

State Of Connecticut  
Department of Energy and Environmental Protection  
Recommended Reasonable Confidence Protocols  
Quality Assurance and Quality Control Requirements  
Toxicity Characteristic Leaching Procedure  
By SW-846 Method 1311  
Version 3.0  
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Written by the Connecticut DEEP QA/QC Workgroup

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***Acronym List***

<b><u>ACRONYM</u></b>	<b><u>DEFINITION</u></b>
CH <sub>3</sub> CH <sub>2</sub> OOH	Acetic acid
°C	Degrees Celsius
cm	centimeters
DEEP	CT Department of Energy and Environmental Protection
EP	Environmental Professional
g	grams
L	Liters
LLOQ	Lower limit of quantitation
µm	micrometers
mL	Milliliters
mm	Millimeters
MS	Matrix spike
MSD	Matrix spike duplicate
N	Normality (measure of concentration)
HCl	Hydrochloric acid
HDPE	High-density polyethylene
HNO <sub>3</sub>	Nitric Acid
H <sub>2</sub> SO <sub>4</sub>	Sulfuric Acid
N/A	Not applicable
NaOH	Sodium hydroxide
ND	Not detected
PCBs	Polychlorinated biphenyls
PP	polypropylene
psig	Pounds per square inch gauge
PVC	Polyvinyl chloride
QA	Quality assurance
QC	Quality control
RCP	Reasonable Confidence Protocol
RL	Reporting limit
RPD	Relative percent difference
RPM	Revolutions per minute
SVOCs	Semivolatile organic compounds
TCLP	Toxicity Characteristic Leaching Procedure
VOCs	Volatile organic compounds
VPH	Volatile petroleum hydrocarbons
ZHE	Zero headspace extractor

## **Quality Assurance and Quality Control Requirements for SW-846 Method 1311**

### **1.0 Overview of Method 1311**

Method 1311 is designed to determine the mobility of both organic and inorganic analytes present in liquids, soils, and wastes. Samples are leached (or extracted) with a dilute acetic acid or acetate solution and filtered. The resulting extract can be analyzed by a variety of methods to determine the pollutant mobility of various analytes or if a given waste is a hazardous waste.

All method references are to the latest promulgated version of the method found in Test Methods for Evaluating Solid Waste, SW-846.

### **2.0 Summary of SW-846 Method 1311**

For liquid samples (i.e., those containing less than 0.5 % dry solid material), the sample, after filtration through a 0.6 to 0.8  $\mu\text{m}$  glass fiber filter is defined as the TCLP extract.

For samples containing greater than 0.5 % solids, the liquid phase, if any, is separated from the solid phase and stored for later analysis; the particle size of the solid phase is reduced, if necessary. The solid phase is extracted with an amount of extraction fluid equal to 20 times the weight of the solid phase. The extraction fluid employed is a function of the region of the country where the sample site is located if the sample is a soil. If the sample is a waste or wastewater, the extraction fluid employed is a pH 4.2 solution. A special extractor vessel is used when testing for volatile analytes. Following extraction, the liquid extract is separated from the solid phase by filtration through a 0.6 to 0.8- $\mu\text{m}$  glass fiber filter.

If compatible (i.e., multiple phases will not form on combination), the initial liquid phase of the waste is added to the liquid extract, and these are analyzed together. If incompatible, the liquids are analyzed separately and the results are mathematically combined to yield a volume-weighted average concentration.

### **3.0 Interferences**

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

Significant losses of volatile organics may be encountered due to improper sample handling, both in the field and in the laboratory. Samples for TCLP volatiles should be collected and stored in “core and store” type sampling devices in accordance with the Connecticut Department of Environmental Protection’s *Guidance on Collecting and Preserving of Soil and Sediment Samples for Laboratory Determination of Volatile Organic Compounds*.

Upon receipt in the laboratory the sample container for volatiles must be stored frozen ( $-12^{\circ}\text{C} \pm 3^{\circ}\text{C}$ ). When preparing the sample for extraction, the laboratory must transfer the sample for extraction from the storage container to the zero headspace extractor (“ZHE”) as quickly as possible after the container is opened. The ZHE must be sealed as soon as the sample is added.

## **4.0 Apparatus and Materials**

### *4.1 Agitation apparatus*

The agitation apparatus must be capable of rotating the extraction vessel in an end-over-end fashion at  $30 \pm 2$  rpm. Suitable devices are identified in SW-846 Method 1311.

### *4.2 Extraction Vessels*

4.2.1 Zero Headspace Extraction Vessel. This device is for use only when the sample is being tested for the mobility of volatile analytes. The ZHE (depicted in SW-846 Method 1311) allows for liquid/solid separation within the device and effectively precludes headspace. This type of vessel allows for initial liquid/solid separation, extraction, and final extract filtration without opening the vessel (see Step 4.3.1). These vessels shall have an internal volume of 500-600 mL and be equipped to accommodate a 90-110 mm filter. The devices contain VITON® O-rings which should be replaced frequently. Suitable ZHE devices known to EPA are identified in SW-846 Method 1311.

For the ZHE to be acceptable for use, the piston within the ZHE should be able to be moved with approximately 15 pounds per square inch gauge (“psig”) or less. If it takes more pressure to move the piston, the O-rings in the device should be replaced. If this does not solve the problem, the ZHE is unacceptable for TCLP analyses and the manufacturer should be contacted. The ZHE should be checked for leaks after every extraction. If the device contains a built-in pressure gauge, pressurize the device to 50 psig, allow it to stand unattended for 1 hour, and recheck the pressure. If the device does not have a built-in pressure gauge, pressurize the device to 50 psig, submerge it in water, and check for the presence of air bubbles escaping from any of the fittings. If pressure is lost, check all fittings and inspect and replace O-rings, if necessary. Retest the device. If leakage problems cannot be solved, the manufacturer should be contacted.

Some ZHEs use gas pressure to actuate the ZHE piston, while others use mechanical pressure (see SW-846 Method 1311). Whereas the volatiles procedure (see Step 7.3) refers to psig, for the mechanically actuated piston, the pressure applied is measured in torque inch- pounds. Refer to the manufacturer's instructions as to the proper conversion.

4.2.2 Bottle Extraction Vessel. When the sample is being evaluated using the nonvolatile extraction, a jar with sufficient capacity to hold the sample and the extraction fluid is needed. Headspace is allowed in this vessel. The extraction bottles may be constructed from various materials, depending on the analytes to be analyzed and the nature of the waste (see Step 4.3.3). It is recommended that borosilicate glass bottles be used instead of other types of glass, especially when inorganics are of concern. Plastic bottles, other than polytetrafluoroethylene (“PTFE”), shall not be used if organics are to be investigated. Bottles are available from a number of laboratory suppliers. When this type of extraction vessel is used, the filtration device discussed in Step 4.3.2 is used for initial liquid/solid separation and final extract filtration.

