Remedial Action Plan Quality Assurance Project Plan Addendum, 43 Hempstead Street, New London, Connecticut

Prepared for: **City of New London**
13 Masonic Street
New London, Connecticut

United States Environmnetnal Protection Agency, Region 1
5 Post Office Square – Suite 100
Boston, Massachusetts

Prepared by:
Vanasse Hangen Brustlin, Inc.
100 Great Meadow Road – Suite 200
Wethersfield, Connecticut

EPA Tracking Number: 24055
Grant Number: 00A00828

March 13, 2025

Table of Contents

SECTION A
Title and Approval Page/Introduction

Project Title

Remedial Action Plan Quality Assurance Project Plan (QAPP)
43 Hempstead Street, New London, Connecticut

Preparer, Dates, and Signatories

Prepared for:

City of New London
13 Masonic Street
New London, Connecticut

And

United States Environmnental Protection Agency, Region 1
5 Post Office Square – Suite 100
Boston, Massachusetts

Prepared by:

Vanasse Hangen Brustlin, Inc. (Neal Hulstein)
100 Great Meadow Road – Suite 200
Wethersfield, Connecticut

Plan Date:
March 13, 2025

Project Manager (VHB): Pamela Lind

Signature

Printed Name/Date: Pamela Lind, LEP, March 13, 2025

Project QA Officer (VHB): Amy Vaillancourt

Signature

Printed Name/Date: Amy Vaillancourt, LEP, March 13, 2025

U.S. EPA QA Officer:

Signature

Printed Name/Date: Emily Ambeliotis

U.S. EPA Project Officer:

Signature

Printed Name/Date: Katy Deng

Introduction

Vanasse Hangen Brustlin, Inc. (VHB) has prepared this Remedial Action Plan (RAP) Project-Specific Quality Assurance Project Plan (QAPP) on behalf of the City of New London (City) as part of their EPA Brownfields Cleanup Grant for 43 Hempstead Street, New London, Connecticut (Site).

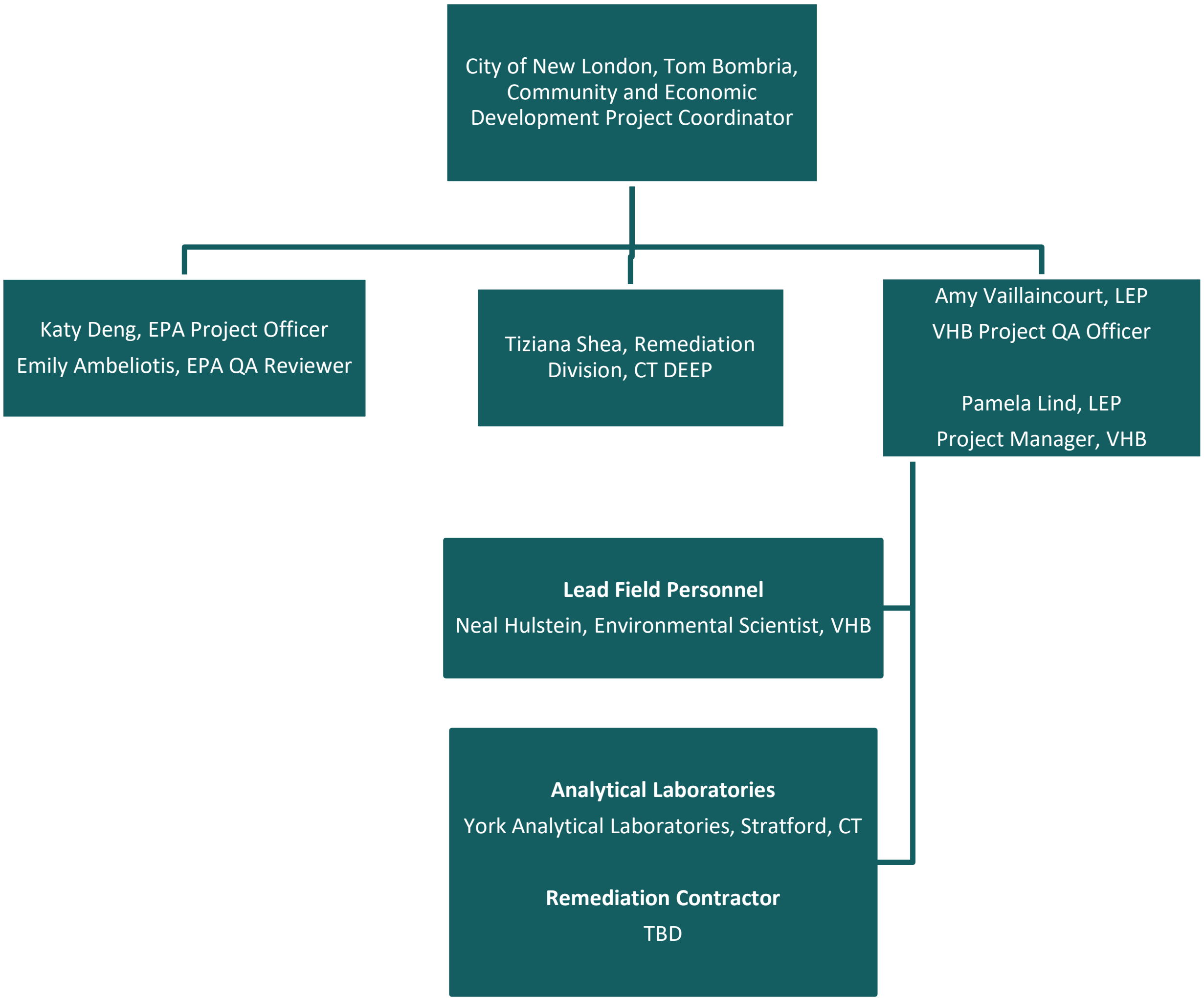
VHB's RAP QAPP was prepared in accordance with the U.S. Environmental Protection Agency (EPA) Brownfields program and associated guidance and technical documents. The RAP QAPP is an addendum to VHB's initial Site Specific QAPP dated December 1, 2023, Revised January 5, 2024.

VHB understands that approval of the RAP QAPP must be given by the EPA Project Officer prior to commencement of any Site work. All the work described in the RAP QAPP will be performed in accordance with the processes and procedures outlined in this RAP QAPP and what is referenced in the Site Specific QAPP previously approved by EPA.

VHB completed a Phase I Environmental Site Assessment (ESA) for the Site in October 2023 and performed a Phase III ESA / Remedial Design Investigation (RDI) in March 2024. Based on the findings of the Phase III ESA / RDI and conversations with EPA and Connecticut Department of Energy and Environmental Protection (CTDEEP), VHB completed a Supplemental Phase III ESA / RDI to further delineate PFAS contamination identified and install additional groundwater monitoring wells. After all investigations were performed, VHB completed a Remedial Action Plan (RAP) and Analysis of Brownfield Cleanup Alternatives (ABCA) dated March 10, 2025. Public outreach and meetings were held in association with the RAP and ABCA to keep the public informed.

SECTION B
Project Organization and Responsibility

The following organizational chart identifies the chain of command of key personnel for the project.



SECTION C
Problem Definition

Site Description

Refer to VHB's Site Specific QAPP dated December 1, 2023, Revised January 5, 2024 for information related to the Site Description.

Environmental Setting

Refer to VHB's Site Specific QAPP dated December 1, 2023, Revised January 5, 2024 for information related to the Environmental Setting.

VHB's Previous Investigations

Refer to VHB's Site Specific QAPP dated December 1, 2023, Revised January 5, 2024 and VHB's Project Specific QAPP dated May 28, 204, Revised June 20, 2024 for information related to the previous VHB Phase I ESA and Phase III Environmental Site Assessment / Remedial Design Investigation performed.

VHB performed an additional **Supplemental Phase III Environmental Site Assessment / Remedial Design Investigation** dated November 15, 2024. Based on the investigation, the following conclusions and recommendations were made:

- No further investigation was recommended.
- Development of a Remedial Action Plan (RAP) and Analysis of Brownfields Cleanup Alternatives (ABCA) was recommended prior to remediation of the Site.
- Removal of the UST(s) and proper tank closure and confirmatory sampling is recommended during the remedial process.

VHB completed a **Remediation Action Plan / Analysis of Brownfields Cleanup Alternatives** dated March 10, 2025. Based on the RAP/ABCA, cost alternative #2 was selected that includes UST removal, targeted hotspot soil removal, and removal of 2 feet of soil site wide with an estimated fee between approximately $1.05 million (non-hazardous soils) and $2.61 million (hazardous soils). The public was engaged during the RAP/ABCA public comment period and VHB completed a public meeting to address any questions. The City of New London addressed various questions during various council meetings. Additionally, answers to questions were included in the finalized RAP prior to submittal to the CTDEEP.

Purpose

The Site has been awarded a $615,000 Brownfield Cleanup Grant from the U.S. EPA to remediate and redevelop the Site into a public park. Additionally, the City of New London has committed $123,000 for the purpose of remediation and redevelopment. It is VHB's understanding, based on a conceptual site plan by Vita Nuova, that the park will consist of a grassy field, playground, pavilion, community garden, and ornamental trees.

Once the RAP QAPP is approved, the City of New London will solicit bids to complete the remediation at 43 Hempstead Street, New London, Connecticut. This RAP QAPP provides information on the remediation to be performed, which was based on alternative #2 from the RAP/ABCA completed. Completing remediation will bring the Site into compliance with CTDEEP Remediation Standard Regulations (RSRs).

SECTION D
Project Description and Project Timeline

Project Objectives

The project objective outlined in this RAP QAPP includes the steps to complete remediation. With completion of remediation at the Site, the Site will be brought into compliance with the CTDEEP RSRs. The intended future use of the Site upon completion of remedial activities will eventually include the development of a public park for the City of New London.

Permits

No state or local permits are required prior to initiating the proposed activities.

Call Before You Dig

Prior to conducting field activities, VHB will coordinate with the remediation subcontractor to ensure that the Call-Before-You-Dig (CBYD) is performed (as required by law). The CBYD marks out underground utilities entering the Site.

Previous investigations, including the 2021 TBA, had a ground penetrating radar (GPR) scan performed. The 2021 TBA report included a copy of the GPR scan performed with all known anomalies. This report will be utilized during the remediation to identify/locate various anomalies.

Remedial Action Plan

This Remedial Action Plan QAPP includes the remediation actions that are slated to be completed including underground storage tank (UST) removals, targeted soil excavations, site wide soil excavation, end point sampling, and waste characterization sampling. Refer to Section E for additional details on the remediation. The goal of the Remedial Action is to complete Site remediation, obtain an LEP Verified Site, to comply with the CTDEEP RSRs, and to have a Site ready for the development into a public park for the City.

Project Reporting

Following the completion of the sampling activities, VHB will prepare a Site Verification report. The report will provide a summary of the completion of remediation performed including summary of the sampling activities, pertinent findings, and comparison to sample analytical results to applicable CTDEEP Remediation Standard Regulation (RSR) criteria. Copies of analytical reports, sample location maps, and summary tables will be provided with the report.

Project Timeline

A project timeline is provided below. No field work will be conducted until approval of the QAPP has been granted by the EPA Project Officer.

Task/Sub-Tasks	**Start -Completion Dates**
RAP QAPP Review/Final Approval	March 13 – April 11, 2025
Contractor Bidding/Selection Approval	April 14 – May 30, 2025
Remedial Action by Contractor	June 2 – July 31, 2025
Post Remediation Groundwater Sampling (Q3)	August 4, 2025
Data Review / Validation	August 4 – 22, 2025
Post Remediation Groundwater Sampling (Q4)	October 6, 2025
Post Remediation Groundwater Sampling (Q1)	January 5, 2026
Post Remediation Groundwater Sampling (Q2)	April 1, 2026
Verification Report	May 1, 2026

Client Review of Report	May 15, 2026
Submit Verification Report to EPA/CTDEEP	June 1, 2026

SECTION E
Sampling Design and Site Figures

The Remedial Action will take place over the course of up to two months plus post-remediation compliance groundwater sampling quarterly for four quarters. The proposed remediation, described in the RAP/ABCA, includes the following work:

- Mobilization and site preparation by selected contractor for remedial work
- Excavation and proper off-site disposal of contaminated soils, closure sampling, and waste characterization sampling for three (3) USTs
- Targeted soil removal, confirmation sampling, and waste characterization sampling of:
  - PFAS impacted soil
  - Lead impacted soil
- Contaminated fill removal (2′ site wide), confirmation sampling (in 50′ increments starting from property boundary), waste characterization sampling, and installation of a demarcation layer
- Surface restoration via placement of clean fill, compaction, and hydroseeding

Confirmation samples will be based on Site specific or release specific constituents of concern. Waste characterization samples will be based on Site specific constituents of concern and requirements set forth by the facility receiving the contaminated soil.

Groundwater samples will be collected based on Site specific constituents of concern from five (5) overburden groundwater monitoring wells.

Field sampling quality assurance will include the collection of duplicate and trip blank samples. Quality control checks on field activities will be performed to ensure collection of data that is representative and valid. Laboratory quality assurance measures will also be provided including use of CTDEEPs Reasonable Confidence Protocols (RCPs).

Once laboratory results are received, they will be compared to CTDEEP’s RSRs which define the cleanup standards applicable for the state. The lowest technically practicable laboratory quantitative detection limits for contaminants will be used for sampling conducted during this project.

SECTION F
Sampling and Analytical Methods Requirements

UST Removal

It is anticipated that three (3) USTs (A1, A2, A3) will be removed. VHB will collect five (5) confirmation soil samples (four sidewall and one grave from each tank grave) for the following constituents of concern (COCs) per soil sample:

> Semi-volatile Organic Compounds (SVOC) Polycyclic Aromatic Hydrocarbons (PAH) Target List via SW-846 Method 8270D and EPA Method 625.1
>
> Connecticut Extractible Total Petroleum Hydrocarbons (ETPH) via CTDEEP ETPH Method
>
> CT 15 Metals via SW-846 Update V Method 6010D and EPA Method 200.8

Lead Impacted Soils

It is anticipated that approximately 60 cubic yards of lead impacted soil will be removed. A total of two (2) confirmation samples will be collected and analyzed for:

> Semi-volatile Organic Compounds (SVOC) Polycyclic Aromatic Hydrocarbons (PAH) Target List via SW-846 Method 8270D and EPA Method 625.1
>
> Connecticut Extractible Total Petroleum Hydrocarbons (ETPH) via CTDEEP ETPH Method
>
> CT 15 Metals via SW-846 Update V Method 6010D and EPA Method 200.8

PFAS Impacted Soils

It is anticipated that approximately 200 cubic yards of PFAS impacted soil will be removed. A total of six (6) confirmation samples will be collected and analyzed for:

> Semi-volatile Organic Compounds (SVOC) Polycyclic Aromatic Hydrocarbons (PAH) Target List via SW-846 Method 8270D and EPA Method 625.1
>
> Connecticut Extractible Total Petroleum Hydrocarbons (ETPH) via CTDEEP ETPH Method

- CT 15 Metals via SW-846 Update V Method 6010D and EPA Method 200.8
- Per- and Polyfluoroalkyl Substances (PFAS) by EPA Method 1633

Site-Wide Polluted Fill Material

It is anticipated that approximately 1,850 cubic yards of contaminated fill will be removed. A total of sixteen (16) confirmation samples will be collected based on collected samples every 50′. Samples will be collected and analyzed for:

- Semi-volatile Organic Compounds (SVOC) Polycyclic Aromatic Hydrocarbons (PAH) Target List via SW-846 Method 8270D and EPA Method 625.1
- Connecticut Extractible Total Petroleum Hydrocarbons (ETPH) via CTDEEP ETPH Method
- CT 15 Metals via SW-846 Update V Method 6010D and EPA Method 200.8

Waste Characterization Sampling

Based on the remedial approach, it is anticipated that twelve (12) waste characterization samples will be collected. Sample analytical is dependent upon acceptance into a soil disposal facility. However, it is anticipated the samples will have to be analyzed for the following:

- Volatile Organic Compounds (VOCs) via SW-846 Method 8260D
- Semi-volatile Organic Compounds (SVOC) Polycyclic Aromatic Hydrocarbons (PAH) Target List via SW-846 Method 8270D and EPA Method 625.1
- Connecticut Extractible Total Petroleum Hydrocarbons (ETPH) via CTDEEP ETPH Method
- CT 15 Metals via SW-846 Update V Method 6010D and EPA Method 200.8
- Polychlorinated Biphenyls (PCBs) via SW-846 Method 3540C: Soxhlet Extraction
- Flashpoint via SW-846 1010A
- Reactivity via SW-846 Ch 7.3.3
- Corrosivity via SW-846 9040C and 9045D and SM 4500 H^+B
- Paint Filter Test via SW-846 9095B
- Toxicity Characteristic Leaching Procedure (TCLP) via SW-846 1311 or 1312, Modified Morgan and ASTM D3987 (analyzed on metals and PCBs)

Compliance Groundwater Sampling

A total of 24 groundwater samples (5 wells per quarter over four quarters plus one duplicate per quarter) are expected to be collected. Samples collected will be analyzed for one or more of the following COCs:

- Volatile Organic Compounds (VOCs) via SW-846 Method 8260D
- Semi-volatile Organic Compounds (SVOC) Polycyclic Aromatic Hydrocarbons (PAH) Target List via SW-846 Method 8270D and EPA Method 625.1
- Connecticut Extractible Total Petroleum Hydrocarbons (ETPH) via CTDEEP ETPH Method
- CT 15 Metals via SW-846 Update V Method 6010D and EPA Method 200.8

- PFAS via EPA Method 1633 Draft 3

Analytical methods are identified on **Table 1**, below.

