameter	Qualifier	Result	DL	RL	Units	DF	AnalystDate	Time	Batch	Method

J The result was greater than the MDL, but less than the RL and is an estimated value. Values below a CRDL are also flagged.
U Indicates the target analyte was analyzed for but not detected above the MDL.
X Lab-specific qualifier-please see case narrative, data summary package or contact your Project Manager for details.
h Sample preparation or preservation holding time exceeded.

The above sample is reported on an "as received" basis.

Where the analytical method has been performed under NELAP certification, the analysis has met all of the requirements of the NELAC standard unless qualified on the Certificate of Analysis.

This data report has been prepared and reviewed in accordance with [redacted] Laboratories of Ohio, LLC standard operating procedures. Please direct any questions to your Project Manager, [redacted]

Reviewed by

00063

[redacted] LABORATORIES OF OHIO, LLC

Certificate of Analysis

Company : Ohio Environmental Protection Agency
Address : P.O. Box 1049
Columbus, Ohio 43216

Report Date: July 24, 2004

Contact:
Project:

Page 1 of 3

Client Sample ID: 002
Sample ID: 115782011
Matrix: Oil
Collect Date: 24-JUN-04 09:30
Receive Date: 25-JUN-04
Collector: Client

Project: OHEP00304
Client ID: OHEPA001

Client Desc.: Oil

Parameter	Qualifier	Result	DL	RL	Units	DF	AnalystDate	Time	Batch	Method
Volatiles Analysis										
GEL 8260B Method List Soil										
1,1,1,2-Tetrachloroethane	U	ND	11.1	97.5	ug/kg	50	MW1 07/08/04	2335	347358	1
1,1,1-Trichloroethane	U	ND	16.8	97.5	ug/kg	50				
1,1,2,2-Tetrachloroethane	U	ND	9.26	97.5	ug/kg	50				
1,1,2-Trichloroethane	U	ND	12.7	97.5	ug/kg	50				
1,1-Dichloroethane	U	ND	15.7	97.5	ug/kg	50				
1,1-Dichloroethylene	U	ND	14.2	97.5	ug/kg	50				
1,1-Dichloropropene	U	ND	15.7	97.5	ug/kg	50				
1,2,3-Trichlorobenzene	U	ND	21.5	97.5	ug/kg	50				
1,2,3-Trichloropropane	U	ND	29.1	97.5	ug/kg	50				
1,2,4-Trichlorobenzene	U	ND	19.0	97.5	ug/kg	50				
1,2,4-Trimethylbenzene	B	670	18.5	97.5	ug/kg	50				
1,2-Dibromo-3-chloropropane	U	ND	18.6	97.5	ug/kg	50				
1,2-Dibromoethane	U	ND	12.5	97.5	ug/kg	50				
1,2-Dichlorobenzene	U	ND	22.6	97.5	ug/kg	50				
1,2-Dichloroethane	U	ND	8.09	195	ug/kg	50				
1,2-Dichloropropane	U	ND	19.2	97.5	ug/kg	50				
1,3,5-Trimethylbenzene		159	11.1	97.5	ug/kg	50				
1,3-Dichlorobenzene	U	ND	14.2	97.5	ug/kg	50				
1,3-Dichloropropane	U	ND	15.6	97.5	ug/kg	50				
1,4-Dichlorobenzene	U	ND	20.6	97.5	ug/kg	50				
2,2-Dichloropropane	U	ND	14.9	97.5	ug/kg	50				
2-Butanone	B	1390	245	975	ug/kg	50				
2-Chlorotoluene	U	ND	13.0	97.5	ug/kg	50				
2-Hexanone	U	ND	88.7	975	ug/kg	50				
4-Chlorotoluene	U	ND	16.3	97.5	ug/kg	50				
4-Isopropyltoluene		246	15.2	97.5	ug/kg	50				
4-Methyl-2-pentanone	U	ND	16.9	975	ug/kg	50				
Acetone		5270	336	1950	ug/kg	50				
Benzene	U	ND	19.7	97.5	ug/kg	50				
Bromobenzene	U	ND	16.0	97.5	ug/kg	50				
Bromochloromethane	U	ND	22.7	97.5	ug/kg	50				
Bromodichloromethane	U	ND	13.0	97.5	ug/kg	50				
Bromoform	U	ND	11.1	195	ug/kg	50				
Bromomethane	UR	ND	21.2	195	ug/kg	50				
Carbon disulfide	U	ND	13.5	195	ug/kg	50				
Carbon tetrachloride	U	ND	10.8	97.5	ug/kg	50				
Chlorobenzene	U	ND	9.26	97.5	ug/kg	50				

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[redacted] LABORATORIES OF OHIO, LLC

Certificate of Analysis

Company : Ohio Environmental Protection Agency
Address : P.O. Box 1049
Columbus, Ohio 43216

Report Date: July 24, 2004

Contact:
Project:

Client Sample ID: 002
Sample ID: 115782011

Project: OHEP00304
Client ID: OHEPA001

Parameter	Qualifier	Result	DL	RL	Units	DF	AnalystDate	Time	Batch	Method
Volatiles Analysis										
GEL 8260B Method List Soil										
Chloroethane	U	ND	20.2	97.5	ug/kg	50				
Chloroform		3050	18.1	97.5	ug/kg	50				
Chloromethane	U	ND	17.3	97.5	ug/kg	50				
Dibromochloromethane	U	ND	11.8	97.5	ug/kg	50				
Dibromomethane	U	ND	15.5	97.5	ug/kg	50				
Dichlorodifluoromethane	U	ND	10.7	97.5	ug/kg	50				
Ethylbenzene	J	89.9	16.6	97.5	ug/kg	50				
Hexachlorobutadiene	U	ND	25.4	97.5	ug/kg	50				
Isopropylbenzene	J	24.2	16.8	97.5	ug/kg	50				
Methylene chloride	U	ND	21.0	97.5	ug/kg	50				
Naphthalene	B	121	28.8	97.5	ug/kg	50				
Styrene	BJ	20.5	12.9	195	ug/kg	50				
Tetrachloroethylene	U	ND	18.3	97.5	ug/kg	50				
Toluene	BJ	159	30.6	195	ug/kg	50				
Trichloroethylene	U	ND	16.2	97.5	ug/kg	50				
Trichlorofluoromethane	U	ND	9.06	97.5	ug/kg	50				
Vinyl chloride	U	ND	14.3	97.5	ug/kg	50				
Xylenes (total)		408	64.4	292	ug/kg	50				
cis-1,2-Dichloroethylene	U	ND	18.0	97.5	ug/kg	50				
cis-1,3-Dichloropropylene	U	ND	13.0	97.5	ug/kg	50				
m,p-Xylenes		290	45.5	195	ug/kg	50				
n-Butylbenzene		158	15.3	97.5	ug/kg	50				
n-Propylbenzene	J	77.2	15.3	97.5	ug/kg	50				
o-Xylene		118	18.9	97.5	ug/kg	50				
sec-Butylbenzene	J	52.8	13.8	97.5	ug/kg	50				
tert-Butyl methyl ether	U	ND	11.4	195	ug/kg	50				
tert-Butylbenzene	U	ND	12.8	97.5	ug/kg	50				
trans-1,2-Dichloroethylene	U	ND	20.5	97.5	ug/kg	50				
trans-1,3-Dichloropropylene	U	ND	13.0	97.5	ug/kg	50				

The following Prep Methods were performed

Method	Description	Analyst	Date	Time	Prep Batch
SW846 5035	5035/8260B Prep Solids	MW1	07/08/04	1505	347357

The following Analytical Methods were performed

Method	Description	Analyst Comments
1	SW846 8260B	

Surrogate/Tracer recovery	Test	Recovery%	Acceptable Limits
1,2-Dichloroethane-d4	[redacted]8260B Method List Soil	103	(74%-121%)

00065

[redacted] LABORATORIES OF OHIO, LLC

Certificate of Analysis

Company : Ohio Environmental Protection Agency
Address : P.O. Box 1049
Columbus, Ohio 43216

Report Date: July 24, 2004

Contact:
Project:

Page 3 of 3

Client Sample ID: 002
Sample ID: 115782011

Project: OHEP00304
Client ID: OHEPA001

Parameter	Qualifier	Result	DL	RL	Units	DF	AnalystDate	Time	Batch	Method
Bromofluorobenzene	[redacted]	8260B Method List Soil			108		(85%-128%)			
Dibromofluoromethane	[redacted]	8260B Method List Soil			99		(86%-114%)			
Toluene-d8	[redacted]	8260B Method List Soil			92		(80%-120%)			

Notes:
The Qualifiers in this report are defined as follows :

* Indicates that a quality control analyte recovery is outside of specified acceptance criteria.
< Result is less than amount reported.
> Result is greater than amount reported.
B Target analyte was detected in the sample as well as the associated blank.
E Concentration of the target analyte exceeds the instrument calibration range.
H Analytical holding time exceeded.
J The result was greater than the MDL, but less than the RL and is an estimated value. Values below a CRDL are also flagged.
P The concentration between the confirmation column and the primary column is >40 RPD for EPA methods 8081A, 8082 & 608.
U Indicates the target analyte was analyzed for but not detected above the MDL.
X Lab-specific qualifier-please see case narrative, data summary package or contact your Project Manager for details.
h Sample preparation or preservation holding time exceeded.

The above sample is reported on an "as received" basis.

Where the analytical method has been performed under NELAP certification, the analysis has met all of the requirements of the NELAC standard unless qualified on the Certificate of Analysis.

This data report has been prepared and reviewed in accordance with [redacted] Laboratories of Ohio, LLC standard operating procedures. Please direct any questions to your Project Manager, [redacted].

Reviewed by

00066

[redacted] LABORATORIES OF OHIO, LLC

QC Summary

Report Date: July 26, 2004
Page 1 of 3

Client: Ohio Environmental Protection Agency
P.O. Box 1049
Columbus, Ohio

Contact:

Workorder: 115782

Parmname	NOM		Sample	Qual	QC	Units	RPD%	REC%	Range	Anlst	Date Time
Volatiles: GC-MS											
Batch 347359											
QC1200658905 LCS											
1,1-Dichloroethylene	0.050				2.19	mg/L		88	(72%-126%)	MWI	07/15/04 19:51
1,2-Dichloroethane	0.050				2.25	mg/L		90	(67%-116%)		
1,4-Dichlorobenzene	0.050				2.08	mg/L		83	(52%-163%)		
2-Butanone	0.050				1.74	mg/L		70	(58%-125%)		
Benzene	0.050				2.37	mg/L		95	(78%-130%)		
Carbon tetrachloride	0.050				2.48	mg/L		99	(67%-120%)		
Chlorobenzene	0.050				2.32	mg/L		93	(79%-112%)		
Chloroform	0.050				2.35	mg/L		94	(69%-117%)		
Tetrachloroethylene	0.050				2.11	mg/L		84	(79%-119%)		
Trichloroethylene	0.050				2.29	mg/L		92	(77%-118%)		
Vinyl chloride	0.050				1.96	mg/L		79	(66%-125%)		
**1,2-Dichloroethane-d4	0.050				2.18	mg/L		87	(78%-110%)		
**Bromofluorobenzene	0.050				2.57	mg/L		103	(95%-108%)		
**Dibromofluoromethane	0.050				2.38	mg/L		95	(79%-115%)		
**Toluene-d8	0.050				2.55	mg/L		102	(94%-107%)		
QC1200658906 115782001 PS											
1,1-Dichloroethylene	50.0	U	ND		41.8	ug/L		84	(72%-126%)		07/15/04 20:29
1,2-Dichloroethane	50.0	U	ND		43.7	ug/L		87	(67%-116%)		
1,4-Dichlorobenzene	50.0	U	ND		42.0	ug/L		84	(52%-163%)		
2-Butanone	50.0	U	ND		32.1	ug/L		64	(58%-125%)		
Benzene	50.0	U	ND		46.7	ug/L		93	(78%-130%)		
Carbon tetrachloride	50.0	U	ND		48.1	ug/L		96	(67%-120%)		
Chlorobenzene	50.0	U	ND		45.8	ug/L		92	(79%-112%)		
Chloroform	50.0	U	ND		46.4	ug/L		93	(69%-117%)		
Tetrachloroethylene	50.0	J	0.226		41.9	ug/L		83	(79%-119%)		
Trichloroethylene	50.0	U	ND		44.6	ug/L		89	(77%-118%)		
Vinyl chloride	50.0	U	ND		38.2	ug/L		77	(66%-125%)		
**1,2-Dichloroethane-d4	50.0		49.3		42.7	ug/L		86	(78%-110%)		
**Bromofluorobenzene	50.0		53.2		49.9	ug/L		100	(95%-108%)		
**Dibromofluoromethane	50.0		48.6		48.0	ug/L		96	(79%-115%)		
**Toluene-d8	50.0		50.0		50.9	ug/L		102	(94%-107%)		
QC1200658907 115782001 PSD											
1,1-Dichloroethylene	50.0	U	ND		42.6	ug/L	2	85	(0%-30%)		07/15/04 21:09
1,2-Dichloroethane	50.0	U	ND		44.1	ug/L	1	88	(0%-30%)		
1,4-Dichlorobenzene	50.0	U	ND		42.7	ug/L	2	85	(0%-30%)		
2-Butanone	50.0	U	ND		32.9	ug/L	2	66	(0%-30%)		
Benzene	50.0	U	ND		48.2	ug/L	3	96	(0%-30%)		
Carbon tetrachloride	50.0	U	ND		50.9	ug/L	6	102	(0%-30%)		
Chlorobenzene	50.0	U	ND		47.0	ug/L	3	94	(0%-30%)		
Chloroform	50.0	U	ND		46.8	ug/L	1	94	(0%-30%)		
Tetrachloroethylene	50.0	J	0.226		43.0	ug/L	3	86	(0%-30%)		
Trichloroethylene	50.0	U	ND		46.5	ug/L	4	93	(0%-30%)		

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QC Summary

Workorder: 115782 **Page 2 of 3**

Parmname	NOM		Sample	Qual	QC	Units	RPD%	REC%	Range	Anlst	Date Time
Volatiles GC-MS											
Batch 347359											
Vinyl chloride	50.0	U	ND		38.9	ug/L	2	78	(0%-30%)		
*1,2-Dichloroethane-d4	50.0		49.3		41.9	ug/L		84	(78%-110%)	MW1	07/15/04 21:09
*Bromofluorobenzene	50.0		53.2		51.0	ug/L		102	(95%-108%)		
*Dibromofluoromethane	50.0		48.6		47.2	ug/L		94	(79%-115%)		
*Toluene-d8	50.0		50.0		50.7	ug/L		101	(94%-107%)		
QC1200654868 TB											
1,1-Dichloroethylene				U	ND	mg/L					07/15/04 23:03
1,2-Dichloroethane				U	ND	mg/L					
1,4-Dichlorobenzene				J	0.0209	mg/L					
2-Butanone				U	ND	mg/L					
Benzene				U	ND	mg/L					
Carbon tetrachloride				U	ND	mg/L					
Chlorobenzene				U	ND	mg/L					
Chloroform				U	ND	mg/L					
Tetrachloroethylene				J	0.013	mg/L					
Trichloroethylene				U	ND	mg/L					
Vinyl chloride				U	ND	mg/L					
**1,2-Dichloroethane-d4	0.050				2.48	mg/L		99	(78%-110%)		
**Bromofluorobenzene	0.050				2.62	mg/L		105	(95%-108%)		
**Dibromofluoromethane	0.050				2.43	mg/L		97	(79%-115%)		
**Toluene-d8	0.050				2.48	mg/L		99	(94%-107%)		

Notes:
The Qualifiers in this report are defined as follows:

- * Indicates that a quality control analyte recovery is outside of specified acceptance criteria.
- < Result is less than amount reported.
- > Result is greater than amount reported.
- B Target analyte was detected in the sample as well as the associated blank.
- E Concentration of the target analyte exceeds the instrument calibration range.
- H Analytical holding time exceeded.
- J The result was greater than the MDL, but less than the RL and is an estimated value. Values below a CRDL are also flagged.
- P The concentration between the confirmation column and the primary column is >40 RPD for EPA methods 8081A, 8082 & 608.
- U Indicates the target analyte was analyzed for but not detected above the MDL.
- X Lab-specific qualifier-please see case narrative, data summary package or contact your Project Manager for details.
- h Sample preparation or preservation holding time exceeded.