### *4.3 Filtration Devices*

It is recommended that all filtrations be performed in a hood.

4.3.1 Zero-Headspace Extraction Vessel When the sample is evaluated for volatiles, the zero-headspace extraction vessel described in Step 4.2.1 is used for filtration. The device shall be capable of supporting and keeping in place the glass fiber filter and be able to withstand the pressure needed to accomplish separation (50 psig).

NOTE: When it is suspected that the glass fiber filter has been ruptured, an in-line glass fiber filter may be used to filter the material within the ZHE.

**4.3.2 Filter Holder.** When the sample is evaluated for other than volatile analytes, a filter holder capable of supporting a glass fiber filter and able to withstand the pressure needed to accomplish separation may be used. Suitable filter holders range from simple vacuum units to relatively complex systems capable of exerting pressures of up to 50 psig or more. The type of filter holder used depends on the properties of the material to be filtered (see Step 4.3.3). These devices shall have a minimum internal volume of 300 mL and be equipped to accommodate a minimum filter size of 47 mm (filter holders having an internal capacity of 1.5 L or greater, and equipped to accommodate a 142 mm diameter filter, are recommended). Vacuum filtration can only be used for wastes with low solids content (<10 %) and for highly granular, liquid-containing wastes. All other types of wastes should be filtered using positive pressure filtration. Suitable filter holders known are listed in of SW-846 Method 1311.

**4.3.3 Materials of Construction.** Extraction vessels and filtration devices shall be made of inert materials that will not leach or absorb sample components of interest. Glass, PTFE, or type 316 stainless steel equipment may be used when evaluating the mobility of both organic and inorganic components. Devices made of high-density polyethylene (“HDPE”), polypropylene (“PP”), or polyvinyl chloride (“PVC”) may be used only when evaluating the mobility of metals. Borosilicate glass bottles are recommended for use over other types of glass bottles, especially when inorganics are analytes of concern.

#### **4.4 Filters**

Filters shall be made of borosilicate glass fiber, shall contain no binder materials, and shall have an effective pore size of 0.6 to 0.8- $\mu$ m. Filters known to EPA which meet these specifications are identified in SW-846 Method 1311. Pre-filters must not be used. When evaluating the mobility of metals, filters shall be acid washed prior to use by rinsing with 1N nitric acid (“HNO<sub>3</sub>”) followed by three consecutive rinses with reagent water (a minimum of 1-L per rinse is recommended). Glass fiber filters are fragile and should be handled with care.

#### **4.5 pH Meters**

The meter should be accurate to  $\pm 0.05$  units at 25° C.

#### **4.6 ZHE Extract Collection Devices**

TEDLAR® bags or glass, stainless steel or PTFE gas-tight syringes are used to collect the initial liquid phase and the final extract when using the ZHE device. These devices listed are recommended for use under the following conditions:

4.6.1 If a waste contains an aqueous liquid phase or if a waste does not contain a significant amount of nonaqueous liquid (i.e., <1 % of total waste), the TEDLAR® bag or a 600 mL syringe should be used to collect and combine the initial liquid and solid extract.

4.6.2 If a waste contains a significant amount of nonaqueous liquid in the initial liquid phase (i.e., >1 % of total waste), the syringe or the TEDLAR® bag may be used for both the initial solid/liquid separation and the final extract filtration. However, analysts should use one or the other, not both.

4.6.3 If the waste contains no initial liquid phase (is 100 %solid) or has no significant solid phase (is <0.5% solid), either the TEDLAR® bag or the syringe may be used. If the syringe is used, discard the first 5 mL of liquid expressed from the device. The remaining aliquots are used for analysis.

#### *4.7 ZHE Extraction Fluid Transfer Devices*

Any device capable of transferring the extraction fluid into the ZHE without changing the nature of the extraction fluid is acceptable (e.g., a positive displacement or peristaltic pump, a gas-tight syringe, pressure filtration unit (see Step 4.3.2), or other ZHE device).

#### *4.8 Laboratory Balance*

Any laboratory balance accurate to within  $\pm 0.01$  grams may be used (all weight measurements are to be within  $\pm 0.1$  grams).

#### *4.9 Glassware*

- Beaker or Erlenmeyer flask, glass, 500 mL.
- Watch glass, appropriate diameter to cover beaker or Erlenmeyer flask.
- Magnetic stirrer.

### **5.0 Reagents**

#### *5.1 Reagent grade 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.

#### *5.2 Reagent Water*

Reagent water is defined as water in which an interferant is not observed at or above the method's detection limit of the analyte(s) of interest. For nonvolatile extractions, ASTM Type II water or equivalent meets the definition of reagent water. For volatile extractions, it is recommended that reagent water be generated by any of the following methods. Reagent water should be monitored periodically for impurities.

5.2.1 Reagent water for volatile extractions may be generated by passing tap water through a carbon filter bed containing about 500 grams of activated carbon (Calgon Corp., Filtrasorb-300 or equivalent).

5.2.2 A water purification system (Millipore Super-Q or equivalent) may also be used to generate reagent water for volatile extractions.

5.2.3 Reagent water for volatile extractions may also be prepared by boiling water for 15 minutes. Subsequently, while maintaining the water temperature at  $90 \pm 5$  degrees C, bubble a contaminant-free inert gas (e.g. nitrogen) through the water for 1 hour. While still hot, transfer the water to a narrow mouth screw-cap bottle under zero-headspace and seal with a Teflon-lined septum and cap.

5.3 Hydrochloric Acid, 1N, made from ACS reagent grade.

5.4 Nitric Acid, 1N, made from ACS reagent grade.

5.5 Sodium Hydroxide, 1N, made from ACS reagent grade.

5.6 Glacial Acetic Acid, CH<sub>3</sub>CH<sub>2</sub>OOH, ACS reagent grade.

#### 5.7 Extraction fluids

##### 5.7.1 Extraction fluid # 1

Add 5.7 mL glacial CH<sub>3</sub>CH<sub>2</sub>OOH to 500 mL of reagent water (See Section 5.2), add 64.3 mL of 1N NaOH, and dilute to a volume of 1 liter. When correctly prepared, the pH of this fluid will be 4.93 ± 0.05.

##### 5.7.2 Extraction fluid # 2

Dilute 5.7 mL glacial CH<sub>3</sub>CH<sub>2</sub>OOH with reagent water (See Section 5.2) to a volume of 1 liter. When correctly prepared, the pH of this fluid will be 2.88 ± 0.05.