During the groundwater sampling event, samples will be collected using a peristaltic pump and stabilization parameters recorded via a YSI water quality meter. Sampling will be conducted in accordance with EPA Low Flow Purging and Sampling Procedures. Physical characteristics of groundwater samples will be recorded along with the stabilization parameters, which are turbidity, dissolved oxygen, specific conductance, temperature, pH, and ORP/Eh.

Standard Operating Procedures (SOPs) pertaining to various sample collection methodologies, field screening protocols, field notes, sample labeling, and handling, and equipment decontamination are provided in subsequent sections of this QAPP.

The following sample holding times shall be used as a guide for specific analytical methods, as shown in **Table 1**.

Table 1: Analytical Methods, Containers, Preservation, and Holding Time Requirements

Analytical Parameters	**Matrix**	**Analytical SOP**	**Containers**	**Preservation Requirements**	**Maximum Holding Times**
VOCs	Aqueous	1b	2 X 40 ml VOA (no headspace)	1:1 HCl, 4ºC	14 Days
	Soil / Solid - Low Level	1b	2 X 40 ml VOA	Stir Bar, Freeze	14 Days
	Soil / Solid - High Level	1b	1 X 40 ml VOA	Methanol, 4ºC	14 Days
PAHs (subset of SVOCs)	Soil / Solid	2b	1 X 8 oz glass	Non-Preserved, 4ºC	14 Days
	Aqueous	2b	1 X 1 amber liter glass bottle	Non-Preserved, 4ºC	7 Days to extract / 40 Days to analyze
CT ETPH	Soil / Solid	2c	1 X 4 oz glass	Non-Preserved, 4ºC	14 Days to extract / 40 Days once extract to analyze
	Aqueous	2c	1 X 1 amber liter glass bottle	Non-Preserved, 4ºC	7 Days to extract / 40 Days to analyze
PCBs	Soil / Solid	4b	1 X 4 oz glass	Non-Preserved, 4ºC	14 Days
	Aqueous	4b	1 X 1 amber liter glass bottle	Non-Preserved, 4ºC	1 Year
Metals (Total)	Soil / Solid	5b, 6b	1 X 8 oz glass	Non-Preserved, 4ºC	180 Days / 28 Days (Mercury)
Metals (Total)	Aqueous	5b, 6b	1 X 250 ml plastic	HNO3, 4ºC	6 Months / 28 Days (Mercury)
Pesticides	Soil / Solid	8b	1 X 4 oz glass	Non-Preserved, 4ºC	14 Days
PFAS	Soil / Solid	10b	1 x 125 ml polypropylene	Non-Preserved, 4ºC	14 Days
	Aqueous	10b	1 X 250 ml high density polyethylene	Non-Preserved, 4ºC	14 Days
1,4-Dioxane	Soil / Solid	11b	1 X 40 ml VOA	Methanol, 4ºC	14 Days
	Aqueous	11b	2 X 40 ml VOA	Non-Preserved, 4ºC	14 Days
Metals (TCLP)	Soil/Solid	12b	1x 9 oz glass	Non-preserved, 4ºC	180 Days / 28 Days (Mercury)
PCBs (TCLP)	Soil/Solid	12b	1 X 9 oz glass	Non-Preserved, 4ºC	14 Days
Paint Filter Test	Soil/Solid	13b	1 x 9 oz glass / 250 mL plastic	Non-Preserved, 4ºC	n/a
Corrosivity	Soil/Solid	14b	1 x 9 oz glass	Non-Preserved, 4ºC	24 Hours
Flashpoint	Soil/Solid	15b	1 x 9 oz glass	Non-Preserved, 4ºC	n/a
Reactivity	Soil/Solid	16b	1 x 9 oz glass	Non-Preserved, 4ºC	14 Days

Refer to Method SOP Reference **Table 2** in **Section G** of VHB’s Site Specific QAPP dated December 1, 2023, Revised January 5, 2024. Information provided in **Table 2** is also applicable to blanks and duplicate samples. Laboratory SOPs have been provided in **Appendix 2** of VHB’s Site Specific QAPP dated December 1, 2023, Revised January 5, 2024.

SECTION G
Method and SOP Reference Tables

Table 2: Method and SOP Reference Tables

Analysis	Analytical Method Reference*	Project Analytical SOPs
VOCs	1a. SW-846 Method 8260D	1b. Analysis of Volatile Organic Compounds by Gas Chromatography/Mass Spectrometry in Aqueous and Soil Samples by EPA SW-846 8260D, Revision 1.1, Effective Date 03/10/2023.
PAHs (subset of SVOCs)	2a. SW-846 Method 8270D and EPA Method 625.1	2b. Analysis of Semi-Volatile Organics (BNAs) by Gas Chromatography/Mass Spectrometry in Aqueous and Soil Samples by EPA SW-846 8270D and EPA 625.1, Revision 08/01/2022, Effective Date 08/01/2022.
CT ETPH	3a. CTDEEP ETPH Method	3b. Analysis of CT-ETPH in Environmental Extracts, Revision 1.9, Effective Date 05/06/2024.
PCBs	4a. SW-846 Method 3540C: Soxhlet Extraction	4b. Soil/Caulk Extraction by Soxhlet Method 3540C, Version Number 4.2, Effective Date 08/07/2023.
Metals – ICP	5a. SW-846 Update V Method 6010D	5b. Analysis of Metals in Digestates Using Inductively Coupled Plasma-Optical Emission Spectrometry (ICP-OES) by EPA SW846 6010D, Revision 1.3, Effective Date 01/02/2023.
Metals – ICP-OES	6a. SW-846 Method 6020A and 6020B and EPA Method 200.8	6b. Analysis of Metals in Digestates Using Inductively Coupled Plasma/Mass Spectrometry by EPA Method 200.8 and SW-846 Method 6020A and 6020B, Revision 2.0, Effective Date 08/20/2021.
Mercury	7a. SW-846 Method 7473	7b. Analysis of Mercury in Aqueous, Soil, and Sludge Samples by EPA SW-846 Method 7473, Revision 1.6, Effective Date 10/31/2019.
Pesticides	8a. SW-846 8081B and EPA Method 608.3	8b. Analysis of Chlorinated Pesticides by Gas Chromatography Using EPA SW-846 Method

Analysis	Analytical Method Reference*	Project Analytical SOPs
		8081B and EPA 608.3, Revision 2.2, Effective Date 04/17/2023.
PFAS	10a. EPA Method 1633 Draft 3	10b. Determination of Target Per- and Polyfluoroalkyl Substances (PFAS) in Aqueous and Solid matrices by Isotope Dilution Analysis by HPLC/MS-MS According to EPA Method 1633 Draft 3, Revision 1.2, Effective Date 06/23/2023.
1,4-Dioxane	11a. SW-846 8270D and is a SIM/isotope dilution method	11b. Analysis of 1,4-Dioxane by Gas Chromatography/Mass Spectrometry/SIM in Soil and Aqueous Extracts by EPA Method 8270E SIM with Isotope Dilution, Revision 1.4, Effective Date 08/28/2020.
TCLP	12a. SW-846 1311 or 1312, Modified Morgan and ASTM D3987	12b. Leaching Procedures for Aqueous and Solid Samples by EPA SW-846 1311 or 1312, Modified Morgan and ASTM D3987, Revision 1.8, Effective Date 12/02/2021.
Paint Filter Test	13a. SW-846 9095B	13b. Determination of Free Liquids in a Waste Samples by EPA SW-846 9095B (Paint Filter Liquids Test), Revision 1.0, Effective Date 03/04/2016.
Corrosivity	14a. SW-846 9040C and 9045D and SM 4500 H$^+$B	14b. Determination of pH (Corrosivity) in Aqueous and Solid Samples by EPA SW-846 9040C and 9045D and SM 4500 H$^+$B, Revision 2.1, Effective Date 03/30/2023.
Flashpoint	15a. SW-846 1010A	15b. Determination of Flashpoint in Liquid and Soil Samples by EPA SW-846 1010A, Revision 1.4, Effective Date 10/03/2012.
Reactivity	16a. SW-846 Ch 7.3.3	16b. Analysis of Reactivity-Cyanide as Releasable Cyanide According to EPA SW-846 Ch 7.3.3, Revision 1.6, Effective Date 04/11/2023. Analysis of Reactivity-Sulfide as Releasable Sulfide According to EPA SW-846 Ch 7.3.3, Revision 1.6, Effective Date 04/11/2023.

Laboratory SOPs (see Appendix 2)

*All analytical methods will be conducted in accordance with the EPA protocols.

Refer to VHB's Site Specific QAPP dated December 1, 2023, Revised January 5, 2024 for information related to VHB Field SOPs located in Section G.

SECTION H

Calibration and Corrective Action - Field Equipment

Refer to VHB's Site Specific QAPP dated December 1, 2023, Revised January 5, 2024 for information related to Section H.

SECTION I

Laboratory Equipment Calibration and Corrective Action

Refer to VHB’s Site Specific QAPP dated December 1, 2023, Revised January 5, 2024 for information related to Section I.

SECTION J

Sample Handling and Custody Requirements

Refer to VHB's Site Specific QAPP dated December 1, 2023, Revised January 5, 2024 for information related to Section J.

SECTION K
Analytical Sensitivity and Project Criteria

Refer to VHB's Site Specific QAPP dated December 1, 2023, Revised January 5, 2024 for information related to Section K.

SECTION L
Field Quality Control Requirements

Refer to VHB’s Site Specific QAPP dated December 1, 2023, Revised January 5, 2024 for information related to Section L.

SECTION M
Laboratory Quality Control Requirements

Refer to VHB's Site Specific QAPP dated December 1, 2023, Revised January 5, 2024 for information related to Section M.

SECTION N
Data Management and Documentation

Refer to VHB's Site Specific QAPP dated December 1, 2023, Revised January 5, 2024 for information related to Section N.

SECTION O

Assessment and Response Actions

Refer to VHB's Site Specific QAPP dated December 1, 2023, Revised January 5, 2024 for information related to Section O.

SECTION P
Project Reports

Following the completion of various tasks, formal and informal reports shall be prepared which include the following:

> Site Verification Report

Reports shall be submitted to the Client/Grantee for draft review. Following all comments, the reports will be finalized and issued to the Client/Grantee and the EPA in electronic format. Project status memorandums shall be prepared on a quarterly basis throughout the course of the project.

SECTION Q
Field Data Evaluation

Refer to VHB's Site Specific QAPP dated December 1, 2023, Revised January 5, 2024 for information related to Section Q.

SECTION R
Laboratory Data Evaluation

Refer to VHB's Site Specific QAPP dated December 1, 2023, Revised January 5, 2024 for information related to Section R.

SECTION S

Data Usability and Project Evaluation

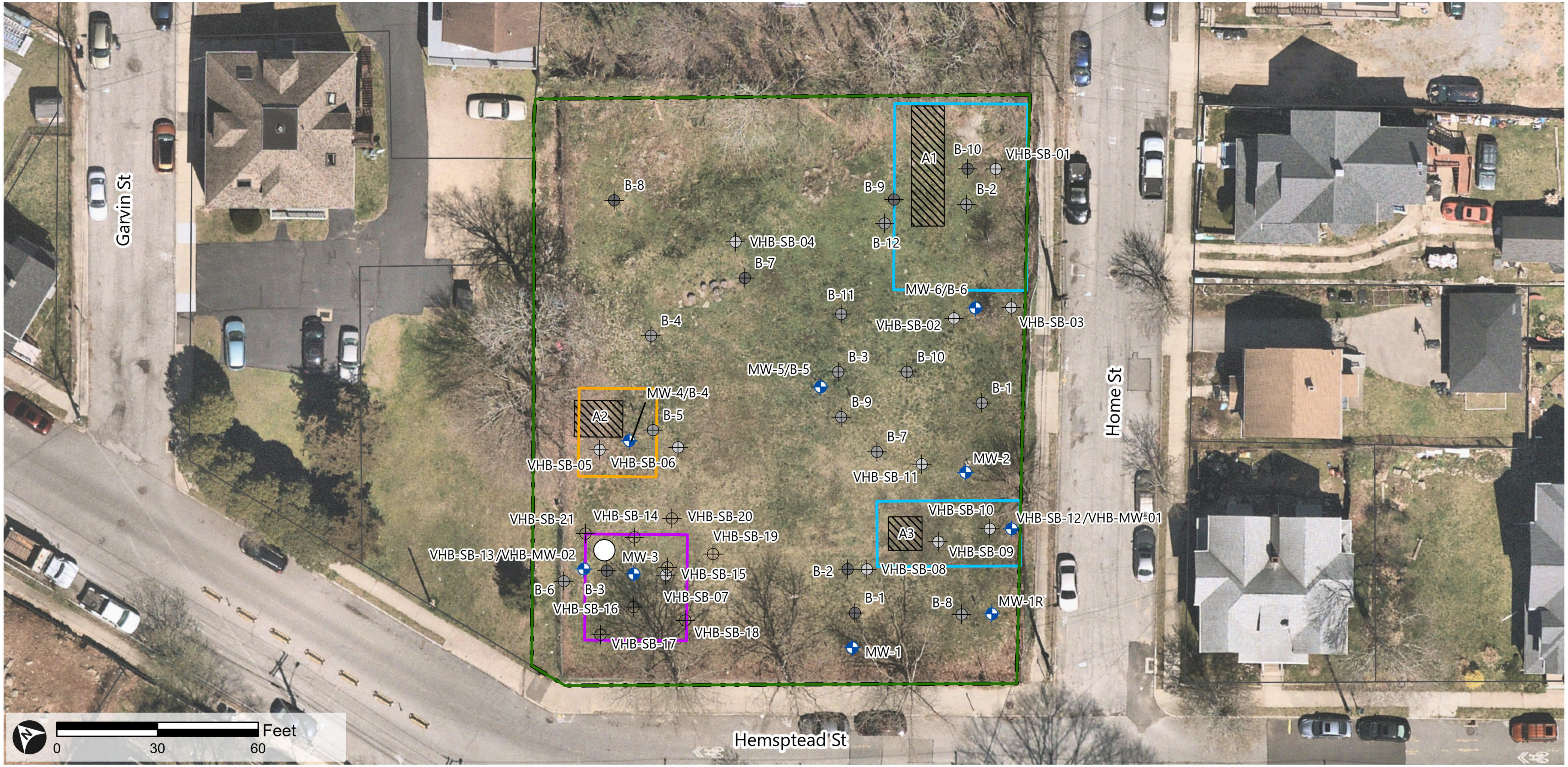
Refer to VHB's Site Specific QAPP dated December 1, 2023, Revised January 5, 2024 for information related to Section S.