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QC Summary

Workorder: 115782

Parmname	NOM	Sample	Qual	QC	Units	RPD%	REC%	Range	Anlst	Date	Time

N/A indicates that spike recovery limits do not apply when sample concentration exceeds spike conc. by a factor of 4 or more.
^ The Relative Percent Difference (RPD) obtained from the sample duplicate (DUP) is evaluated against the acceptance criteria when the sample is greater than five times (5X) the contract required detection limit (RL). In cases where either the sample or duplicate value is less than 5X the RL, a control limit of +/- the RL is used to evaluate the DUP result.
For PS, PSD, and SDILT results, the values listed are the measured amounts, not final concentrations.

Where the analytical method has been performed under NELAP certification, the analysis has met all of the requirements of the NELAC standard unless qualified on the QC Summary.

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QC Summary

Report Date: July 24, 2004
Page 1 of 7

Client: Ohio Environmental Protection Agency
P.O. Box 1049
Columbus, Ohio

Contact:

Workorder: 115782

Parmname	NOM	Sample	Qual	QC	Units	RPD%	REC%	Range	Anist	Date	Time
Volatiles GC-MS											
Batch 347358											
QC1200658897 LCS											
1,1,1,2-Tetrachloroethane	5000			5980	ug/kg		120	(64%-122%)	MW1	07/08/04	17:38
1,1,1-Trichloroethane	5000			5420	ug/kg		108	(60%-131%)			
1,1,2,2-Tetrachloroethane	5000			4120	ug/kg		82	(36%-143%)			
1,1,2-Trichloroethane	5000			5090	ug/kg		102	(62%-123%)			
1,1-Dichloroethane	5000			5250	ug/kg		105	(59%-133%)			
1,1-Dichloroethylene	5000			4210	ug/kg		84	(45%-129%)			
1,1-Dichloropropene	5000			5600	ug/kg		112	(63%-136%)			
1,2,3-Trichlorobenzene	5000			5540	ug/kg		111	(37%-126%)			
1,2,3-Trichloropropane	5000			4810	ug/kg		96	(58%-131%)			
1,2,4-Trichlorobenzene	5000			5800	ug/kg		116	(52%-126%)			
1,2,4-Trimethylbenzene	5000		B	5720	ug/kg		114	(64%-118%)			
1,2-Dibromo-3-chloropropane	5000			4080	ug/kg		82	(62%-129%)			
1,2-Dibromoethane	5000			4980	ug/kg		100	(69%-124%)			
1,2-Dichlorobenzene	5000			5130	ug/kg		103	(71%-123%)			
1,2-Dichloroethane	5000			4920	ug/kg		98	(58%-134%)			
1,2-Dichloropropane	5000			5550	ug/kg		111	(57%-139%)			
1,3,5-Trimethylbenzene	5000			5700	ug/kg		114	(64%-122%)			
1,3-Dichlorobenzene	5000			5450	ug/kg		109	(68%-121%)			
1,3-Dichloropropane	5000			4930	ug/kg		99	(65%-129%)			
1,4-Dichlorobenzene	5000			5160	ug/kg		103	(69%-116%)			
2,2-Dichloropropane	5000			5590	ug/kg		112	(62%-135%)			
2-Butanone	5000		B	4200	ug/kg		84	(55%-149%)			
2-Chlorotoluene	5000			5280	ug/kg		106	(62%-128%)			
2-Hexanone	5000			4490	ug/kg		90	(57%-126%)			
4-Chlorotoluene	5000			5450	ug/kg		109	(67%-127%)			
4-Isopropyltoluene	5000			5750	ug/kg		115	(59%-118%)			
4-Methyl-2-pentanone	5000		B	4520	ug/kg		90	(58%-128%)			
Acetone	5000			5360	ug/kg		107	(44%-181%)			
Benzene	5000			5300	ug/kg		106	(56%-133%)			
Bromobenzene	5000			5490	ug/kg		110	(68%-123%)			
Bromochloromethane	5000			5450	ug/kg		109	(68%-141%)			
Bromodichloromethane	5000			5650	ug/kg		113	(53%-138%)			
Bromoform	5000			5430	ug/kg		109	(40%-146%)			
Bromomethane	5000			1550	ug/kg		31*	(41%-163%)			
Carbon disulfide	5000			4120	ug/kg		82	(45%-135%)			
Carbon tetrachloride	5000			5430	ug/kg		109	(58%-132%)			
Chlorobenzene	5000			5300	ug/kg		106	(59%-125%)			
Chloroethane	5000			985	ug/kg		20*	(51%-145%)			
Chloroform	5000			5410	ug/kg		108	(59%-133%)			
Chloromethane	5000			4520	ug/kg		90	(58%-134%)			
Dibromochloromethane	5000			5510	ug/kg		110	(62%-125%)			
Dibromomethane	5000			5060	ug/kg		101	(74%-133%)			

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QC Summary

Workorder: 115782

Parmname	NOM	Sample	Qual	QC	Units	RPD%	REC%	Range	Anlst	Date Time
Volatiles GC-MS										
Batch 347358										
Dichlorodifluoromethane	5000			4650	ug/kg		93	(35%-135%)		
Ethylbenzene	5000			5400	ug/kg		108	(56%-129%)	MW1	07/08/04 17:38
Hexachlorobutadiene	5000			5750	ug/kg		115	(56%-121%)		
Isopropylbenzene	5000			5770	ug/kg		115	(55%-134%)		
Methylene chloride	5000			5140	ug/kg		103	(58%-131%)		
Naphthalene	5000		B	4420	ug/kg		88	(45%-127%)		
Styrene	5000		B	5770	ug/kg		115	(60%-129%)		
Tetrachloroethylene	5000			5290	ug/kg		106	(65%-129%)		
Toluene	5000		B	4680	ug/kg		94	(56%-124%)		
Trichloroethylene	5000			5660	ug/kg		113	(43%-143%)		
Trichlorofluoromethane	5000			3760	ug/kg		75	(39%-147%)		
Vinyl chloride	5000			4890	ug/kg		98	(46%-135%)		
Xylenes (total)	15000			16300	ug/kg		109	(54%-129%)		
cis-1,2-Dichloroethylene	5000			5450	ug/kg		109	(46%-150%)		
cis-1,3-Dichloropropylene	5000			5930	ug/kg		119	(69%-133%)		
m,p-Xylenes	10000			10700	ug/kg		107	(54%-129%)		
n-Butylbenzene	5000			5880	ug/kg		118*	(56%-115%)		
n-Propylbenzene	5000			5600	ug/kg		112	(64%-126%)		
o-Xylene	5000			5530	ug/kg		111	(59%-128%)		
sec-Butylbenzene	5000			5780	ug/kg		116	(59%-124%)		
tert-Butyl methyl ether	5000			4850	ug/kg		97	(53%-147%)		
tert-Butylbenzene	5000			5730	ug/kg		115	(59%-125%)		
trans-1,2-Dichloroethylene	5000			5240	ug/kg		105	(39%-138%)		
trans-1,3-Dichloropropylene	5000			5650	ug/kg		113	(56%-130%)		
**1,2-Dichloroethane-d4	5000			4460	ug/kg		89	(74%-121%)		
**Bromofluorobenzene	5000			5080	ug/kg		102	(85%-128%)		
**Dibromofluoromethane	5000			4890	ug/kg		98	(86%-114%)		
**Toluene-d8	5000			4650	ug/kg		93	(80%-120%)		
QC1200658896 MB										
1,1,1,2-Tetrachloroethane			U	ND	ug/kg					07/08/04 21:25
1,1,1-Trichloroethane			U	ND	ug/kg					
1,1,2,2-Tetrachloroethane			U	ND	ug/kg					
1,1,2-Trichloroethane			U	ND	ug/kg					
1,1-Dichloroethane			U	ND	ug/kg					
1,1-Dichloroethylene			U	ND	ug/kg					
1,1-Dichloropropene			U	ND	ug/kg					
1,2,3-Trichlorobenzene			U	ND	ug/kg					
1,2,3-Trichloropropane			U	ND	ug/kg					
1,2,4-Trichlorobenzene			U	ND	ug/kg					
1,2,4-Trimethylbenzene			J	27.1	ug/kg					
1,2-Dibromo-3-chloropropane			U	ND	ug/kg					
1,2-Dibromoethane			U	ND	ug/kg					
1,2-Dichlorobenzene			U	ND	ug/kg					
1,2-Dichloroethane			U	ND	ug/kg					
1,2-Dichloropropane			U	ND	ug/kg					
1,3,5-Trimethylbenzene			U	ND	ug/kg					
1,3-Dichlorobenzene			U	ND	ug/kg					
1,3-Dichloropropane			U	ND	ug/kg					

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[redacted] LABORATORIES OF OHIO, LLC

QC Summary

Workorder: 115782

Parmname	NOM	Sample	Qual	QC	Units	RPD%	REC%	Range	Anlst	Date Time
Volatiles: GC-MS										
Batch 347358										
1,4-Dichlorobenzene			U	ND	ug/kg					
2,2-Dichloropropane			U	ND	ug/kg				MW1	07/08/04 21:25
2-Butanone			J	788	ug/kg					
2-Chlorotoluene			U	ND	ug/kg					
2-Hexanone			U	ND	ug/kg					
4-Chlorotoluene			U	ND	ug/kg					
4-Isopropyltoluene			U	ND	ug/kg					
4-Methyl-2-pentanone			J	27.3	ug/kg					
Acetone			U	ND	ug/kg					
Benzene			U	ND	ug/kg					
Bromobenzene			U	ND	ug/kg					
Bromochloromethane			U	ND	ug/kg					
Bromodichloromethane			U	ND	ug/kg					
Bromoform			U	ND	ug/kg					
Bromomethane			U	ND	ug/kg					
Carbon disulfide			U	ND	ug/kg					
Carbon tetrachloride			U	ND	ug/kg					
Chlorobenzene			U	ND	ug/kg					
Chloroethane			U	ND	ug/kg					
Chloroform			U	ND	ug/kg					
Chloromethane			U	ND	ug/kg					
Dibromochloromethane			U	ND	ug/kg					
Dibromomethane			U	ND	ug/kg					
Dichlorodifluoromethane			U	ND	ug/kg					
Ethylbenzene			U	ND	ug/kg					
Hexachlorobutadiene			U	ND	ug/kg					
Isopropylbenzene			U	ND	ug/kg					
Methylene chloride			U	ND	ug/kg					
Naphthalene			J	31.4	ug/kg					
Styrene			J	13.5	ug/kg					
Tetrachloroethylene			U	ND	ug/kg					
Toluene			J	46.0	ug/kg					
Trichloroethylene			U	ND	ug/kg					
Trichlorofluoromethane			U	ND	ug/kg					
Vinyl chloride			U	ND	ug/kg					
Xylenes (total)			U	ND	ug/kg					
cis-1,2-Dichloroethylene			U	ND	ug/kg					
cis-1,3-Dichloropropylene			U	ND	ug/kg					
m,p-Xylenes			U	ND	ug/kg					
n-Butylbenzene			U	ND	ug/kg					
n-Propylbenzene			U	ND	ug/kg					
o-Xylene			U	ND	ug/kg					
sec-Butylbenzene			U	ND	ug/kg					
tert-Butyl methyl ether			U	ND	ug/kg					
tert-Butylbenzene			U	ND	ug/kg					
trans-1,2-Dichloroethylene			U	ND	ug/kg					
trans-1,3-Dichloropropylene			U	ND	ug/kg					
**1,2-Dichloroethane-d4	50.0			5030	ug/kg		101	(74%-121%)		

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[redacted] LABORATORIES OF OHIO, LLC

QC Summary

Workorder: 115782

Parmname	NOM		Sample	Qual	QC	Units	RPD%	REC%	Range	Anlst	Date Time
Volatiles: GC-MS											
Batch 347358											
**Bromofluorobenzene	50.0				5330	ug/kg		107	(85%-128%)		
**Dibromofluoromethane	50.0				4930	ug/kg		99	(86%-114%)	MW1	07/08/04 21:25
**Toluene-d8	50.0				4550	ug/kg		91	(80%-120%)		
QC1200658898 115782011 PS											
1,1,1,2-Tetrachloroethane	50.0	U	ND		52.5	ug/L		105	(64%-122%)		07/08/04 19:15
1,1,1-Trichloroethane	50.0	U	ND		47.5	ug/L		95	(60%-131%)		
1,1,2,2-Tetrachloroethane	50.0	U	ND		38.9	ug/L		78	(36%-143%)		
1,1,2-Trichloroethane	50.0	U	ND		44.2	ug/L		88	(62%-123%)		
1,1-Dichloroethane	50.0	U	ND		47.3	ug/L		95	(59%-133%)		
1,1-Dichloroethylene	50.0	U	ND		38.0	ug/L		76	(45%-129%)		
1,1-Dichloropropene	50.0	U	ND		48.8	ug/L		98	(63%-136%)		
1,2,3-Trichlorobenzene	50.0	U	ND		41.2	ug/L		83	(57%-126%)		
1,2,3-Trichloropropane	50.0	U	ND		42.9	ug/L		86	(58%-131%)		
1,2,4-Trichlorobenzene	50.0	U	ND		44.7	ug/L		90	(52%-126%)		
1,2,4-Trimethylbenzene	50.0	B	6.88	B	58.0	ug/L		102	(64%-118%)		
1,2-Dibromo-3-chloropropane	50.0	U	ND		40.2	ug/L		80	(62%-129%)		
1,2-Dibromoethane	50.0	U	ND		43.6	ug/L		87	(69%-124%)		
1,2-Dichlorobenzene	50.0	U	ND		46.9	ug/L		94	(71%-123%)		
1,2-Dichloroethane	50.0	U	ND		43.6	ug/L		87	(58%-134%)		
1,2-Dichloropropane	50.0	U	ND		49.3	ug/L		99	(57%-139%)		
1,3,5-Trimethylbenzene	50.0		1.63		52.3	ug/L		101	(64%-122%)		
1,3-Dichlorobenzene	50.0	U	ND		49.3	ug/L		99	(68%-121%)		
1,3-Dichloropropane	50.0	U	ND		44.2	ug/L		89	(65%-129%)		
1,4-Dichlorobenzene	50.0	U	ND		46.6	ug/L		93	(69%-116%)		
2,2-Dichloropropane	50.0	U	ND		48.6	ug/L		97	(62%-135%)		
2-Butanone	50.0	B	14.3	B	38.1	ug/L		48*	(55%-149%)		
2-Chlorotoluene	50.0	U	ND		48.4	ug/L		97	(62%-128%)		
2-Hexanone	50.0	U	ND		38.1	ug/L		76	(57%-126%)		
4-Chlorotoluene	50.0	U	ND		50.0	ug/L		100	(67%-127%)		
4-Isopropyltoluene	50.0		2.53		52.1	ug/L		99	(59%-118%)		
4-Methyl-2-pentanone	50.0	U	ND	B	38.0	ug/L		76	(58%-128%)		
Acetone	50.0		54.1		65.8	ug/L		24*	(44%-181%)		
Benzene	50.0	U	ND		48.2	ug/L		97	(56%-133%)		
Bromobenzene	50.0	U	ND		49.6	ug/L		99	(68%-123%)		
Bromochloromethane	50.0	U	ND		48.4	ug/L		97	(68%-141%)		
Bromodichloromethane	50.0	U	ND		49.0	ug/L		98	(53%-138%)		
Bromoform	50.0	U	ND		47.3	ug/L		95	(40%-146%)		
Bromomethane	50.0	U	ND		14.7	ug/L		29*	(41%-163%)		
Carbon disulfide	50.0	U	ND		37.7	ug/L		75	(45%-135%)		
Carbon tetrachloride	50.0	U	ND		47.4	ug/L		95	(58%-132%)		
Chlorobenzene	50.0	U	ND		47.9	ug/L		96	(59%-125%)		
Chloroethane	50.0	U	ND		9.31	ug/L		19*	(51%-145%)		
Chloroform	50.0		31.3		73.8	ug/L		85	(59%-133%)		
Chloromethane	50.0	U	ND		40.5	ug/L		81	(58%-134%)		
Dibromochloromethane	50.0	U	ND		47.3	ug/L		95	(62%-125%)		
Dibromomethane	50.0	U	ND		44.2	ug/L		88	(74%-133%)		
Dichlorodifluoromethane	50.0	U	ND		39.9	ug/L		80	(35%-135%)		
Ethylbenzene	50.0	J	0.922		49.9	ug/L		98	(56%-129%)		

