

THE TOXICITY CHARACTERISTIC LEACHING PROCEDURE EPA METHOD 1311

TABLE OF CONTENTS

1. SCOPE AND APPLICATION	1
2. SUMMARY OF THE METHOD	1
3. APPARATUS AND EQUIPMENT	2
4. REAGENTS AND CHEMICALS	2
5. SAMPLE COLLECTION, PRESERVATION, AND HANDLING	3
6. SAMPLE PREPARATION PROCEDURE	4
7. QUALITY CONTROL	14
8. SAFETY/HAZARDOUS WASTE MANAGEMENT	14
9. REFERENCES	14
Appendices	
A. Maximum Concentration of Contaminants for Toxicity Characteristic	
B. Totals Thresholds for possible TCLP violations	
C. Appendix of Significant Changes	

1. SCOPE AND APPLICATION

- 1.1. The toxicity characteristic leaching procedure (TCLP) is designed to determine the mobility of both organic and inorganic analytes in liquid, solid, and multiphasic waste under conditions that simulate those found in a landfill. This SOP applies to TCLP for inorganic and organic analytes.
- 1.2. This method is applicable to soil, sediments and chemical waste. Samples are extracted according to EPA Method 1311 and for inorganic analyses the TCLP extract is digested in preparation for inductively coupled plasma-atomic emission spectroscopy (ICP-AES), inductively coupled plasma mass spectrometry (ICPMS), or cold vapor atomic absorption spectroscopy (CVAAS, for mercury analysis). For organic analyses the TCLP extract goes through the organic extraction process in preparation for gas chromatography mass spectrometry (GC/MS) or gas chromatography with electron capture detection (GC/ECD).

2. SUMMARY OF THE METHOD

- 2.1. The sample undergoes a preliminary evaluation, which may include determination of percent solids as well as particle size reduction. For samples with $< 0.5\%$ dry solids, the sample filtrate is defined as the extract. For samples with $\geq 0.5\%$ dry solids, the solid and liquid phases are separated and the solid is extracted with the appropriate extraction fluid. The volume of extraction fluid used (in mL) is 20 times the mass of the solid (in grams).
- 2.2. The type of extraction fluid used is determined by the sample pH. After the sample has been extracted in the appropriate fluid, the solid and liquid phases are separated by filtration.

3. APPARATUS AND EQUIPMENT

- 3.1. Agitation apparatus (agitator).
- 3.2. Accumet pH meter.
- 3.3. Millipore pressure filtration device.
- 3.4. Ohaus GT-410 top-loading balance.
- 3.5. MSI glass fiber filters -- 0.7 um pore diameter. Filters should be acid washed using 1N HNO₃ followed by 3 consecutive rinses with DI water prior to use.
- 3.6. Thermolyne heating/stirring plate.
- 3.7. 250 mL beakers.
- 3.8. 2000 mL beakers.
- 3.9. Jack stand.
- 3.10. Compressed nitrogen hookup for the filtration device.
- 3.11. Vented oven.
- 3.12. 9.5 mm (0.375 inch) standard sieve, preferably Teflon coated.
- 3.13. Watch glass large enough to cover a 250 mL beaker.
- 3.14. Polypropylene extraction vessels.
- 3.15. Teflon extraction vessels.
- 3.16. Aluminum foil.
- 3.17. 1000 mL polypropylene bottles for storing extracted samples.
- 3.18. Disposable stir bars
- 3.19. 6" wide Teflon tape.
- 3.20. Separatory funnel.
- 3.21. Narrow range pH paper, 0 - 5.0 pH units.
- 3.22. 10 mL volumetric flasks.
- 3.23. 1000 mL graduated cylinder.
- 3.24. 1.0 mm standard sieve
- 3.25. Disposable plastic spoons

4. REAGENTS AND CHEMICALS

- 4.1. Deionized (DI) Water.
- 4.2. 1.0 N Hydrochloric Acid, commercially available or dilute 83 mL of concentrated HCl to 1000 mL.