APPENDIX 1

Figures

Figure 1: Remedial Action Plan

Phase III ESA / RDI | 43 Hempstead St, New London, CT



2023 VHB Soil Boring Location

2024 VHB Soil Boring Location

2000/2001 Delta Environmental Services, Inc Soil Borings

2021 USEPA Targeted Brownsfields Assessment Soil Borings

Monitoring Well Location

○ Former Gas Tank

▨ Potential UST

Subject Property

☐ Parcel Boundary

Approximate Release Areas

🟧 RA-1: Historic Lead Release

🟦 RA-2: Underground Storage Tanks (USTs)

🟪 RA-3: PFAS Release

🟩 RA-4: Site-wide Polluted Fill Material

Areas of Concern	Description
AOC-1	Site-Wide Historic Operations
AOC-2	Underground Storage Tanks
AOC-3	Site-Wide Polluted Fill

Source: 2024 Aerial Basemap - NearMap, New York State, State of Connecticut, Maxar , Microsoft

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APPENDIX 2

Laboratory Standard Operating Procedures

Standard Operating Procedure

Determination of Flashpoint in Liquid and Soil Samples by EPA SW-846 1010A

Approvals

Laboratory Director ______________________
Robert Q. Bradley

QA/QC Officer Teresa Weikel ______________________
Teresa Weikel

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Controlled Copy No. WC FP 040795-Rev 1.4- ______

Issued to: ____________________________

Determination of Flash Point

1. SCOPE AND APPLICATION

This method is applicable for the determination of flash points of liquids, excluding biodiesel (B100), and soils.

The temperature range covered is 50 - 200^{o}F. (RL = 50^{o}F)

This method is to be performed only by a trained analyst who has completed a Demonstration of Capability within the last 12 months. Please refer to York SOP Adm080206 for additional training instructions.

2. SUMMARY

This procedure is based upon EPA SW-846 1010A and ASTM-D93-Rev 79, 80 and later revisions Manual Pensky-Martens Closed-Cup Tester.

A refrigerated sample is heated and stirred at a controlled constant rate while a flame is applied to the sample vapor at appropriate intervals. The temperature is increased to 200^{o}F, or until a flash is observed.

3. DEFINITIONS

Analytical Batch Client samples (20 or fewer) and associated QC samples (SRM, sample duplicate) analyzed at one time.

Flash Point: The lowest temperature (corrected to a barometric pressure of 760 mm Hg) at which application of an ignition source causes the vapors of a sample to ignite.

Sample Duplicate A client sample analyzed a second time in the same analytical batch. This is used to determine method reproducibility and matrix homogeneity.

Standard Reference Material (SRM) A purchased material, with a known analyte concentration, which is designed to replicate a given sample matrix.

4. INTERFERENCES

Improper storage of samples may cause loss of volatiles and lead to erroneous results.

5. SAMPLE HANDLING

Samples are collected in glass jars with minimum headspace and refrigerated at 4 ± 2°C.

The recommended holding time from sampling to analysis of preserved samples is 10 days.

6. APPARATUS

- Precision Scientific Model 74537 Manual Pensky-Marten Closed Cup Flash Point apparatus, with propane canister, and integrated NIST traceable thermometer
- Barometer for recording and correcting data to 760 mm Hg.

7. REAGENTS AND STANDARDS

- **SRM -** n-Decane, 99+% Alpha Aesar #A14732 or L03747 (or equivalent)
  Alternately: p-Xylene, 99+% (FP - 80.6 F) or mixed xylenes (FP – 84.2 F)
- **Acetone -** technical grade
- **Methanol** - technical grade
- **1**:**1 Nitric acid**

8. PROCEDURE

8.1 It is MANDATORY to wear GOGGLES and GLOVES for this procedure.

8.2 This procedure CANNOT be conducted in a fume hood as high air flows may influence test.

8.3 Ensure that propane tank is set-up properly and no bubble in propane line is evident. If bubble is seen, abort procedure.

8.4 Enter the batch information in the Flash Point Log Book and in the LIMS system. Record barometric pressure at this time. Record the starting temperature of the unheated sample in the log.

8.5 Place correct amount of sample (up to line) into cup. Close and place cup in apparatus. Frequently stir sample using stirrer on apparatus.

8.6 Stop stirring sample and apply test flame. If sample flashes: Record flash point as < temperature measured in step 8.4 above, and proceed to step 8.9.

8.7 Turn heating assembly setting to 40 to cause slow gradual heating, while again stirring sample. At approximately 2^{o}F intervals, stop stirring sample and apply test flame. When sample flashes, record thermometer reading to nearest whole degree. Proceed to step 8.9.

8.8 If no flash point occurs before 200^{o}F is reached, conclude test and record flash point as >200^{o}F.

8.9 The flash point temperature reading must be corrected if barometric pressure is different than 760 mm Hg. See conversion chart. (Attachment 1)

8.10 Clean the flash point cup between samples using soap and water, 1:1 nitric acid, acetone and/or methanol as necessary.

9. CALIBRATION

The thermometer must be annually recertified against a NIST thermometer and labeled with temperature reading correction.

10. QA/QC

Each analytical batch must contain an SRM and a sample duplicate. (If sufficient client sample is not available for a duplicate, an additional SRM may be substituted.)

The SRM must be successfully run at the beginning and end of each batch and must flash between 96.5-103.5% of the expected value (115 + 4^{o}F for n-decane). (Or values currently in LIMS system, if different)

> **Corrective action:** Thoroughly clean system. Check that thermometer is immersed properly and flame appears constant. Rerun standard. If rerun fails to produce correct result, see group leader.

The sample duplicate should have a relative percent difference (RPD) of $\leq$ 20% (Or the value currently in LIMS system, if different)

> **Corrective action:** Rerun sample and duplicate. Due to sample variability, these results may not be achievable. If RPD is not achievable, the data must be flagged with “FP-D” in the LIMS.

11. CALCULATIONS AND REPORTING

Results of <50 ^{o}F and >200 ^{o}F are reported as such.

Report uncorrected data to nearest whole degree Fahrenheit (i.e. 135.6 is reported as 136) in the Flashpoint Log.

Perform the following barometric correction calculation and report to the nearest whole degree.

Corrected Flash Point = F + 0.06(760-P)

Where:
F = Measured (observed) flash point in ^{o}F
P = barometric pressure (mm Hg) measured at time of test

EXAMPLE: Flashpoint observed 122.5 ^{o}F, barometric pressure= 731 mm Hg:
Corrected FP = 122.5 + 0.06(760-731) = 124.2 ^{o}F

12. HEALTH & SAFETY

General safety considerations and requirements are detailed in the York Laboratory Safety and Health Standard Operating Procedure No. Safety011600.

Specific safety rules applying to the conduct of this analysis include the following:

- GOGGLES are mandatory when conducting this test.
- When handling standards and samples, latex gloves are required.
- While the flash point apparatus and test is conducted OUTSIDE a fume hood, handling of highly odorous samples should be done inside the fume hood.
- Refer to MSDSs for specific safety/health information for all chemicals.

The analysts must exercise normal care and be supervised and trained to work in an analytical chemistry laboratory. The analysts will be handling fragile glassware, needles, syringes, volatile and flammable chemicals, toxic chemicals and corrosive chemicals.

No smoking or open flames are allowed.
No food or food products may be brought into the laboratory.

Hood doors must be kept in the position which yields approx. 100 fpm face velocity.

Safety glasses are provided and must be worn at all times in the laboratory.

Latex gloves are provided and must be worn when working with chemicals.

Laboratory coats are provided and should be worn to protect the analysts' clothes.

13. WASTE MANAGEMENT/POLLUTION PREVENTION

Neat Materials

Waste management procedures require the prudent use of neat materials. The ordering of neat standards and materials must be done to minimize unused material which would result in storage or handling of excess material. Quantities ordered should be sufficient to provide for necessary standards with consideration to shelf life. When ordering a unique material for a standard, be sure to order the smallest practical quantity.

Samples

Unused or remaining soil and water samples are returned to the sample control room for continued storage for proper disposal by the sample control group.

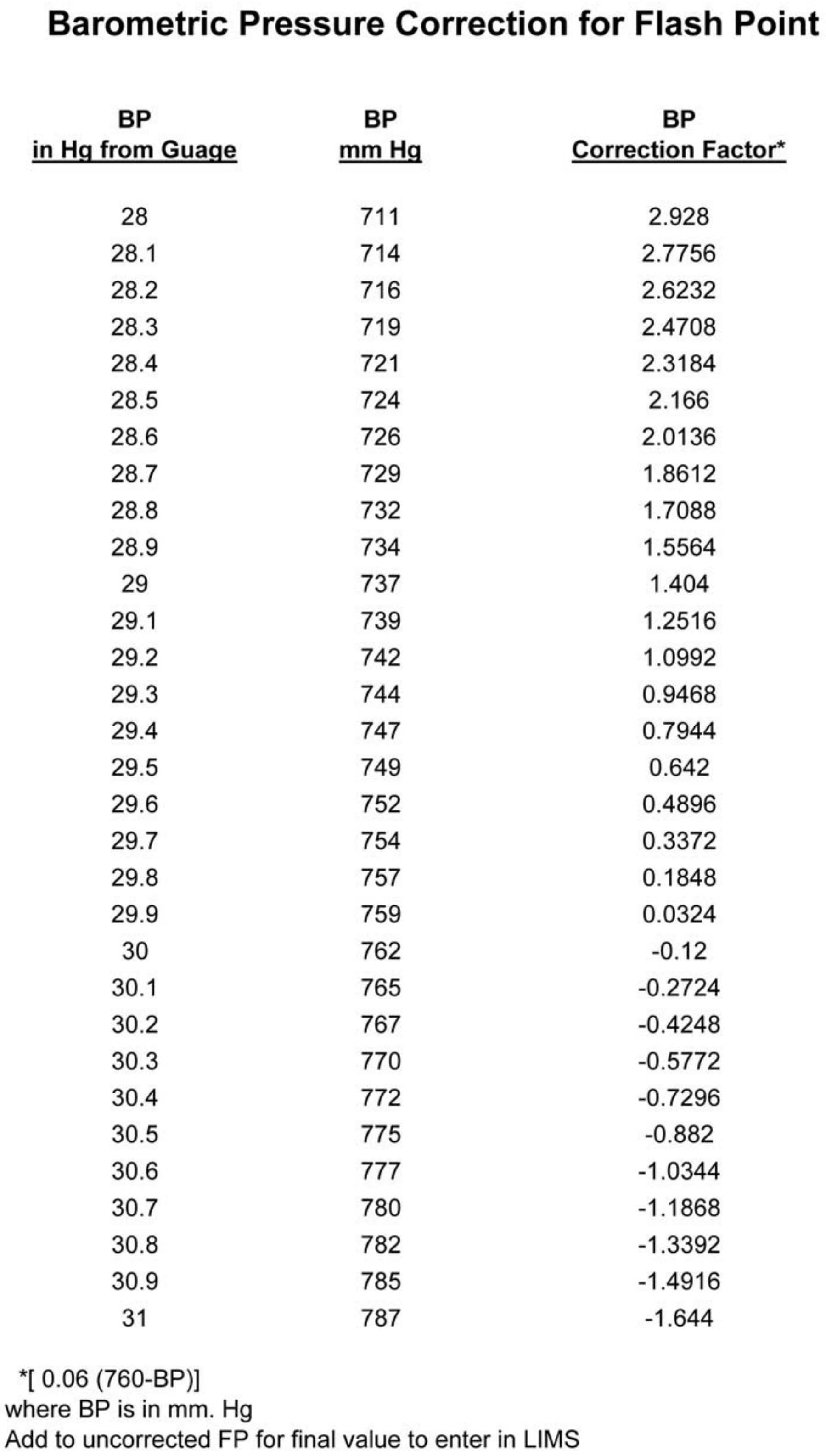
14. REFERENCES

USEPA SW-846 Test Method 1010A

15. REVISION HISTORY

04/07/1995	Revision 1.0	Original Document
04/11/2000	Revision 1.1	Added Waste Management section
04/19/2010	Revision 1.2	Added QC requirements and LIMS entry info
06/02/2012	Revision 1.3	Corrected n-decane FP to 115 deg F, updated format
10/03/2012	Revision 1.4	Updated format, added corrective actions, referenced SW-846 1010A method

Attachment 1



Barometric Pressure Correction for Flash Point

BP in Hg from Guage	BP mm Hg	BP Correction Factor*
28	711	2.928
28.1	714	2.7756
28.2	716	2.6232
28.3	719	2.4708
28.4	721	2.3184
28.5	724	2.166
28.6	726	2.0136
28.7	729	1.8612
28.8	732	1.7088
28.9	734	1.5564
29	737	1.404
29.1	739	1.2516
29.2	742	1.0992
29.3	744	0.9468
29.4	747	0.7944
29.5	749	0.642
29.6	752	0.4896
29.7	754	0.3372
29.8	757	0.1848
29.9	759	0.0324
30	762	-0.12
30.1	765	-0.2724
30.2	767	-0.4248
30.3	770	-0.5772
30.4	772	-0.7296
30.5	775	-0.882
30.6	777	-1.0344
30.7	780	-1.1868
30.8	782	-1.3392
30.9	785	-1.4916
31	787	-1.644

*[0.06 (760-BP)]
where BP is in mm. Hg
Add to uncorrected FP for final value to enter in LIMS

Standard Operating Procedure

Determination of Free Liquids in a Waste Samples by EPA SW-846 9095B (Paint Filter Liquids Test)

Approvals

Laboratory Director ________________________ Ben Gulizia

Sr. Scientist/Technical Director ________________________ Robert Bradley

QA/QC Officer ________________________ Magdalena Szymczuk

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Controlled Copy No. WC Free Liquids Rev 1.0-_______

Issued to: ______________________________

Free Liquids – Pain Filter Liquids Test

1. SCOPE AND APPLICATION

This method is applicable to the determination of free liquids in sample of waste.

This method is to be performed only by a trained analyst. Please refer to York SOP Adm080206 for additional training instructions.

2. SUMMARY

The procedure is based upon EPA SW-846 method 9095B.
The method is used to determine compliance with 40 CFR 264.314 and 265.314

A predetermined amount of material is placed in a paint filter. If any portion of the material passes through and drops from the filter within the 5 minute test period, the material is deemed to contain free liquids.

3. DEFINITIONS

Analytical Batch Client samples (20 or fewer) and associated QC sample (duplicate) analyzed at one time.

Sample Duplicate A client sample analyzed a second time in the same analytical batch. This is used to determine method reproducibility and matrix homogeneity.

4. INTERFERENCES

Filter media may separate from the filter cone on exposure to alkaline materials (no problem if sample is not disturbed).
Temperature can affect the test results if the test is performed below the freezing point if any liquids in the sample. Tests must be performed above the freezing point

5. SAMPLE HANDLING

A 100 ml (liquids) or 100 g (other) sample is required for the test.
If it's not possible to obtain a sample of 100 ml or 100 g that is sufficiently representative of the waste, the analyst may use larger size samples in multiples of 100 ml or 100 g, i.e., 200, 300, 400 ml or g.
However, when larger samples are used, analyst should divide the sample into 100 ml or 100 g and test each portion separately. If any portion contains free liquids, the entire

sample is considered to have free liquids. If the sample is measured volumetrically, it should not have any air bubbles.