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LABORATORIES OF OHIO, LLC

QC Summary

Workorder: 115782 **Page 5 of 7**

Parmname	NOM		Sample	Qual	QC	Units	RPD%	REC%	Range	Anlst	Date Time
Volatiles: GC-MS											
Batch 347358											
Hexachlorobutadiene	50.0	U	ND		32.9	ug/L		66	(56%-121%)		
Isopropylbenzene	50.0	J	0.249		51.8	ug/L		103	(55%-134%)	MWI	07/08/04 19:15
Methylene chloride	50.0	U	ND		47.4	ug/L		95	(58%-131%)		
Naphthalene	50.0	B	1.24	B	38.9	ug/L		75	(45%-127%)		
Styrene	50.0	BJ	0.210	B	52.5	ug/L		105	(60%-129%)		
Tetrachloroethylene	50.0	U	ND		48.6	ug/L		97	(65%-129%)		
Toluene	50.0	BJ	1.63	B	45.1	ug/L		87	(56%-124%)		
Trichloroethylene	50.0	U	ND		49.1	ug/L		98	(43%-143%)		
Trichlorofluoromethane	50.0	U	ND		31.6	ug/L		63	(39%-147%)		
Vinyl chloride	50.0	U	ND		43.1	ug/L		86	(46%-135%)		
Xylenes (total)	150		4.19		152	ug/L		98	(54%-129%)		
cis-1,2-Dichloroethylene	50.0	U	ND		48.8	ug/L		98	(46%-150%)		
cis-1,3-Dichloropropylene	50.0	U	ND		51.2	ug/L		102	(69%-133%)		
m,p-Xylenes	100		2.98		101	ug/L		98	(54%-129%)		
n-Butylbenzene	50.0		1.62		51.1	ug/L		99	(56%-115%)		
n-Propylbenzene	50.0	J	0.792		50.9	ug/L		100	(64%-126%)		
o-Xylene	50.0		1.21		51.3	ug/L		100	(59%-128%)		
sec-Butylbenzene	50.0	J	0.542		50.4	ug/L		100	(59%-124%)		
tert-Butyl methyl ether	50.0	U	ND		42.3	ug/L		85	(53%-147%)		
tert-Butylbenzene	50.0	U	ND		50.6	ug/L		101	(59%-125%)		
trans-1,2-Dichloroethylene	50.0	U	ND		47.2	ug/L		94	(39%-138%)		
trans-1,3-Dichloropropylene	50.0	U	ND		50.1	ug/L		100	(56%-130%)		
**1,2-Dichloroethane-d4	50.0		51.6		42.9	ug/L		86	(74%-121%)		
**Bromofluorobenzene	50.0		54.0		51.3	ug/L		103	(85%-128%)		
**Dibromofluoromethane	50.0		49.7		47.5	ug/L		95	(86%-114%)		
**Toluene-d8	50.0		46.2		49.1	ug/L		98	(80%-120%)		
QC1200658899 115782011 PSD											
1,1,1,2-Tetrachloroethane	50.0	U	ND		55.2	ug/L	5	110	(0%-30%)		07/08/04 19:48
1,1,1-Trichloroethane	50.0	U	ND		50.1	ug/L	5	100	(0%-30%)		
1,1,2,2-Tetrachloroethane	50.0	U	ND		46.6	ug/L	18	93	(0%-30%)		
1,1,2-Trichloroethane	50.0	U	ND		49.6	ug/L	12	99	(0%-30%)		
1,1-Dichloroethane	50.0	U	ND		49.9	ug/L	5	100	(0%-30%)		
1,1-Dichloroethylene	50.0	U	ND		38.7	ug/L	2	78	(0%-30%)		
1,1-Dichloropropene	50.0	U	ND		51.9	ug/L	6	104	(0%-30%)		
1,2,3-Trichlorobenzene	50.0	U	ND		39.5	ug/L	4	79	(0%-30%)		
1,2,3-Trichloropropane	50.0	U	ND		52.7	ug/L	21	105	(0%-30%)		
1,2,4-Trichlorobenzene	50.0	U	ND		42.5	ug/L	5	85	(0%-30%)		
1,2,4-Trimethylbenzene	50.0	B	6.88	B	60.2	ug/L	4	107	(0%-30%)		
1,2-Dibromo-3-chloropropane	50.0	U	ND		47.8	ug/L	17	96	(0%-30%)		
1,2-Dibromoethane	50.0	U	ND		49.7	ug/L	13	99	(0%-30%)		
1,2-Dichlorobenzene	50.0	U	ND		49.1	ug/L	5	98	(0%-30%)		
1,2-Dichloroethane	50.0	U	ND		47.2	ug/L	8	94	(0%-30%)		
1,2-Dichloropropane	50.0	U	ND		52.2	ug/L	6	104	(0%-30%)		
1,3,5-Trimethylbenzene	50.0		1.63		53.6	ug/L	3	104	(0%-30%)		
1,3-Dichlorobenzene	50.0	U	ND		51.0	ug/L	3	102	(0%-30%)		
1,3-Dichloropropane	50.0	U	ND		48.7	ug/L	10	97	(0%-30%)		
1,4-Dichlorobenzene	50.0	U	ND		48.1	ug/L	3	96	(0%-30%)		
2,2-Dichloropropane	50.0	U	ND		51.4	ug/L	6	103	(0%-30%)		

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QC Summary

Workorder: 115782 Page 6 of 7

Parmname	NOM		Sample	Qual	QC	Units	RPD%	REC%	Range	Anlst	Date Time
Volatiles: GC-MS											
Batch 347358											
2-Butanone	50.0	B	14.3	B	50.1	ug/L	40*	72	(0%-30%)		
2-Chlorotoluene	50.0	U	ND		51.0	ug/L	5	102	(0%-30%)	MWI	07/08/04 19:48
2-Hexanone	50.0	U	ND		48.2	ug/L	24	97	(0%-30%)		
4-Chlorotoluene	50.0	U	ND		52.0	ug/L	4	104	(0%-30%)		
4-Isopropyltoluene	50.0		2.53		50.5	ug/L	3	96	(0%-30%)		
4-Methyl-2-pentanone	50.0	U	ND	B	49.0	ug/L	25	98	(0%-30%)		
Acetone	50.0		54.1		89.1	ug/L	99*	70	(0%-30%)		
Benzene	50.0	U	ND		50.9	ug/L	3	102	(0%-30%)		
Bromobenzene	50.0	U	ND		54.4	ug/L	9	109	(0%-30%)		
Bromochloromethane	50.0	U	ND		53.1	ug/L	9	106	(0%-30%)		
Bromodichloromethane	50.0	U	ND		51.9	ug/L	6	104	(0%-30%)		
Bromoform	50.0	U	ND		56.3	ug/L	17	113	(0%-30%)		
Bromomethane	50.0	U	ND		15.0	ug/L	2	30*	(0%-30%)		
Carbon disulfide	50.0	U	ND		38.1	ug/L	1	76	(0%-30%)		
Carbon tetrachloride	50.0	U	ND		50.3	ug/L	6	101	(0%-30%)		
Chlorobenzene	50.0	U	ND		50.3	ug/L	5	101	(0%-30%)		
Chloroethane	50.0	U	ND		9.18	ug/L	1	18*	(0%-30%)		
Chloroform	50.0		31.3		87.5	ug/L	28	112	(0%-30%)		
Chloromethane	50.0	U	ND		44.1	ug/L	8	88	(0%-30%)		
Dibromochloromethane	50.0	U	ND		51.8	ug/L	9	104	(0%-30%)		
Dibromomethane	50.0	U	ND		48.7	ug/L	10	97	(0%-30%)		
Dichlorodifluoromethane	50.0	U	ND		43.9	ug/L	10	88	(0%-30%)		
Ethylbenzene	50.0	J	0.922		52.1	ug/L	4	102	(0%-30%)		
Hexachlorobutadiene	50.0	U	ND		26.0	ug/L	23	52*	(0%-30%)		
Isopropylbenzene	50.0	J	0.249		54.8	ug/L	6	109	(0%-30%)		
Methylene chloride	50.0	U	ND		50.0	ug/L	5	100	(0%-30%)		
Naphthalene	50.0	B	1.24	B	41.0	ug/L	6	80	(0%-30%)		
Styrene	50.0	BJ	0.210	B	54.6	ug/L	4	109	(0%-30%)		
Tetrachloroethylene	50.0	U	ND		50.6	ug/L	4	101	(0%-30%)		
Toluene	50.0	BJ	1.63	B	47.2	ug/L	5	91	(0%-30%)		
Trichloroethylene	50.0	U	ND		52.1	ug/L	6	104	(0%-30%)		
Trichlorofluoromethane	50.0	U	ND		32.2	ug/L	2	64	(0%-30%)		
Vinyl chloride	50.0	U	ND		47.2	ug/L	9	94	(0%-30%)		
Xylenes (total)	150		4.19		159	ug/L	4	103	(0%-30%)		
cis-1,2-Dichloroethylene	50.0	U	ND		51.7	ug/L	6	103	(0%-30%)		
cis-1,3-Dichloropropylene	50.0	U	ND		54.7	ug/L	6	109	(0%-30%)		
m,p-Xylenes	100		2.98		105	ug/L	5	102	(0%-30%)		
n-Butylbenzene	50.0		1.62		47.4	ug/L	8	92	(0%-30%)		
n-Propylbenzene	50.0	J	0.792		52.7	ug/L	4	104	(0%-30%)		
o-Xylene	50.0		1.21		53.5	ug/L	4	105	(0%-30%)		
sec-Butylbenzene	50.0	J	0.542		49.2	ug/L	2	97	(0%-30%)		
tert-Butyl methyl ether	50.0	U	ND		47.1	ug/L	11	94	(0%-30%)		
tert-Butylbenzene	50.0	U	ND		51.0	ug/L	1	102	(0%-30%)		
trans-1,2-Dichloroethylene	50.0	U	ND		50.2	ug/L	6	100	(0%-30%)		
trans-1,3-Dichloropropylene	50.0	U	ND		54.5	ug/L	8	109	(0%-30%)		
**1,2-Dichloroethane-d4	50.0		51.6		45.8	ug/L		92	(74%-121%)		
**Bromofluorobenzene	50.0		54.0		53.5	ug/L		107	(85%-128%)		
**Dibromofluoromethane	50.0		49.7		48.8	ug/L		98	(86%-114%)		

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QC Summary

Workorder: 115782 **Page 7 of 7**

Parmname	NOM	Sample	Qual	QC	Units	RPD%	REC%	Range	Anlst	Date	Time
Volatiles: GC-MS											
Batch 347358											
**Toluene-d8	50.0	46.2		48.3	ug/L		97	(80%-120%)			

Notes:
The Qualifiers in this report are defined as follows:

- * *Indicates that a quality control analyte recovery is outside of specified acceptance criteria.*
- < Result is less than amount reported.
- > Result is greater than amount reported.
- B Target analyte was detected in the sample as well as the associated blank.
- E Concentration of the target analyte exceeds the instrument calibration range.
- H Analytical holding time exceeded.
- J The result was greater than the MDL, but less than the RL and is an estimated value. Values below a CRDL are also flagged.
- P The concentration between the confirmation column and the primary column is >40 RPD for EPA methods 8081A, 8082 & 608.
- U Indicates the target analyte was analyzed for but not detected above the MDL.
- X Lab-specific qualifier-please see case narrative, data summary package or contact your Project Manager for details.
- h Sample preparation or preservation holding time exceeded.

N/A indicates that spike recovery limits do not apply when sample concentration exceeds spike conc. by a factor of 4 or more.
^ The Relative Percent Difference (RPD) obtained from the sample duplicate (DUP) is evaluated against the acceptance criteria when the sample is greater than five times (5X) the contract required detection limit (RL). In cases where either the sample or duplicate value is less than 5X the RL, a control limit of +/- the RL is used to evaluate the DUP result.
For PS, PSD, and SDILT results, the values listed are the measured amounts, not final concentrations.

Where the analytical method has been performed under NELAP certification, the analysis has met all of the requirements of the NELAC standard unless qualified on the QC Summary.

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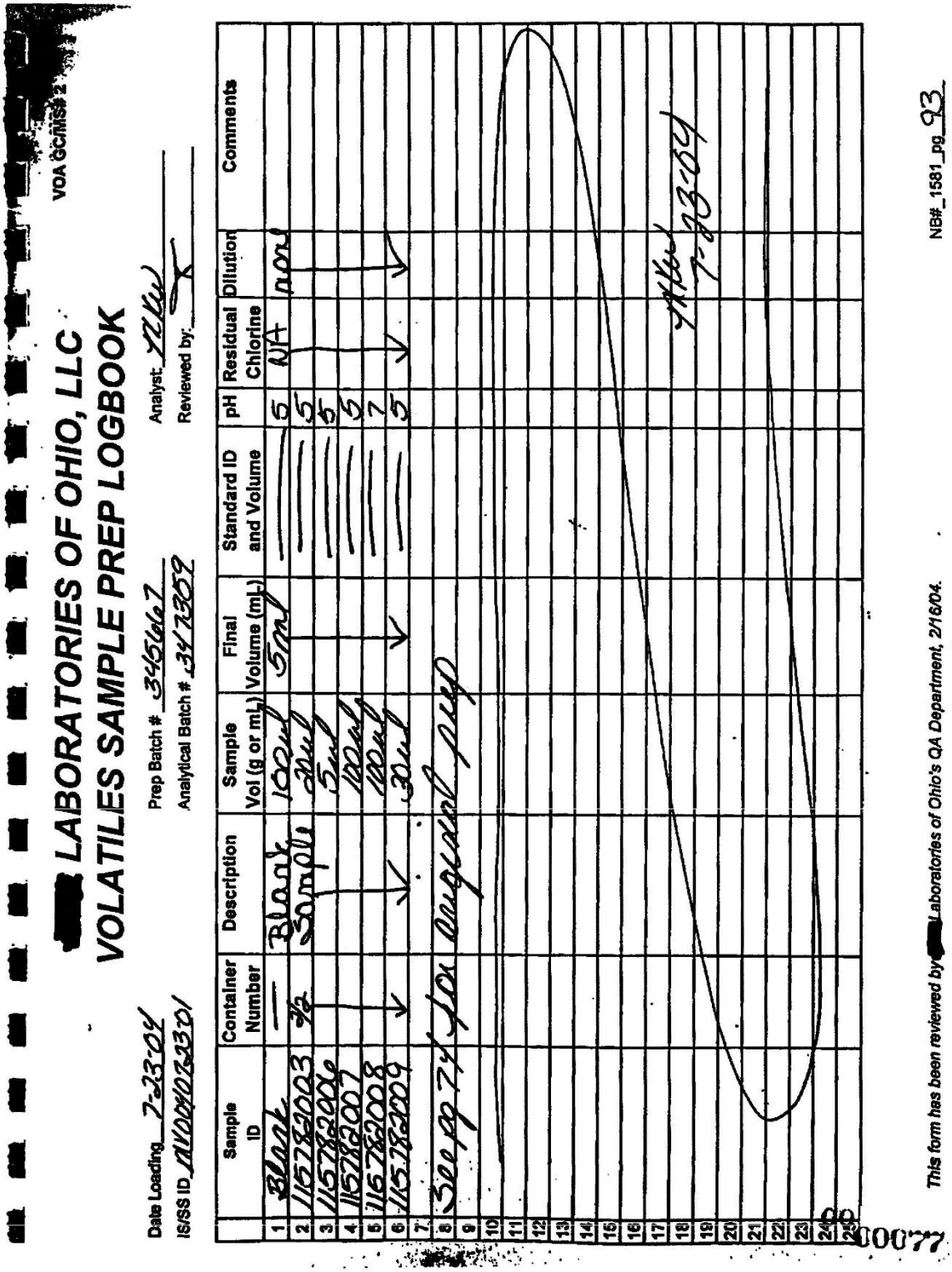

[redacted] LABORATORIES OF OHIO, LLC

VOLATILES SAMPLE PREP LOGBOOK

VOA GC/MS 2

Date Loading 7-23-04

IS/SS ID AVOA0723301

Prep Batch # 345067

Analytical Batch # 34723B

Analyst: [redacted]

Reviewed by: [redacted]

	Sample ID	Container Number	Description	Sample Vol (g or mL)	Final Volume (mL)	Standard ID and Volume	pH	Residual Chlorine	Dilution	Comments
1	Blank	—	Blank	100ml	5ml	———	5	NA	none	
2	115782003	72	50 mOU	20ml		———	5			
3	115782004			5ml		———	5			
4	115782007			100ml		———	5			
5	115782008			100ml		———	7			
6	115782009	↓	↓	30ml	↓	———	5	↓	↓	
7										
8	Seep 72 hr duplicate prep									
9										
10										
11										
12										
13										
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This form has been reviewed by [redacted] Laboratories of Ohio's QA Department, 2/16/04.