- 4.3. 1.0 N Sodium Hydroxide: Dilute 40 g solid NaOH to 1000 mL with DI water. This solution is used in the preparation of extraction fluid #1 (section 4.5.1).
- 4.4. Glacial Acetic Acid-used in the preparation of the extraction fluids.
- 4.5. Extraction Fluids.
 - 4.5.1. Extraction Fluid #1: Add 11.4 mL of glacial acetic acid to 1000 mL DI water. To this solution add 128.6 mL of the 1.0 N sodium hydroxide solution prepared according to section 4.3 above. Dilute this solution to 2 liters with DI water. The pH must be 4.93 pH units \pm 0.05 pH units (4.88 - 4.98 pH units). Add more acid or base to adjust the pH if necessary. If the pH of any old extraction fluid is not within this range, then discard the old solution and remake.
 - 4.5.2. Extraction Fluid #2: Add 11.4 mL of glacial acetic acid to 1000 mL of DI water. Dilute this solution to 2 liters. The pH must be 2.88 pH units \pm 0.05 pH units (2.83 - 2.93 pH units). Add more acid to adjust the pH if necessary. If the pH of old extraction fluid is not within this range, then discard the old solution and remake.

5. SAMPLE COLLECTION, PRESERVATION, AND HANDLING

- 5.1. Samples are stored in the metals room or refrigerator, located in the Receiving/Custody Room of the laboratory. If samples are also to be analyzed for organics they will be stored in a refrigerator. Allow the chilled samples to come to room temperature before determining pH or beginning an extraction.
- 5.2. All glassware should be cleaned thoroughly according to FLDEP SOP MT-007-2.11: Procedure for Cleaning Metals Lab. The filtration unit should be cleaned by disassembling the apparatus (especially by removing the o-rings), soaking in soapy water, scrubbing gently with a brush (be careful not to harm the Teflon coating), and thoroughly rinsing with DI water. **NOTE:** If the filtration unit does not come clean because of an organic residue, use an organic solvent (hexanes, acetone, methanol, or methylene chloride) to dissolve the residue and then repeat this step if necessary. Be sure to dump the organic waste in the appropriate waste container.
- 5.3. Samples are not preserved prior to extraction. After separating the solid and liquid phases of the extract, **divide the final extract into two portions of equal size and** acidify one portion of the extract's liquid **phase** by adding trace metal grade nitric acid until the liquid phase's pH is $<$ 2. **WARNING: DO NOT ACIDIFY ANY NON-AQUEOUS PORTION OF THE SAMPLE.** Nitric acid should not be mixed with organic compounds because of the possibility of a dangerous reaction. Mark the acidified extract's bottle as "preserved."

6. SAMPLE PREPARATION PROCEDURE

NOTE: Be sure to record all of the necessary information neatly and correctly in the TCLP logbook. Any information that needs to be recorded in the logbook is shown in boldface.

6.1. Preliminary Evaluations

6.1.1. Visually observe the original sample. Note in the logbook the number of sample phases, whether the sample is homogeneous or heterogeneous, whether the sample is 100% solid and describe the appearance of the sample.

6.1.2. Determination of Percent Solids

6.1.2.1. Percent solids are defined as that fraction of a waste sample from which no liquid may be forced out by an applied pressure. If the sample is sand or soil and obviously contains no liquid, the sample may be assumed to be 100 % solids and there is no need to determine the percent solids. If it is uncertain whether the sample is 100% solid, then percent solids must be determined.

6.1.2.2. Weigh a clean, dry, acid rinsed filter that will be used in sample filtration along with a piece of aluminum foil and record as **Mass of Clean Filter**. Clean the filter by rinsing it with 1N nitric acid followed by 3 consecutive rinses of DI water. Write the sample ID # on the aluminum foil for identification purposes. Weigh the beaker that will receive the filtrate and record weight of this beaker as the **Mass of Clean Receiving Vessel**. Set up the filtration device using the pre-weighed filter. Place the filtrate-receiving beaker beneath the filtration device outlet. Use a jack, if necessary, to raise the receiving beaker so that the beaker mouth is at the same level as the outlet. This will insure that the receiving beaker catches all of the filtrate.