6. APPARATUS

- Conical paint filter – Mesh number 60 +/- 5% (Fine meshed size) – Available at the local paint store, such as Barnum Hardware.
- Glass funnel – the funnel should be fluted or have a large open mouth in order to support the paint filter and not interfere with the movement to the graduated cylinder.
- Ring stand and ring, or tripod
- Scissors and knife for cutting materials as required.
- Graduated cylinder or beaker – 100 ml.

7. REAGENTS AND STANDARDS

This procedure does not require any reagents and standards.

8. PROCEDURE

8.1 Assemble test apparatus as shown in Figure 1.

8.2 Place sample in the filter. A funnel may be used to provide support for the paint filter. If sample has a light appearance (bulk density) that it overflows the filter, then the sides of the filter can be extended upward by taping filter paper to the inside of the filter and above the mesh. Settling the sample into the paint filter may be facilitated by lightly tapping the side of the filter as it is being filled.

8.3 To assure uniformity and standardization of the test, materials which don't conform to the shape of the paint filter should be cut into small pieces (approx. <0.4 in.) and poured into the filter. Sample size reduction can be done by cutting the sorbent material using scissors, or a knife as to preserve as much of the original integrity of the sorbent fabric as possible. Sorbents enclosed in a fabric should be mixed with the resultant fabric pieces. Grinding sorbent material should be avoided as this may destroy the integrity of the sorbent and may produce " fine particles" which would normally not be present.

8.4 For brittle materials larger than 1 cm that don't conform to the filter, light crushing to reduce oversized particles is employed if it is not possible to cut the material (clay, silica gel and some polymers can fall into this category).

8.5 Allow the sample to drain for 5 min. into the graduated cylinder or beaker.

8.6 If any portion of the test material is collected in the graduated cylinder or beaker in the 5 minute period, then the material is considered to contain free liquids.

9. CALIBRATION

This procedure does not require calibration.

10. QA/QC

Each analytical batch should contain a duplicate sample.

11. Data Reporting

If Free Liquid is not present – use qualifier "PF-01".
If Free Liquid is present – use qualifier "PF-02".

12. HEALTH AND SAFETY

General safety considerations and requirements are detailed in the York Laboratory Safety and Health Standard Operating Procedure No. Safety011600.

Specific safety rules applying to the conduct of this analysis requiring the following:

- When handling standards and samples, latex gloves are required.
- When handling samples, gloves and glasses are required.
- Highly odorous samples must be handled in a fume hood.

The analysts must exercise normal care and be supervised and trained to work in an analytical chemistry laboratory. The analysts will be handling fragile glassware, needles, syringes, volatile and flammable chemicals, toxic chemicals and corrosive chemicals.

No smoking or open flames are allowed.
No food or food products may be brought into the laboratory.

Hood doors must be kept in the position which yields approx. 100 fpm face velocity.

Strong acids and bases must be handled with gloves and appropriate eye wear.

Concentrated acids must be used and kept under fume hoods designated for acids.

Safety glasses are provided and must be worn at all times in the laboratory.

Laboratory coats are provided and should be worn to protect the analysts' clothes.

13. WASTE MANAGEMENT/POLLUTION PREVENTION

Acids/Bases

Our laboratory employs the use of concentrated acids (hydrochloric, nitric and sulfuric acids). These acids are utilized for sample preparation, glassware preparation and standards preparation. All acid rinsing of glassware is done to minimize the amount of acid used. The amount of the particular acid used is minimized to only the volume necessary to reduce the amount of acid waste after processing samples.

Samples

Unused or remaining soil and water samples are returned to the sample control room for continued storage for proper disposal by the sample control group.

14. REFERENCES

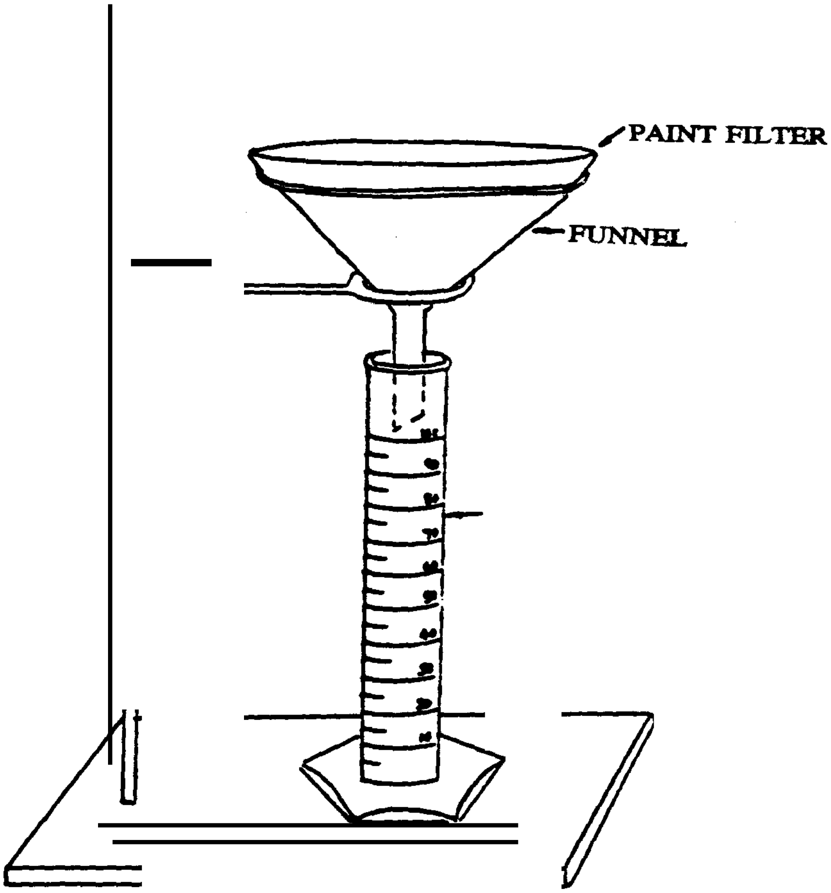
USEPA SW-846 Test Method 9095 B, Revision 2, November 2004

15. REVISION HISTORY

Revision 1.0	03/07/2016	Original Document

Figure 1

Paint Filter Test Apparatus



Standard Operating Procedure

Determination of pH (Corrosivity) in Aqueous and Solid Samples by EPA SW-846 9040C and 9045D and SM 4500 H^+B

Approvals

Laboratory Director
Cassie Mosher

Technical Director
Robert Q. Bradley

QA/QC Officer
Sarah Widomski

Controlled Copy No. WC pH-Rev 2.1- ______

Issued to: ____________________________

pH (Corrosivity)

1. SCOPE AND APPLICATION

This method is applicable to the determination of pH in drinking, surface, and saline waters, domestic and industrial waste waters, soils and wastes.

The range of pH covered by this method is 0.5-14. (RL = 0.5)

This method is to be performed only by a trained analyst who has completed a Demonstration of Capability within the last 12 months. Please refer to York SOP Adm080206 for additional training instructions.

2. SUMMARY

The procedure is based upon EPA SW-846 methods 9040C and 9045D and Standard Method 4500 H^+ B.

The pH of a sample is determined electrometrically using a single combination electrode with automatic temperature compensation, OR using a separate temperature electrode.

3. DEFINITIONS

pH The hydrogen ion activity in a solution at a given temperature, or the intensity of its acidic or basic character.

Analytical Batch Client samples (20 or fewer) and associated QC samples (CCV, sample duplicate) analyzed at one time.

Calibration Curve The relationship between response and analyte concentration determined for a series of calibration standards. The calibration curve is made by graphing the response-vs-pH and performing a regression analysis of the data.

Calibration Standards A series of buffer solutions (pH 4, 7, 10) used by the analyst for preparation of the calibration curve.

ICV A second source standard (pH 7 buffer) used to verify the calibration accuracy

CCV (Continuing Calibration Verification) A calibration standard (pH 7 buffer) run at the beginning and end of each analytical batch to confirm the validity of the curve.

Reporting Limit (RL) The lowest pH value reported.

Sample Duplicate A client sample analyzed a second time in the same analytical batch. This is used to determine method reproducibility and matrix homogeneity.

4. INTERFERENCES

Oily materials may coat the probe, and should be gently wiped off and rinsed. Also oily materials can be checked with pH strips and flagged with “ pH-Est

5. SAMPLE HANDLING

Samples must be unpreserved be in either glass or plastic containers.

Samples must be stored at 4 ± 2° C until analyzed.

The EPA recommended holding time from sampling to analysis samples is 15 minutes.

Samples should be analyzed as soon as possible upon entering the lab. All pH measured on samples in the lab have exceeded holding times and must be flagged with “HT-pH” in the LIMS.

6. APPARATUS

- pH meter, Orion Dual Star (or equivalent)
- Combination pH electrode (e.g. Ross electrode)
- Temperature compensation probe
- Analytical balance
- Beakers 50, 150 ml
- Glass filter funnels
- Qualitative filter paper
- Centrifuge
- Kimwipes
- pH strips

7. REAGENTS AND STANDARDS

ALL BUFFER INFORMATION (i.e. manufacturer, lot#, date opened, date finished) MUST BE ENTERED INTO THE FRONT OF THE pH CALIBRATION LOG BOOK.

In addition, at the bottom each page, the CCV buffer used that day must be listed and traceable to the information in the front of the pH calibration log book.

- **pH 4.0 buffer solution**, Hach #2283449 (or equivalent)
- **pH 7.0 buffer solution**, Hach #22835-49 (or equivalent)
- **pH 10.0 buffer solution**, Hach #2283649 (or equivalent)
- **pH 2.0 buffer solution**, LabChem LC122201 (or equivalent)
- **pH 12.0 buffer solution,** LabChem LC125602 (or equivalent)
- **pH 7.0 buffer, 2nd source, Thermo Scientific Orion 910107 7.00 pH buffer solution, 475 mL bottle**
- **Electrode filling solution**, Orion #810007 (or equivalent)
- **Deionized water**

8. PROCEDURE

8.1 Perform instrument calibration (Section 9.0) daily before use. Verify the curve with a 2nd source pH = 7.0 buffer (ICV) to be ± 0.05 of true value. If not, recalibrate and repeat.

8.2 Aqueous Samples

8.2.1 Place pH electrode and temperature probe into beaker of sample. Beaker should contain enough sample to cover the bulb of the pH probe.

8.2.2 When reading stabilizes, record pH to nearest 0.01 unit in pH log book.

8.2.3 Note: If the pH of a sample is above or below the initial calibration points, use the appropriate buffer check (2 if below cal. and 12 if above cal.) to ensure linearity is still intact. Also check all containers to confirm and inform supervisor.

8.2.4 Rinse and blot electrode.

8.2.5 Refer to electrode manual for storage of electrode when not in use.

8.3 Soil or Solid Samples

8.3.1 Place 20g soil into a 50 ml beaker.

8.3.2 Add 20 ml DI water.

8.3.3 Cover and continuously stir the suspension for 5 minutes. Add additional DI water if necessary to allow effective stirring. (ex: Hygroscopic samples may absorb all 20 ml of DI water resulting in a semisolid, rather than the aqueous and solid phases necessary.)

8.3.4 Centrifuge suspension, pour through qualitative filter paper in a filter funnel, or allow to stand for 1 hour, to separate solids from the aqueous phase.

8.3.5 Using the aqueous phase only, follow steps 8.2.1-8.2.3 above.

8.3.6 Note: If the pH of a sample is above or below the initial calibration points, use the appropriate buffer check (2 if below cal. and 12 if above cal.) to ensure linearity is still intact.

8.4 Waste Samples

8.4.1 Place 20g soil into a 50 ml beaker.

8.4.2 Add 20 ml DI water.

8.4.3 If sample is hygroscopic and absorbs all water, repeat experiment with 20 g waste and 40 ml water.

8.4.4 Cover and continuously stir the suspension for 5 minutes

8.4.5 Centrifuge suspension, pour through qualitative filter paper in a filter funnel, or allow to stand for 15 minutes, to separate solids from the aqueous phase.

8.4.6 If the liquid phase has an oily layer, decant this layer.

8.4.7 Using the aqueous phase only, follow steps 8.2.1-8.2.3 above.

8.4.8 Note: If the pH of a sample is above or below the initial calibration points, use the appropriate buffer check (2 if below cal. and 12 if above cal.) to ensure linearity is still intact.

9. CALIBRATION

9.1 Check the electrode for proper filling solution level. The solution should be just below opening in probe.

9.2 Fill buffer containers with fresh pH 4.0, 7.0 and 10.0 buffers.

9.3 Place temperature and pH probes in the pH 4 buffer.

9.4 On orion dual star press cal (F2)

9.5 Press select for the channel being calibrated.

9.6 Press start (F3).

9.7 When reading is stable, adjust value to 4.0 and press accept (F2)

9.8 Press next (F2).

9.9 Repeat process for buffers 7 and 10.

9.10 Press cal done (F3).

9.11 Press “Yes.” (“pH electrode placed in buffer 2?”)

9.12 Record slope % in calibration logbook.

9.13 Press log print (F2) to get to main screen.

10. QA/QC

Each analytical batch must contain a sample duplicate, and two CCVs.

An ICV (2nd source pH buffer) must be run after calibration to verify the accuracy of the pH standards used.

> **Corrective Action**: Recalibrate and rerun ICV, if still out, contact your supervisor

A CCV (pH 7 Buffer) must be run at the beginning and at the end of each batch. They must both fall within 6.95-7.05.

> **Corrective action:** Analytical batch must be repeated and/or meter must be recalibrated.

The sample duplicate should have a relative percent difference (RPD) of <10%. (Or the value currently in LIMS system, if different)

> **Corrective action:** Rerun sample and duplicate. Due to sample variability, these results may not be achievable.

Outside initial calibration: Each batch with a sample outside of the initial calibration range must have a secondary CCV (pH 2 or pH 12 buffer) recorded to ensure linearity of the calibration.

11. Data Reporting

pH is reported to three significant figures.

12. HEALTH AND SAFETY

General safety considerations and requirements are detailed in the York Laboratory Safety and Health Standard Operating Procedure No. Safety011600.

Specific safety rules applying to the conduct of this analysis requiring the following:

- When handling standards and samples, latex gloves are required.
- When handling samples, gloves and glasses are required.
- Highly odorous samples must be handled in a fume hood.

The analysts must exercise normal care and be supervised and trained to work in an analytical chemistry laboratory. The analysts will be handling fragile glassware, needles, syringes, volatile and flammable chemicals, toxic chemicals and corrosive chemicals.

No smoking or open flames are allowed.
No food or food products may be brought into the laboratory.

Hood doors must be kept in the position which yields approx. 100 fpm face velocity.

Strong acids and bases must be handled with gloves and appropriate eye wear.

Concentrated acids must be used and kept under fume hoods designated for acids.

Safety glasses are provided and must be worn at all times in the laboratory.

Laboratory coats are provided and should be worn to protect the analysts’ clothes.

13. WASTE MANAGEMENT/POLLUTION PREVENTION

Acids/Bases

Our laboratory employs the use of concentrated acids (hydrochloric, nitric and sulfuric acids). These acids are utilized for sample preparation, glassware preparation and standards preparation. All acid rinsing of glassware is done to minimize the amount of acid used. The amount of the particular acid used is minimized to only the volume necessary to reduce the amount of acid waste after processing samples.

<u>Samples</u>

Unused or remaining soil and water samples are returned to the sample control room for continued storage for proper disposal by the sample control group.