NOTE: These extraction fluids should be monitored frequently for impurities. The pH should be checked prior to use to ensure that these fluids are made up accurately. If impurities are found or the pH is not within the above specifications, the fluid shall be discarded and fresh extraction fluid prepared.

5.8 Analytical standards shall be prepared according to the appropriate analytical method.

## 6.0 Sample Collection, Preservation, and Handling

All samples shall be collected using an appropriate sampling plan.

There may be requirements on the minimal size of the field sample depending upon the physical state or states of the waste and the analytes of concern. An aliquot is needed for the preliminary evaluations of the percent solids and the particle size. An aliquot may be needed to conduct the nonvolatile analyte extraction procedure. If volatile organics are of concern, another aliquot may be needed. Quality control ("QC") measures may require additional aliquots. Further, it is always wise to collect more sample just in case something goes wrong with the initial attempt to conduct the test.

Preservatives shall not be added to samples before extraction.

Samples may be refrigerated unless refrigeration results in irreversible physical change to the waste. If precipitation occurs, the entire sample (including precipitate) should be extracted. Soil/sediment samples for volatile organics should be collected in "core and store" type containers and frozen at -12° ± 3° C.

When the sample is to be evaluated for volatile analytes, extreme care shall be taken to minimize the loss of volatiles. Samples shall be collected and stored in a manner intended to prevent the loss of volatile analytes (e.g., waste samples should be collected in Teflon-lined septum capped vials and stored at 4° C. Soil/sediment samples for volatile organics should be collected in "core and store" type containers and frozen at -12° ± 3° C. Samples should be opened only immediately prior to extraction).

TCLP extracts should be prepared for analysis and analyzed as soon as possible following extraction. Extracts or portions of extracts for metallic analyte determinations must be acidified with



HNO<sub>3</sub> to a pH < 2, unless precipitation occurs (see Step 7.2.14 if precipitation occurs). Extracts should be preserved for other analytes according to the guidance given in the individual analysis methods. Extracts or portions of extracts for organic analyte determinations shall not be allowed to come into contact with the atmosphere (i.e., no headspace) to prevent losses. See Table 2.0 of this RCP for acceptable holding times.

## **7.0 Procedure**

### *7.1 Preliminary Evaluations*

Perform preliminary TCLP evaluations on a minimum 100-g aliquot of sample. This aliquot may not actually undergo TCLP extraction. These preliminary evaluations include: (1) determination of the percent solids (Step 7.1.1); (2) determination of whether the waste contains insignificant solids and is, therefore, its own extract after filtration (Step 7.1.2); (3) determination of whether the solid portion of the waste requires particle size reduction (Step 7.1.3); and (4) determination of which of the two extraction fluids are to be used for the nonvolatile TCLP extraction of the sample (Section 7.1.4).

7.1.1 Preliminary determination of percent solids: Percent solids is defined as that fraction of a waste sample (as a percentage of the total sample) from which no liquid may be forced out by an applied pressure, as described below.

7.1.1.1 If the sample will obviously yield no free liquid when subjected to pressure filtration (i.e., is 100% solid), weigh out a representative subsample (100 g minimum) and proceed to Step 7.1.3.

7.1.1.2 If the sample is liquid or multiphasic, liquid/solid separation to make a preliminary determination of percent solids is required. This involves the filtration device discussed in Step 4.3.2, and is outlined in Steps 7.1.1.3 through 7.1.1.9.

7.1.1.3 Pre-weigh the filter and the container that will receive the filtrate.

7.1.1.4 Assemble filter holder and filter following the manufacturer's instructions. Place the filter on the support screen and secure.

7.1.1.5 Weigh out a subsample of the waste (100 g minimum) and record the weight.

7.1.1.6 Allow slurries to stand to permit the solid phase to settle. Samples that settle slowly may be centrifuged prior to filtration. Centrifugation is to be used only as an aid to filtration. If used, the liquid should be decanted and filtered followed by filtration of the solid portion of the waste through the same filtration system.

7.1.1.7 Quantitatively transfer the sample to the filter holder (liquid and solid phases). Spread the sample evenly over the surface of the filter. If filtration of the waste at 4° C reduces the amount of expressed liquid over what would be expressed at room temperature, then allow the sample to warm up to room temperature in the device before filtering. Gradually apply vacuum or gentle pressure of 1-10 psig, until air or pressurizing gas moves through the filter. If this point is not reached under 10 psig, and if no additional liquid has passed through the filter in any 2-minute interval, slowly increase the pressure in 10 psig increments to a maximum of 50 psig. After each incremental increase of 10 psig, if the pressurizing gas has not moved through the filter, and if no additional liquid has passed through the filter in any 2-minute interval, proceed to the next 10-psig increment. When the pressurizing gas begins to move through the filter, or when liquid

flow has ceased at 50 psig (i.e., filtration does not result in any additional filtrate within any 2-minute period), stop the filtration.

NOTE: If sample material (>1 % of original sample weight) has obviously adhered to the container used to transfer the sample to the filtration apparatus, determine the weight of this residue and subtract it from the sample weight determined in Step 7.1.1.5 to determine the weight of the sample that will be filtered.

NOTE: Instantaneous application of high pressure can degrade the glass fiber filter and may cause premature plugging.

7.1.1.8 The material in the filter holder is defined as the solid phase of the sample, and the filtrate is defined as the liquid phase.

NOTE: Some samples, such as oily wastes and some paint wastes, will obviously contain some material that appears to be a liquid, but even after applying vacuum or pressure filtration, as outlined in Step 7.1.1.7, this material may not filter. If this is the case, the material within the filtration device is defined as a solid. Do not replace the original filter with a fresh filter under any circumstances. Use only one filter.

7.1.1.9 Determine the weight of the liquid phase by subtracting the weight of the filtrate container (see Step 7.1.1.3) from the total weight of the filtrate-filled container. Determine the weight of the solid phase of the sample by subtracting the weight of the liquid phase from the weight of the total sample, as determined in Step 7.1.1.5 or 7.1.1.7. Record the weight of the liquid and solid phases. Calculate the percent solids as follows:

$$\text{Percent Solids} = \left( \frac{\text{Wt. solid (Sec. 7.1.1.9)}}{\text{Total wt. waste (Sec. 7.1.1.5 or 7.1.1.7)}} \right) \times 100$$

7.1.2 If the percent solids determined in Step 7.1.1.9 is equal to or greater than 0.5%, then proceed either to Step 7.1.3 to determine whether the solid material requires particle size reduction or to Step 7.1.2.1 if it is noticed that a small amount of the filtrate is entrained in wetting of the filter. If the percent solids determined in Step 7.1.1.9 is less than 0.5%, then proceed to Step 7.2.9 if the nonvolatile TCLP analysis is to be performed, and to Step 7.3 with a fresh portion of the waste if the volatile TCLP analysis is to be performed.

