




LABORATORY OPERATIONS



January 10, 2022



AGENDA

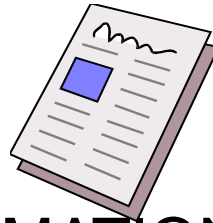
- ❑ LABORATORY OPERATIONS OVERVIEW
- ❑ SAMPLING
- ❑ SAMPLE PREPARATION
- ❑ ANALYTICAL METHODS
- ❑ ANALYTICAL QUALITY CONTROL
- ❑ ANALYTICAL REPORT
- ❑ TCLP – EPA METHOD 1311 - “Rule of 20”

LABORATORY OPERATIONS OVERVIEW



LABORATORY OPERATIONS OVERVIEW

❑ ANALYSIS REQUESTED



- INFORMATION REQUIRED
- ACCEPTED/ NOT ACCEPTED

LABORATORY OPERATIONS OVERVIEW



❑ **SAMPLE RECEIPT**

- PROCESS

❑ **CHAIN OF CUSTODY**

- INFORMATION REQUIRED

❑ **PRESERVATIVES**

- TEMPERATURE; CHEMICAL



LABORATORY OPERATIONS OVERVIEW

Toxicity Characteristic

- A solid waste (except manufactured gas plant waste) exhibits the characteristic of toxicity if, using the Toxicity Characteristic Leaching Procedure (TCLP), SW-846 Test Method 1311, the extract from a representative sample of the waste contains any of the contaminants listed in 40 CFR §261.24 Table 1 at the concentration equal to or greater than the respective regulatory level listed.
- TCLP is used to determine if a waste material will leach these chemicals into the soil or groundwater. A waste material has the characteristic of “toxicity” if it is able to leach specific metals and organic compounds into the soil or groundwater resulting in specific levels.
- A Material Safety Data Sheet (MSDS), prepared by the supplier or manufacturer of a material, provides health and safety information on the potential hazards (health, physical, chemical environmental) and how to work safely with the material. An MSDS cannot be used to assess if a material is hazardous as defined in 40 CFR §261.24.

LABORATORY OPERATIONS OVERVIEW

SAMPLE PREPARATION

- ❑ TCLP Method 1311 for Metals, Volatiles and Semi-Volatiles
- Followed by
 - EXTRACTION for semi-Volatiles (SW-846 3500 series)
 - DIGESTION for Metals (SW-846 3000 series)



LABORATORY OPERATIONS OVERVIEW

SAMPLE PREPARATION

■ **GOAL**

THE MAIN OBJECTIVE OF ANY SAMPLE PREPARATION TECHNIQUE IS TO SEPARATE THE CHEMICAL ANALYTE(S) OF INTEREST FROM OTHER CHEMICAL ANALYTES THAT COULD INTERFERE WITH THE ANALYTE DETERMINATION



LABORATORY OPERATIONS OVERVIEW

SAMPLE PREPARATION

❑ **INTERFERENCES**

A CHEMICAL INTERFERANT IS ONE THAT WILL CAUSE THE FINAL ANALYTICAL RESULT TO BE TOO HIGH OR TOO LOW COMPARED TO ITS "TRUE" VALUE



LABORATORY OPERATIONS OVERVIEW

SAMPLE PREPARATION

❑ **EXTRACTION**

- AN ORGANIC SOLVENT
(e.g., METHYLENE CHLORIDE, HEXANE)
IS ADDED TO APPROXIMATELY ONE
LITER OF WATER SAMPLE. THE
ORGANIC ANALYTES "LEAVE" THE
WATER PHASE AND "ENTER" THE
SOLVENT PHASE
- OLD CHEMISTRY PRINCIPLE: "LIKE
DISSOLVES LIKE"

SAMPLE PREPARATION

■ **DIGESTION - METALS**

- MIXTURE OF ACIDS ARE ADDED TO ABOUT 50 mL OF SAMPLE AND REFLUXED AT A TEMPERATURE OF ABOUT 90°C

- THE DIGESTION PROCESS PRODUCES "FREE" DISSOLVED METALS IN SOLUTION

LABORATORY OPERATIONS OVERVIEW

SAMPLE ANALYSIS

❑ MEASURES THE INSTRUMENT RESPONSE OF SEVERAL ANALYTES, SEQUENTIALLY OR SIMULTANEOUSLY, AND REPORTS A SEPARATE VALUE FOR EACH ANALYTE

- Volatiles (SW-846 8260D)
- Semi-Volatiles (SW-846 8270E)
- Pesticides (SW-846 8081B)
- Metals (SW-846 6010D/6020B)
- Mercury (SW-846 7470A)



LABORATORY OPERATIONS OVERVIEW

Quality Control

- Laboratory Control Sample
- Matrix Spike (required by EPA TCLP 1311)
- Sample Duplicate
- Surrogates
- Method Blank (required by EPA TCLP 1311)