14. REFERENCES

USEPA SW-846 Test Methods 9040C, 9045D

Standard Methods for the Examination of Water and Wastewater. 20th Edition, 1999. Method 4500 H^+ B

15. REVISION HISTORY

Revision 1.0	04/06/1995	Original Document
Revision 1.1	05/11/2000	Added sections 9.0 and 10.0
Revision 1.2	09/14/2006	Revised section 5.0
Revision 1.3	09/15/2008	Revised language in section 6.0
Revision 1.4	04/30/2010	Modified Section 2, 4 and 5 to reflect current practices and temperature compensation
Revision 1.5	10/11/2012	Updated format, deleted method blank, updated soil method, added corrective actions
Revision 1.6	01/05/2014	Apply “HT-pH” flag to all samples, Delete original date from title
Revision 1.7	03/27/2017	Modified sections 7 and 10 to reflect inclusion of an ICV.
Revision 1.8	07/18/2017	Added pH 2 and 12 buffer checks if sample is out of initial calibration range
Revision 1.9	04/03/2018	Updated Quality Assurance Manager position
Revision 2.0	12/02/2021 Sbw	Updated QA Officer, Lab Director, headers and footers.
Revision 2.1	3/30/2023	Updated section 2 for temp. electrode. Updated sec 4. Updated sec 6 for meter and strips. Sec 8.2.3 added comment for checking all containers and to inform supervisor. Updated sec 9 for calibration of Orion dual star meter.

Standard Operating Procedure

Analysis of Reactivity-Cyanide as Releasable Cyanide According to EPA SW-846 Ch 7.3.3

Approvals

Laboratory Director ______________________
Cassie Mosher

Sr. Scientist/Technical Director ______________________
Robert Bradley

QA/QC Officer ______________________
Sarah Widomski

Controlled Copy No. WC CNR Rev1.6-_______

Issued to: ______________________________

REACTIVE-CYANIDE (Releaseable Cyanide)

1. SCOPE AND APPLICATION

1.1 Background

The regulation in 40 CFR 261.23 defines reactive wastes to include wastes that have any of the following properties: (1) readily undergo violent chemical change; (2) react violently or form potentially explosive mixtures with water; (3) generate toxic fumes when mixed with water or, in the case of cyanide- or sulfide-bearing wastes, when exposed to mild acidic or basic conditions; (4) explode when subjected to a strong initiating force; (5) explode at normal temperatures and pressures; or (6) fit within the Department of Transportation's forbidden explosives, Class A explosives, or Class B explosives classifications.

This SOP only addresses item (3) above: It is a cyanide- or sulfide-bearing waste which, when exposed to pH conditions between 2 and 12.5, can generate toxic gases, vapors, or fumes in a quantity sufficient to present a danger to human health or the environment.

1.2 This method is applicable to all wastes, with the condition that wastes that are combined with acids do not form explosive mixtures.

1.3 This method provides a way to determine the specific rate of release of hydrocyanic acid upon contact with an aqueous acid. The interim guidance for reactive cyanide level is: Total releasable cyanide: 250 mg HCN/kg waste. The reporting limit of the method as defined herein is 0.50 mg/kg reactive cyanide (releasable cyanide).

1.4 This test measures only the hydrocyanic acid evolved at the test conditions. It is not intended to measure forms of cyanide other than those that are evolvable under the test conditions.

1.5 It is noted that this method has been withdrawn by EPA. Many disposal facilities are still requiring this therefore it is necessary until otherwise noted.

2. SUMMARY

An aliquot of weak sulfuric acid is added to a fixed weight of waste (10g.) in a closed system. The generated gas is swept into a sodium hydroxide scrubber. The cyanide in the scrubber solution is quantitated. The procedure for quantitating the cyanide is Method 9014 or EPA 335.2 / SM 18-20 4500-CN C/E (manual pyridine/barbituric acid spectrophotometric method). Refer to York SOP WC CNT 070900.

This method is based upon EPA SW-846 Chapter 7, Section 7.3.3, Revision 3 December 1996.

3. DEFINITIONS

Reactivity-Cyanide- the hydrocyanic acid evolved at the test conditions described herein. The test determines the specific rate of release of hydrocyanic acid upon contact with an aqueous acid. The interim guidance for reactive cyanide level is: Total releasable cyanide: 250 mg HCN/kg waste. The reporting limit of the method as defined herein is 0.25 mg/kg reactive cyanide (releasable cyanide).

Analytical Batch Client samples (20 or fewer) and associated QC samples (method blank and sample duplicate) analyzed at one time.

Method Blank Deionized water (for aqueous matrices) or Ottawa sand, (for solid matrices) treated exactly as a sample, and run with each analytical batch.

Reporting Limit (RL) The lowest point of quantitation based upon the lowest point in the calibration curve. This ideally is less than or equal to desired regulatory action levels.

Sample Duplicate A client sample analyzed a second time in the same analytical batch. This is used to determine method reproducibility and matrix homogeneity.

4. INTERFERENCES

No interferences are noted.

5. SAMPLE HANDLING

5.1 Samples containing, or suspected of containing, sulfide or a combination of sulfide and cyanide wastes should be collected with a minimum of aeration. The sample bottle should be filled completely, excluding all head space, and closed tightly.

5.2 Storage of samples should be under refrigeration (2-6C) and in the dark.

5.3 Testing may be performed in a ventilated hood.

5.4 Containers recommended are 2 or 4 oz. glass with teflon lined lids.

5.5 The holding times are not established, however it is recommended that analysis should commence as soon as possible, and samples should be kept in a cool, dark place until analysis begins. Holding time should be considered as 14 days from collection.

6. APPARATUS (See Figure 1)

6.1 Round-bottom flask - 500-mL, three-neck, with 24/40 ground-glass joints.

6.2 Gas scrubber - 50 mL scrubber-either calibrated or add 50 mL with a Class A pipet to the scrubber

6.3 Magnetic Stirring apparatus - To achieve approximately 30 rpm. This may be either a rotating magnet and stirring bar combination

6.4 Addition funnel - With pressure-equalizing tube and 24/40 ground-glass joint and Teflon sleeve.

6.5 Flexible tubing - For connection from nitrogen supply to apparatus.

6.6 Prepurified nitrogen gas - With two-stage regulator.

6.7 Rotometer - For monitoring nitrogen gas flow rate. 0-200 ml/min or equivalent

6.8 Top loading balance - capable of weighing to 0.01 g.

6.9 Ottawa Sand or cyanide/sulfide free sand-can be created by muffling fine sand at 650C for 2 hours and stored in a sealed glass container until use.

7. REAGENTS AND STANDARDS

Reagent grade inorganic chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

7.1 Reagent water. All references to water in this method refer to organic free deinoized water delivered by the York water purification system.

7.2 Sulfuric acid (1.0 N), H_2SO_4. Add 28 mL concentrated H2SO4 to Deionized water and dilute to 1 L.

7.3 Potassium cyanide, AR grade

7.4 Sodium Hydroxide, AR grade

7.5 Sodium hydroxide scrubber solution (0.25N), NaOH. Dissolve 10.0 g of NaOH in DI water and dilute to 1 liter with DI water.

8. PROCEDURE

Set up the apparatus as shown in Figure 1.0.

8.1 Add 50 mL of 0.25N NaOH solution to a calibrated scrubber or use a Class A pipet.

8.2 Close the system and adjust the flow rate of nitrogen, using the rotometer. Flow should be 60 mL/min.

8.3 Add 10.0 g of the waste to be tested to the system. Close the system immediately.

8.4 With the nitrogen flowing, add enough 1.00 N sulfuric acid to fill the flask approximately half full. Start the sir bar to mildly stir the contents with no vortex created. Start the 3 minute test period. The stirring speed must remain constant throughout the test.

8.5 After 3 minutes, close off the nitrogen and disconnect the scrubber. Determine the amount of cyanide in 25 mls of the 50 mL scrubber solution using York SOP WC CNT 070900. If desired, RETAIN the remaining 25 mL of scrubber solution for Reactive Sulfide determination-see SOP WC ReactiveSulf

8.6 Run a method blank and a duplicate with each batch of 20 or fewer/day.

9. CALIBRATION

Calibration does not apply specifically to this procedure. It does apply to cyanide analysis steps as defined in York SOP WC CNT 070900.

10. QA/QC

The QA/QC measures associated with this procedure include a method blank and sample duplicate.

10.1 Method Blank

The method blank is simply 10 g. of Ottawa sand (or cyanide/sulfide-free sand) in the flask and then treated exactly like a sample as dictated in section 8.0. The acceptance criteria is < 0.25 mg/kg releasable cyanide.

Corrective Action: If < 0.25 mg/kg releasable cyanide is not achieved the system must be cleaned and the method blank re-prepared and analyzed.

10.2 Sample Duplicate

For sample duplicate results > 10 x the RL of 0.5 mg/kg (5.0 mg/kg), the RPD should be < 50%.

Corrective Action: If the RPD is $\geq$ 50, rerun the duplicate. If this problem still persists, flag the data accordingly. If samples are "Not detected" no precision value is meaningful.

11. CALCULATIONS AND REPORTING

11.1 Determine the specific rate of release of HCN, using the following parameters:

X = Concentration of HCN in diluted scrubber solution (mg/L)
(This is obtained from the spectrophotometric method)

L = Volume of solution in scrubber (L) (50 mL = 0.050 liters but we are using 25 mL so 0.025 L

W = Weight of waste used (kg)-we use 10.0 g. or 0.010 kg

S = Time of measurement (sec.) = Time N stopped - Time N started =should be around 180 seconds (3 minutes x 60sec/min)

R = specific rate of release (mg/kg/sec.) = $\frac{X \bullet L}{W \bullet S}$

Total releasable HCN (mg/kg) = R x S

Example: if we used 10.5 g. of sample and the cyanide in 25 mL of scrubber was determined to be 1.25 mg/L and the procedure was conducted for 29 minutes (1740 seconds) the results would be:

$$R = \frac{1.25 \text{ x } 0.025 \text{ L}}{0.015 \text{ x } 1740} = 0.001197 \text{ mg/kg/sec}$$

Total Releasable HCN (mg/kg) = 0.001197 x 1740 = 2.08 mg/kg

This would be classified as non-reactive (< 250 mg/kg)

11.2 This values would then be entered in LIMS as is.

12. HEALTH AND SAFETY

General safety considerations and requirements are detailed in the York Laboratory Safety and Health Standard Operating Procedure No. Safety011600.

Specific safety rules applying to the conduct of this analysis requiring the following:

- When handling standards and samples, latex gloves are required.
- Also, when handling neat materials, a fume hood and safety glasses are required.
- When handling samples, gloves and glasses are required.
- Highly odorous samples must be handled in a fume hood.
- Refer to MSDSs for specific safety/health information

The analysts must exercise normal care and be supervised and trained to work in an analytical chemistry laboratory. The analysts will be handling fragile glassware, needles, syringes, volatile and flammable chemicals, toxic chemicals and corrosive chemicals.

No smoking or open flames are allowed.
No food or food products may be brought into the laboratory.

Solvents should not be left uncovered on the laboratory benches.

All solvent transfers should be done in the hoods.

Hood doors must be kept in the position which yields approx. 100 fpm face velocity.

Venting of separatory funnels must be done in the hood.

Solvent evaporation must be done in the hood with exhaust elevated and in the rear.

Waste containers that had solvents must be vented to a hood until all solvents have evaporated.

Strong acids and bases must be handled with gloves and appropriate eye ware.

Concentrated acids must be used and kept under fume hoods designated for acids.

Safety glasses are provided and must be worn at all times in the laboratory.

Latex gloves are provided and must be worn when working with chemicals.

Laboratory coats are provided and should be worn to protect the analysts’ clothes.

Syringes and needles must be kept in their original cases when not in use.

Care must be exercised in using and handling syringes to avoid injury.

Report any sticking with a needle immediately to your supervisor.

13. WASTE MANAGEMENT/POLLUTION PREVENTION

Neat Materials

Waste management procedures require the prudent use of neat materials. The ordering of neat standards and materials must be done to minimize unused material which would result in storage or handling of excess material. Quantities ordered should be sufficient to provide for necessary standards with consideration to shelf life. When ordering a unique material for a standard, be sure to order the smallest practical quantity.

Acids/Bases

Our laboratory employs the use of concentrated acids (Hydrochloric, Nitric and Sulfuric acids). These acids are utilized for sample preparation, glassware preparation and standards preparation. All acid rinsing of glassware is done to minimize the amount of acid used. The amount of the particular acid used is minimized to only the volume necessary to reduce the amount of acid waste after processing samples.

The concentrated bases used at York include Sodium Hydroxide and Ammonium Hydroxide. These bases are used to prepare various molar or normal solutions for specific analyses such as cyanide, etc. The amounts prepared are minimized to that needed for a particular application.

Samples

Unused or remaining soil and water samples are returned to the sample control room for continued storage for proper disposal by the sample control group.

14. REFERENCES

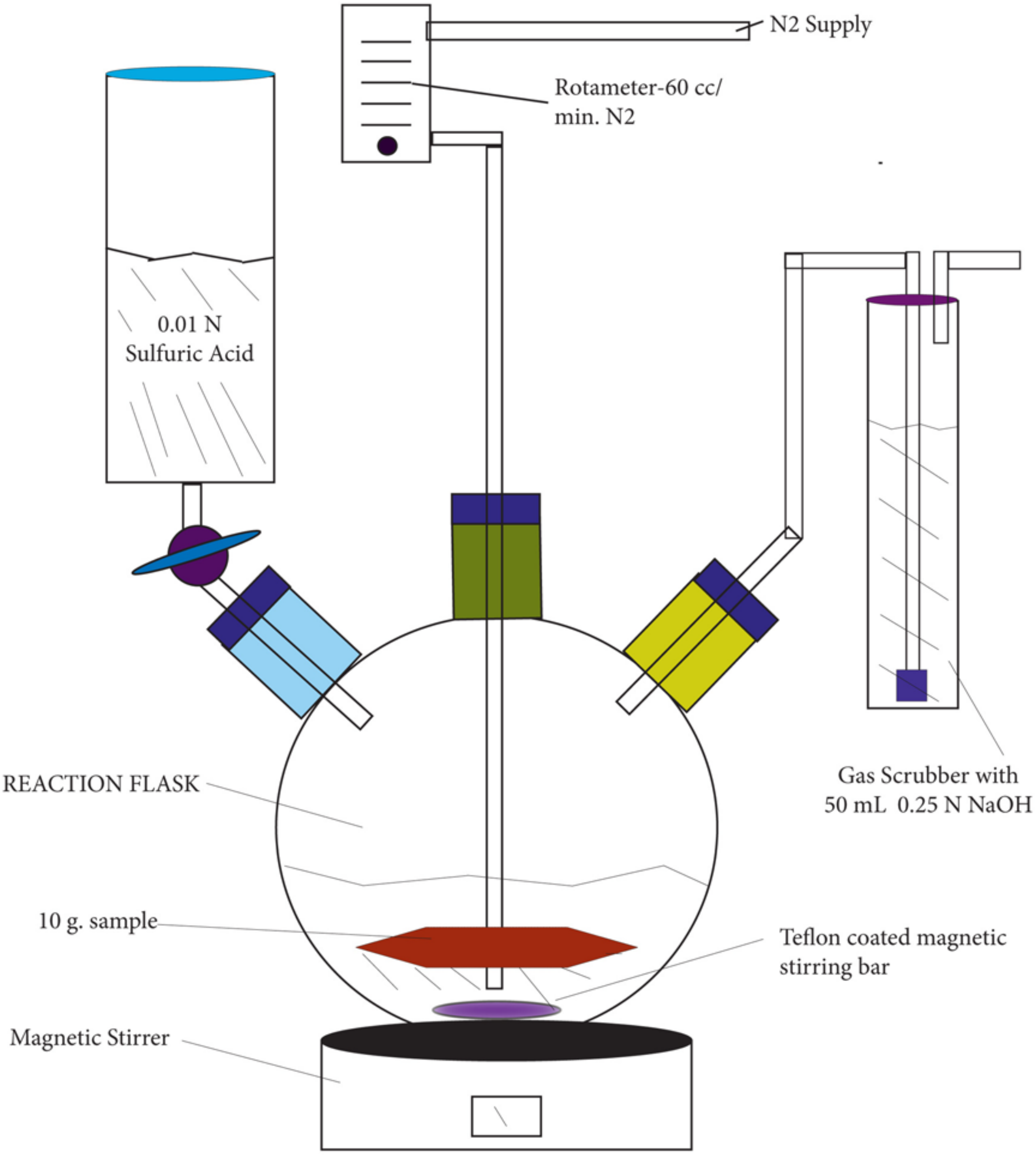
U.S. EPA SW846 Chapter 7.3- Reactivity- Revision 3, December 1996

YORK SOP WCCNT070900

15. REVISION HISTORY

08/08/2000	Revision 1.0	Original Document
11/05/2000	Revision 1.1	Added sections 7.0 and 8.0
10/16/2012	Revision 1.2	Revised all sections to meet NELAC 23 points and clarified procedural items including materials and methodology
10/22/2015	Revision 1.3	Reagents concentration updated, Original date removed from SOP title
04/03/2018	Revision 1.4	Updated Quality Assurance Manager position and removed LCS requirements
12/02/2021	Revision 1.5	Updated QA Officer, Lab Director, headers and footers. Sbw
4/11/2023	Revision 1.6	General clean-up of some misspellings

Figure 1.0- Releasable Cyanide and Sulfide Apparatus



Standard Operating Procedure

Analysis of Reactivity-Sulfide as Releasable Sulfide According to EPA SW-846 Ch 7.3.3

Approvals

Laboratory Director ______________________
Cassie Mosher

Sr. Scientist/Technical Director ______________________
Robert Bradley

QA/QC Officer ______________________
Sarah Widomski

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Issued to: ______________________________

REACTIVE-Sulfide (Releasable Sulfide)

1. SCOPE AND APPLICATION

This method is applicable to all wastes, with the condition that wastes that are combined with acids do not form explosive mixtures.