7.1.2.1 Remove the solid phase and filter from the filtration apparatus.

7.1.2.2 Dry the filter and solid phase at  $100 \pm 20^\circ \text{C}$  until two successive weighings yield the same value within  $\pm 1\%$ . Record the final weight.

Caution: The drying oven should be vented to a hood or other appropriate device to eliminate the possibility of fumes from the sample escaping into the laboratory. Care should be taken to ensure that the sample will not flash or violently react upon heating.

7.1.2.3 Calculate the percent dry solids as follows:

$$\text{Percent dry solids} = \left( \frac{[\text{Wt. dry sample} + \text{filter}] - \text{tared filter wt.}}{\text{Initial sample wt. (Sec. 7.1.1.5 or 7.1.1.7)}} \right) \times 100$$

7.1.2.4 If the percent dry solids is less than 0.5%, then proceed to Step 7.2.9 if the nonvolatile TCLP analysis is to be performed, and to Step 7.3 if the volatile TCLP

analysis is to be performed. If the percent dry solids is greater than or equal to 0.5%, and if the nonvolatile TCLP analysis is to be performed, return to the beginning of this Step (7.1) and, with a fresh portion of sample, determine whether particle size reduction is necessary (Step 7.1.3). Also determine the appropriate extraction fluid (Section 7.1.4). If only volatile TCLP is to be performed, see the note in Section 7.1.4.

7.1.3 Determination of whether the sample requires particle-size reduction (particle-size is reduced during this step): Using the solid portion of the sample, evaluate the solid for particle size. Particle size reduction is required, unless the solid has a surface area per gram of material equal to or greater than 3.1 cm<sup>2</sup>, or is smaller than 1 cm in 2 its narrowest dimension (i.e., is capable of passing through a 9.5 mm (0.375 inch) standard sieve). If the surface area is smaller or the particle size larger than described above, prepare the solid portion of the sample for extraction by crushing, cutting, or grinding the waste to a surface area or particle size as described above. If the solids are prepared for organic volatiles extraction, special precautions must be taken (see Step 7.3.6).

NOTE: Surface area criteria are meant for filamentous (e.g., paper, cloth, and similar) waste materials. Actual measurement of surface area is not required, nor is it recommended. For materials that do not obviously meet the criteria, sample-specific methods would need to be developed and employed to measure the surface area. Such methodology is currently not available.

7.1.4 Determination of appropriate extraction fluid: If the solid content of the waste is greater than or equal to 0.5% and if the sample will be extracted for nonvolatile constituents (Section 7.2), determine the appropriate fluid (Section 5.7) for the nonvolatiles extraction as follows:

TCLP extraction for volatile constituents uses only extraction fluid #1 (Section 5.7.1). Therefore, if TCLP extraction for nonvolatiles is not required, proceed to Section 7.3.

7.1.4.1 Weigh out a small subsample of the solid phase of the waste, reduce the solid (if necessary) to a particle size of approximately 1 mm in diameter or less, and transfer 5.0 grams of the solid phase of the waste to a 500 mL beaker or Erlenmeyer flask.

7.1.4.2 Add 96.5 mL of reagent water to the beaker, cover with a watch glass, and stir vigorously for 5 minutes using a magnetic stirrer. Measure and record the pH. If the pH is <5.0, use extraction fluid #1. Proceed to Section 7.2.

7.1.4.3 If the pH from Step 7.1.4.2 is >5.0, add 3.5 mL 1N HCl, slurry briefly, cover with a watchglass, heat to 50° C, and hold at 50° C for 10 minutes.

7.1.4.4 Let the solution cool to room temperature and record the pH. If the pH is <5.0, use extraction fluid #1. If the pH is >5.0, use extraction fluid #2. Proceed to Section 7.2.

7.1.5 If the aliquot of the sample used for the preliminary evaluation (Steps 7.1.1 - 7.1.4) was determined to be 100% solid at Step 7.1.1.1, then it can be used for the Step 7.2 extraction (assuming at least 100 g remain). The aliquot of the waste subjected to the procedure in Step 7.1.1.7 might be appropriate for use for the Step 7.2 extraction if an adequate amount of solid (as determined by Step 7.1.1.9) was obtained. The amount of solid necessary is dependent upon whether a sufficient amount of extract will be produced to support the analyses. If an adequate amount of solid remains, proceed to Step 7.2.10 of the nonvolatile TCLP extraction.

## 7.2 Procedure When Volatiles Are Not Involved

A minimum sample size of 100 g (solid and liquid phases) is required, unless the amount of sample is unavailable or the sample has such a low density that 100 g would be unmanageable. In some cases, a larger sample size may be appropriate, depending on the solids content of the waste sample (percent solids, See Step 7.1.1), whether the initial liquid phase of the waste will be miscible with the aqueous extract of the solid, and whether inorganics, semivolatile organics (“SVOCs”), pesticides, and herbicides are all analytes of concern. Enough solids should be generated for extraction such that the volume of TCLP extract will be sufficient to support all of the analyses required. If the amount of extract generated by a single TCLP extraction will not be sufficient to perform all of the analyses, more than one extraction may be performed and the extracts from each combined and aliquoted for analysis.

7.2.1 If the sample will obviously yield no liquid when subjected to pressure filtration (i.e., is 100 % solid, see Step 7.1.1), weigh out a subsample of the sample (100 g minimum) and proceed to Step 7.2.9.

7.2.2 If the sample is liquid or multiphasic, liquid/solid separation is required. This involves the filtration device described in Step 4.3.2 and is outlined in Steps 7.2.3 to 7.2.8.

7.2.3 Pre-weigh the container that will receive the filtrate.

7.2.4 Assemble the filter holder and filter following the manufacturer's instructions. Place the filter on the support screen and secure. Acid wash the filter if evaluating the mobility of metals (see Step 4.4).

NOTE: Acid washed filters may be used for all nonvolatile extractions even when metals are not of concern.

7.2.5 Weigh out a subsample of the sample (100 g minimum) and record the weight. If the waste contains <0.5 % dry solids (Step 7.1.2), the liquid portion of the waste, after filtration, is defined as the TCLP extract. Therefore, enough of the sample should be filtered so that the amount of filtered liquid will support all of the analyses required of the TCLP extract. For wastes containing >0.5 % dry solids (Steps 7.1.1 or 7.1.2), use the percent solids information obtained in Step 7.1.1 to determine the optimum sample size (100 gram minimum) for filtration. Enough solids should be generated by filtration to support the analyses to be performed on the TCLP extract.

7.2.6 Allow slurries to stand to permit the solid phase to settle. Samples that settle slowly may be centrifuged prior to filtration. Use centrifugation only as an aid to filtration. If the sample is centrifuged, the liquid should be decanted and filtered followed by filtration of the solid portion of the waste through the same filtration system.