FINAL REPORT

❑ FORMAT OF FINAL REPORT

- SUMMARY OF RAW DATA
- ELECTRONIC VERSION
- RAW DATA



LABORATORY OPERATIONS OVERVIEW

FINAL REPORT

- Method(s) Used
- Field Sample ID & location
- Sample Collection Date/Time
- Unique Laboratory ID
- Results
- Measurement Units, e.g., mg/L, mg/Kg
- Reporting Limit – RL, DL
- Qualifiers (data flags)
- Case Narrative



LABORATORY OPERATIONS OVERVIEW

Data Qualifiers

- Data Qualifiers are added by the laboratory
- Data Qualifiers are generally applied when sample preservation or quality control results were not within specifications.
- Common Laboratory Qualifiers:
 - U/ND = Not Detected
 - B = Analytes are detected in the blank
 - J = Estimated Result
 - Q = Bias High/Bias Low Result

CASE NARRATIVE

- Identifies any issues with sample receipt
- Identifies any anomalies with sample preparation and analysis
- Identification of any QC failures and impact to results
- Any other factors that could affect the sample results (e.g., Unique characteristics of sample matrix, multi-phasic samples,



UNITED STATES ENVIRONMENTAL PROTECTION AGENCY

Region 2 Laboratory
2890 Woodbridge Avenue
Edison , New Jersey 08837
732-906-6886 Phone
732-906-6165 Fax

December 02, 2015



RE: [REDACTED]

Enclosed are the results of analyses for samples received by the laboratory on 10/29/2015. The signature below reflects the laboratory's approval of the reported results. If you have any questions concerning this report, please refer to Project Number 1510057 and contact the laboratory.

Sincerely,

A handwritten signature in black ink, reading "John R. Bourbon", is positioned above the typed name.

John R. Bourbon
Chief, DESA/LB



UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
Region 2 Laboratory

Final Report

Project: [REDACTED] - 1510057

Project Number: 1510057

Project Narrative:

The National Environmental Laboratory Accreditation Conference Institute (TNI) is a voluntary environmental laboratory accreditation association of State and Federal agencies. TNI established and promoted a National Environmental Laboratory Accreditation Program (NELAP) that provides a uniform set of standards for the generation of environmental data that are of known and defensible quality. The EPA Region 2 Laboratory is NELAP accredited. The Laboratory tests that are accredited have met all the requirements established under the TNI Standards.

Condition Comments

None

Comment(s):

The samples were not maintained at 4 degrees C as the original analysis was completed within the standard analysis time and they were placed on a shelf at room temperature for disposal 30 days after the initial report was submitted.

The "Sample Analysis Date and Time" is included in the results section for any analyte with a prescribed holding time of 72 hours or less.

Data Qualifier(s):

- U- The analyte was not detected at or above the Reporting Limit.
- J- The identification of the analyte is acceptable; the reported value is an estimate.
- K- The identification of the analyte is acceptable; the reported value may be biased high.
- L- The identification of the analyte is acceptable; the reported value may be biased low.
- NJ- There is presumptive evidence that the analyte is present; the analyte is reported as a tentative identification. The reported value is an estimate.

Reporting Limit(s):

The Laboratory was able to achieve the appropriate limits for each analyte requested.



UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
Region 2 Laboratory

Final Report

Project: [REDACTED] - 1510057

Project Number: 1510057

SUMMARY REPORT FOR SAMPLES

Field ID	Laboratory ID	Matrix	Date Sampled	Date Received
P001-J15-S-0001-01	1510057-01	Solid	05/19/2015 09:59	10/29/2015 17:40
P001-A05-S-1824-01	1510057-02	Solid	05/18/2015 09:40	10/29/2015 17:40
P001-G15-S-0612-01	1510057-03	Solid	05/19/2015 11:14	10/29/2015 17:40
P001-B02-S-1824-01	1510057-04	Solid	05/18/2015 11:50	10/29/2015 17:40



UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
Region 2 Laboratory

Final Report

Project: [REDACTED] - 1510057

Project Number: 1510057

SUMMARY REPORT FOR METHODS

Analysis	Method	Certification	Matrix
Mercury	EPA 245.1 SOP C-110 Rev2.4	NELAP	Solid
Metals ICP TCLP	EPA 200.7 SOP C-109 Rev3.3	NELAP	Solid



UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
Region 2 Laboratory

Final Report

Project: [REDACTED] - 1510057

Project Number: 1510057

Analyte	Result	Qualifier	Reporting Limit	Units	Date and Time of Analysis*
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Field ID: P001-J15-S-0001-01

Sample ID: 1510057-01

Metals TCLP ICP

Arsenic	---	U	0.08 0	mg/L
Barium	---	U	1.0	mg/L
Cadmium	0.056		0.03 0	mg/L
Chromium	---	U	0.05 0	mg/L
Lead	0.24	J	0.08 0	mg/L
Selenium	---	U	0.20	mg/L
Silver	---	U	0.05 0	mg/L