6.1.2.3. Weigh approximately 103 g homogenized sample into the transfer beaker and record the weight of the sample as the **Mass of Beaker + waste**. The additional 3 grams is to account for the sample that may stick to the transfer beaker. Transfer the sample into the filtration device and weigh the dirty transfer beaker; record the weight as the **Mass of Dirty Beaker**. Determine the **Mass of Waste** (Mass of Beaker + Waste – Mass of Dirty Beaker) and record.

6.1.2.4. Re-tighten the fixtures holding the top on the filtration device. Place the sealing white O-ring on the top and attach the gas line. Gradually apply a pressure of 1 - 10 psi. (Note: Applying instantaneous pressure can prematurely plug the filter). If no filtrate comes out or the filtrate flow slows to less than 1 drip per 2 minutes, slowly increase pressure in 10 psi increments to a

maximum of 50 psi. It is important that pressure be increased slowly so that the filter does not prematurely plug. When pressurizing gas passes through the filter or when the filtrate flow is less than 1 drip per 2 minutes, stop filtration. Stop the gas flow, vent the excess pressure and remove the gas line from the filtration device.

NOTE: If the sample contains mostly aqueous liquids with fine particulate solids, it **MAY BE** necessary to centrifuge the sample **PRIOR** to filtration to prevent the sample from clogging the TCLP filter. If the sample needs to be centrifuged, then use the following steps:

- 6.1.2.4.1 The centrifuge must be operated with great care. The minimum number of centrifuge containers that may be used is 2. Each centrifuge container must be balanced with a twin centrifuge container. Place opposing containers side by side after filling each with sample and use a disposable transfer pipette to balance the liquid level between opposing containers.
- 6.1.2.4.2 Cover each centrifuge container with a small piece of aluminum foil to prevent spills. Wipe up any excess fluid on the exterior of each container. The centrifuge should be kept dry.
- 6.1.2.4.3 Wrap one paper towel around each container to ensure a snug fit into the rotor and place the containers in the centrifuge making sure that samples that were balanced to one another are opposite each other in the centrifuge rotor.
- 6.1.2.4.4 Close and secure the centrifuge lid.
- 6.1.2.4.5 Turn the knob to 10 to start the rotor turning. After a few seconds turn the speed knob to 30. Allow the rotor to accelerate for about 30 seconds and observe the centrifuge for any excessive shaking. If the centrifuge remains fairly steady turn the knob to 60 on the dial. Stay with the centrifuge until it stops accelerating, about 1 to 2 minutes. **DO NOT LEAVE THE CENTRIFUGE UNATTENDED WHILE IT IS ACCELERATING.**
- 6.1.2.4.6 Allow the centrifuge to run for at least 30 minutes. Samples that are very cloudy may need a centrifugation time of 60 to 120 minutes to be effective. When the determined time has passed stop the rotor by turning the control knob until it points to zero. The rotor will take several minutes to come to a stop.
- 6.1.2.4.7 After centrifuging the sample, decant off the liquid phase into the filtration device. Place the collection vessel

under the filtration device and collect the filtrate according to 6.1.2.4.

6.1.2.4.8 Open the filtration device and transfer as much of the solids as possible to the filtration device. You may add some of the filtered liquids back to the solids to loosen them. **DO NOT RINSE THE SOLID WITH DI WATER!!!** This will dilute the sample and it will not be useable.

6.1.2.4.9 Reassemble the filtration device and return the receiving vessel back under the filtration device. Apply pressure again as in 6.1.2.4. Proceed with determining percent solids.

6.1.2.5. Weigh the receiving beaker and filtrate and record weight of the **Mass of Vessel + filtrate**. Determine the weight of the **filtrate** (wt. of receiving beaker & filtrate – wt. of receiving beaker) and record. Determine the weight of the **solid** (wt. of sample – wt. of filtrate) and record. Determine the **percent solids** ([wt. of solid/wt. of subsample] * 100) and record as a percentage. This filtrate may have both an aqueous and non-aqueous phase.