7.2.7 Quantitatively transfer the sample (liquid and solid phases) to the filter holder (see Step 4.3.2). Spread the waste sample evenly over the surface of the filter. If filtration of the waste at 4° C reduces the amount of expressed liquid over what would be expressed at room temperature, then allow the sample to warm up to room temperature in the device before filtering. Gradually apply vacuum or gentle pressure of 1-10 psig, until air or pressurizing gas moves through the filter. If this point is not reached under 10 psig, and if no additional liquid has passed through the filter in any 2-minute interval, slowly increase the pressure in 10-psig increments to maximum of 50 psig. After each incremental increase of 10 psig, if the pressurizing gas has not moved through the filter, and if no additional liquid has passed through the filter in any 2-minute interval, proceed to the next 10-psig increment. When the pressurizing

gas begins to move through the filter, or when the liquid flow has ceased at 50 psig (i.e., filtration does not result in any additional filtrate within a 2-minute period), stop the filtration.

NOTE: If waste material (>1 % of the original sample weight) has obviously adhered to the container used to transfer the sample to the filtration apparatus, determine the weight of this residue and subtract it from the sample weight determined in Step 7.2.5, to determine the weight of the waste sample that will be filtered.

NOTE: Instantaneous application of high pressure can degrade the glass fiber filter and may cause premature plugging.

7.2.8 The material in the filter holder is defined as the solid phase of the sample, and the filtrate is defined as the liquid phase. The liquid phase may now be either analyzed (see Step 7.2.12) or stored at 4° C until time of analysis.

NOTE: Some wastes, such as oily wastes and some paint wastes, will obviously contain some material which appears to be a liquid. Even after applying vacuum or pressure filtration, as outlined in Step 7.2.7, this material may not filter. If this is the case, the material within the filtration device is defined as a solid and is carried through the extraction as a solid. Do not replace the original filter with a fresh filter under any circumstances. Use only one filter.

7.2.9 If the sample contains <0.5% dry solids (see Step 7.1.2), proceed to Step 7.2.13. If the sample contains >0.5 % dry solids (see Step 7.1.1 or 7.1.2), and if particle-size reduction of the solid was needed in Step 7.1.3, proceed to Step 7.2.10. If the sample as received passes a 9.5 mm sieve, quantitatively transfer the solid material into the extractor bottle along with the filter used to separate the initial liquid from the solid phase and proceed to Step 7.2.11.

7.2.10 Prepare the solid portion of the sample for extraction by crushing, cutting, or grinding the waste to a surface area or particle size as described in Step 7.1.3. When the surface area or particle-size has been appropriately altered, quantitatively transfer the solid material into an extractor bottle. Include the filter used to separate the initial liquid from the solid phase.

NOTE: Sieving of the waste is not normally required. Surface area requirements are meant for filamentous (e.g., paper, cloth) and similar waste materials. Actual measurement of surface area is not recommended. If sieving is necessary, a Teflon-coated sieve should be used to avoid contamination of the sample.

7.2.11 Determine the amount of extraction fluid to add to the extractor vessel as follows:

$$Wt. \text{ extraction fluid} = \left( \frac{[20 \times \% \text{ solids (Sec. 7.1.1)}] \times wt. \text{ waste filtered (Sec 7.2.5 or 7.2.7)}}{100} \right)$$

Slowly add this amount of appropriate extraction fluid (see Step 7.1.4) to the extractor vessel. Close the extractor bottle tightly (it is recommended that Teflon tape be used to ensure a tight seal), secure in rotary extractor device, and rotate at 30 ± 2 rpm for 18 ± 2 hours. Ambient temperature (i.e., temperature of room in which extraction takes place) shall be maintained at 23 ± 2° C during the extraction period.

NOTE: As agitation continues, pressure may build up within the extractor bottle for some types of sample (e.g., limed or calcium carbonate-containing sample may evolve gases such as carbon dioxide). To relieve excess pressure, the extractor bottle may be periodically opened (e.g., after 15 minutes, 30 minutes, and 1 hour) and vented into a hood.

7.2.12 Following the  $18 \pm 2$  hour extraction, separate the material in the extractor vessel into its component liquid and solid phases by filtering through a new glass fiber filter, as outlined in Step 7.2.7. For final filtration of the TCLP extract, the glass fiber filter may be changed, if necessary, to facilitate filtration. Filter(s) shall be acid-washed (see Step 4.4) if evaluating the mobility of metals.

7.2.13 Prepare the TCLP extract as follows:

7.2.13.1 If the sample contained no initial liquid phase, the filtered liquid material obtained from Step 7.2.12 is defined as the TCLP extract. Proceed to Step 7.2.14.

7.2.13.2 If compatible (e.g., multiple phases will not result on combination), combine the filtered liquid resulting from Step 7.2.12 with the initial liquid phase of the sample obtained in Step 7.2.7. This combined liquid is defined as the TCLP extract. Proceed to Step 7.2.14.

7.2.13.3 If the initial liquid phase of the waste, as obtained from Step 7.2.7, is not or may not be compatible with the filtered liquid resulting from Step 7.2.12, do not combine these liquids. Analyze these liquids, collectively defined as the TCLP extract, and combine the results mathematically, as described in Step 7.2.14.

7.2.14 Following collection of the TCLP extract, the pH of the extract should be recorded. Immediately aliquot and preserve the extract for analysis. Metals aliquots must be acidified with nitric acid to  $\text{pH} < 2$ . If precipitation is observed upon addition of nitric acid to a small aliquot of the extract, then the remaining portion of the extract for metals analyses shall not be acidified and the extract shall be analyzed as soon as possible. All other aliquots must be stored under refrigeration ( $4^{\circ}\text{C}$ ) until analyzed. The TCLP extract shall be prepared and analyzed according to appropriate analytical methods. TCLP extracts to be analyzed for metals shall be acid digested except in those instances where digestion causes loss of metallic analytes. If an analysis of the undigested extract shows that the concentration of any regulated metallic analyte exceeds the regulatory level, then the waste is hazardous and digestion of the extract is not necessary. However, data on undigested extracts alone cannot be used to demonstrate that the waste is not hazardous. If the individual phases are to be analyzed separately, determine the volume of the individual phases (to  $+ 0.5\%$ ), conduct the appropriate analyses, and combine the results mathematically by using a simple volume-weighted average:

$$\text{Final analyte concentration} = \left( \frac{[(V_1 C_1) + (V_2 C_2)]}{[V_1 + V_2]} \right)$$

where:

$V_1$  = The volume of the first phase (L).

$C_1$  = The concentration of the analyte of concern in the first phase (mg/L).