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VOA GC/MS# 2

VOLATILES SAMPLE PREP LOGBOOK

Date Loading 7-15-04 Prep Batch # 3456607 Analyst: [illegible]

IS/SS ID IVV040712-02 Analytical Batch # ~~345~~ 347359 Reviewed by: VV

NLW 7-15-04

	Sample ID	Container Number	Description	Sample Vol (g or mL)	Final Volume (mL)	Standard ID and Volume	pH	Residual Chlorine	Dilution	Comments
1	1200054808	—	Blank	100ul	5ml	[illegible]	5	NA	none	
2	1200058905	—	LCS			IVV040712-03/100ul				
3	115782001	3x1/2	Sample				6			
4	1200058906	NLW 7-16-04	MS			IVV040712-03/100ul				
5	1200058907		MSD							
6	115782002		Sample	50ul			6			
7	115782003			50ul			5			
8	115782004			100ul			5			
9	115782005			100 25ul			5			
10	115782006			NLW 7-16-04 25ul			5			
11	115782007			100ul			5			
12	115782008			100ul			7			
13	115782009			25ul			5			
14										
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19				NLW 7-16-04						
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24										
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This form has been reviewed by [redacted] Laboratories of Ohio's QA Department, 2/16/04.

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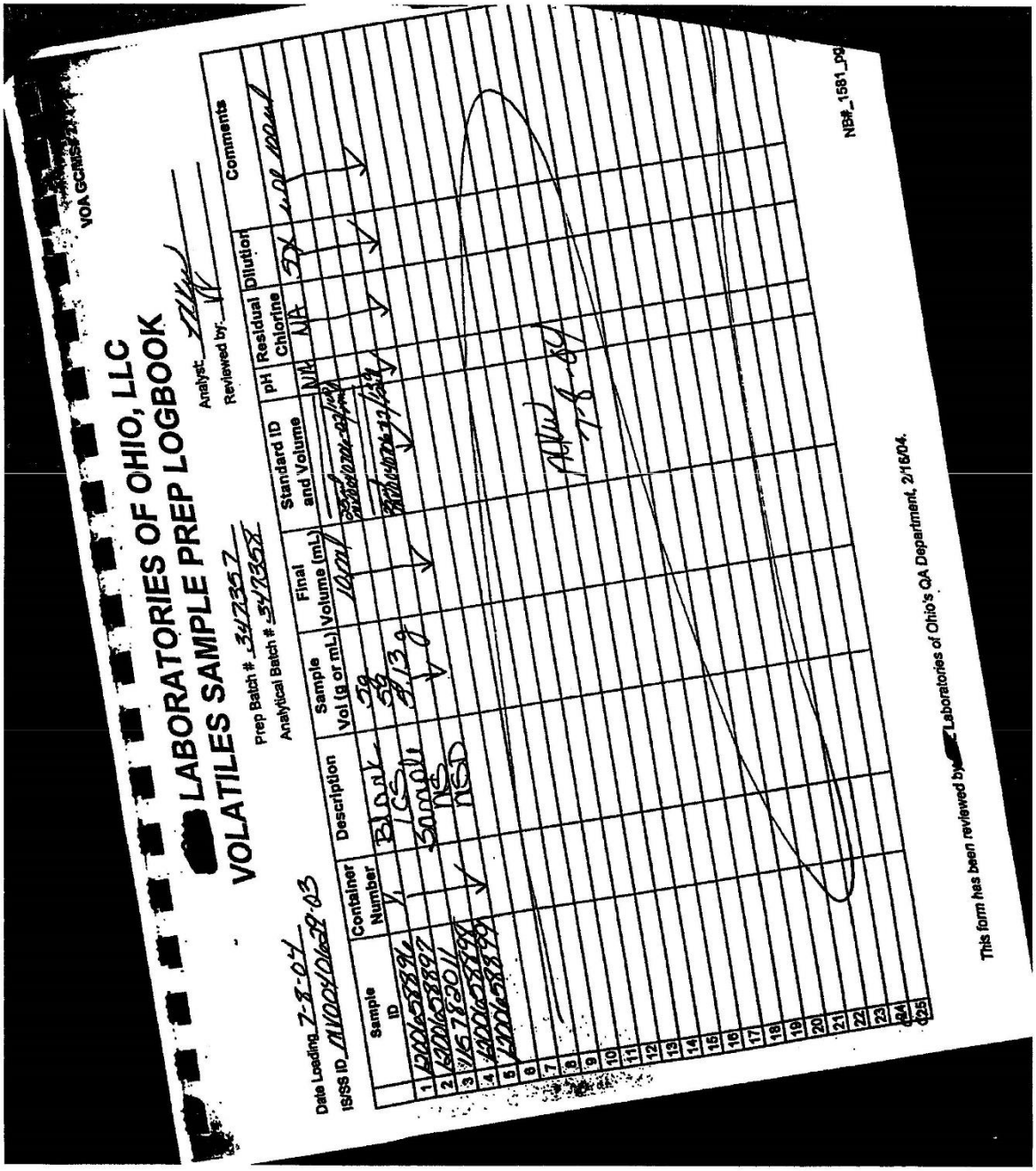

LABORATORIES OF OHIO, LLC
VOLATILES SAMPLE PREP LOGBOOK

VOA GCMS 2

Prep Batch # 342357
Analytical Batch # 342357

Analyst: [signature]
Reviewed by: [signature]

Date Loading 7-8-04
IS/SS ID MIXOX/DCB-03

	Sample ID	Container Number	Description	Sample Vol (g or mL)	Final Volume (mL)	Standard ID and Volume	pH	Residual Chlorine	Dilution	Comments
1	[illegible]	1	Blank	5g	10ml	[illegible]	NA	NA	5X	100ml
2	[illegible]		LCS	5g						
3	[illegible]		Sample	5g						
4	[illegible]		MS	5g						
5	[illegible]		MSD	5g						
6										
7										
8										
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This form has been reviewed by [illegible] Laboratories of Ohio's QA Department, 2/16/04.

NB#_1581_p0

GC079

Data Validation Plan Review Form

Tier I

This Plan Review Form is #	1	of	1	forms completed in the review of this closure plan.

Facility Name	Blue Knell Industries	Validator/DO	Anyone
ID Number	OHDXXX123	Date of Plan	Dec. 2, 2004
Date Review of Plan Completed	Dec. 2, 2004	Plan is: New, Amended, Revised	NEW
Document Title:	Blue Knell Industries, compliance sampling		
Lab Name: GEL Laboratories	Media Type(s): Waste Water (WW): Solid Waste (SS): Oil (O):	Analyses Requested: TCLP VOC, TCLP Metals; TCLP VOC, TCLP Metals; pH, Flashpoint, PCBs, Total VOCs, Total RCRA Metals	Notes:

Note: The criteria used in the Tier I Data Validation checklist are derived primarily from SW-846 method requirements and
U.S. EPA's National Functional Guidelines (NFGs) for Organic and Inorganic Data Review. Criteria from methods are considered preferable as they are specific to that procedure. Where the method is silent, criteria from the NFGs, or other sources when necessary, are adopted. For flashpoint (which uses ASTM methods dictated by the OAC rules), ASTM methodcriteria are used.

The Tier I data validation manual is the primary reference for this checklist. It explains and gives examples for the questions inthis checklist. The Tier II methodology and terminology builds on that established in the Tier I checklist and its associated data validation manual. There is no Tier II manual, only the checklist and completed example checklists. Additional information is also available by referring to the specific methods.

Data Qualifiers and their meanings used throughout the Tier I Checklist	
J	Estimated
J+	Estimated High (results are likely reported higher than the true value)
J-	Estimated Low (results are likely reported lower than the true value)
R	Rejected
UJ	Undetected Estimated
NJ	Tentatively Identified, Quantitation Estimated

Checklist #1

Report Completeness and Technical Holding Times

1.0 Report Completeness and Technical Holding Times	
1.1 Sample Package Completeness and Deliverables	
Completeness **This section provides a checklist of important components of data reports. If a report is incomplete, it may be necessary to halt data validation procedures until all the missing information is provided. Please, refer to the Tier I Data Validation Manual for additional assistance in completing the checklist.**	
1.1.1 Are Chains of Custody (COC) forms present for all samples? *Action: If not, contact the applicable party for a replacement of missing or illegible copies.*	**Indicate yes or no:** Yes. The COC is found on page 4 and lists sample analysis for TCLP VOCs, TCLP metals, pH, flashpoint, PCBs, Total VOCs, and Total RCRA metals. **If no, explain action taken:** N/A
1.1.2 Is a signed statement from the laboratory present that attests to the validity of the data? *Action: Take no further action, contact the applicable party, and have the lab submit a valid data report. If no response is received, qualify all data as unusable and STOP DATA VALIDATION until resolved.*	**Indicate yes or no:** Yes. A signed completeness statement is found on page 2. **If no, explain action taken:** N/A
1.1.3 Is a case narrative present that summarizes QA/QC discrepancies and/or other problems? *Action: No action is necessary, but this information is useful to focus data validation efforts.*	**Indicate yes or no:** Yes. The narrative is presented as part of the analytical resultsfor each of the requested methods.
1.1.4 Are all the requested analyses accounted for in the data report? Describe any omissions between the COC and the submitted sampling data. *Action: If there are discrepancies, contact the laboratory for any missing deliverables and/or an explanation.*	**Indicate yes or no:** No. **Describe any omissions and actions taken:** PCB analysis is listed on the COCs, but data is not present in the lab report. (Pgs. 44-53 are missing). The lab or the facility should be contacted and a new data report containing the required information should be submitted to the Agency.

1.1.5 Is a sample receipt form present? If so, does it contain information on the condition of sample containers, proper preservatives used (cross-check with the COC), canister pressure, and temperature of the cooler? **Note: Waste samples may not require cooling prior to receipt by the laboratory. Best professional judgment should be used to determine if qualification of the data is warranted.** *Action: If the sample receipt form does not contain the necessary information, contact the laboratory or applicable party. Describe any comments or abnormal conditions. Actions may be taken for the following special conditions:* *A. For samples analyzed for volatiles that were not properly cooled (temperature >6° C), all detected results should be qualified as "J-" and all non-detects qualified as "UJ."* *B. For all liquid Volatile Organic Compound (VOC) samples or vials with air bubbles, detected results should be qualified as "J-" and non-detects as "UJ" or "R" depending on professional judgment (considering other quality control information such as sample cooler temperature and other site-specific data quality objectives (DQOs).* *C. If aqueous samples for VOCs were not preserved, check that technical holding times were met (see Table 1-2). If they were not met, qualify all associated sample results.* *D. If canister samples are not within ±3.5 kPA (0.5 psi) of the measured pressure recorded upon retrieval, qualify any sub-slab or soil gas results as "R" if the pressure change exceeds approximately 5% of the total pressure, unless the pressure difference can be decisively attributed to a temperature difference. For indoor air samples, qualify any detects as "J" and non-detects as "UJ" unless the pressure difference can be decisively attributed to a temperature difference.*	**Indicate yes or no:** Yes – The receipt form is present with information about the condition of the samples. **Summarize problems identified:** The sample receipt form does not indicate whether preserved samples were checked for pH, so it is unknown whether mercury was property preserved in nitric acid. Otherwise, the sample receipt form indicates that all samples arrived in good condition. (page 5) **Describe actions taken:** Detects of mercury should be qualified with "J-" and non-detects with "UJ".

E. If liquid TCLP samples were preserved, qualify all associated results as rejected and flag the data with an "R."	
1.1.6 Do the COC forms, sample receipt form, or the case narrative indicate any problems with sample receipt, condition of samples, analytical problems or special circumstances affecting the quality of the data? *Action: Use the information to focus data validation efforts.*	**Indicate yes or no:** No. No problems are directly stated, but there is no indication of preservation within the COC or sample receipt form, except for cooling the samples. This is not acceptable because aqueous mercury samples need to be preserved in nitric acid with a pH of <2. **If yes, list any problems that were found:**
1.1.7 Are custody seals present and intact?	**Indicate yes, no, N/A**: Yes.

Batch-Specific Data Validation Worksheet										
Sample ID	**Lab ID**	**Matrix**	**Sample Date**	**Date Received by the Lab**	**Parameter**	**Extraction Date**	**Preparation Date**	**Analysis Date**	**QA/QC Data Present (Yes or No)**	**Batch ID#**
028	115782001	Waste Water	06-23-04	06-25-04	TCLP Metals	07-01-04 #346201	07-02-04 #346201	07-06-04	YES	346202
					TCLP Hg	07-01-04 #345991	07-06-04 #346228	07-06-04	Yes	346229
					TCLP VOCs	07-06-04 #345667	NA	07-16-04	Yes	347359
RO-02	115782002	Solid	06-24-04	06-25-04	TCLP Metals	07-01-04 #345991	07-02-04 #346201	07-06-04	Yes	346202
					TCLP Hg	07-01-04 #345991	07-06-04 #346228	07-06-04	Yes	346229
					TCLP VOCs	07-06-04 (missing)	NA	07-08-04	Yes	347359
002	115782011	Oil	06-24-04	06-25-04	pH	NA	NA	06-30-04	Yes	345466
					Flashpoint	NA	NA	07-21-04	Yes	350989
					PCBs	Not found	Not found	Not found	Not found	Not found
					Total VOCs		07-07-04 #347357	07-08-04	Yes	347358
					Total Metals	NA	07-07-04 #346534	07-06-04	Yes	346537
					Total Hg	NA	06-30-04 #344887	07-06-04	Yes	344889

Note: Use this worksheet to evaluate report completeness and verify all appropriate batch-specific QA/QC requirements are present. To fill out this worksheet, list one sample ID# then list all sample parameters on one line each with their associated analysis dates, batch ID#s, etc. (*e.g.*, put mercury on a separate line from the other metals since it will have its own prep. dates, analysis dates, and batch ID#s.) For the QA/QC Data Present column, indicate whether the appropriate batch-specific QA/QC is present for each batch of samples. Batch specific QA/QC requirements for Tier I data validation for organic data consists of blank data, Matrix Spike/Matrix Spike Duplicate data, Laboratory Control Sample (LCS) data, and surrogate data. For inorganic data, the QA/QC data includes a Matrix Spike/Matrix Spike Duplicate, LCS data and blank data. Additional QA/QC data may include ICP serial dilution results and post-digestion spike data.