6.1.2.5.1. If the percent solids are > 0.5% observe the filter and if it is damp, determine percent dry solids. The % dry solids may be determined by weighing the used filter (from filtration device) and corresponding foil. Place in an oven set at 100 °C ± 20 °C and dry until two successive weighings differ by less than 1%. Record both weights and compute the % dry solids (use the first weight recorded of the two measurements that indicated stability).

6.1.2.5.1.1.If the percent DRY solids are < 0.5%, go to the sample filtration step (6.3.2.2). The sample will be filtered through a Millipore, or equivalent, pressure filtration device; the resulting filtrate is defined as the TCLP extract.

6.1.2.5.1.2.If the percent DRY solids are ≥ 0.5% or if the filter is dry, proceed to the particle size reduction step (6.1.3).

6.1.2.5.2. If the percent solids are < 0.5%, then proceed to the sample filtration step (6.3.2.2). In this case, the sample will be filtered with a filtration device and the resulting filtrate will be defined as the TCLP extract.

NOTE: If the 100 g sample portion used in the determination of percent solids is 100% solid, then this portion can be used for the

extraction. If the 100 g percent solids sample portion yielded greater than 25g of solid (determined by percent solids), then this solid can be used for extraction. Twenty-five (25) g of solid is extracted with 500 mL extraction fluid, which should yield sufficient fluid for the digestion procedure.

6.1.3. Particle Size Reduction

6.1.3.1. This step determines if a sample requires particle size reduction. This is necessary if:

- a). The solid portion of sample does not pass through a 9.5 mm (0.375 in) standard sieve.

OR

- b). The surface area per gram of material is not equal to or greater than 3.1 cm^2

6.1.3.2. Surface area criteria apply only to filamentous waste materials such as paper or cloth (See Method 1311, section 7.1.3 for a more detailed description). The method states that there is no method for particle size reduction for materials that do not obviously meet the criteria.

6.1.3.3. To reduce particle size, crush, cut, or grind until particles are small enough. If particle size of the sample is reduced, note on extraction sheet in the sample notes section that particle size reduction was performed.

6.2. Determination of the Extraction Fluid to be used:

6.2.1. If the percent dry solid content of the sample is greater than or equal to 0.5%, the solid portion of the sample will need to be extracted. Determine the appropriate extraction fluid to be used for the sample as follows:

6.2.2. Place a 250 mL beaker on the scale and tare. Weigh 4.9 to 5.1 g of sample into the beaker and record as the **Mass of Subsample**. Pour the subsample into a 1mm sieve. Any sample that does not pass through sieve, break-up until the entire sample passes through. If it is obvious the sample will pass through the sieve without size reduction it is not necessary to pass through the sieve. Add 96.5 mL of DI water, record the **volume of DI water**, cover with a watch glass or parafilm, and stir vigorously on a stir plate for 5 minutes using a magnetic stirrer. Measure and record the pH with a calibrated pH meter as the **initial pH**. A solid state pH probe is preferred due to the interference from TCLP samples and standard pH electrodes.

6.2.2.1. If the pH of the sample is less than 5.0 pH units, extraction fluid #1 is used for the sample extraction.

6.2.2.2. If the pH is greater than 5.0 pH units, add 3.5 mL 1.0 N HCl and record as 3.5 mL **HCl added?** Swirl the sample and heat at 50 °C for 10 minutes in a water bath. Let the solution cool to room temperature, and measure/record the pH as the **pH after HCl + Heat**.

6.2.2.2.1. If the pH is less than 5.0, use extraction fluid #1.

6.2.2.2.2. If the pH is greater than 5.0, use extraction fluid #2. Be sure to record which extraction fluid will be used for each sample under the heading of **extraction fluid**.

6.3. Sample Extraction

NOTE: If the sample requires analysis for organics, tumble the sample in a glass or PTFE extraction vessel.

6.3.1. Each time a reusable extraction vessel is used; its identification number must be recorded in the TCLP glass extraction vessel log book. Following twenty (20) extractions in a particular vessel, that vessel must be used for a blank extraction in order to assess carryover contamination. To properly clean a glass or PTFE extraction vessel, first rinse with dilute HCl (20-50%). Then after rinsing with DI water 3x, wash glassware with an organic solvent, such as methanol (CH₃OH), followed by rinsing with DI water 3x.