Mercury CVAA

Mercury	---	U	0.00 2	mg/L
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Field ID: P001-A05-S-1824-01

Sample ID: 1510057-02

Metals TCLP ICP

Arsenic	---	U	0.08 0	mg/L
Barium	---	U	1.0	mg/L
Cadmium	0.85		0.03 0	mg/L
Chromium	---	U	0.05 0	mg/L
Lead	16		0.08 0	mg/L
Selenium	---	U	0.20	mg/L
Silver	---	U	0.05 0	mg/L

Mercury CVAA

Mercury	---	U	0.00 2	mg/L
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Field ID: P001-G15-S-0612-01

Sample ID: 1510057-03

Metals TCLP ICP

Arsenic	---	U	0.08 0	mg/L
Barium	1.4		1.0	mg/L
Cadmium	---	U	0.03 0	mg/L
Chromium	---	U	0.05 0	mg/L

LABORATORY OPERATIONS OVERVIEW

TCLP METHOD 1311

From the Method:


- 1.1 The TCLP is designed to determine the mobility of both organic and inorganic analytes present in liquid, solid, and multiphasic wastes.
- 1.2 If a Total Analysis of the waste demonstrates that individual analytes are not present in the waste, or that they are present but at such low concentrations that the appropriate regulatory levels could not possibly be exceeded, the TCLP need not be run.
- Total Analysis: The “total” waste analysis determines the total concentration of a contaminant present in a sample. This analysis can be used as a screening tool during a hazardous waste determination and may eliminate the need for additional testing in some cases.

LABORATORY OPERATIONS OVERVIEW

TCLP METHOD 1311

“Rule of 20”

- For solid materials/samples, The TCLP procedure specifies to mix a measured amount of solid sample with a volume of extraction fluid that equals 20 times the weight of the sample
- If the contaminant in the sample is completely leached into the extraction fluid during the tumbling cycle, then the concentration of the contaminant in the TCLP extract will be 20 times less than its original concentration in the sample, because it is diluted to 1/20th of its original concentration.
- This required 20:1 dilution ratio of the extraction fluid to the sample allows generators to use another option to determine if a waste is hazardous, using the total waste analysis result and the “Rule of 20” concept from the TCLP procedure



LABORATORY OPERATIONS OVERVIEW

TCLP METHOD 1311

“Rule of 20”

- The total concentration results can be divided by 20 and compared to the regulatory concentrations on the TCLP list. If the result is less than the TCLP regulatory limit (for each respective TCLP constituent of concern) then the waste is not a “toxicity characteristic” hazardous waste
- If your total analysis results are equal to more than 20 times the TCLP limits the waste may be a hazardous waste and should be handled as such until further testing, using the TCLP analysis, confirms the waste’s toxicity characteristic results.




LABORATORY OPERATIONS OVERVIEW

TCLP METHOD 1311

“Rule of 20”

METAL	TC Regulatory Limit (mg/L)	TC Regulatory Limit x20 (mg/Kg)
Arsenic	5.0	100
Barium	100	2000
Cadmium	1.0	20
Chromium	5.0	100
Lead	5.0	100
Mercury	0.2	4
Selenium	1.0	20
Silver	5.0	100




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TCLP METHOD 1311

“Rule of 20”

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- If your total analysis results are equal to more than 20 times the TCLP limits the waste may be a hazardous waste and should be handled as such until further testing, using the TCLP analysis, confirms the waste’s toxicity characteristic results.



LABORATORY OPERATIONS OVERVIEW

TCLP METHOD 1311

“Rule of 20”

- Lead - Total Analysis result of 50 ppm. Applying the “Rule of 20”, 50 ppm (mg/Kg) is divided by 20 resulting in a TC level of 2.5 ppm (mg/L). TC level for Lead is 5.0 ppm (mg/L). The converted result is well below the TC regulatory level; the TCLP need not be run.
- Lead - Total Analysis result of 200 ppm. Applying the “Rule of 20”, 200 ppm (mg/Kg) is divided by 20 resulting in a TC level of 10.0 ppm (mg/L). TC level for Lead is 5.0 ppm (mg/L). The converted result is well above the TC regulatory level; the TCLP needs to be run
- Lead - Total Analysis result of 90 ppm. Applying the “Rule of 20”, 90 ppm (mg/Kg) is divided by 20 resulting in a TC level of 4.5 ppm (mg/L). TC level for Lead is 5.0 ppm (mg/L). The converted result is very close to the TC regulatory level. Does TCLP need to be run? You decide
- From the method ... or that they are present but at such low concentrations that the appropriate regulatory levels **could not possibly be exceeded**, the TCLP need not be run.



LABORATORY OPERATIONS OVERVIEW

Questions?

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