$V_2$  = The volume of the second phase (L).

$C_2$  = The concentration of the analyte of concern in the second phase (mg/L).

7.2.15 Compare the analyte concentrations in the TCLP extract with the levels identified in the appropriate regulations. Refer to Section 8.0 of this RCP for quality assurance ("QA") requirements.

### 7.3 Procedure When Volatiles Are Involved

Use the ZHE device to obtain TCLP extract for analysis of volatile compounds only. Extract resulting from the use of the ZHE shall not be used to evaluate the mobility of non-volatile analytes

(e.g., metals, pesticides, etc.). The ZHE device has approximately a 500 mL internal capacity. The ZHE can thus accommodate a maximum of 25 grams of solid (defined as that fraction of a sample from which no additional liquid may be forced out by an applied pressure of 50 psig), due to the need to add an amount of extraction fluid equal to 20 times the weight of the solid phase. Charge the ZHE with sample only once and do not open the device until the final extract (of the solid) has been collected. Repeated filling of the ZHE to obtain 25 grams of solid is not permitted. Do not allow the sample, the initial liquid phase, or the extract to be exposed to the atmosphere for any more time than is absolutely necessary. Any manipulation of these materials should be done when cold (4° C) to minimize loss of volatiles.

7.3.1 Pre-weigh the (evacuated) filtrate collection container (see Step 4.6) and set aside. If using a TEDLAR® bag, express all liquid from the ZHE device into the bag, whether for the initial or final liquid/solid separation, and take an aliquot from the liquid in the bag for analysis. The containers listed in Step 4.6 are recommended for use under the conditions stated in Steps 4.6.1-4.6.3.

7.3.2 Place the ZHE piston within the body of the ZHE (it may be helpful first to moisten the piston O-rings slightly with extraction fluid). Adjust the piston within the ZHE body to a height that will minimize the distance the piston will have to move once the ZHE is charged with sample (based upon sample size requirements determined from Step 7.3, Step 7.1.1 and/or 7.1.2). Secure the gas inlet/outlet flange (bottom flange) onto the ZHE body in accordance with the manufacturer's instructions. Secure the glass fiber filter between the support screens and set aside. Set liquid inlet/outlet flange (top flange) aside.

7.3.3 If the sample is 100% solid (see Step 7.1.1), weigh out a subsample (25 gram maximum) of the waste, record weight, and proceed to Step 7.3.5.

7.3.4 If the sample contains <0.5% dry solids (Step 7.1.2), the liquid portion of waste, after filtration, is defined as the TCLP extract. Filter enough of the sample so that the amount of filtered liquid will support all of the volatile analyses required. For samples containing >0.5% dry solids (Steps 7.1.1 and/or 7.1.2), use the percent solids information obtained in Step 7.1.1 to determine the optimum sample size to charge into the ZHE. The recommended sample size is as follows:

7.3.4.1 For samples containing <5% solids (see Step 7.1.1), weigh out a 500-subsample of waste and record the weight.

7.3.4.2 For wastes containing >5% solids (see Step 7.1.1), determine the amount of waste to charge into the ZHE as follows:

$$Wt. \text{ waste to charge ZHE} = \left( \frac{25}{\% \text{ solids (Sec. 7.1.1)}} \right) \times 100$$

Weigh out a subsample of the waste of the appropriate size and record the weight.

7.3.5 If particle-size reduction of the solid portion of the sample was required in Step 7.1.3, proceed to Step 7.3.6. If particle size reduction was not required in Step 7.1.3, proceed to Step 7.3.7.

7.3.6 Prepare the sample for extraction by crushing, cutting, or grinding the solid portion of the waste to a surface area or particle size as described in Step 7.1.3.1. Wastes and appropriate reduction equipment should be refrigerated, if possible, to 4° C prior to particle-size reduction. The means used to effect particle-size reduction must not generate heat in and of itself. If

reduction of the solid phase of the waste is necessary, exposure of the waste to the atmosphere should be avoided to the extent possible.

NOTE: Sieving of the waste is not recommended due to the possibility that volatiles may be lost. The use of an appropriately graduated ruler is recommended as an acceptable alternative. Surface area requirements are meant for filamentous (e.g., paper, cloth) and similar waste materials. Actual measurement of surface area is not recommended. When the surface area or particle-size has been appropriately altered, proceed to Step 7.3.7.

7.3.7 Waste slurries need not be allowed to stand to permit the solid phase to settle. Do not centrifuge samples prior to filtration.

7.3.8 Quantitatively transfer the entire sample (liquid and solid phases) quickly to the ZHE. Secure the filter and support screens into the top flange of the device and secure the top flange to the ZHE body in accordance with the manufacturer's instructions. Tighten all ZHE fittings and place the device in the vertical position (gas inlet/outlet flange on the bottom). Do not attach the extraction collection device to the top plate.

NOTE: If sample material (>1% of original sample weight) has obviously adhered to the container used to transfer the sample to the ZHE, determine the weight of this residue and subtract it from the sample weight determined in Step 7.3.4 to determine the weight of the waste sample that will be filtered. Attach a gas line to the gas inlet/outlet valve (bottom flange) and, with the liquid inlet/outlet valve (top flange) open, begin applying gentle pressure of 1-10 psig (or more if necessary) to force all headspace slowly out of the ZHE device into a hood. At the first appearance of liquid from the liquid inlet/outlet valve, quickly close the valve and discontinue pressure. If filtration of the waste at 4° C reduces the amount of expressed liquid over what would be expressed at room temperature, then allow the sample to warm up to room temperature in the device before filtering. If the waste is 100 % solid (see Step 7.1.1), slowly increase the pressure to a maximum of 50 psig to force most of the headspace out of the device and proceed to Step 7.3.12.

7.3.9 Attach the evacuated pre-weighed filtrate collection container to the liquid inlet/outlet valve and open the valve. Begin applying gentle pressure of 1-10 psig to force the liquid phase of the sample into the filtrate collection container. If no additional liquid has passed through the filter in any 2-minute interval, slowly increase the pressure in 10-psig increments to a maximum of 50 psig. After each incremental increase of 10 psig, if no additional liquid has passed through the filter in any 2-minute interval, proceed to the next 10-psig increment. When liquid flow has ceased such that continued pressure filtration at 50 psig does not result in any additional filtrate within a 2-minute period, stop the filtration. Close the liquid inlet/outlet valve, discontinue pressure to the piston, and disconnect and weigh the filtrate collection container.

NOTE: Instantaneous application of high pressure can degrade the glass fiber filter and may cause premature plugging.

7.3.10 The material in the ZHE is defined as the solid phase of the sample and the filtrate is defined as the liquid phase.