6.3.2. A minimum “solid” sample size of 100 g is recommended. If the sample has a low percent solid, a large sample volume will be needed to produce the required amount of “solid” sample. Enough solids should be generated for extraction so that the volume of the TCLP extract is sufficient to support all necessary analyses. If the amount of extract generated by a single TCLP extraction is not sufficient to perform all analyses, more than one extraction may be performed and the extracts from each combined.

6.3.2.1. If the sample is 100% solid, weigh out approximately 103 g homogenized sample into a large weighing boat and record as the **Mass of Waste + Beaker**. The extra sample takes into account the sample loss occurring from the adherence of sample to the walls of the transfer vessel. Transfer the sample into the extraction vessel and reweigh the dirty beaker and record the weight as the **Mass of Dirty Beaker**. Determine the **Mass of Waste** (wt. of waste & transfer vessel – wt. of dirty transfer beaker) and record. Record this weight as the **Weight of Solid**. Determine the volume of extraction fluid to be used (i.e., weight of solid in grams x 20 = volume of extraction fluid to be used in mL). Record this volume as the **Extraction Fluid Volume Used**. Go to step 6.3.3.

- 6.3.2.2. If sample is liquid or multiphasic, liquid/solid separation is necessary. Filter the sample through a filtration device. The liquid filtrate is collected and stored for later processing. The solid remaining on the filter paper is extracted. To filter:
- 6.3.2.2.1. Assemble the filtration device using a clean filter that has been rinsed with 1N nitric acid followed by 3 consecutive rinses of DI water.
 - 6.3.2.2.2. Weigh the filtrate receiving beaker (the beaker that will receive the filtrate) and record the weight as the **Mass of Clean Beaker**. Place the filtrate receiving beaker beneath the filtration device outlet. Use a stand, if necessary, to raise the receiving beaker so that the beaker mouth is at the same level as the outlet. This insures the receiving beaker catches all of the filtrate.
 - 6.3.2.2.3. Weigh an appropriate amount (see section 6.3.2) of sample and record the weight as the **Mass of Waste + Beaker**.
 - 6.3.2.2.3.1. If the sample is $< 0.5\%$ dry solids, the filtrate is defined as the extract. Be sure to filter enough sample to get at least 500 mL of extract if possible -- this will be enough for metals and mercury digestions. Go to section 6.4.2.
 - 6.3.2.2.3.2. If the sample is $\geq 0.5\%$ dry solids, divide 100 g by the percent solids (in decimal form) to determine the amount of sample needed to filter to get 100 g of solid phase. **NOTE:** Do NOT use the percent DRY solid results for this calculation; use the percent solid results. Transfer the sample into the filtration device from the transfer beaker. Weigh the dirty transfer beaker and record as the Mass **Dirty Beaker**. Re-tighten the fixtures holding the top on the filtration device. Place the sealing white O-ring on the top and attach the gas line. Gradually apply a pressure of 1 - 10 psi. (Note: Applying instantaneous pressure can prematurely plug the filter!) If no filtrate comes out or the filtrate flow slows to less than 1 drip per 2 minutes, slowly increase pressure in 10 psi increments to a

maximum of 50 psi. It is important that pressure be increased slowly so that the filter does not prematurely plug. When pressurizing gas passes through the filter or when filtrate flow is less than 1 drip per 2 minutes, stop filtration. Stop gas flow and remove the gas line from the filtration device.