1.2 Technical Holding Times

Technical holding time is the time, in days, from sample acquisition in the field to either laboratory preparation or analysis. Technical holding times are established from information contained in the laboratory report, COC, and raw analytical bench sheets (if available). Technical holding times also depend upon whether samples were preserved. The recommended technical holding times for volatile compounds, semi-volatile compounds, metals, Hexavalent Chromium, Mercury, Ammonia, Cyanide, pH and TCLP analyses are listed in Table 1-2 below.

Table 1-2: Technical Holding Times[1] for Volatile, Semi-Volatile, Metals, Ammonia, Cyanide and pH Samples

Analytes (Method) (Media phase)	Preserved?	From field collection to extraction	From extraction to preparation	From extraction to analysis	Max holding times	Common preservative
VOCs (8260) (aqueous)	Yes	NA	NA	14 days	14 days	Cool to 0-6˚C, HCl
VOCs (8260) (aqueous)	No	NA	NA	7 days	7 days	Cool to 0-6˚C
VOCs (8260) Acrolein and Acrylonitrile - only (aqueous)	Yes	NA	NA	7 days	7 days	Cool to 0-6˚C, pH 4-5
VOCs (8260) (liquid/waste)	No	NA	NA	14 days	14 days	Cool to 0-6˚C
VOCs (8260) (soil/waste)	No	NA	NA	NA	14 days	Cool to 0-6˚C or no preservative
VOCs (5035/8260) (soil/waste)	Yes	2 days	NA	12 days	14 days	Encore Sampler or equivalent, Cool to approximately 4°C
VOCs (TO-15) (air)	NA	NA	NA	NA	30 days	NA
SVOCs (8270)	Yes	7-14 days	NA	40 days	47 days	Cool to ≤ 6° C
TPH (8015)	No	NA	NA	14 days	14 days	Cool to 4 °C ±2

(GRO) (solid)						
TPH (8015) (GRO) (aqueous)	Yes	NA	NA	14 days	14 days	Cool to 4 °C ±2; HCl
TPH (8015) (DRO) (solid and aqueous)	No	7-14 days	NA	40 days	47 days	Cool to 4 °C ±2; Keep away from light
Total Metals (6000/7000) (Except Cr^{6+} and Hg)	Yes	NA	NA	180 days	180 days	Nitric Acid (pH<2-aqueous); cool to 4±2˚C - solid samples
Hexavalent Chromium (7196) (aqueous)	No		NA	24 hours	24 hours	Cool to ≤ 4 °C
Hexavalent Chromium (3060A/7196) (solid)	No	30 days	NA	7 days		≤4±2 °C
Mercury (7470 aqueous and 7471B solid)	Yes	NA	NA	28 days	28 days	Nitric Acid (pH<2-aqueous); cool to ≤ 6ºC
TCLP VOCs (1311/8260)	No	14 days	NA	14 days	28 days	no preservative
TCLP SVOCs (1311/8270)	No	14 days	7 days	40 days	61 days	no preservative
TCLP Metals (except mercury) (1311/6010)	No	180 days	NA	180 days	360 days	no preservative
TCLP Mercury (1311/7470)	No	28 days	NA	28 days	56 days	no preservative

pH (9040)	No	24 hours	NA	NA	1 day	no preservative
Ammonia (Liquid, SM 4500-N)	No	NA	NA	7 days	7 days	Cool to 4˚C
Ammonia (Liquid, SM 4500-N)	Yes	NA	NA	28 days	28 days	Cool to 4˚C; H_2SO_4 to pH <2
Cyanide (Solid, Liquid, Multi-Phase; 9010c)	Yes	NA	NA	14 days	14 days	Cool to 4˚C ±2; NaOH ≥ pH 12

1.2 Technical Holding Times	
Technical holding time evaluation is important to ensure the data is valid and not biased from inappropriate handling procedures. Technical holding times are judged by assessing the lapsed time from field sampling to extraction and then to analysis. There are specific technical holding time requirements for specific classes of compounds. In addition, holding times may vary due to the presence or absence of preservatives. The validator should refer to specific criteria for holding times listed in Table 1 and in the Tier I Data Validation Manual. Use sampling information found on the COC, and extraction and analysis dates (found in the data report, examined in section 1.1) to determine whether technical holding times comply with criteria listed above in Table 1-2. Complete the following section to determine if any violations of technical holding time exist and qualify all associated sampling data.	
Technical Holding Times - Volatile Organic Compounds	
1.2.1 Were samples properly preserved? Check preservation requirements, COC, and sample receipt form for discrepancies. *Action: Note any problems and use the information to qualify results.*	**Indicate yes or no:** Yes. TCLP samples should not be preserved and there is no indication of preservation. All samples were chilled to 6°C, which is acceptable. **If no, list any problems:**
1.2.2 Were any technical holding times exceeded? *Action: If samples were improperly preserved or unpreserved, if applicable, and the technical holding times were exceeded, qualify all detected results for affected samples as "J-" and all non-detected results as "UJ."*	**Indicate yes or no:** No. **If yes, list sample ID(s) and summarize actions taken:**
1.2.3 Were any technical holding times greater than 2x the time requirement? *Action: If technical holding times are greatly exceeded (> 2x the time requirement) upon analysis or re-analysis then the validator may use professional judgment to qualify all non-detected*	**Indicate yes or no:** No. **If yes, list sample ID(s) and summarize any actions taken:**

compounds as "UJ" or "R" based upon professional judgment and on DQOs.	

Technical Holding Times - Inorganic Compounds	
1.2.11 Were samples properly preserved (4°C for solids; acid preservation for aqueous samples or unpreserved for TCLP)? Check preservation requirements, COC, and sample receipt form for discrepancies. *Action: Note any problem and use the information to qualify results in the next step.*	**Indicate yes or no:** No. **List any problems:** As stated in the above sections, aqueous samples require acid preservation for mercury analysis, but there is no indication that nitric acid was used to preserve samples at a pH of <2. Otherwise, TCLP samples were not preserved, and all other samples were chilled to 6°C. This is acceptable.
1.2.12 Were any technical holding times exceeded? *Action: If samples were improperly preserved or properly preserved and the technical holding times shown in Table 1-2 were exceeded, qualify all detected results for affected samples as estimated ("J-") and all non-detected results as "UJ" or rejected ("R") depending on DQOs.*	**Indicate yes or no:** No. **If yes, list sample ID(s) and summarize actions taken:**
1.2.13 Were any technical holding times greater than 2x the time requirement? *Action: If technical holding times are greatly exceeded (> 2x the time requirement), the validator may use professional judgment and the project's DQOs to qualify all non-detected compounds as "R" and all detected results as "J-" or "R," depending on DQOs.*	**Indicate yes or no:** No. **If yes, list sample ID(s) and summarize actions taken:**

Technical Holding Times - Mercury	
1.2.14 Were samples properly preserved (pH <2 for aqueous samples, ≤ 6ºC for solid or aqueous samples, or unpreserved for TCLP)? Check preservation requirements, COC, and sample receipt form for discrepancies. *Action: Note any problem and use the information to qualify results in the next step.*	**Indicate yes or no:** No. **List any problems:** The sample receipt form does not state whether aqueous samples were preserved in nitric acid with a pH of <2.

1.2.15 Were any technical holding times exceeded? *Action: If samples were improperly preserved or properly preserved and the technical holding times shown in Table 1-2 were exceeded, qualify all detected results for affected samples as estimated ("J-") and all non-detected results as "UJ" or rejected ("R") depending on DQOs.*	**Indicate yes or no:** No. **If yes, list sample ID(s) and summarize actions taken:** Technical holding times were not exceeded but aqueous samples were not properly preserved. Detects of mercury should be qualified with "J-" and non-detects with "UJ". Aqueous sample 028 did not have a detection of mercury, so it should be flagged with "UJ".
1.2.16 Were any technical holding times greater than 2x the time requirement? *Action: If technical holding times are greatly exceeded (> 2x the time requirement), the validator may use professional judgment and the project's DQOs to qualify all non-detected compounds as "R" and all detected results as "J-" or "R," depending on DQOs.*	**Indicate yes or no:** No. **If yes, list sample ID(s) and summarize actions taken:**

Technical Holding Times – pH	
1.2.25 Were any technical holding times exceeded? **Note: For ground water samples, pH should be evaluated in the field within 15 minutes of sampling. For waste samples, the technical holding time is more flexible and requires an examination of the type of waste and the project's DQOs. If technical holding times exceed 24 hours, consider qualification. If wastes exhibit the characteristic of corrosivity (*i.e.*, <pH 2 or >pH 12.5), samples should not be qualified.** *Action: If technical holding times are exceeded, the data validator may use professional judgment and DQOs to qualify data as "R" or "J-."*	**Indicate yes or no:** Yes. **If yes, list sample ID(s) and summarize actions taken:** Sample 002 (Page 8 and 10). The technical holding times were exceeded by 5 days. For ground water samples, the technical holding time requirement is for field analysis (i.e., immediately). For RCRA compliance samples, no set technical holding time requirements are required. A 24-hour technical holding time would be acceptable, but for some wastes, a longer holding time may be warranted. If results indicate that a waste is corrosive, the results should not be flagged. All other exceedances of technical holding times (>24 hours) could merit qualification based upon the type of waste and the DQOs for the project. Professional judgement should be used to qualify

	data as “R” or “J-“.

Checklist #2

VOC Data Validation

2.0 VOC Data Validation	
2.1 Volatile Data Review - Blank Data	
Laboratory blanks are used to assess whether contamination from the laboratory, reagents, or other samples exists and whether this contamination can bias sample results. The qualification of sample results will depend upon the magnitude of blank contamination.	
2.1.1 Is the method blank data present for each batch (matrix and sample number dependent), including TCLP? *Action: If not present, request information from the applicable party or laboratory. If the required method blank(s) was not analyzed, sample results may be qualified as estimated ("J," for detected results and "UJ," for non-detected compounds) based upon the validator's judgment. Additional qualification may be warranted based upon other QA/QC information.*	**Indicate yes or no:** Yes. A TCLP blank (TB) for VOCs is present for samples 028 and RO-02 (batch 347359, Page 68). A blank for total VOCs (MB) is present for sample 002 (batch 347358, Page 71).
2.1.2 Is there an indication that the samples associated with the method blank were diluted? **Note: The dilution factor (DF) can be found in the data report (a dilution factor of 1 indicates no dilution).**	**Indicate yes or no:** Yes. **If yes, list the sample ID(s) and dilution factor(s):** TCLP VOCs for 028 and RO-02 are diluted: • DF for RO-02 = 100 (page 62) • DF for 028 and 002 = 50 (pages 60 and 64-65)
2.1.3 Do any method/field/trip/equipment blanks have any detected results for any volatile target analytes? Were the same target compounds found in the samples? **Note: A list of samples associated with each of the contaminated blanks should be prepared. Trip blanks are used to qualify samples based on potential contamination during shipment and are not required for non-aqueous matrices. If analytes are detected in a blank but not in the sample of interest, then qualification of those analytes is not necessary.**	**Indicate yes or no:** Yes. **If yes, list those analytes and results found in both the blanks and samples:** Batch 347359: PCE was present at a concentration of 0.013 mg/L in the blank (page 68). Associated samples: • Sample ID 028: 0.0113 mg/L PCE (page 60) • Sample ID RO-02: 0.0361 mg/L PCE (page 62)

Use the information from 2.1.3 to determine whether a dilution factor should be used to determine qualification. When a dilution is applied to samples, the contaminant concentrations in the samples are divided by the dilution factor, then use the criteria listed in the following table to qualify blanks and sample data. *Action: Use the criteria in Table 2-1 below to qualify sample results due to blank contamination. Use the largest value from all associated blanks. If any blanks are grossly contaminated, all associated data may be qualified as "R", based upon professional judgment and the project's DQOs.*	Batch 347358: 1,2,4 TMB, Naphthalene, Styrene, Toluene, and 2-Butanone were present in the blank at: • 1,2,4 TMB: 27.1 • Naphthalene: 31.4 • Styrene: 13.5 • Toluene: 46.0 • 2-Butanone: 788 These values are all less than the analytes' respective reporting limits. Associated sample: Sample ID 002: • 1,2,4 TMB: 670 • Naphthalene: 121 • Styrene: 20.5 • Toluene: 159 • 2-Butanone: 1390 **Summarize sample result qualifications based on blank results:** Based upon the dilution factor and the criteria in Table 2-1, sample results for all the constituents listed above should be flagged with a "U". PCE in Samples 028 and RO-02 should be flagged with "U" and reported at the QL. Constituents listed above in Sample 002 should be flagged with "U" and reported at the QL.

Table 2-1: Blank Actions for VOC Analyses

Qualification		
Blank Result	Sample Result	Action
Detection	Non-detect	No Action
< QL	< QL	Report at QL and qualify U
< QL	≥ QL but < 2x Blank Result for common laboratory contaminants	Report at QL and qualify U

Table 2-1: Blank Actions for VOC Analyses		
Qualification		
< QL	≥ QL (≥ 2x Blank Result for common laboratory contaminants)	Report at sample result and qualify J+
≥ QL	< QL	Report at QL and qualify U
≥ QL	≥ QL but < Blank Result	Report at sample result and qualify U
≥QL	≥ QL and ≥ Blank Result or 2x Blank Result for common laboratory contaminants	Report at sample result and qualify J+
Gross contamination*	Detection	Report at sample result and qualify R

* Gross contamination is when blank results are greater than the initial calibration high-point standard concentration.

2.2 Volatile Data Review - Laboratory Control Sample (LCS)	
An LCS should be included with each batch of samples (approximately 20). The LCS consists of an aliquot of a clean (control) matrix similar to the matrix type of the sample and at the same weight or volume. The LCS is spiked with the same analytes at the same concentration as the matrix spike. When the results of the matrix spike indicate a potential problem due to the sample matrix itself, the LCS verifies that the laboratory can perform analyses in a clean matrix. Suggested surrogate recovery limits are 70% - 130% or acceptance criteria set by the lab.	
2.2.1 Was an LCS prepared, extracted, analyzed, and reported once per batch of 20 samples? **Note: This information should be included in the QA package provided by the lab. If not, contact the laboratory and request that the information be submitted. This information should be found in the injection log.** *Action: If LCS information cannot be found, contact the applicable party or laboratory for re-submittal of the data package. If LCS information is not present, qualify all detected results as "J" and all non-detect results as "UJ" or reject all results based on best professional judgment.*	**Indicate yes or no:** Yes. For samples 028 and RO-02, the LCS results are found on page 67. For sample 002, the LCS results are found on pages 70-71. **Summarize any actions taken:** N/A
2.2.2 Does the LCS contain the following volatile target compounds in addition to the required surrogates? 1,1-Dichloroethene Toluene Trichloroethene Benzene	**Indicate yes or no:** Yes. The LCS in both batches contains these compounds.