The regulation in 40 CFR 261.23 defines reactive wastes to include wastes that have any of the following properties: (1) readily undergo violent chemical change; (2) react violently or form potentially explosive mixtures with water; (3) generate toxic fumes when mixed with water or, in the case of cyanide- or sulfide-bearing wastes, when exposed to mild acidic or basic conditions; (4) explode when subjected to a strong initiating force; (5) explode at normal temperatures and pressures; or (6) fit within the Department of Transportation's forbidden explosives, Class A explosives, or Class B explosives classifications.

This SOP only addresses item (3) above: It is a cyanide- or sulfide-bearing waste which, when exposed to pH conditions between 2 and 12.5, can generate toxic gases, vapors, or fumes in a quantity sufficient to present a danger to human health or the environment.

This method provides a way to determine the specific rate of release of hydrogen sulfide upon contact with an aqueous acid. The interim guidance for reactive sulfide level is: Total releasable sulfide: 500 mg H_2S/kg waste. The reporting limit of the method as defined is 15.0 mg/kg reactive sulfide.

1.4 This test measures only the hydrogen sulfide evolved at the test conditions. It is not intended to measure forms of cyanide other than those that are evolvable under the test conditions.

1.5 It is noted that this method has been withdrawn by EPA. Many disposal facilities are still requiring this therefore it is necessary until otherwise noted.

2. SUMMARY

An aliquot of weak sulfuric acid is added to a fixed weight of waste (10g.) in a closed system. The generated gas is swept into a sodium hydroxide scrubber. The sulfide in the scrubber solution is quantitated. The procedure for quantitating the sulfide is Method 9034. The sulfide is oxidized to sulfur by adding a known excess amount of iodine. The excess iodine is determined by titration with a standard solution of phenyl arsine oxide (PAO) or sodium thiosulfate until the blue iodine starch complex disappears. As the use of standard sulfide solutions is not possible because of oxidative degradation, quantitation is based on the PAO or sodium thiosulfate. Refer to York SOP WC Sulfide.

This method is based upon EPA SW-846 Chapter 7, Section 7.3.3, Revision 3 December 1996.

3. DEFINITIONS

Reactivity-Sulfide- the hydrogen sulfide (H2S) evolved at the test conditions described herein. The test determines the specific rate of release of H2S upon contact with an aqueous acid. The interim guidance for reactive Sulfide level is: Total releasable Sulfide: 500 mg H2S/kg waste. The reporting limit of the method as defined herein is 2.0 mg/kg reactive sulfide (releasable sulfide).

Analytical Batch Client samples (20 or fewer) and associated QC samples (method blank and sample duplicate) analyzed at one time.

Method Blank Deionized water (for aqueous matrices) or Ottawa sand, (for solid matrices) treated exactly as a sample, and run with each analytical batch.

Reporting Limit (RL) The lowest point of quantitation based upon the lowest point in the calibration curve, if applicable. This ideally is less than or equal to desired regulatory action levels.

Sample Duplicate A client sample analyzed a second time in the same analytical batch. This is used to determine method reproducibility and matrix homogeneity.

4. INTERFERENCES

The iodometric method suffers interference from reducing substances that react with iodine, including thiosulfate, sulfite, and various organic compounds.

5. SAMPLE HANDLING

Samples containing, or suspected of containing, sulfide or a combination of sulfide and cyanide wastes should be collected with a minimum of aeration. The sample bottle should be filled completely, excluding all head space, and closed tightly.

Storage of samples should be under refrigeration (2-6C) and in the dark.

Testing may be performed in a ventilated hood.

Containers recommended are 2 or 4 oz. glass with Teflon lined lids.

The holding times are not established, however it is recommended that analysis should commence as soon as possible, and samples should be kept in a cool, dark place until analysis begins. Holding time should be considered as 14 days from collection.

6. APPARATUS (See Figure 1)

- Round-bottom flask - 500-mL, three-neck, with 24/40 ground-glass joints.

- Gas scrubber - 50 mL scrubber-either calibrated or add 50 mL with a Class A pipet to the scurbber

- Magnetic Stirring apparatus - To achieve approximately 30 rpm. This may be either a rotating magnet and stirring bar combination

- Addition funnel - With pressure-equalizing tube and 24/40 ground-glass joint and
- Teflon sleeve.

- Flexible tubing - For connection from nitrogen supply to apparatus.

- Prepurified nitrogen gas - With two-stage regulator.

- Rotometer - For monitoring nitrogen gas flow rate. 0-200 ml/min or equivalent

- Top loading balance - capable of weighing to 0.01 g.

- Ottawa Sand or cyanide/sulfide free sand-can be created by muffling fine sand at 650C for 2 hours and stored in a sealed glass container until use.

7. REAGENTS AND STANDARDS

Reagent grade inorganic chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

Reagent water- All references to water in this method refer to organic free deinoized water delivered by the York water purification system.

Sulfuric acid (1.0 N)- Add 28 mL concentrated H2SO4 to Deionized water and dilute to 1 L.

Sodium hydroxide scrubber solution (0.25N)- Dissolve 10.0 g of NaOH in DI water and dilute to 1 liter with DI water.

Hydrochloric acid (6N)- J.T Baker

Iodine solution (0.05 N)- Standard Material, J.T Baker

Method Blank-10 g Ottawa sand

Starch indicator solution - BDH

Sodium Thiosulfate (0.025 N)- BDH #7227-1 (or equivalent) purchased with certificate of analysis

Ottawa sand or cyanide/sulfide free sand (can be prepared by taking any sand and muffling for 2 hours @ 650C).

8. PROCEDURE

Set up the apparatus as shown in Figure 1.0.

8.1 Add 50 mL of 0.25N NaOH solution to a calibrated scrubber or use a Class A pipet.

8.2 Close the system and adjust the flow rate of nitrogen, using the rotometer. Flow should be 60 mL/min.

8.3 Add 10.0 g of the waste to be tested to the system. Close the system immediately.

8.4 With the nitrogen flowing, add 100 mL 1.00 N sulfuric acid to the flask. Start the sir bar to mildly stir the contents with no vortex created. Start the 3 minute test period. The stirring speed must remain constant throughout the test.

8.5 After 3 minutes, close off the nitrogen and disconnect the scrubber. Determine the amount of H2S in 25 mls of the 50 mL scrubber solution using the titrimetric procedure described below in Section 8.7 et seq.. If desired, RETAIN the remaining 25 mL of scrubber solution for Reactive Cyanide determination- see SOP WC CNR

8.6 Run a method blank and a duplicate with each batch of 20 or fewer/day.

8.7 <u>Determination of sulfide in scrubber solution</u>- Using a Class A pipet, transfer 25 ml scrubber solution into a 150 ml flask.

  8.7.1 Pipet a 2.5 mL of standardized 0.05 N iodine solution in a 500-mL flask.

  8.7.2 Add 2 mL of 6N HCl to the flask. If at any point the amber color of the iodine disappears or fades to yellow, more (exact known amount) 0.05 N iodine must be added and recorded in the log book. This additional amount must be added to the amount from Step 8.7.1 for calculations. Record the total volume of standardized 0.05 N iodine solution used.

8.7.3 Titrate the solution in the flask with standard 0.025N sodium thiosulfate solution (swirl the flask often to mix) until the amber color fades to yellow.

8.7.4 Add enough starch indicator for the solution to turn dark blue and continue to titrate until the blue disappears. Record the volume of titrant used to reach this endpoint.

9. CALIBRATION

The materials for the titrimetric procedure are purchased as standard materials of known, certified normalities. A method blank and duplicate are run with each batch to verify the efficacy of the standard materials.

10. QA/QC

Each Analytical Batch must include a method blank and sample duplicate.

The method blank must have releasable sulfide <10.0 mg/kg for soils or <2.0 mg/L for waters.

Corrective Action: Clean system as needed. Repeat analytical batch.

The sample duplicate RPD should be < 50%.

Corrective Action: Rerun the sample and duplicate. If this problem still persists, flag the data accordingly. If samples are "Not detected" no precision value is meaningful.

11. CALCULATIONS AND REPORTING

All data are entered into the laboratory logbook.

Determine the specific rate of release of H2S, using the following parameters:

From the titrimetric data:

Calculate the concentration of sulfide in the scrubber solution 25 mL as follows:

mg/L Sulfide in distillate = (“X”) =

[(mL of I_2 x N of I_2) - (mL of 0.025N $Na_2S_2O_3$ x N of $Na_2S_2O_3$)] x (16000)

Volume of distillate titrated (mls) (25 ml)

For example, if the N of I2 = 0.050N and 5.0 mL was used; and it took 2.1 mL of 0.025N $Na2S_2O3$ and 25 mL was used as volume of distillate then the mg/L sulfide would be:

$$\frac{[(5.0 \times 0.050) - (2.1 \times 0.025)] \times 16000}{25.0 \text{ mL}} = 126.4 \text{ mg/L Sulfide}$$

This number is then used in the equation below and is "X"

X = Concentration of H2S in scrubber solution (mg/L)
(This is obtained from the Titrimetric method)

L = Volume of solution in scrubber (L) (50 mL = 0.050 liters but we are using 25 mL so 0.025 L)

W = Weight of waste used (kg)-we use 10.0 g. or 0.010 kg

S = Time of measurement (sec.) = Time N stopped - Time N started =should be around 180 seconds (3 minutes x 60 sec/min)

R = specific rate of release (mg/kg/sec.) = $\frac{X \bullet L}{W \bullet S}$

Total releasable H2S (mg/kg) = R x S

Example: if we used 10.0 g. of sample and the cyanide in 25 mL of scrubber was determined to be 126.4 mg/L and the procedure was conducted for 30 minutes (1800 seconds) the results would be:

$$R = \frac{126.4 \times 0.025 \text{ L}}{0.010 \times 1800} = 0.17556$$

Total Releasable Sulfide (mg/kg) = 0.17556 x 1800 = 316.0 mg/kg

This would be classified as non-reactive (< 500 mg/kg)

This value would is entered in LIMS.

12. HEALTH AND SAFETY

General safety considerations and requirements are detailed in the York Laboratory Safety and Health Standard Operating Procedure No. Safety011600.
Specific safety rules applying to the conduct of this analysis requiring the following:

- When handling standards and samples, latex gloves are required.
- Also, when handling neat materials, a fume hood and safety glasses are required.
- When handling samples, gloves and glasses are required.
- Highly odorous samples must be handled in a fume hood.
- Refer to MSDSs for specific safety/health information

The analysts must exercise normal care and be supervised and trained to work in an analytical chemistry laboratory. The analysts will be handling fragile glassware, needles, syringes, volatile and flammable chemicals, toxic chemicals and corrosive chemicals.

No smoking or open flames are allowed.
No food or food products may be brought into the laboratory.

Solvents should not be left uncovered on the laboratory benches.
All solvent transfers should be done in the hoods.
Hood doors must be kept in the position which yields approx. 100 fpm face velocity.
Venting of separatory funnels must be done in the hood.
Solvent evaporation must be done in the hood with exhaust elevated and in the rear.

Waste containers that had solvents must be vented to a hood until all solvents have evaporated.
Strong acids and bases must be handled with gloves and appropriate eye ware.
Concentrated acids must be used and kept under fume hoods designated for acids.
Safety glasses are provided and must be worn at all times in the laboratory.
Latex gloves are provided and must be worn when working with chemicals.
Laboratory coats are provided and should be worn to protect the analysts' clothes.
Syringes and needles must be kept in their original cases when not in use.
Care must be exercised in using and handling syringes to avoid injury.
Report any sticking with a needle immediately to your supervisor.

13. WASTE MANAGEMENT/POLLUTION PREVENTION

Neat Materials

Waste management procedures require the prudent use of neat materials. The ordering of neat standards and materials must be done to minimize unused material which would result in storage or handling of excess material. Quantities ordered should be sufficient to provide for necessary standards with consideration to shelf life. When ordering a unique material for a standard, be sure to order the smallest practical quantity.

Acids/Bases

Our laboratory employs the use of concentrated acids (Hydrochloric, Nitric and Sulfuric acids). These acids are utilized for sample preparation, glassware preparation and standards preparation. All acid rinsing of glassware is done to minimize the amount of acid used. The amount of the particular acid used is minimized to only the volume necessary to reduce the amount of acid waste after processing samples.

The concentrated bases used at York include Sodium Hydroxide and Ammonium Hydroxide. These bases are used to prepare various molar or normal solutions for specific analyses such as cyanide, etc. The amounts prepared are minimized to that needed for a particular application.

Samples

Unused or remaining soil and water samples are returned to the sample control room for continued storage for proper disposal by the sample control group.