- 6.3.2.2.4. Determine and record **Mass of Waste** (wt. of waste & transfer vessel – wt. dirty transfer vessel). Weigh the receiving beaker and filtrate and record the **Mass of Beaker + Filtrate**. Determine the **Mass of Filtrate** (wt. of receiving beaker & filtrate – wt. of receiving beaker) and record. Determine the **Mass of Solid** (which is that part of the sample which did not filter through the filtration device, wt. of sample – wt. of filtrate) and record. Determine the volume of extraction fluid to be used (wt. of solid in grams x 20 = volume in mL) and record as **Volume of Extraction Fluid Used**.
 - 6.3.2.2.5. Transfer the filtrate into a clean sampling container, the size of which will depend on the amount of filtrate collected. If doing several extractions for one sample, use a larger container to begin with and store all filtrate from all extractions in that container.
 - 6.3.2.2.6. Carefully transfer the dirty filter from the filtration device to the extraction vessel using a clean spatula if necessary. Also, if the solid is thick and/or viscous, place a clean filter atop the dirty one to soak up the solid. This allows for cleaner transfer of the solid from the filtration device to the extraction vessel.
- 6.3.3. An Extraction Blank must be prepared for each set of TCLP samples that are extracted together. The Extraction Blank is prepared in the same manner as the samples. If both types of extraction fluid are used, prepare two blanks - one for each type of extraction fluid.
 - 6.3.4. Slowly add the appropriate amount of extraction fluid to the solid in the extraction vessel. Be sure to record the **pH of Extraction Fluid**. Screw the original extraction vessel lid. Finally, stretch Parafilm around the vessel lid to provide additional protection from leaks. Secure the extraction vessel into the rotator/tumbler. Check carefully for correct attachment. It may appear secure but can fall out of the agitator when touched. Make sure that the samples are evenly balanced and secure.

6.3.5. Turn on the tumbler and let the samples turn over a few times. Turn off the tumbler and check around the vessel lips for any leaks or bulging. Try to squeeze the vessel. If any leaks are noticed, remove the vessel and replace the Teflon tape as necessary. If bulging is observed or the vessel cannot be squeezed, unscrew the vessel and vent into a hood. Securely replace the vessel in the tumbler. Repeat the above steps while checking and fixing any leaks. If no leaks are observed, the extraction may proceed. The samples must rotate for 18 hours \pm 2 hours. **Record the temperature of room, the revolutions per minute of the agitator, time & date extraction started and time & date extraction complete.**

6.4. Extract Filtration:

6.4.1. After the 18 \pm 2 hour rotation period, the material in the vessel must be separated into liquid and solid phases by filtering through a clean filtration device with a clean, acid rinsed filter. Clean the filter by rinsing it with 1N nitric acid followed by 3 consecutive rinses of DI water.

6.4.2. If the extraction fluid is very cloudy with suspended solids and will be difficult to filter it may be centrifuged prior to filtration (see 6.1.2.4.1) Pour the non-filtered extract into the filtration device through the top intake port. Place the sealing white O-ring on the top and attach the gas line. Gradually apply a pressure of 1 psi, (filtrate should come out of the filtration device). **NOTE:** Applying instantaneous pressure can prematurely plug the filter! If no filtrate comes out or the filtrate flow slows to less than 1 drip per 2 minutes, slowly increase the pressure in 10 psi increments to a maximum of 50 psi. It is important that the pressure be increased **slowly** so that the filter does not prematurely plug. When pressurizing gas passes through the filter or when filtrate flow is less than 1 drip per 2 minutes, stop filtration. Stop the gas flow and remove the gas line from the filtration device.

6.4.3. If there are two phases in either the liquid extract or the filtrate, use a clean, acid-rinsed separatory funnel to separate the two phases. Record the volumes of the aqueous and non-aqueous phases of the extract. Record the weights of the aqueous and non-aqueous phases of the filtrate. Weigh 10.00 mL each of the aqueous and non-aqueous phases of the filtrate and use these to compute the density of each layer. The densities can then be used to compute the volumes of the aqueous and non-aqueous filtrate. Store each phase in a separate container and label. Combine the aqueous phase of the extract (normally the extract consists of a single aqueous phase) with the aqueous phase of the filtrate (if one was present). If applicable, combine the non-aqueous phase of the extract with the non-aqueous phase of the filtrate.

NOTE: If both non-aqueous extract and non-aqueous filtrate phases are present, the density of the combined mixture needs to be measured. Weigh a 10.00 mL portion of the combined non-aqueous phase and compute the density.

Record this value on the TCLP log sheets. Similarly, if a non-aqueous extract phase is present, and there is no non-aqueous filtrate, the density of the non-aqueous extract needs to be measured and recorded.