2.2 Volatile Data Review - Laboratory Control Sample (LCS)	
Chlorobenzene **Note: Method 8260D calls for the LCS to be spiked at the same level as the matrix spike. See Section 5.5.1 of Method 5000 for the recommended purgeable matrix spiking solution for 8260. When the results of the matrix spike indicate a problem due to sample matrix, the LCS should be checked to determine whether the laboratory can perform the analysis on a clean matrix.**	
2.2.3 Do the percent recoveries (%R) meet the suggested QC limits or limits provided by the lab? **Note: The laboratory should use 70 - 130% as interim acceptance criteria for recoveries of spiked analytes, until in-house LCS limits are developed.** *Action: Follow the directions in Table 2-2 below for qualifying results.*	**Indicate yes or no:** For batch 347359 –Yes. For batch 347358 – No. **If no, list compounds and sample IDs that do not meet QC limits and summarize actions taken:** 3 LCS compounds were outside of the QC limits for Sample 002 in batch 347358. Bromomethane %R = 31%, below limits of 41-163 (page 70) Chloroethane %R = 20%, below limits of 51-145 (page 70) N-Butyl Benzene %R = 118%, above limits of 56-115 (page 71) The results for bromomethane and chloroethane in the samples were non-detect. These results should be qualified as rejected "R". N-Butyl Benzene was detected in sample 002 at 158 ug/kg. This result should be qualified as 158 J+ ug/kg.
2.2.4 Verify the calculations for at least one %R. $$\%R = \left(\frac{Measured\ Concentration}{Spiked\ Amount}\right) \times 100$$ *Action: If the %R is not calculated correctly, verify the other %R calculations and/or contact the lab for re-submittal. If the re-calculated %R values fall within the QC limits,*	**Show results of verified %R calculation:** Using the LCS result for 2-butanone for batch 347358 (sample 002), the %R was reported as 84%. (Page 70-71) To verify this result, the value listed under the NOM heading (LCS spike concentration) and the measured result for 2-butanone were used. %R = 4200/5000 X 100 = 84%

2.2 Volatile Data Review - Laboratory Control Sample (LCS)	
the validator should use professional judgment to determine if the lab should be contacted for re-submittal or if the data should be flagged.	

Table 2-2: LCS Actions for VOC Analyses		
Qualification		
LCS Result	Sample Result	Action
> Upper acceptance limit	Detection	J+
< Lower acceptance limit	Detection	J-
< Lower acceptance limit	Non-detect	R
≥ Half target compounds not within recovery criteria	All detections	J
≥ Half target compounds not within recovery criteria	All non-detects	R

2.3 Volatile Data Review - Matrix Spike/Matrix Spike Duplicates	
Matrix spike and matrix spike duplicates are performed to assess method precision for VOC analyses. Matrix spikes and duplicates are required for every batch of samples (approximately every 20 samples). The validator should be aware that the MS/MSD are batch specific QA/QC samples, not sample specific. For example, the MS/MSD information may be analyzed with any sample in the batch, but not necessarily a sample being validated. Because of this, matrix spike and matrix spike duplicate data alone usually are not used to qualify results, but the information is used with other QA/QC data to qualify data.	
2.3.1 Is matrix spike/matrix spike duplicate recovery data present? Note: MS/MSD recovery data is more important for aqueous samples, so it is recommended that projects include review of MS/MSD recovery data for water samples in project DQOs. *Action: If the matrix spike/spike duplicate data are required by the project-specific QAPP or DQOs but missing, the laboratory should be contacted for a re-submittal.*	**Indicate yes or no:** Yes. In this lab report, the matrix spike, matrix spike duplicate is listed as prep-spike, prep-spike duplicate (i.e., PS and PSD). For batch 347359 (samples 028 and RO-02) the QC results are found on pages 67-68. For batch 347358 (sample 002) the QC results are found on pages 73-76.
2.3.2 How many VOC spike recoveries are outside the QC limits?	**Record the spike recovery and control limits:** For batch 347358 (sample 002) 4 compounds are out of criteria in the matrix spike and 2 of the same compounds are outside of QC limits in the matrix spike duplicate. PS %R PSD %R Limits 2-Butanone 48 55-149

2.3 Volatile Data Review - Matrix Spike/Matrix Spike Duplicates	
	Acetone 24 44-181 Bromomethane 29 30 44-163 chloroethane 19 18 51-145 hexachlorobutadiene 52 56-121 No compounds are out of control limits for batch 347359.
2.3.3 Verify the calculations for at least one %R. Matrix Spike Recovery $\%R=\frac{SSR-SR}{SA} \times 100$ Where: SSR= spiking analyte result in the spiked sample SR= Result of the same analyte in the original sample SA= spike added in the spiked sample	**Show results of verified %R calculation:** For 2-butanone: %R = (38.1-14.3) / 50 x100 = 48
2.3.4 How many relative percent differences (RPDs) for matrix spike and matrix spike duplicate recoveries are outside the QC limits for VOCs (≤20%)? **Note: The MS/MSD results may be used in conjunction with other QC criteria to determine the need for data qualification. Outliers should be identified.**	**Record the recovery data out of criteria and control limits. Review surrogate and LCS data to determine if qualification is necessary:** For batch 347358 (sample 002), 2 compounds had RPDs that exceeded the control limits. (Page 75) 2-butanone: RPD: 40, above laboratory limits of 0-30 Acetone: RPD: 99, above laboratory limits of 0-30
2.3.5 Verify the calculations for at least one RPD. RPD $RPD=\frac{\lvert MSR-MSDR\rvert}{\left(\frac{MSR+MSDR}{2}\right)} \times 100$ Where: MSR= Matrix spike result for the spiking analyte in the MS sample MSDR= Matrix spike result for the spiking analyte in the MSD sample	**Show results of verified RPD calculation:** Calculations could not be verified. While RPD is shown, the duplicate results are not provided.

2.4 Volatile Data Review - Surrogate Recovery
Surrogate compounds are spiked compounds of known composition and concentration that are added to samples and blanks. Surrogates are compounds that mimic target analytes but are

either compounds that are not commonly found in the environment or that have been altered (*e.g.*, "deuterized") so that they can be identified as quality QA analytes. The recovery of surrogate compounds allows an assessment of matrix interference. VOC surrogate recoveries are also used with other QA/QC data to qualify sample results and to justify laboratory re-analysis. Specific examples are listed in the data validation guidance document. **Common VOC surrogates include the following:** **<u>Recommended Surrogate Compound</u>[a]** **4-Bromofluorobenzene** **Toluene-d8** **1,2-Dichloroethane-d4** **a See SW-846 Method 8260D, Table 1A for additional acceptable surrogates.**	
2.4.1 Are the surrogate recovery data present for each batch (method and matrix), including TCLP? **Note: Samples may be included in different batches. When this is the case, separate surrogate recoveries should be provided.** *Action: If no, then contact the laboratory for an explanation and report re-submittal.*	**Indicate yes or no:** Yes, each result page contains surrogate recovery information (*e.g.*, page 62).
2.4.2 Are any surrogate recoveries are outside the QC limits? **Note: Suggested surrogate recovery limits are 70 to 130% until laboratory or project-specific criteria are developed. QC limits will depend on the surrogates chosen, levels used, and instrument conditions. Acceptance criteria is guidance.** *Action: Identify samples with recoveries outside QC limits.*	**Indicate yes or no:** Yes. **If yes, list the sample ID(s), matrix(-ces) and parameter(s):** Batch 347359 on page 62 has surrogate recoveries for Bromofluorobenzene and toluene-d8 in RO-02 that out of limits. Bromofluorobenzene %R: 118, while limits are 95-108 Toluene-d8 %R: 116, while limits are 94-107
2.4.3 Verify the calculations for at least one %R. $Recovery\ \% = \frac{(Concentration\ found)}{(Concentration\ added)} \times 100$	**Show results of verified %R calculation:** A %R calculation could not be verified. While %R values for surrogates were provided, the found and added concentrations are not shown.

2.4.4 If any surrogate compound was out of compliance was re-analysis performed to confirm a matrix interference? **Note: Check the report narrative for an indication of re-analysis. Additionally, qualification may not be appropriate for TCLP data. Best professional judgment should be used to qualify data.** *Action: Based on the findings, qualify data using the following criteria in Table 2-3 below.*	**Indicate yes or no:** No. There is no evidence of re-analysis for sample RO-02. The results (Page 62) for this sample are subject to qualification. In this sample, two surrogates were above the upper control limit. Therefore, all detected results should be qualified as estimated and flagged with a "J+." Non-detected results should not be qualified. **If yes, list sample ID(s) for surrogate compounds out of compliance and criteria:**

Table 2-3: Surrogate Actions for VOC Analyses

	Action	
	Detect	**Non-detect**
Surrogate not present or not at specified concentration	J or R	UJ or R
%R < Expanded Lower Acceptance Limit (10%, excluding surrogates with 10% as a lower acceptance limit, undiluted sample analysis)	J-	R
Expanded Lower Acceptance Limit (10%) ≤ %R (excluding surrogates with 10% as a lower acceptance limit, undiluted sample analysis) < specified Lower Acceptance Limit	J-	UJ
%R < specified Lower Acceptance Limit (diluted sample analysis)	Use professional judgment	Use professional judgment
%R within specified Acceptance Limits	No qualification	No qualification
%R > specified Upper Acceptance Limit	J+	No qualification

Checklist #6

Metals Data Validation

6.0 Metals Data Validation	
6.1 Metals Data Review – Blank Data Analysis	
Laboratory or preparation blanks are used to assess whether contamination from the laboratory, reagents, or other samples exists and whether this contamination can bias sample results. The qualification of sample results will depend upon the magnitude of blank contamination. Metals, excluding mercury, are typically analyzed using Method 6010D: Inductively Coupled Plasma – Atomic Emission Spectrometry (ICP-AES). Direct analysis should be conducted on only relatively clean, aqueous materials. Other, more complex aqueous and/or solid samples may be analyzed using this method but require acid digestion prior to analysis.	
6.1.1 Was a method/preparation blank included with each batch of samples (for each matrix), including TCLP? *Action: If not present, request information from the laboratory or applicable party. If the required method blanks were not analyzed, sample results may be qualified as "J" for detected results and "UJ" for non-detected compounds. Qualification should consider other QA/QC information and the DQOs.*	**Indicate yes or no:** Yes. Batch 346202 (samples 028 and RO-02), Pages 36-37 Batch 346537 (sample 002), Page 37 **Summarize any actions taken:** N/A
6.1.2 Were any samples diluted? *Action: Record the sample ID and dilution factor(s).*	**Indicate yes or no:** No. **Record sample ID(s) and dilution factor(s):** Batch 346202 (samples 028 and RO-02), page 30 and 32 Batch 346537 (sample 002), page 34 Dilution Factor = 1 (no dilution) for all samples
6.1.3 Were metals detected in the blank? Were the same target analytes found in the samples? **Note: Use the information from 6.1.2 to determine whether a dilution factor should be used to determine qualification. When a dilution factor is applied to samples the contaminant concentration in the samples is divided by the dilution factor. The criteria in Table 6-1 are used to qualify sample results.** *Action: For those metals identified in both the blank and sample, follow the directions in Table 6-1 below for qualifying data based on blank results.*	**Indicate yes or no:** Yes. **If yes, list those analytes and results found in both the blanks and samples and summarize any actions taken:** Batch 346202 (samples 028 and RO-02), page 36: Barium detected in the blank at 0.0233 Barium in Sample 028: 0.169 Barium in Sample RO-02: 2.65 Both are greater than 10x the blank result, so no action is needed. Batch 346537 (Sample 002), page 37: Arsenic detected in the blank at 1.90

6.0 Metals Data Validation	
6.1 Metals Data Review – Blank Data Analysis	
	Arsenic in Sample 002: ND No action is needed.

Table 6-1: Blank Actions for Metals		
Qualification		
Blank Result	**Sample Result**	**Qualification**
>QL	>10X Blank Result	No Action
>MDL but <QL	>MDL but <QL	U
>MDL	<10X Blank Result	U or J-
<QL	<10X Blank Result	U

6.2 Metals Data Review – Laboratory Control Samples	
Laboratory Control Samples (LCSs) are analyte-free water or solid, clean control matrixes similar to the sample matrix, spiked with target analytes at known concentrations. LCSs are analyzed using the same sample preparation, reagents, and analytical methods employed for the samples received. Method 6010D calls for the LCS to be spiked at the same levels and using the same spiking materials as the corresponding matrix spike (MS) and matrix spike duplicate (MSD). When the results of the MS analysis indicate a potential problem due to the sample matrix itself, the LCS results are used to verify that the laboratory can acceptably perform unbiased analysis in a clean matrix. LCS criteria listed in this section are determined from Method 6010D and U.S. EPA's National Functional Guidelines for Inorganic Data Review.	
6.2.1 Was an LCS prepared, extracted, analyzed, and reported once per batch of 20 samples? **Note: This information should be included in the QA package provided by the lab. If not, contact the laboratory and request that the information be submitted. This information should be found in the injection log.** *Action: If LCS information cannot be found, contact the applicable party or laboratory for re-submittal of the data package. If LCS information is not present, qualify all detected results as "J" and all non-detect results as "UJ" or reject all results based on best professional judgment.*	**Indicate yes or no:** Yes. (Pages 36 and 37) **Summarize any actions taken:** N/A
6.2.2 Was an LCS analyzed per batch and within suggested QC limits (80% - 120%) or limits provided by the lab?	**Indicate yes or no:** Yes – all LCS results were within limits. The %R for silver was less than 80%, but was still within the laboratory's QC limits of 75-120%.

Note: Use 80% - 120% unless appropriate lab-specific LCS limits have been developed. The results for solid and aqueous LCSs should always be within the control limits. Digestion method 3050B includes optional steps for constituents that are difficult to recover, such as Ag (See Section 7.5). If out of limits, the laboratory should terminate the analysis, correct the problem, and the samples should be re-digested and re-analyzed. If still unacceptable, then all samples after the last acceptable method blank must be re-prepared and reanalyzed, along with all other appropriate analysis batch QC samples. *Action: Refer to Table 6-2 below to determine whether data needs to be qualified. If >120%, qualify all detect data as "J+". If <80%, qualify detect data as "J-" and non-detect data as "UJ".*	**Summarize any actions taken:** N/A
6.2.3 Verify the calculations for at least one %R. $\%R = \left(\frac{Measured\ Concentration}{Spiked\ Amount}\right) \times 100$ *Action: If the %R is not calculated correctly, verify the other %R calculations and/or contact the lab for re-submittal. If the re-calculated %R values fall within the QC limits, the validator should use professional judgment to determine if the lab should be contacted for re-submittal or if the data should be flagged.*	**Show results of verified %R calculation:** For cadmium (page 36): %R = 0.488 / 0.500 x 100 = 98

Table 6-2: LCS Actions for Metals

Criteria	Action	
	Detect	**Non-detect**
LCS not prepared with samples	J	UJ
LCS not prepared at specified concentrations	J	UJ
Aqueous/water and soil/sediment/waste %R < 40% (<20% Ag, Sb)	J-	R
Aqueous/water and soil/sediment/waste %R 40-79% (20 – 49% Ag, Sb)	J-	UJ

Aqueous/water and soil/sediment/waste %R 80-120% (50-150% Ag, Sb)	No qualification	No qualification
Aqueous/water and soil/sediment/waste %R 121-150% (151-170% Ag, Sb)	J+	No qualification
Aqueous/water and soil/sediment/waste %R > 150% (>150% Ag, Sb)	R	No qualification

6.3 Metals Data Review – Matrix Spike/Matrix Spike Duplicate	
Spikes are elements of known composition that are added to blanks and to samples that measure accuracy and precision of the analyses. At least one spike [termed a matrix spike or prep spike] should be included for each batch of 20 samples or less. Specifically, at least one MS, one unspiked sample, and one MSD for each batch of samples should be analyzed. Spike recovery criteria listed in this section are determined from U.S. EPA's National Functional Guidelines for Inorganic Data Review and Method 6010D. The criteria applied by an individual laboratory may vary. The laboratory should be consulted and have its QA/QC criteria supplied to the validator.	
6.3.1 Was at least one pre-digestion spiked sample (matrix spike) analyzed per batch, matrix type, or concentration or sample delivery group? *Action: If not present, flag detections "J", non-detections "UJ", and contact the applicable party for re-submittal.*	**Indicate yes or no:** Yes. Batch 346202 (TCLP metals; spl. 028 and RO-02) on page 36. Batch 346537 (total metals; spl 002), page 37 **If no, describe any actions taken:**
6.3.2 Are all spike recoveries within control limits (*e.g.*, 75% to 125%)? **Note: Digestion method 3050B includes optional steps for constituents that are difficult to recover, such as Ag (See Section 7.5). When the spike sample result is less than the instrument detection limit, the percent recovery calculation should use a value of zero (not the detection limit) for the sample result.** *Action: Is the sample concentration ≥ 4 times the spiked concentration? If yes, spike recovery limits do not apply, and data is unqualified. If no, identify those analytes whose concentration is < 4 times the spike added (these would be analytes that should potentially be qualified using professional judgement and other QC results).*	**Indicate yes or no:** Yes. **If no, list analytes < 4 times the spike added:**
6.3.3 Verify the calculations for at least one %R. Spike Percent Recovery (%R)	**Show results of verified %R calculation:** For Barium on page 36:

6.3 Metals Data Review – Matrix Spike/Matrix Spike Duplicate	
$$\%\ Recovery\ (\%R) = \frac{SSR - SR}{SA} X100$$ Where: SSR=Spiked sample result SR=Sample result SA=Sample added	%R = ((0.981-0.0621) / 1.00) x100 = 92
6.3.4 Based on the results of 6.3.2, if the spike recoveries are outside the control limits and the sample results were <4x the spike amount, a post-digestion spike should be analyzed at 2x the indigenous level or QL, whichever is greater. **Note: Post-digestion spikes are not required for Ag or Sb. However, one is typically run if the LCS was out of control. The post-digestion spike confirms a matrix interference and should not be used for qualification.** *Action: Contact the applicable party or laboratory for an explanation if a post-digestion spike was not performed and analyzed if the LCS was out of control. If a satisfactory explanation is not available, use professional judgment to qualify sample results.*	**Summarize results of any post-digestion spikes and actions taken:** N/A
6.3.5 Is the %R (pre and post digestion) for any matrix type: 1. Less than 30%? 2. Between 30% and 74%? 3. Greater than 125%? **Note: The criteria in the table below are method requirements for spike samples of any matrix type. However, for technical review purposes only, the QAPP or project-specific DQOs for data review may allow the use of less restrictive criteria (*e.g.*, 10 %R and 150 %R for the lower and upper limits) to be assessed against spike and post-digestion spike soil samples.** *Action: Use the criteria in Table 6-3 below to determine whether the data needs to be qualified. If qualification is needed, take the listed necessary actions.*	**Indicate yes or no:** No. **Summarize any actions taken:** N/A – all %Recoveries are within criteria.
6.3.6 Was at least one MS and one duplicate unspiked sample, or one matrix spike/matrix	**Indicate yes or no:**

6.3 Metals Data Review – Matrix Spike/Matrix Spike Duplicate	
spike duplicate (MS/MSD) pair analyzed for each batch of samples processed? **Note: If samples are expected to contain target analytes, laboratories may choose to use an MS and a duplicate analysis of an unspiked field sample. If samples are not expected to contain target analytes, the laboratories should use an MS/MSD pair. Duplicate samples are not required for wipe samples.** *Action: Verify that at least one duplicate sample was prepared and analyzed from each group of samples of a similar matrix type or for each data package.*	Yes. One MS/MSD pair was analyzed, as well as one unspiked duplicate sample for each batch. **Summarize any actions taken:** N/A
6.3.7 Are all relative percent difference (RPD) values within control limits? **Note: Acceptance criteria for RPD should be a set of laboratory-derived limits; however, acceptance limits must not exceed 20% for original and duplicate sample values ≥ 5x the QL. For samples analyzed under the Statement of Work (SOW), a control limit of the Quantitation Limit (QL) should be used if either the sample or duplicate value is < 5x the QL.** *Action: Determine whether RPD values exceed laboratory-derived control limits. If control limits have not been developed, use ≤20% as the acceptance criteria.*	**Indicate yes or no:** Yes. **Summarize any actions taken:** N/A
6.3.8 RPD is calculated to evaluate the spike values for precision using the following equation: $$\text{RPD} = \frac{\lvert S - D \rvert}{(\frac{S + D}{2})} \text{x}100$$ Where: S = Sample Result (original) D = Duplicate Result When the sample or duplicate result is reported as a non-detect, use a value of zero (0) for calculating the RPD. This will always yield an RPD of 200%.	**Show results of one verified RPD calculation:** RPD calculation for Barium on page 36: Sample result = 0.0621 Duplicate result = 0.0612 RPD = |0.0621-0.0612| / ((0.0621+0.0612)/2) x100 = 1.45 1.45 rounds to 1. The RPD given is 2, indicating a calculation error or a difference due to rounding. 1 is still within criteria, so no actions are necessary.

6.3 Metals Data Review – Matrix Spike/Matrix Spike Duplicate	
Action: Verify an RPD calculation for one set of MS/MSD samples. Contact the applicable party or laboratory for an explanation if RPD was not calculated. If a satisfactory explanation is not available, use professional judgment to qualify sample results.	

Table 6-3: Matrix Spike Actions for Metals

Criteria	Action	
	Detect	**Non-detect**
Matrix Spike %R < 30% Post-digestion spike %R < 75%	J-	R
Matrix Spike %R < 30% Post-digestion spike %R ≥ 75%	J	UJ
Matrix Spike %R 30-74% Post-digestion Spike %R < 75%	J-	UJ
Matrix Spike %R 30-74% Post-digestion spike %R ≥ 75%	J	UJ
Matrix Spike %R 75-125% No Post-digestion spike required	No qualification	No qualification
Matrix Spike %R > 125% Post-digestion spike %R > 125%	J+	No qualification
Matrix Spike %R > 125% Post-digestion spike %R ≤ 125%	J	No qualification
Matrix Spike %R < 30% No post-digestion spike performed (not required for Ag and Sb)	J-	R
Matrix Spike %R 30-74% No post-digestion spike performed (not required for Ag and Sb)	J-	UJ
Matrix Spike %R > 125% No post-digestion spike performed (not required for Ag and Sb)	J+	No qualification

Checklist #7

Mercury Data Validation

7.0 Mercury Data Validation	
7.1 Mercury Data Review – Blank Data	
Mercury is analyzed using SW-846 Method 7470A for liquid samples and Method 7471B for solid samples. These methods utilize a manual cold vapor atomic adsorption (AA) technique to quantify mercury. These methods have slightly different acceptance criteria than other AA methods and therefore are separated into a separate section of the checklist.	
7.1.1 Was a method/preparation blank included with each batch of samples (for each matrix)? *Action: If no method blank was included, consult the laboratory or applicable facility and, if possible, have the data submitted. If the data is not available, the data validator may apply best professional judgment to qualify the sample results.*	**Indicate yes or no:** Yes. Batch 346229 (Hg samples 028 and RO-02), page 38 Batch 346537 (Hg sample 002), page 38
7.1.2 Were any samples diluted? *Action: Record the sample ID and dilution factor(s).*	**Indicate yes or no:** No. **Sample ID(s) and dilution factor(s):** The dilution factor is 1.
7.1.3 Did the method blank contain mercury above detectable levels? Was mercury also detected in the sample results? If so, these results are subject to qualification. **Note: If mercury is discovered in the method blank above or equal to the quantitation limit, the lowest concentration of any sample in that batch must be greater than or equal to 10x the method blank concentration (after dilution is accounted for). If this is not the case, all samples in that batch should have been redigested and reanalyzed.** *Action: Review the blank data. Use Table 7-1 below to qualify results. If the sample results are detected at concentrations greater than or equal to the QL but less than 10 x the concentration in the blank, the results should have been redigested and reanalyzed.*	**Indicate yes or no:** No, mercury was not detected in either blank. **Summarize any actions taken:** N/A

Table 7-1: Blank Actions for Mercury

Blank Result	Sample Result	Action
Not analyzed at specified frequency	Non-detect	UJ
	Detect	J

Detect < QL	Non-detect	No qualification
	Detect < QL	Report at QL and qualify U
	Detect > QL	J+ or no qualification
≤ (-MDL) but > (-QL)	Non-detect	UJ
	Detect	J- or no qualification
≥ QL	Non-detect	No qualification
	Detect < QL	Report at QL and qualify U
	≥ QL but < 10x the Preparation Blank Result	Report at Preparation Blank Result and qualify J+ or R
	≥ 10x the Preparation Blank Result	No qualification
≤ (-QL)	Non-detect	UJ
	Detect < QL	J-
	≥ QL but < 10x QL	J-
	≥ 10x QL	No qualification

7.2 Mercury Data Review – Laboratory Control Sample	
Laboratory Control Samples (LCSs) are analyte-free water or solid, clean control matrixes similar to the sample matrix, spiked with target analytes at known concentrations. LCSs are analyzed using the same sample preparation, reagents, and analytical methods employed for the received samples. LCS criteria listed in this section are determined from Method 7471B.	
7.2.1 Was an LCS prepared, extracted, analyzed, and reported once per batch of 20 samples? **Note: This information should be included in the QA package provided by the lab. If not, contact the laboratory and request that the information be submitted. This information should be found in the injection log.** *Action: If LCS information cannot be found, contact the applicable party or laboratory for re-submittal of the data package. If LCS information is not present, qualify all detected results as "J" and all non-detect results as "UJ", or reject all results based on best professional judgment.*	**Indicate yes or no:** Yes. LCS data for both batches are found on page 38. **Summarize any actions taken:** N/A
7.2.2 Was the LCS within suggested QC limits (80 to 120%) or limits provided by the lab? **Note: Use 80% - 120% unless appropriate lab-specific LCS limits have been developed. The results for an LCS should always be within the**	**Indicate yes or no:** Yes. Both are within 80-120% **Summarize any actions taken:** N/A

control limits. If out of limits, the laboratory should terminate the analysis, correct the problem, and the samples should be re-digested and re-analyzed for mercury. *Action: If the LCS is outside of the control limit, qualify all positive results as estimated ("J+" or "J-").* *If the LCS results are higher than control limits and the sample results are below the detection limit, the results are acceptable.* *If the LCS result is below the lower control limit, initially qualify all results below the detection limit as "UJ". Non-detected compounds may be qualified as rejected, "R" based upon professional judgment and the project's DQOs.*	

7.3 Mercury Data Review – Matrix Spike Recovery	
7.3.1 Was a matrix spike analyzed at the required frequency (one pre-digestion spike for each group of samples with a similar matrix type or for each data package), and was each matrix spike within limits? **Note: Post-digestion spikes are not required for Mercury. However, one typically is run if the LCS was out of control to show matrix interference.** *Use the criteria in Table 7-2 below to determine whether the data needs to be qualified. If qualification is needed, take the necessary actions listed in table.*	**Indicate yes or no:** Yes. Hg data for both batches is on page 38. **Summarize any actions taken:** N/A
7.3.2 Documenting the effect of the matrix should include the analysis of at least one matrix spike/matrix spike duplicate (MS/MSD) pair for each batch of samples processed. A separate spike sample and a separate duplicate sample may be analyzed in lieu of the MS/MSD at the analyst's discretion. *Action: Verify that at least one MS/MAD pair or an MS and a duplicate sample were prepared and analyzed from each group of samples of a similar matrix type or for each data package.*	**Indicate if an MS/MSD pair, or a MS and a duplicate were analyzed:** An MS and MSD pair were analyzed. A duplicate sample was also analyzed.

7.3.3 Are all relative percent difference (RPD) values within control limits? **Note: Acceptance criteria for RPD should be a set of laboratory-derived limits; however, acceptance limits must not exceed 20%.** *Action: Determine whether RPD values exceed laboratory-derived control limits. If control limits have not been developed, use ≤20% as the acceptance criteria.*	**Indicate yes or no:** Yes. MS/MSD pair RPDs were within control limits. The duplicate results were both ND, so an RPD was not analyzed for duplicates from either batch.
7.3.4 RPD is calculated to evaluate the spike values for precision using the following equation: $$\text{RPD} = \frac{\lvert S - D \rvert}{(\frac{S + D}{2})} \text{x}100$$ Where: S = Sample Result (original) D = Duplicate Result When the sample or duplicate result is reported as a non-detect, use a value of zero (0) for calculating the RPD. This will always yield an RPD of 200%. *Action: Verify an RPD calculation for one set of MS/MSD samples. Contact the facility or laboratory for an explanation if RPD was not calculated. If a satisfactory explanation is not available, use professional judgment to qualify sample results.*	**Show results of one verified RPD calculation:** Mercury MS: 0.0049 mg/kg Mercury MSD: 0.0052 mg/kg RPD = (|0.0049-0.0052|) / (((0.0049 + 0.0052)/2)) x100 RPD = 6 This RPD is within control limits.

Table 7-2: Matrix Spike Actions for Mercury

Criteria	Action	
	Detect	Non-detect
Matrix Spike analysis not performed at the specified frequency	J	UJ
Matrix Spike not prepared from field sample	J	UJ
Matrix Spike %R < 80%	J-	UJ
Matrix Spike %R 80-120%	No qualification	No qualification

Matrix Spike %R > 120%	J+	No qualification

Checklist #11

TCLP Data Validation

11.0 TCLP Data Validation	
Toxicity Characteristic Leaching Procedure (TCLP) is used to determine whether wastes exhibit the toxicity characteristic or whether Land Disposal Restrictions have been met. The TCLP test is specified in OAC 3745-51-24 and defined in SW-846, Method 1311. TCLP data validation requires specific data concerning preparation of the extraction procedure in addition to the usual data submitted for organic and inorganic analytical methods. In most cases, a laboratory will have to supply bench sheet data to complete data validation. The validator should consult the Tier I Data Validation Guidance Manual for specific information and examples.	
11.1 Did the laboratory calculate TCLP filterable solids? Based on the percent solid calculations, were the correct analytical procedures followed? **Note: TCLP requires that solid samples, semi solid samples and liquid samples be prepared based upon the amount of solids in the sample. For a sample that has greater than 99.5% solids, the sample is considered solid, and 100 grams of material are extracted with 20 times this weight of extraction fluid. For a sample that is equal to or less than 0.5% solids, the sample is considered a liquid and the liquid itself is considered the extract (no additional extraction fluid or tumbling is necessary). If the sample contains both solids and liquids, the solid portion, trapped by filtering, is extracted with 20 times its weight of extraction fluid and then analyzed. In addition, an aliquot of the liquid portion of the sample is analyzed. The results are then mathematically combined. Alternately, the multiphase components may be physically recombined prior to analysis.** *Action: If percent solids were not calculated, contact the applicable party for the proper information.* *If, based on the percent solids calculations, the appropriate preparation methods were not used, qualify analytical results using the following criteria:* *All detected results above the regulatory level should not be qualified.* *All results above the detection limits but below the regulatory level should be qualified based on*	**Indicate yes or no:** Yes, the laboratory calculated TCLP filterable solids. However, the correct analytical procedures were not followed. See below for more information. **Summarize any actions taken:** Percent liquids are listed on Page 41 in the TCLP logbook. Sample 028 had greater than 0.5% solids, but the laboratory treated the samples as a liquid. This is incorrect and the data is subject to qualification. Sample 028: A detected result for Cadmium (page 30) was recorded at 0.787 mg/L. This result is just below the regulatory limit of 1.0 mg/L. Since metal extraction may be significant in the solids portion of the waste, the results for Cadmium should be qualified as rejected and flagged with an “R” based on professional judgement. Barium was also detected in sample 028 (page 30); however, its results are significantly less than the regulatory limit. Based upon professional judgement, the barium result will not be qualified.

professional judgment. It may be necessary to communicate with the Tier II validator regarding qualifiers. *All non-detected results should be qualified based on professional judgment.*	
11.2 Was the proper amount of material extracted? **Note: For samples to be analyzed for metals or SVOCs (in the solid portion), a minimum of 100 grams is required. For samples to be analyzed for volatile compounds, approximately 20-25 grams of sample is required.** **Liquid samples are directly analyzed as the TCLP extract, no extraction fluid is added to the sample.** *Action: If improper sample mass is used, qualify analytical results using the following criteria:* *All detected results above the regulatory level should not be qualified.* *All results above the detection limits, but below the regulatory level should initially be qualified as "J" estimated. Based on professional judgment, qualification of data as "R" may be warranted.* *Based on professional judgment, all non-detected results should be qualified as either "J" estimated or "R."*	The proper amount was not extracted. **List sample IDs and sample mass(es) used for the extraction:** The following information is found on page 41 (TCLP Logbook): For sample 028 (1157892001), the solids portion was not separated and extracted. Batch 346202 for metals analysis: (samples 028 and RO-02) slightly more than 100 g of material was extracted. This is acceptable. Batch 347359 for VOC analysis: (sample 028 and RO-02) no information is presented on the volume of material used. The lab should be contacted and the required information for TCLP VOCs presented to the Agency. If this information is not available, the data validator should use professional judgement in qualifying sample results. The data validator may wish to reject results until information is presented from the laboratory.
11.3 Was the correct extraction fluid used? **Notes:** **Fluid # 1 is always used for VOC analysis.** **Fluid #1 should be used if the final pH of the pre-test sample is below 5.0.** **If the pH is above 5.0, hydrochloric acid should be added to the pre-test sample (refer to the method for specifics) and re-analyzed for pH.** **Fluid #2 should be used if the final pH of the pre-test is above 5.0.**	**List sample IDs and fluid type(s) used for the extraction:** Metals: Page 41 For Sample RO-02, the only solid sample, the initial pH was determined to be 8. After acidification, the final pH was 3.567. This indicates that a TCLP fluid #1 is correct. The lab used TCLP fluid #1 for extraction. This is correct. However, the pH of the buffer solution (pH 5.09) was outside of the methods requirements.