14. REFERENCES

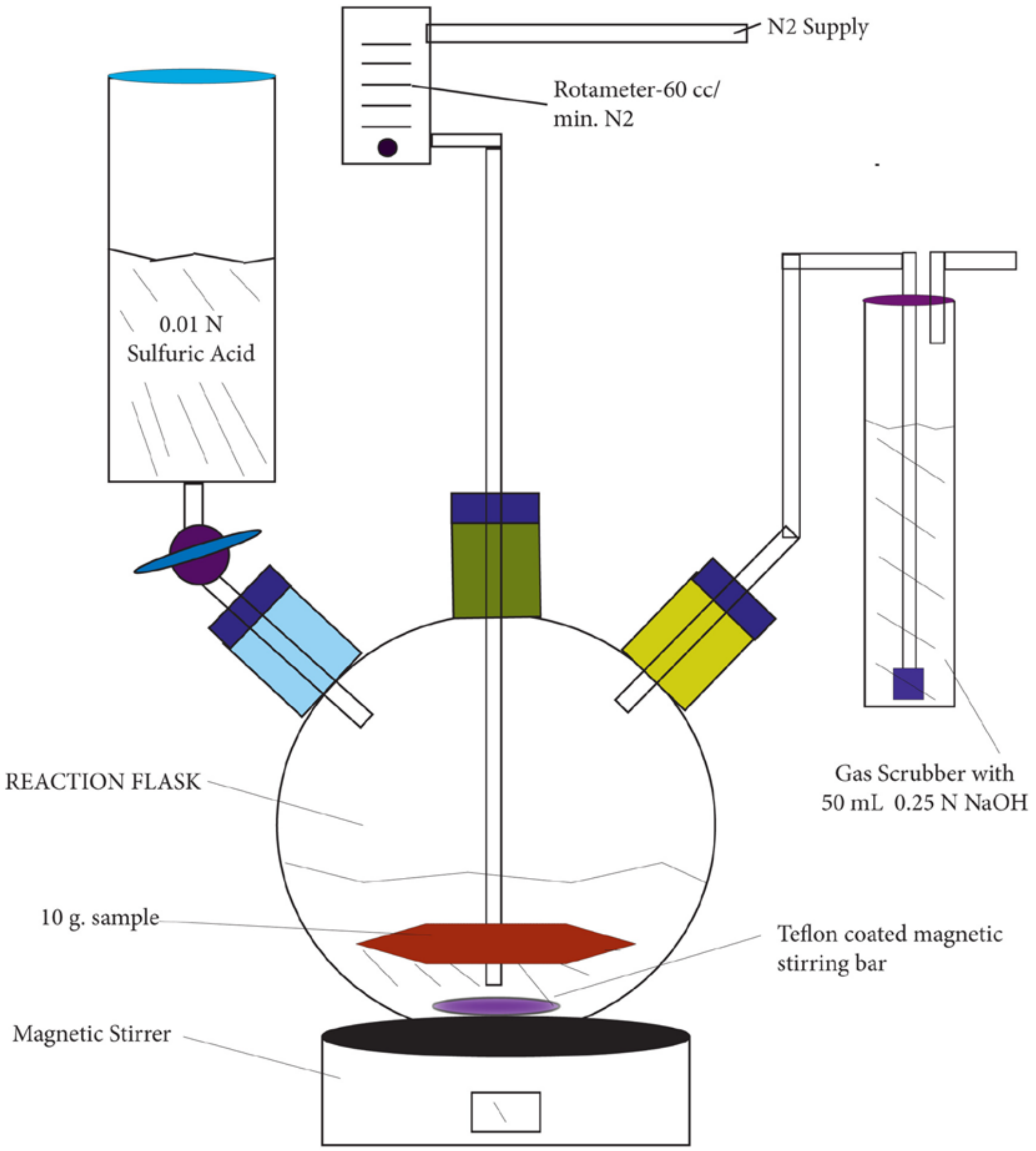
U.S. EPA SW846 Chapter 7.3- Reactivity- Revision 3, December 1996

U.S. EPA SW846 Method 9034-Titrimetric Procedure for determination of Acid soluble and Acid Insoluble Sulfides-Revision 0, December, 1996.

15. REVISION HISTORY

08/08/2000	Revision 1.0	Original Document
11/05/2000	Revision 1.1	Added sections 7.0 and 8.0
10/16/2012	Revision 1.2	Revised all sections to meet NELAC 23 points and clarified procedural items including materials and methodology. Also added specifics to procedural items for acid concentration, etc., Figures.
10/19/2012	Revision 1.3	Reagents concentration updated, Date removed from SOP title.
04/03/2018	Revision 1.5	Updated Quality Assurance Manager position and removed LCS requirements
12/02/2021	Revision 1.6	Updated QA Officer, lab Director, headers and footers. Sbw
4/11/2023	Revision 1.7	sec 1.5 removed " due to inaccuracies "

Figure 1.0 Sulfide/Cyanide Reactivity Apparatus



Standard Operating Procedure

Leaching Procedures for Aqueous and Solid Samples by EPA SW-846 1311 or 1312, Modified Morgan and ASTM D3987

Approvals

Laboratory Director ______________________
Cassie Mosher

Sr. Scientist/Technical Director ______________________
Robert Bradley

QA/QC Officer ______________________
Sarah Widomski

Controlled Copy No. WC TCLPEX-Rev 1.8- ______

Issued to: ___________________________

Leaching Procedures

1. SCOPE AND APPLCATION

This method is designed to determine the mobility of organic and inorganic analytes present in liquid, solid, and multiphasic wastes.

The concentration levels of analyte suitable for this method vary by analysis type and analyte. Refer to specific analyses in the LIMS to determine reporting limits

This method is to be performed only by a trained analyst who has completed a Demonstration of Capability within the last 12 months. Please refer to York SOP Adm080206 for additional training instructions.

2. SUMMARY

This procedure is based upon EPA SW-846 methods 1311 and 1312.

After an initial evaluation of the solids content of a waste sample, 100 g (25 g for volatiles evaluation) are combined with extraction fluid and tumbled at 30 rpm for 18 hours. This procedure is designed to imitate the environmental potential of the waste to leach toxics into groundwater. In the TCLP procedure, the extraction fluid used is a function of the alkalinity of the sample. In the SPLP procedure, the extraction fluid used is a function of the region of the country where the sample site is located. Samples from east of the Mississippi River are subjected to extraction fluid with a lower pH than samples from west of the Mississippi River. Following extraction, the liquid extract is separated from the sold phase by filtration and analyzed. A Zero Headspace Extractor is used when testing for volatile analytes.

3. DEFINITIONS

SPLP Synthetic Precipitation Leaching Procedure

TCLP Toxicity Characteristic Leaching Procedure

Analytical Batch Client samples (20 or fewer) and associated QC samples (method blank, sample duplicate) analyzed at one time.

Method Blank Extraction fluid treated exactly as a sample, and run with each analytical batch. Use the extraction fluid most common in that batch.

Reporting Limit (RL) The lowest point of quantitation. This ideally is less than or equal to desired regulatory action levels.

Sample Duplicate A client sample analyzed a second time in the same analytical batch. This is used to determine method reproducibility and matrix homogeneity.

4. INTERFERENCES

Potential interferences that may be encountered during analysis are discussed in the individual analytical methods.

5. SAMPLE HANDLING

Samples must be unpreserved and be in either glass or plastic containers.

Samples must be stored at 4 ± 2° C until analyzed unless refrigeration results in irreversible physical change to the waste.

Maximum Holding Times (in days)

Item	From collection to leaching	From leaching to extraction	From leaching or extraction to analysis
Volatiles	14	NA	14
Semi-volatiles	14	7	40
Mercury	28	NA	28
Metals (except Hg)	180	NA	180

Note: NA = not applicable

6. APPARATUS

- Zero-Headspace Extraction (ZHE) vessel capable of in-line filtration **(Attachment 1)**
- Pressurization tank **(Attachment 2)**
- Extraction vessels, Scientific Specialties #134064 (or equivalent) or HDPE Daniel's Scientific, 2 liter (or less) or equivalent for metals; Environmental Express BP-2000-1 2.2 liter glass extraction vessels with Teflon-lined lids for semi-volatiles (PEST/BNA/Herb)/metals extractions.
- Tumbling apparatus capable of rotating @ 30 ± 2 rpm **(Attachment 2)**
- Positive pressure filtration device **(Attachment 3)**
- TCLP glass fiber filters, 0.7µm nominal, 142 and 90 mm
- Analytical balance

7. REAGENTS AND STANDARDS

ALL STANDARD AND REAGENT INFORMATION (i.e. manufacturer, lot#, date opened, date finished) MUST BE ENTERED INTO THE FRONT OF THE LOG BOOK.

In addition, at the bottom each page, the standards and reagents used that day must be listed and traceable to the information in the front of the log book.

Reagent water - Water in which an analyte is not observed at or above its method detection limit

Hydrochloric acid (1N) – Dilute 20.75 ml HCl (reagent grade) to 250 ml with reagent water.

Sodium hydroxide (10N) - Dilute 400g NaOH (reagent grade) to 1 liter with reagent water

TCLP Extraction fluid #1 - Add 285 ml glacial acetic acid (reagent grade) to approximately 10 liters of reagent water. Add 322 ml *10N NaOH*. Dilute to 50 liters. The pH of this fluid must be 4.93 ± 0.05. Add additional acid or base if necessary.

TCLP Extraction fluid #2 - Dilute 5.7 ml glacial acetic acid (reagent grade) to 1 liter. The pH of this fluid should be 2.88 +/-0.05. Add additional acid or base if necessary.

SPLP Extraction fluid #1 - Adding a 60/40 weight percent mixture of sulfuric and nitric acids (reagent grades), or a suitable dilution, to reagent water until the pH is 4.20 ± 0.05. The fluid is used to determine the leachability of soil from a site that is east of the Mississippi River, and the leachability of wastes and wastewaters.

SPLP Extraction fluid #2 - Follow procedure as for SPLP fluid #1, except take to a pH of 5.00 ± 0.05. This fluid is typically not required in Connecticut.

SPLP Extraction fluid #3 – Reagent water, used to determine cyanide and volatiles leachability.

Modified Morgan fluid – Dilute 143.7 mL glacial acetic acid and 92 mL ammonium hydroxide to one liter with DI water. Adjust pH to 4.8.

8. PROCEDURE

8.0 Vessel Handling Procedure

Per Section 8.1A of EPA SW846-1311, a vessel must be challenged as a blank after 20 uses:

`"A minimum of one blank (using the same extraction fluid as used for the samples) must be analyzed for every 20 extractions that have been conducted in an extraction vessel."`

In order to accomplish this, each extraction vessel numbered. This number is entered in the TCLP Extraction log with each extraction batch. Once the vessel has been used for 20 extractions, this vessel becomes the method blank vessel and is documented as such in the TCLP Extraction log, a copy of the log is attached as Attachment 4.

8.1 Preliminary Evaluation

8.1.1 Percent solid evaluation

The solids fraction of a waste sample is that from which no liquid may be forced out by an applied pressure. If the waste will obviously yield no free liquid upon filtration, go to step 8.1.2.

If the waste is mostly liquid or multi-phasic, separate liquid and solids by filtering through a pre-weighed filter. Store liquid at 4°C until step 8.4. With the solid phase, proceed to step 8.1.2. Record percent solids in TCLP/SPLP log book.

Samples exhibiting percent solids of less than 0.5% are defined as their own extract. Proceed to step 8.6.

8.1.2 Particle size reduction

A solid waste that exhibits measurements greater than one centimeter in any dimension must be crushed or cut so it will pass through a 9.5 mm (0.375 inch) standard sieve. If size reduction is required, note in TCLP/SPLP log book.

8.1.3 Determination of extraction fluid

For TCLP extraction of non-volatile constituents, perform the determination of the appropriate fluid.

Note: TCLP extraction of volatile constituents uses extraction fluid #1 ONLY

Weigh out 7 gm of sample into an Erlenmeyer flask.

Add 96.5 ml reagent water. Cover with watch glass and stir for 5 minutes. Measure and record pH in TCLP/SPLP logbook. If pH is <5.0, use extraction fluid #1. B

If the pH is >5.0, add 3.5 ml 1N HCl and stir for 5 minutes.

Record pH in TCLP logbook. If pH is < 5.0 use extraction fluid #1. If the pH is >5.0 use extraction fluid #2.

Note the extraction fluid used in TCLP/SPLP log book.

For SPLP extraction of soils from a site east of the Mississippi River, SPLP fluid #1 should be used. From a site west of the Mississippi, use fluid #2.

For SPLP extraction of wastes and wastewater, use SPLP fluid #1.

For SPLP extraction of cyanide containing wastes and/or soils, use extraction fluid #3 to prevent formation of hydrogen cyanide gas.

For ASTM 3987 extraction of solid wastes, use reagent grade water.

Note the extraction fluid used in TCLP/SPLP log book.

8.2 Extraction (metals and semi-volatiles):

Add 100 g sample and 2 liters of the appropriate fluid to the extraction vessel. If less than 100 g of sample was provided, the amount of fluid must be reduced proportionately. An exception to this may be made *only* with analyses of *homogeneous soil samples* requiring one liter or less extract. (i.e. for SVOA analysis only, 50 g may be extracted with 1 liter fluid, or for metals only, 12.5 g may be extracted with 250 ml fluid.) Enter the sample weight and extraction fluid type and volume into the TCLP/SPLP log book. Close the vessel and secure in rotary agitation apparatus. Vessel should be rotated end-over-end for 18 ± 2 hours at ambient temperature (23 ± 2°C). The extraction vessel may be vented as necessary throughout the extraction. Take the pH of the fluid after the extraction and record in TCLP/SPLP log book.

8.3 Extraction (volatiles):

Samples should be opened only immediately prior to extraction to prevent loss of volatile compounds.

Place a glass fiber filter between the two support screens in the top plate of the ZHE vessel. Adjust the piston until it is near the top of the barrel. Add sample using a 25 g Encore sample if available. Place top plate securely on top of the barrel. Tighten hand wheels as securely as possible using the hand wheel wrench. Pour a volume of TCLP #1 fluid into the pressurization tank equal to 20 times the weight of the sample. (i.e. 25 g sample/500 ml fluid) Enter the sample weight and extraction fluid volume into the TCLP/SPLP log book. Connect fitting from pressurization tank to top of ZHE. Dispense extraction fluid into ZHE.

Disconnect fitting from top of ZHE. Eliminate headspace through vent/release valve.

Secure ZHE in the rotary agitation apparatus. Vessel should be rotated end-over-end for 18 ± 2 hours at ambient temperature (23 ± 2°C). Do not subject the sample to the atmosphere any more than is absolutely necessary.

8.4 Filtration (Metals and Semi-volatiles)

Quantitatively transfer contents of the extraction vessel to the positive pressure filtration device equipped with glass (GF) filter. Gradually apply pressure. Collect filtrate/extract.

Any liquid obtained from filtration in step 8.1.1, should be combined with the extract at this point. If liquid is incompatible with extract, analyze both fractions separately and mathematically combine results reflecting liquid/solid proportion established by the percent solid evaluation.

Extract or combined extract must be preserved as follows:

> Metals: Extracts to be analyzed for metals, excluding Hg, shall be acid digested prior to analysis.
>
> Semi-volatiles: Refrigerate at ≤6°C until analysis.

8.5 Filtration (Volatiles from ZHE)

Prepare in-line glass fiber filter. Using pressure from ZHE, filter sample into a gas-tight syringe or directly into a 40 mL VOA vial. This extract can be combined with any filtrate from step 8.1.1 if applicable.

Extract must be stored without headspace @ ≤6°C until analysis.

8.6 Modified Morgan Extraction

Add 20 g soil to 100 mL Modified Morgan fluid. Tumble 15 minutes. Filter.

8.7 Analysis

Extracts prepared for metals, semi-volatiles, and volatiles should be analyzed in accordance with applicable extraction and analysis SOPs.

9. CALIBRATION

The max/min thermometer must be annually recertified against a NIST thermometer and labeled with temperature reading correction, if applicable.

Record pH meter calibration daily, or with each use, in pH calibration log.

10. QUALITY CONTROL

Each analytical batch must contain a method blank and a sample duplicate.

11. CALCULATIONS AND REPORTING

No calculations are required for this procedure.

12. HEALTH AND SAFETY

Each chemical should be regarded as a potential health hazard and should be as low as reasonably achievable.

General safety considerations and requirements are detailed in the York Laboratory Safety and Health Standard Operating Procedure No. Safety011600.

Specific safety rules applying to the conduct of this analysis require the following:

- Sulfuric acid has the potential to be highly hazardous. Please consult MSDS.
- When handling standards and samples, latex gloves are required.
- When handling neat materials, a fume hood and safety glasses are required.
- When handling samples, gloves and glasses are required.
- Highly odorous samples must be handled in a fume hood.
- Refer to MSDSs for specific safety/health information.

The analysts must exercise normal care and be supervised and trained to work in an analytical chemistry laboratory. The analysts will be handling fragile glassware, needles, syringes, volatile and flammable chemicals, toxic chemicals and corrosive chemicals.

> No smoking or open flames are allowed.
> No food or food products may be brought into the laboratory.

Hood doors must be kept in the position which yields approx. 100 fpm face velocity.

Strong acids and bases must be handled with gloves and appropriate eye wear.

Concentrated acids must be used and kept under fume hoods designated for acids.
Safety glasses are provided and must be worn at all times in the laboratory.

Latex gloves are provided and must be worn when working with chemicals.
Laboratory coats are provided and should be worn to protect the analysts' clothes.