Record on the log sheet which phases were combined. The resulting aqueous and non-aqueous solutions should be labeled as either aqueous or non-aqueous. These two phases will be prepared and analyzed separately. The aqueous solution will be digested (diluted 1/10) as a water sample for TCLP analysis while the non-aqueous sample will be prepared as a WASTE sample. The results for the 2 phases will be suitably combined into a single result to be reported to the customer by the analysis group supervisor as a volume weighted average.

NOTE: NOTIFY YOUR SUPERVISOR AND THE ANALYSIS GROUP SUPERVISOR IMMEDIATELY IF THERE ARE BOTH AQUEOUS AND NON-AQUEOUS PHASES.

6.4.4. Determine and record:

- a. **Volume of aqueous extract** is the total aqueous volume of recovered extraction fluid.
- b. **Volume of non-aqueous extract** is the total volume of the non-aqueous phase of the recovered extraction fluid.
- c. **Volume of aqueous filtrate** is the volume of the aqueous phase of the sample filtrate
- d. **Volume of non-aqueous filtrate** is the volume of the non-aqueous phase of the sample filtrate.
- e. **Total aqueous volume** is the sum of the volumes of the aqueous extract and the aqueous filtrate.
- f. **Total non-aqueous volume** is the sum of the volumes of the non-aqueous extract and the non-aqueous filtrate.

6.4.5. Label the Extraction Blank with the ID numbers of the samples with which it was extracted and the extraction batch ID. Record the sample ID numbers in the comments section of the log sheet.

6.4.6. Determine and record the pH of the extract as **pH of recovered extraction fluid.**

6.4.7. If organic analyses are required on the sample separate approximately 3/4 of the sample for organic analysis (about 1600 mL of extraction fluid) and store in an amber glass bottle. Mark the organic sample number on the side of the bottle and give it to the organic prep group. Approximately 300-400 mL should be kept for metals preparation.

6.4.8. **Check for precipitation by adding acid to a small sub-sample.** Regardless of the formation of a precipitate, DO NOT ACIDIFY THE

ENTIRE SAMPLE! Separate the remaining sample intended for metals and/or mercury analysis into two separate containers and acidify one of the two separate containers of sample extract with trace metal grade nitric acid to a pH of < 2. Clearly label each portion of the TCLP extract as “preserved” or “not preserved”. If the samples are going to be digested the same day as filtration, then the samples can be acidified after the preparation for digestion. Record in the log sheet that the extract has been preserved under **preserved?**

6.4.9. Determine and record on the extraction sheet:

- a. Is the **filtrate multiphasic?**
- b. If so, record the **# of phases** in the filtrate.
- c. **Total filtrate weight** -- combined weight of all filtrates.
- d. **Total weight of aqueous phase** -- combined weight of aqueous phases of all filtrates.
- e. **Total weight of non-aqueous phase** -- combined weight of non-aqueous phases of all filtrates.
- f. **Weight of 10 mL aqueous filtrate** -- determine using a clean, acid-rinsed graduated cylinder.
- g. **Density of aqueous filtrate** -- gram weight of 10 mL aqueous filtrate/10 mL.
- h. **Volume of aqueous filtrate** --gram weight of aqueous filtrate / density.
- i. **Weight of 10 mL non-aqueous filtrate** -- determine by tarring a 10 mL volumetric flask, adding 10 mL to the flask, and weighing the flask and non-aqueous filtrate.
- j. **Density of non-aqueous filtrate** -- gram weight of 10 mL non-aqueous filtrate / 10 mL.
- k. **Volume of non aqueous filtrate** -- gram weight of non-aqueous filtrate / density.

NOTE FOR METALS PREPARATION:

The aqueous TCLP extract should be digested in the same manner as water samples for total recoverable metals analysis (MT-024) except that they need to be diluted 10-fold (1:10). For each type of extraction fluid, spike one of the samples it represents. Furthermore, for batching purposes like samples should be with like samples. For example, do not batch paint samples with soil samples. Create as many batches as necessary to ensure an accurate representation of the matrices present. The QC should contain a set of matrix spikes, set of replicates and an LFB per batch and only one

digestion blank is necessary per tray. Be sure to include the TCLP blanks in the digestion.