Action: Consult with the applicable party and have the extraction fluid information submitted. If the improper fluid is used, qualify analytical results using the following criteria: *All results above the regulatory level should not be qualified.* *All results above the detection limits, but below the regulatory level, should initially be qualified as "J." Rejection of data may be warranted if other preparatory procedures are outside of criteria.* *All non-detected results should be qualified as "R."*	For sample 028, no extraction fluid addition was necessary because this sample was treated as a liquid. VOCs: For VOCs, no information is presented. TCLP fluid #1 should always be used for VOC extraction. This information should be requested from the lab. If it is not available, the data validator should use best professional judgement to qualify sample results.
11.4 Did the extraction fluid have the proper pH? Fluid #1 has a pH range of 4.88 to 4.98 Fluid #2 has a pH range of 2.83 to 2.93. *Action: If an improperly prepared extraction fluid is used, qualify analytical results using the following criteria:* *All results above the regulatory level should not be qualified.* *All results above the detection limits, but below regulatory levels, should initially be qualified as estimated and flagged with a "J-." Rejection of data may be warranted if other preparatory procedures are outside of criteria.* *All results below the detection limits should be qualified as "R."*	**List incorrect fluid pH(s):** No, extraction fluid did not have the correct pH. For sample 028, the pH of the buffer solution was reported as 5.09. This is outside of the acceptance range for TCLP fluid #1. (Page 41.) All detected results below the regulatory limit should initial qualified as estimated "J-". All data below the detection limit should be rejected and results flagged with an "R." Upon review, this qualification may be changed and all detected results can be rejected.
11.5 Was the correct weight of extraction fluid used? Laboratory bench sheets may be needed to complete this section. *Action: If the extraction fluid weight is not more than + 15% of the correct value (20X the sample weight or ~2,000 grams for metals; 400 to 500 grams for VOCs), qualify all results as estimated "J" or "UJ". These values may be re-qualified if additional problems with TCLP preparation exist.*	**Indicate yes or no:** Yes. Greater than 2000g was used for TCLP metals (i.e. 20 X 100+ grams) (Page 41). There is no information on VOCs. **Summarize any actions taken:** N/A

If the extraction fluid weight is less than 70% of the proper weight, qualify all non-detect compounds and detected results below the regulatory level, as "R." All positive results above the regulatory limit will not be qualified. *If the extraction fluid weight is more than 30% greater than the proper weight, qualify all non-detect compounds and positive results below the regulatory level, as "R." All detected results above the regulatory limit will not be qualified.*	
11.6 Was a TCLP blank analyzed with every batch of samples? **Note: TCLP blanks should be prepared using the same extraction fluid as is used for the associated sample's extraction. A minimum of one blank should be analyzed for every 20 extractions.** *Action: Contact the applicable party for submittal of missing data. If a blank was not analyzed, qualify all detected results as "J+." If data is available, qualify TCLP data using the appropriate checklist for the target analytes (i.e., VOCs, SVOCs, metals, etc.).*	**List IDs of affected samples:** Yes, a TCLP blank was analyzed with each batch. TCLP VOCs blank – page 68 TCLP metals blank – page 37-38 TCLP Hg blank – page 38
11.7 Was the tumbling time within 18 +/- 2 hours? **Note: Tumbling time (evaluated based on the day and time tumbling begins/is completed) should be noted on the bench sheets. The laboratory should be contacted if this information isn't present.** *Action: If the tumbling time is not within 18 +/- 2 hours, qualify all data as "J."*	**Indicate yes or no:** Yes – 16 hours. **Summarize any actions taken:** N/A
11.8 Was the tumbler speed within 30 +/-2 revolutions per minute (RPM)? **Note: Tumbler speed should be noted on the bench sheets. The laboratory should be contacted if this information isn't present.** *Action: If the tumbler speed is not within 30 +/-2 RPM, qualify all data as "J."*	**Indicate yes or no:** Yes. Tumbling speed was 30 RPM. **Summarize any actions taken:** N/A

* Note: VOC, SVOC and Metal results from the TCLP test should meet the sample QA/QC criteria outlined in applicable checklist for each constituent.

Checklist #12

Ignitibility Data Valid

12.0 – Ignitability Data Validation	
12.1 Pensky-Martens (SW-846 Method 1010A, ASTM D93, ASTM D8174-18, and ASTM D8175-18) - Procedure A for "Ordinary Liquids"	
SW-846 Method 1010A (Pensky-Martens Closed Cup) is one way of testing methods that may be used to determine this hazardous waste characteristic. Method 1010A is the flashpoint method most often used by the RCRA program. It is used for "fuel oils, lube oils, suspensions of solids, liquids that tend to form a surface film under test conditions, and other liquids." This may include matrices like paint wastes, parts cleaners, etc. To test the flash point, "the sample is heated at a slow, constant rate with continual stirring. A small flame is directed into the cup at regular intervals with simultaneous interruption of stirring. The flash point is the lowest temperature at which application of the test flame causes the vapor above the sample to ignite." Method 1010A has two options, termed "A" and "B". Procedure A, the basic procedure, is used unless the material being tested is a suspension of solids or a highly viscous material. Those materials require the use of Procedure B. There are specific requirements and apparatus for method 1010B that are not included in this check list. These items include the recording of barometric pressure, thermometers, stirrer rates, wind shields and drying of wastes that contain free water. If necessary, specific testing requirements that are used should be discussed with the laboratory and appropriate qualifications of the data should be made.	
Pensky-Martens (SW-846 Method 1010B, ASTM D93)	
12.1.1 Was p-xylene used to calibrate the instrument?	It's not known, and p-Xylene is not listed. The LCS information is for 77°F. This is sometimes listed in MSDS sheets for p-xylene.
12.1.2 Was the flashpoint for the calibration standard p-xylene within 81 +/- 2 °F? **Note: The method specifies p-xylene with an expected flashpoint of 81°F.**	**Record the p-xylene calibration flashpoint(s):** The chemical used to calibrate the instrument had a flashpoint of 77°F. This flashpoint is just below the acceptance criteria specified within the method.
12.1.3 If the calibration standard was outside of this range (see 12.1.2), was corrective action taken? *Action: If no corrective measures were performed, determine whether a significant bias has been imparted to the samples and qualify the results using professional judgment. If sample is still available, notify the laboratory. Consult Tier II evaluator regarding requests for re-analysis.*	**Record IDs of samples that are qualified:** Although the calibration standard was out of the acceptable range, there is no indication that corrective action was taken. The lab should be notified and the Tier II evaluator should be consulted to decide whether re-analysis or qualification of the results should be performed.
12.1.4 Based on 12.1.3, if corrective measures were taken, was the p-xylene calibration flashpoint within 81 +/- 2 °F? **Note: Corrective measures should have continued until this flashpoint calibration range was attained.**	N/A

Action: If these procedures were not followed and documented, contact the laboratory for an explanation. Lack of an adequate explanation may justify qualifying the data.	
12.1.5 If a sample has an expected flashpoint, based on field or site information, measurements should begin at least 30-50°F below the expected flashpoint of the material. If the expected flashpoint is unknown, the initial measurements should begin at the ambient temperature of the laboratory. **Note: Information of the expected flashpoint of a sample should be shared with the laboratory prior to analysis.** *Action: If these procedures were not followed and documented, contact the laboratory for an explanation. Lack of an adequate explanation may justify qualifying the data.*	The result for the sample was 138°F. Assuming a room temperature of 75°F, the laboratory was in compliance. Page 14 or 16.
12.1.6 Was heat applied to raise the temperature of the sample at a rate of 9-11°F per minute? **Note: Laboratory bench sheets may be required to show the starting temperature, the starting time, the flash point (or end) temperature and the time when the flash occurred. These materials should be requested from the laboratory if not present. Documentation of start time is not specifically required per the method but should be adequately demonstrated or explained by the laboratory if not presented.** *Action: If these procedures were not followed and documented, contact the laboratory for an explanation. Lack of an adequate explanation may justify qualifying the data.*	There is insufficient information. The laboratory should be contacted. If no information is available, the review may qualify data based upon best professional judgement and the project's DQOs.
Pensky-Martens (SW-846 Method 1010B, ASTM D8174-18, and ASTM D8175-18)	
12.1.7 Was n-decane or n-undecane used to calibrate the instrument?	**Indicate yes or no:**

	It is not known. The compound used to calibrate the instrument is not provided.
12.1.8 Was the flashpoint for the calibration standard within the limits of accuracy and precision defined in the method? n-decane: Flashpoint 52.8 +/- 2.6 °C or 127°F +/- 4.7°F. n-undecane: Flashpoint 68.7 +/- 3.4 °C or 155.7°F +/- 6.1 °F. **Note: The method specifies n-decane or n-undecane. However, the method allows the use of other Certified Reference Material (CRM) or working standards. The laboratory may be consulted for information on other CRMs and working standards.**	**Indicate yes or no:** It is not known. Calibration standard information is not provided. The lab or applicable party should be contacted for this information. **Record the calibration flashpoint(s):**
12.1.9 If the calibration standards are outside or the acceptance range, was corrective action taken? *Action: If no corrective measures were performed, determine whether a significant bias has been imparted to the samples and qualify the results using professional judgment. If sample is still available, notify the laboratory regarding requests for re-analysis.*	**Indicate yes or no:** N/A **Record IDs of samples that are qualified:** N/A
12.1.10 Based on 12.1.9, if corrective measures were taken, was the n-decane or n-undecane flashpoint within the acceptance range specified in 12.1.8? **Note: Corrective measures should have continued until this flashpoint calibration range was attained.** *Action: If these procedures were not followed and documented, contact the laboratory for an explanation. Lack of an adequate explanation may justify qualifying the data. Consider rejecting data if the sample flashpoints were outside of the regulatory range (I.e., 40 to 140* °F)	**Indicate yes or no:** N/A **Summarize any action taken:**
12.1.11 Was a run log of analyses associated with the sample(s) provided?	**Indicate yes or no:** Yes, see page 16.

Note: The RCRA program requires a run log of analyses associated with the sample(s), including the certified reference material standard data, working standards, initial sample flashpoint and confirmation sample flashpoint. Additional data should include verification of heating rate and stirring RPMs. If this information is not present, contact the laboratory for additional data. *Action: If documentation is not forthcoming, lack of an adequate explanation may justify qualifying the data. If the flashpoint is outside the regulatory range, consider reanalysis if there is sufficient sample remaining or qualify data based on professional judgement. If data is within the regulatory range, no qualification is necessary.*	**Summarize any action taken:** N/A
12.1.12 Was heat applied to raise the temperature of the sample at a rate of 10-16°F per minute? **Note: Laboratory bench sheets may be required to show the starting temperature, the starting time, the flash point (or end) temperature and the time when the flash occurred. These materials should be requested from the laboratory if not present. Documentation of start time is not specifically required per the method but should be adequately demonstrated or explained by the laboratory if not presented.** *Action: If these procedures were not followed and documented, contact the laboratory for an explanation. Lack of an adequate explanation may justify qualifying the data.*	**Indicate yes or no:** There is insufficient information. **Summarize any action taken:** The laboratory should be contacted. If no information is available, the review may qualify data based upon best professional judgement and the project's DQOs
12.1.13 Was the flashpoint of the unknown sample(s) repeated and within the precision limits set by the laboratory?	**Indicate yes or no:** No. There is no indication based on the results table and run log that the flashpoint was repeated. Sample 002 flashed at 138°F

Note: Samples that flash below 140 °F must be verified. The laboratory should allow the device to cool down below 20 °F of the first flashpoint and the analysis repeated. The second flashpoint should be within the analytical precision criteria set by the laboratory. If the first results cannot be verified, additional analytical runs should be performed for verification. *Action: If these procedures were not followed and documented, contact the laboratory for an explanation. Lack of an adequate explanation may justify qualifying the data.*	**Summarize any action taken:** The laboratory should be contacted to explain why the sample was not verified by completing a second flashpoint.

Note: The Setaflash section of Checklist #12 is not shown because this laboratory report does not include results from that ignitability method.

Appendix C
Lab Inquiry Template Letter

Date

Name
Address

City, State, Zip

Dear ____;

The Ohio EPA Division of Environmental Response and Revitalization (DERR) reviews analytical data as part of its quality assurance program. This program requires that analytical data that is submitted to the agency be reviewed for completeness and to determine whether the data quality objectives for which the data was generated have been met. One aspect of this program is an assessment of the validity of analytical data. Data validation at the Tier I level is performed using the procedures and methods outlined in Ohio EPA's Tier I Data Validation Manual and Tier I Data Validation Checklists.

The Ohio EPA has identified that certain information is absent from the following data report thatprevents us from completing a data review. The data report in question contains the following information that will allow you to identify the correct data package and to identify the necessary information.

Facility Name: Sample Report:

Sampling Date: Sample ID(s):

Lab Sample ID(s): _____

Please provide the following data or information was not included within the data report.

We wish to thank you for your prompt attention to this matter so that the Ohio EPA can complete this data review. Please don't hesitate to call us at (Phone #) if you need any clarification on our data requirements.

Thank you.

The following list of commonly omitted data quality parameters can assist the Ohio EPA personnel in preparing the boilerplate letter.

Completeness

- missing chain of custody
- missing sample receipt form or no indication that samples were received intact andproperly preserved.
- missing data results
- missing quality control criteria, such as reporting limit, detection limit, surrogaterecovery criteria, etc.
- missing laboratory QA officer signature
- missing batch ID (analytical and/or preparatory)
- missing data narrative
- missing field ID/laboratory ID correspondence sheet
- missing analytical method numbers (*i.e*., from SW-846, ASTM, etc.)

TCLP Preparation

- missing TCLP fluid determination data.
- missing TCLP Fluid pH
- missing sample volume
- missing extraction Fluid Volume
- missing time on and off the tumbler
- missing temperature data
- missing final extraction pH
- missing % solids and/or multi-phasic wastes information

Flashpoint

- missing bench sheet documentation, possibly including:
    - start and finish times of the analytical run
    - flashpoint duplicate results for samples
    - indication of standard used and/or results obtained
- pH
    - calibration standard and results information

Batch Quality Control

- missing blank data
- missing matrix spike/matrix spike duplicate data
- missing laboratory control sample (LCS) results
- missing dilution factor

Organic Analysis Criteria

- missing surrogate recovery data (or acceptable recovery ranges)
- missing required Base/Neural or Acid spikes in the LCS

Inorganic Analysis Criteria

- missing pre-digestion spike recovery data
- missing post-digestion spike data

- missing data for the Method of Standard Additions (MSA)