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Method SW846-1311 Toxicity Characteristic Leaching Procedure

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1 Scope and Application

1.2 Method SW846-1311, Toxicity Characteristic Leaching Procedure (TCLP) is used to determine the mobility of analytes in liquid, solid, and multiphasic samples. Samples, which undergo TCLP are digested after the extraction and then analyzed by either method SW846-6010B or SW846-6020.

Compound	CAS No.
Aluminum	7429-90-5
Antimony	7440-36-0
Arsenic	7440-38-2
Barium	7440-39-3
Beryllium	7440-41-7
Cadmium	7440-43-9
Calcium	7440-70-2
Chromium	7440-47-3
Cobalt	7440-48-4
Copper	7440-50-8
Iron	7439-89-6
Lead	7439-92-1
Magnesium	7439-95-4
Manganese	7439-96-5
Mercury	7439-97-6
Molybdenum	7439-98-7
Nickel	7440-02-0
Potassium	7440-09-7
Selenium	7782-49-2
Silver	7440-22-4
Sodium	7440-23-5
Strontium	7440-24-6
Thallium	7440-28-0
Compound	CAS No.

Tin	7440-31-5
Vanadium	7440-62-2
Zinc	7440-66-6

1.3 Restricted Procedure

This procedure is restricted to use by an analyst experienced in the handling of hazardous materials. Additionally, the analyst must complete the requirements of the GAEPD Initial Demonstration of Analyst Proficiency prior to the analysis of actual samples. Analysts are further warned that performance of this analysis involves the use of potentially hazardous chemicals; refer to the GAEPD Chemical Hygiene Plan for additional information regarding chemicals required by this method.

2 Definitions

2.1 Refer to Chapter 3 of the Georgia EPD Laboratory Quality Assurance Manual for Quality Control Definitions.

3 Interferences

3.1 Method interferences may be caused by contaminants in reagents or sample processing apparatus that lead to discrete artifacts or metallic contaminants. All reagents and apparatus must be routinely demonstrated to be free from interferences under the conditions of the analysis by running laboratory reagent blanks.

3.1.1 Glassware must be scrupulously cleaned. Any glassware used must be triple washed in reagent grade 1:1 nitric acid followed by a triple rinse in 18 MΩ water. After washing, all glassware is dried in a clean environment then covered with parafilm and stored in a closed cabinet until used. Virgin plasticware (HDPE) is used as much as possible and discarded after use.

4 Safety

Refer to Laboratory Chemical Hygiene Plan, online revision.

5 Apparatus and Equipment

5.1 Sample Container: 2L HDPE bottles with screw caps.
5.2 Agitation device capable of rotating the extraction vessel end-over-end at 30 +/- 2 rpm.
5.3 Teflon lined TCLP filtration device.
5.4 Compressed nitrogen for filtration.
5.5 Acid washed 47 mm diameter 0.6-0.8µm pore size borosilicate glass fiber filters.
5.6 Top loading balance accurate to +/- 0.01 grams and capable of weighing 2.5Kg. All weights will be recorded to +/- 0.1 grams.
5.7 500mL beaker.
5.8 Watch glass capable of covering the 500mL beaker.
5.9 Magnetic stir bar
5.10 Mortar and pestle
5.11 pH meter accurate to +/- 0.05 units at 25^{O}C

5.12 9.5 mm standard sieve.

6 Reagents and Standards

6.1 Reagent grade 1N hydrochloric acid.
6.2 Reagent grade 1N nitric acid.
6.3 Reagent grade 1N sodium hydroxide.
6.4 Reagent grade glacial acetic acid.
6.5 18MΩ water.
6.5 <u>Extraction fluid #1</u>
6.5.1 Add 11.4mL glacial acetic acid to 1L of 18MΩ water.
6.5.2 Add 128.6mL 1N sodium hydroxide, add enough 18MΩ water to bring the final volume up to 2L.
6.5.3 Measure the pH and record on the TCLP prep sheet. The pH must be 4.93 +/- 0.05 units, if not, the solution must be remade.
6.5.4 Check the pH log to determine if the pH meter has been calibrated for that day. If not, calibrate the pH meter with pH 4, 7, and 10 buffers before measuring the pH. Record the meter readings from each buffer on the pH log. The meter readings must agree with the buffer pH to within +/- 0.05 units at 25ºC.
6.6 <u>Extraction fluid #2</u>
6.6.1 Dilute 11.4mL glacial acetic acid to 2L with 18MΩ water.
6.6.2 Measure the pH and record on the TCLP prep sheet. The pH must be 2.88 +/- 0.05 units, if not, the solution must be remade.
6.7 1:1 HCl.