NOTE: Some samples, such as oily wastes and some paint wastes, will obviously contain some material which appears to be a liquid. Even after applying pressure filtration, this material will not filter. If this is the case, the material within the filtration device is defined as a solid, and is carried through the TCLP extraction as a solid. If the original waste contained <0.5 % dry solids (see Step 7.1.2), this filtrate is defined as the TCLP extract and is analyzed directly. Proceed to Step 7.3.15.



7.3.11 The liquid phase may now be either analyzed immediately (see Steps 7.3.13 through 7.3.15) or stored at 4° C under minimal headspace conditions until time of analysis.

Determine the weight of extraction fluid #3 to add to the ZHE as follows:

$$Wt. \text{ extraction fluid} = \left( \frac{20 \times \% \text{ solids (Sec. 7.1.1)} \times wt. \text{ waste filtered (Sec. 7.3.4 or 7.3.8)}}{100} \right)$$

7.3.12 The following steps detail how to add the appropriate amount of extraction fluid to the solid material within the ZHE and agitation of the ZHE vessel. Extraction fluid #1 is used in all cases (see Section 5.7).

7.3.12.1 With the ZHE in the vertical position, attach a line from the extraction fluid reservoir to the liquid inlet/outlet valve. The line used shall contain fresh extraction fluid and should be pre-flushed with fluid to eliminate any air pockets in the line. Release gas pressure on the ZHE piston (from the gas inlet/outlet valve), open the liquid inlet/outlet valve, and begin transferring extraction fluid (by pumping or similar means) into the ZHE. Continue pumping extraction fluid into the ZHE until the appropriate amount of fluid has been introduced into the device.

7.3.12.2 After the extraction fluid has been added, immediately close the liquid inlet/outlet valve and disconnect the extraction fluid line. Check the ZHE to ensure that all valves are in their closed positions. Manually rotate the device in an end-over-end fashion 2 or 3 times. Reposition the ZHE in the vertical position with the liquid inlet/outlet valve on top. Pressurize the ZHE to 5-10 psig (if necessary) and slowly open the liquid inlet/outlet valve to bleed out any headspace (into a hood) that may have been introduced due to the addition of extraction fluid. This bleeding shall be done quickly and shall be stopped at the first appearance of liquid from the valve. Re-pressurize the ZHE with 5-10 psig and check all ZHE fittings to ensure that they are closed.

7.3.12.3 Place the ZHE in the rotary extractor apparatus (if it is not already there) and rotate at  $30 \pm 2$  rpm for  $18 \pm 2$  hours. Ambient temperature (i.e., temperature of room in which extraction occurs) shall be maintained at  $23 \pm 2^\circ$  C during agitation.

7.3.13 Following the  $18 \pm 2$  hour agitation period, check the pressure behind the ZHE piston by quickly opening and closing the gas inlet/outlet valve and noting the escape of gas. If the pressure has not been maintained (i.e., no gas release observed), the ZHE is leaking. Check the ZHE for leaking as specified in Step 4.2.1, and perform the extraction again with a new sample of waste. If the pressure within the device has been maintained, the material in the extractor vessel is once again separated into its component liquid and solid phases. If the waste contained an initial liquid phase, the liquid may be filtered directly into the same filtrate collection container (i.e., TEDLAR® bag) holding the initial liquid phase of the waste. A separate filtrate collection container must be used if combining would create multiple phases, or there is not enough volume left within the filtrate collection container. Filter through the glass fiber filter, using the ZHE device as discussed in Step 7.3.9. All extracts shall be filtered and collected if the TEDLAR®

bag is used, if the extract is multiphasic, or if the waste contained an initial liquid phase (see Steps 4.6 and 7.3.1).

NOTE: An in-line glass fiber filter may be used to filter the material within the ZHE if it is suspected that the glass fiber filter has been ruptured.

7.3.14 If the original sample contained no initial liquid phase, the filtered liquid material obtained from Step 7.3.13 is defined as the TCLP extract. If the sample contained an initial liquid phase, the filtered liquid material obtained from Step 7.3.13 and the initial liquid phase (Step 7.3.9) are collectively defined as the TCLP extract.

7.3.15 Following collection of the TCLP extract, immediately prepare the extract for analysis and store with minimal headspace at 4° C until analyzed. Analyze the TCLP extract according to the appropriate analytical methods. If the individual phases are to be analyzed separately (i.e., are not miscible), determine the volume of the individual phases (to 0.5%), conduct the appropriate analyses, and combine the results mathematically by using a simple volume-weighted average:

$$\text{Final analyte concentration} = \left( \frac{[(V_1 C_1) + (V_2 C_2)]}{[V_1 + V_2]} \right)$$

where:

V<sub>1</sub> = The volume of the first phase (L).

C<sub>1</sub> = The concentration of the analyte of concern in the first phase (mg/L).

V<sub>2</sub> = The volume of the second phase (L).

C<sub>2</sub> = The concentration of the analyte of concern in the second phase (mg/L).

7.3.16 Compare the analyte concentrations in the TCLP extract with the levels identified in the appropriate regulations. Refer to Section 8.0 of this RCP for QA requirements.

## **8.0 Quality Control Requirements for SW-846 Method 1311**

This protocol is restricted to use by, or under the supervision of, analysts who are experienced in using extraction and filtration equipment and skilled in the correction of equipment malfunction described in this method.

Refer to SW-846 Chapter One for general QC procedures for all analytical and preparation methods methods, including SW-846 Method 1311. These requirements ensure that each laboratory maintain a formal QA program and records to document the quality of all inorganic data and be certified by the Connecticut Department of Public Health for the analysis performed.

Due to the nature of the procedure, an Initial Demonstration of Capability (“IDOC”) is not required for this method. Laboratories must have on hand a standard operating procedure (“SOP”) for this method and keep detailed records of the preparation for at least five (5) years.

### **8.1 Specific QA/QC Requirements and Standards for SW-846 Method 1311**

Nominal sample weights as described in the method (25 g for volatiles, 100 g for all other analytes) must be used whenever possible. Using smaller amounts of sample for extraction is not allowed unless there is not enough sample available or if the sample’s density is so low as to make the volume of material too large to handle. In such cases the laboratory must note in the narrative that lower sample weights were used and provide a detailed explanation as to why.

In addition to leached blank/spike, all QC measures in the analytical method must be followed per SW-846 Method 1311.

While optional, parties electing to utilize these protocols will be assured that agency reviewers will, generally, accept “Reasonable Confidence”. To achieve “Reasonable Confidence” parties must:

1. Comply with the applicable QC analytical requirements prescribed in Table 1A for this test procedure;
2. Evaluate and narrate all protocol non-compliances and implement, as necessary, required corrective actions and analytical response actions for all non-conforming performance standards; and
3. Retain reported and unreported analytical data and information for a period of 5 years or as required under applicable accreditation criteria.