For the non-aqueous phase digest as a waste by hot block, hot plate, or microwave (MT-060).

For either type of digestion, aqueous or non-aqueous, print-out and send with the digestion sheet a TCLP flow chart. Also, be sure to note on the digestion sheet which phase is being digested.

7. QUALITY CONTROL

A minimum of 1 extraction fluid blank per batch of 20 samples or less is extracted using the required extraction fluid. If both extraction fluids are used, include two extraction blanks one for each extraction fluid type. A sample must be extracted in duplicate at least every 20 samples.

8. SAFETY/HAZARDOUS WASTE MANAGEMENT

8.1. Review the Laboratory Safety Manual and the Contingency Plans and Emergency Procedures for a Hazardous Waste Generator.

8.2. Use **CAUTION** with strong irritants such as acids, bases. Avoid breathing the fumes of these irritants by using them in a hood when possible and keeping the face away from open containers of these chemicals. Avoid contact of these irritants with skin and clothing by appropriate use of gloves, apron, face-mask, hood shield, etc. Safety glasses must be worn all the time in the Lab.

8.3. Store any wastes that cannot be disposed of down the sink (see Contingency Plans and Emergency Procedures, Hazardous Waste Generator) in a 2 L bottle (properly labeled with **Hazardous Wastes** labels) in the hood next to the instrument. Make sure the bottle stays capped after use and place the bottle in the cabinet below the hood in the metals lab when it is **NO MORE THAN 90% FULL**. When two bottles reach 90% full, dump them immediately into the Hazardous Waste drum located in Bldg C.

8.4. All the non-hazardous wastewater can be dumped into sink if its pH range is 10 - 5. If it is not, the wastewater has to be neutralized before dumping.

9. REFERENCES

9.1. EPA Method 1311, revision 0, November 1992.

9.2. TCLP.XLT -- the TCLP Excel template for recording data.

9.3. MT-007: SOP for cleaning glassware.

9.4. MT-024: SOP for waters metals digestion EPA Method 200.2 (modified).

9.5. MT-060: SOP for Hot Block Digestion of Soil, Sediment, Waste, and Tissue Samples for Total Recoverable Metals

Appendix A

Maximum Concentrations of Contaminates For Toxicity Characteristics

Contaminant	Regulatory Level (mg/L)
Arsenic	5.0
Barium	100.0
Cadmium	1.0
Chromium	5.0
Lead	5.0
Selenium	1.0
Silver	5.0
Mercury	0.2
Benzene	0.5
Carbon Tetrachloride	0.5
Chlordane	0.03
Chlorobenzene	100.0
Chloroform	6.0
o-Cresol	200.0
m-cresol	200.0
p-cresol	200.0
Cresol	200.0
2,4-D	10.0
1,4-Dichlorobenzene	7.5
1,2-Dichloroethane	0.5
1,1-Dichloroethylene	0.7
2,4-Dinitrotoluene	0.13 ²
Endrin	0.02
Heptachlor (and its hydroxide)	0.008
Hexachlorobenzene	0.13 ²
Hexachloro-1,3-butadiene	0.5

Hexachloroethane	3.0
Lindane	0.4
Methoxychlor	10.0
Methyl Ethyl Ketone	200.0
Nitrobenzene	2.0
Pentachlorophenol	100.0
Pyridine	5.0 ²
Tetrachloroethylene	0.7
Toxaphene	0.5
Trichloroethylene	0.7
2,4,5-Trichlorophenol	400.0
2,4,6-Trichlorophenol	2.0
2,4,5-TP (Silvex)	1.0
Vinyl Chloride	0.2

Appendix B

Totals Threshold for Possible TCLP Violations

Note: Only applicable to samples which are 100% solid.

Contaminant	Threshold (mg/kg)
Arsenic	100
Barium	2000
Cadmium	20
Chromium	100
Lead	100
Mercury	4
Selenium	20
Silver	100

Appendix C – Appendix of Significant Changes

Section 1 Edited to apply this SOP of TPLP Extraction to entire lab.

Appendices A and B added.