All work must be stopped in the event of a known or potential compromise to the health and safety of a York associate. The situation must be reported immediately to a laboratory supervisor and/or the Safety Officer.

13. WASTE MANAGEMENT /POLLUTION PREVENTION

Acids/Bases

Our laboratory employs the use of concentrated acids (hydrochloric, nitric and sulfuric acids). All acid rinsing of glassware is done to minimize the amount of acid used. The amount of the particular acid used is minimized to only the volume necessary to reduce the amount of acid waste after processing samples.

The concentrated bases used at York include sodium hydroxide and ammonium hydroxide. These bases are used to prepare various molar or normal solutions for specific analyses such as cyanide, etc. The amounts prepared are minimized to that needed for a particular application.

Samples

Unused or remaining soil and water samples are returned to the sample control room for continued storage for proper disposal by the sample control group.

14. REFERENCES

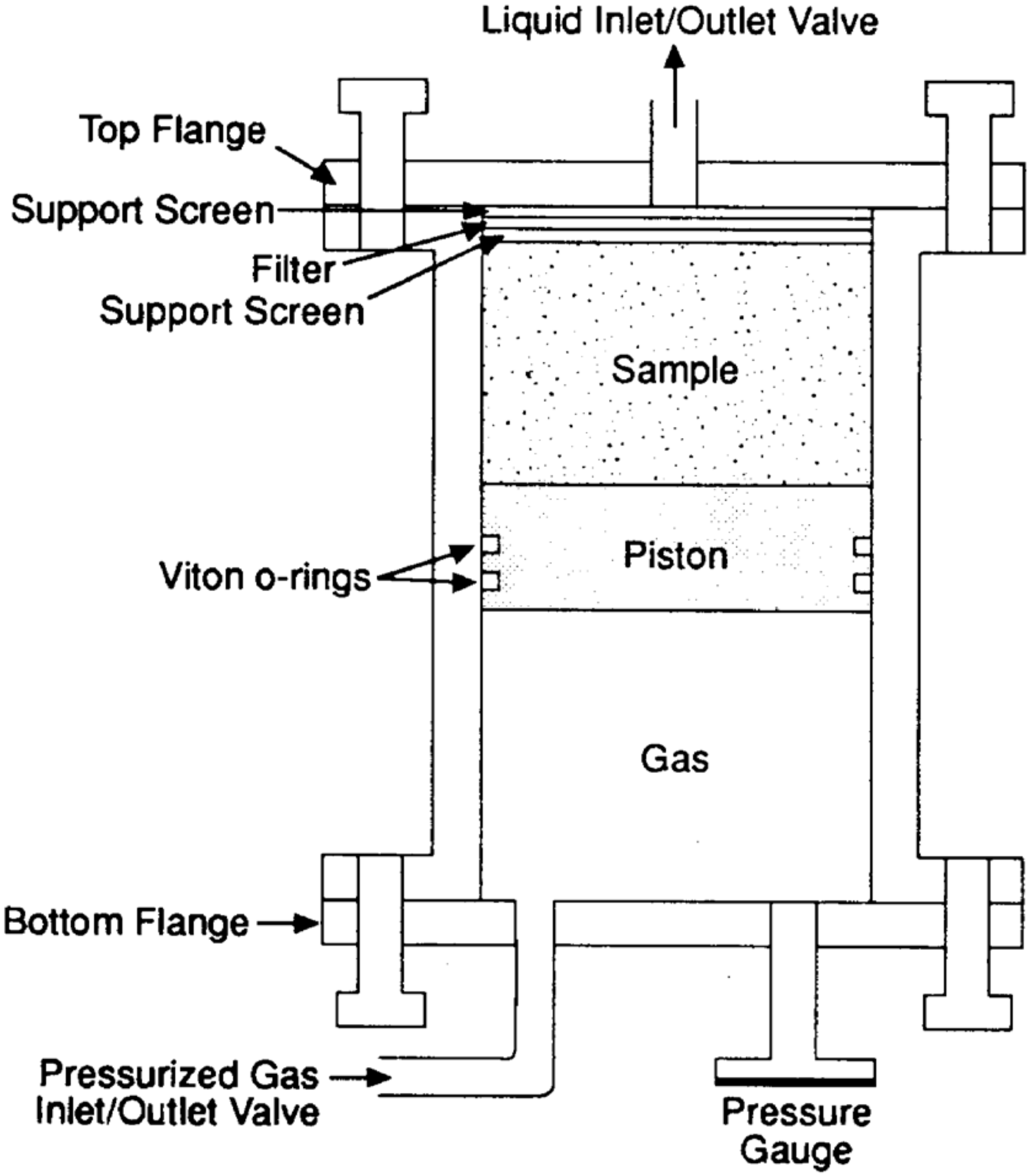
USEPA SW-846 Test Methods 1311 and 1312
ASTM D3987

15. REVISION HISTORY

Revision 1.0	01/04/2000	Original Issue
Revision 1.1	09/04/2006	Added SPLP extraction
Revision 1.2	02/29/2012	Corrected format numbering
Revision 1.3	10/11/2012	Updated format, combined TCLP/SPLP instructions, added equipment diagrams
Revision 1.4	01/05/2014	Added Modified Morgan procedure, Corrected metals holding times. Deleted original date from title
Revision 1.5	11/20/2014	Temperature corrected
Revision 1.6	03/28/2017	Modified Section 8 to add requirement to use each vessel as blank after 20 uses
Revision 1.7	06/04/2018	Added ASTM D3987- DiH2O Leaching and updated Quality Assurance Manager
Revision 1.8	12/02/2021 Sbw	Updated QA Officer, Lab Director, headers and footers.

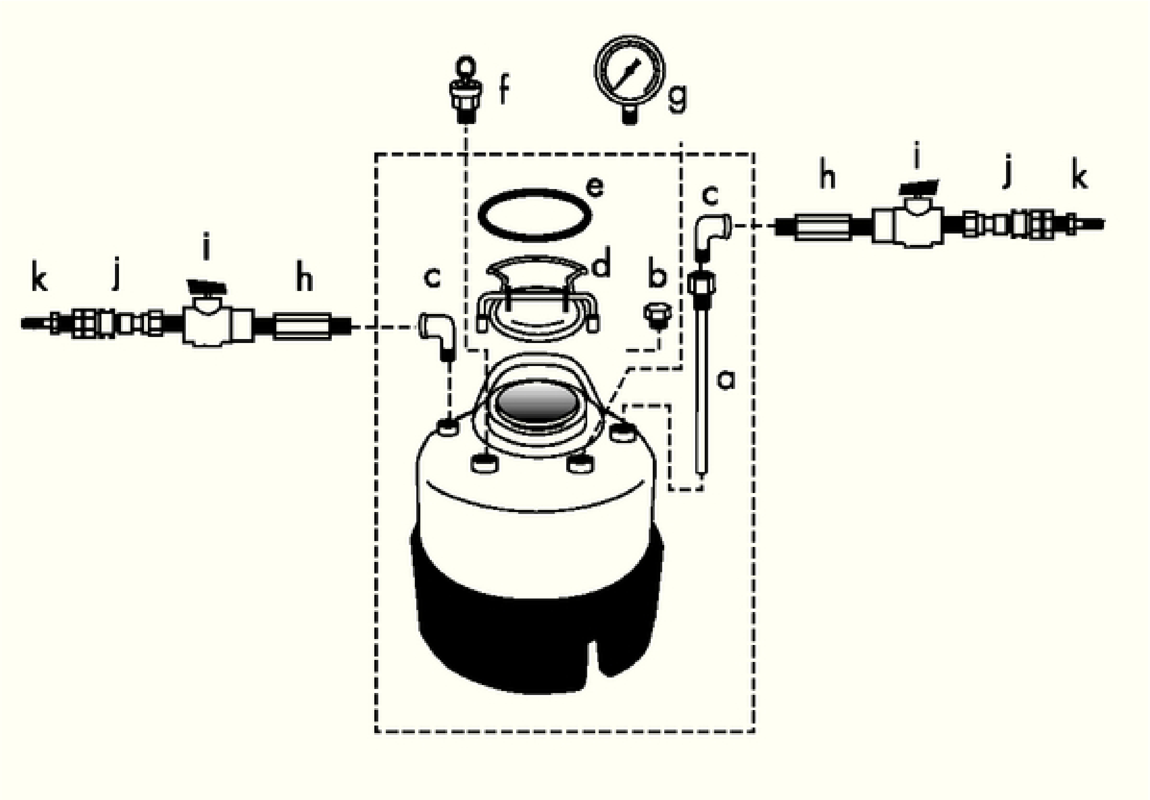
Attachment 1

Zero-Headspace Extractor (ZHE)

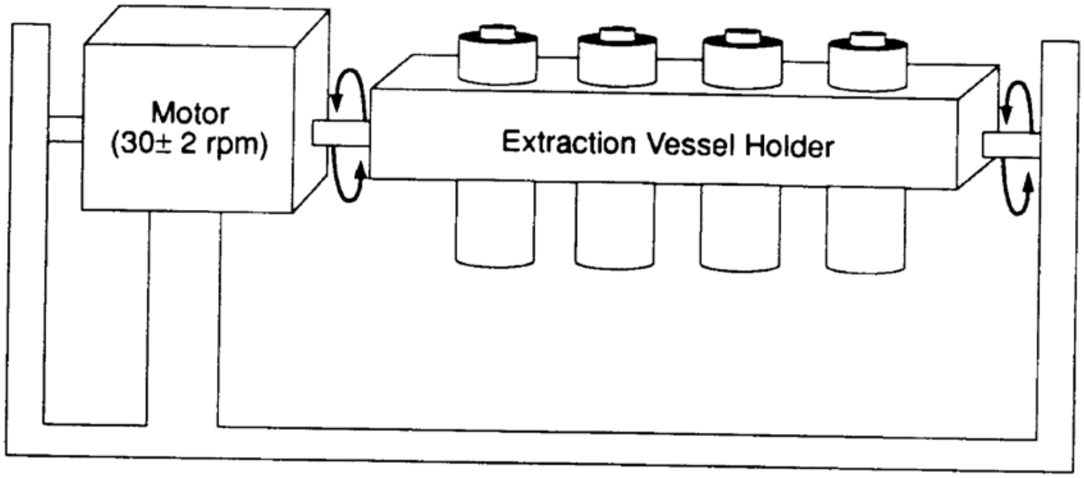


Attachment 2

Pressurization Tank

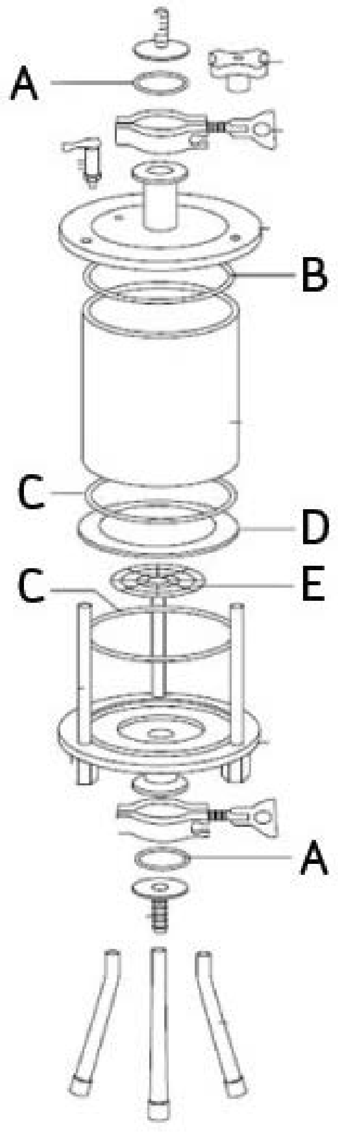


Tumbling Apparatus



Attachment 3

Pressure Filtration Device



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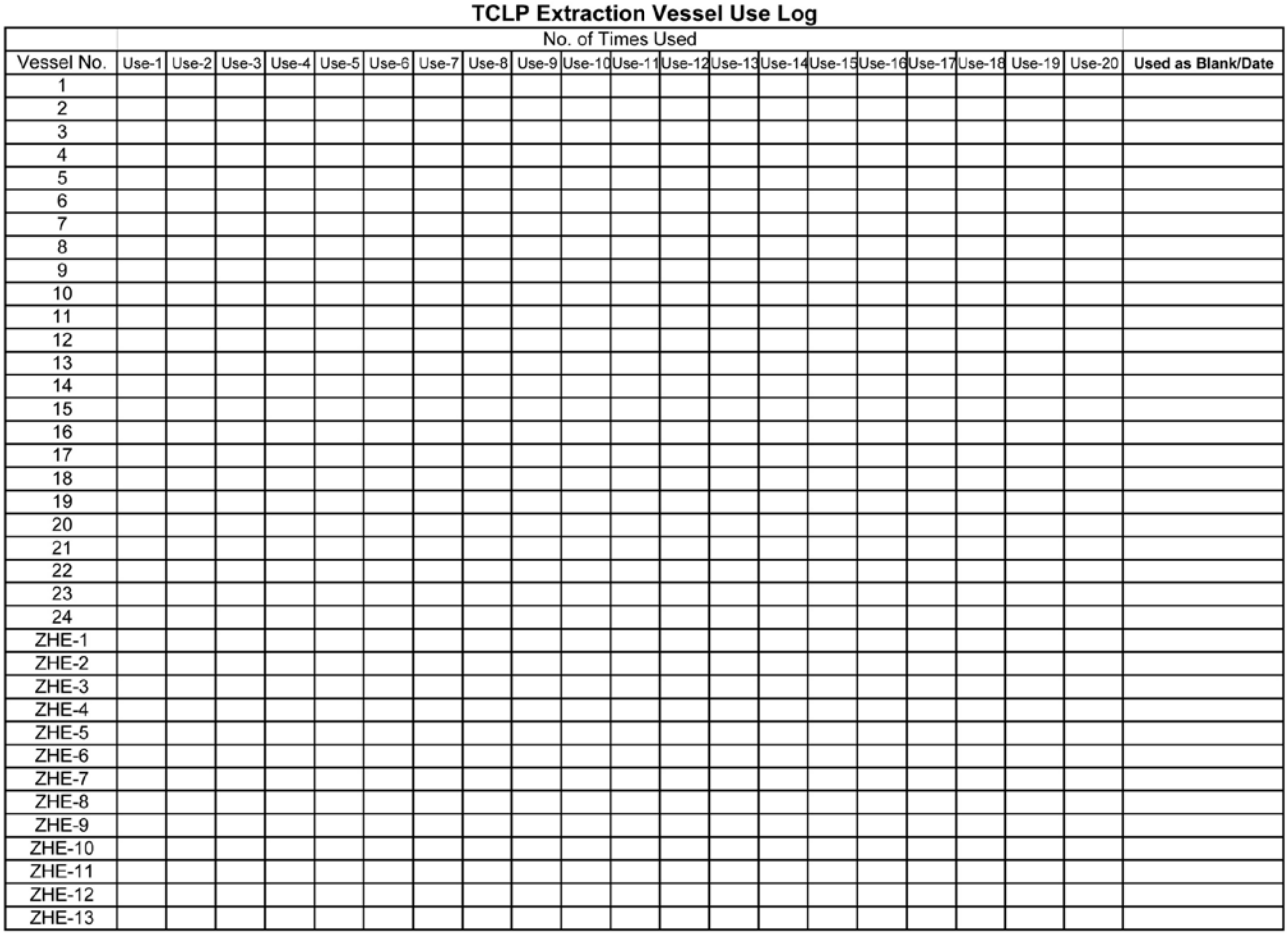

TCLP Extraction Vessel Use Log

	No. of Times Used																				
Vessel No.	Use-1	Use-2	Use-3	Use-4	Use-5	Use-6	Use-7	Use-8	Use-9	Use-10	Use-11	Use-12	Use-13	Use-14	Use-15	Use-16	Use-17	Use-18	Use-19	Use-20	Used as Blank/Date
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