*8.2 Site Specific Matrix Spike (MS), Matrix Spike Duplicate (MSD), and Matrix Duplicate (MD) Samples*

It is strongly recommended that site-specific MS/MSD or MS/MD samples be analyzed from each site and each matrix type sampled. Evaluation of sample matrix effects on analyte recovery is key to making informed decisions. Percent recovery data from site-specific (and matrix-specific) samples allow the environmental professional (“EP”) to make informed decisions regarding contamination levels at the site. Batch MS/MSD or MS/MD results do not give any indication of site-specific matrix interferences or analytical problems related to the specific site matrices. Field, rinsate, or other blanks should not be used for MS/MSD/MDs. A laboratory may substitute a MS/MSD in lieu of the MS/MD.

**Table 1A: Specific QA/QC Requirements and Performance Standards for Method 1311**

Required QC Parameter	Data Quality Objective	Required Performance Standard	Required Deliverable	Recommended Corrective Action	Required Analytical Response Action
Preparation of Samples	Accuracy and Representativeness	Use nominal sample weights for extraction – 25 grams for volatiles, 100 grams for other analytes	NO	N/A	Note discrepancies in narrative.
Method/ Extraction Blank	Contamination	Every 20 samples or a change in extraction fluid, whichever is more frequent.	YES	(1) If concentration of contaminant in sample is <10x concentration in blank, locate source of contamination; correct problem; reanalyze method blank and associated samples. (2) No corrective action required if concentration of contaminant in sample is >10x concentration in blank or if contaminant not detected in sample.	Note non-conformances in laboratory report narrative.
Matrix Spike/Matrix Spike Duplicate (“MS/MSD”)	Method Accuracy & Precision in Sample Matrix	(1) Every ≤20 field samples per matrix (at discretion of lab or at request of data user) (2) Matrix specific (e.g., aqueous, soil). (3) Concentration level near midpoint of curve. (4) Field blanks, trip blanks, etc. cannot be used for MS/MSDs	YES (ONLY when requested by data user)	Recovery criteria per applicable RCP. Samples spiked after extraction and filtration of extract, and before preservation.	Note non-conformances in laboratory report narrative.
Matrix Duplicate (“MD”)	Precision and Accuracy	Every ≤20 field samples	YES (ONLY when requested by data user)	RPD criteria per applicable RCP.	Note non-conformances in laboratory report narrative.
Sample Analysis	N/A	Sample extracts prepared and analyzed per applicable RCPs.	N/A	N/A	N/A

### 9.0 Routine Reporting Deliverables for SW-846 Method 1311

The following table (Table 1.0) lists the routine report deliverables. Note that while laboratory are not required to report certain items, they must keep the data on file and may be required to report all items in special circumstances.

**Table 1.0: Report Deliverables**

PARAMETER	DELIVERABLE	COMMENTS
Sample Preparation Data	NO	Note non-conformances in laboratory report narrative
Method Blanks	YES	Note non-conformances in laboratory report narrative. Flag all positive sample results above RL with “B” flag.
Site Specific Matrix Spike/ Matrix Spike Duplicate or Matrix Duplicate	YES (If requested)	Note non-conformances in laboratory report narrative
General Reporting Issues	YES	Note non-conformances in narrative
QA/QC Certification Form	YES	Signed by laboratory director or their designee.
Chain-of-Custody Form	YES	Signed by sample collector, courier, and laboratory

### 10.0 Sample Containers, Preservations, and Holding Times

Table 2.0 identifies the types of containers, preservation requirements, and holding times dependent upon analyte and matrix.

**Table 2.0: Sample Containers, Preservations, and Holding Times**

ANALYSIS (MATRIX)	CONTAINER <sup>1,2</sup>	PRESERVATIVE	HOLDING TIME <sup>3</sup>
VOC's (soil/sediment)	Core and Store (i.e., En Core® type) sampler <sup>4</sup>	Freeze or extract within 48 hours of collection. Freezer temperature $-12^{\circ} \pm 3^{\circ} \text{C}$	14 days to extraction 14 days from prep to analysis
Other organic parameters <sup>5</sup> (soil/sediment)	250-mL glass jar.	Cool to $4^{\circ} \pm 2^{\circ} \text{C}$	14 days from collection to extraction 7 days from TCLP extraction to preparation extraction 40 days from preparation extraction to analysis

Connecticut DEEP RCPs  
Quality Assurance and Quality Control Requirements  
Toxicity Characteristic Leaching Procedure by Method 1311, SW-846  
Version 3.0  
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<b>ANALYSIS (MATRIX)</b>	<b>CONTAINER<sup>1,2</sup></b>	<b>PRESERVATIVE</b>	<b>HOLDING TIME<sup>3</sup></b>
PCBs (soil/sediment)	250 mL amber glass jar with Teflon-lined cap	Cool to 4° ± 2° C	1 year to extraction
Metals, except mercury (soil/sediment)	250 mL glass or plastic jar with Teflon lined cap <sup>6</sup>	Cool to 4° ± 2° C	180 days to extraction 180 days from extraction to analysis
Mercury (soil/sediment)	250 mL glass or plastic jar with Teflon lined cap <sup>6</sup>	Cool to 4° ± 2° C	28 days to extraction 28 days from extraction to analysis
Cyanide (soil/sediment)	250 mL glass or plastic jar†	Cool to 4° ± 2° C	14 days to extraction 14 days from extraction to analysis
VOC's (High concentration waste)	2-ounce septa capped jar	Freeze or extract within 48 hours of collection. Freezer temperature -12° ± 3° C	14 days to extraction 14 days from extraction to analysis
Other analytes (High concentration waste)	250 mL glass jar	Cool to 4° ± 2° C	14 days to extraction 14 days from extraction to analysis
<p><sup>1</sup>Excepting volatiles, the same container may be used for other analytes.</p> <p><sup>2</sup>The number of sample containers is optional. Laboratories should supply enough containers to allow for any reanalysis or breakage.</p> <p><sup>3</sup>Holding time from sample collection to preparation of extract. Extract holding time would be the same as listed for aqueous samples in the applicable RCP.</p> <p><sup>4</sup>The sampler for VOC's must contain the nominal sample weight (25 g) and be designed so that the sample may be transferred to the ZHE with a minimum of sample handling by the laboratory. Septa capped jars do not meet this requirement. If only TCLP volatiles are to be determined, an additional 250-mL jar must be supplied for pre-leaching analyses.</p> <p><sup>5</sup>Other organic parameters include: EPH, ETPH, Pesticides, Herbicides, and SVOCs</p> <p><sup>6</sup>Plastic bottles must be acid rinsed and either high-density polyethylene, or Teflon.</p>			