7 Sample Collection

7.1 Refer to Chapter 5 of the Georgia EPD Laboratory Quality Assurance Manual for Sample Container, Sample Preservation, and Sample Holding Times
7.2 Preservatives shall not be added to samples before extraction.

8 Calibration

8.1 Calibration Standards
8.2 Analytical standards are prepared according to the appropriate analytical method.
8.2 Calibration Curve
Not applicable
8.3 Calibration Verification
Not applicable

9 Quality Control

9.1 Refer to Table 13.1 for Reporting Limits (PQLs), Table 13.2 for Quality Control Acceptance Criteria, and Table 13.3 for Quality Control Procedures associated with this method.

10 Procedure

10.1 Preliminary Evaluations

10.1.1 Perform preliminary evaluations with a minimum 100g aliquot of sample. This aliquot may or may not undergo the actual extraction procedure. Record the weight of the aliquot on the TCLP prep sheet.

10.2 Preliminary determination of percent solids.

10.2.1 Percent solids is defined as the fraction of a sample from which no liquid may be forced out by an applied pressure using the procedure described below.

10.2.2 If the sample is liquid or multiphasic, filtering the sample using a TCLP filtration device with compressed nitrogen is required. This section may be skipped if the sample obviously does not contain any liquid.

10.2.3 Weigh the filter and container that will receive the filtrate. Record these weights on the TCLP log sheet.

10.2.4 Assemble the filter holder and filter according to the manufacturer's instructions. Place the filter on the holder and secure. Use acid washed filters; otherwise, wash the filter with 1L 1N HNO_3 followed by three 1L washings with 18MΩ water.

10.2.5 Weight out 100 grams of sample and record the weight on the TCLP prep sheet.

10.2.6 Quantitatively transfer the sample to the filter holder. Evenly spread the sample across the filter.

10.2.7 If >1% of the sample has stuck to the sample container, determine the weight of this residue, and subtract if from the original sample weight recorded earlier.

10.2.8 Close the filtration apparatus and connect to a compressed nitrogen tank. Gradually apply approximately 1-10psi pressure until no liquid moves through the filter for 2 minutes. Instantaneous application of pressure may degrade the filter or cause the filter pores to become clogged before all the liquid phase has been filtered. If no liquid moves through the filter after two minutes increase the pressure in 10psi increments. Continue to increase the pressure to a maximum of 50psi. Stop the filtration when either the applied pressure is 50psi or when the pressurizing gas moves through the filter.

10.2.9 Material retained on the filter is defined as the solid phase of the sample, the liquid that passed through the filter is defined as the liquid phase of the sample. Some samples, such as oily materials and paints will have some material retained on the filter that appears to be a liquid even after filtering under pressure to 50psi. This material is defined as the solid phase.

10.2.10 Only one filter is used to determine the percent solids. Under no circumstance is the filter to be removed or replaced.

10.2.11 Weigh the filtrate container and record on the TCLP prep sheet.

10.2.12 Determine the weight of the filtrate by subtracting the weight of the filtrate plus container from the weight of the empty container. Record this weight on the TCLP prep sheet.

10.2.13 Determine the weight of the solid phase by subtracting the weight of the liquid phase from the total sample weight. Record this weight on the TCLP prep sheet.

10.2.14 Calculate the percent solids as follows:

Percent Solids = [Weight of solid (10.2.13) /Total weight of aliquot (10.1.1)] * 100

10.2.15 If the percent solids from 10.2.14 is equal to or greater than 0.5% determine whether particle size reduction is required (section 10.3).
10.2.16 If the percent solids are < 0.5% and it appears that liquid has been entrained in the filter then dry the filter at 100ºC +/- 20ºC for a minimum of one hour. Cool in a desiccator for at least one hour, weigh the dried filter. If this weight is ≤ original weight, record and use to determine percent solids in step 10.5.14, otherwise continue drying and cooling until two successive weights agree to within one percent of each other. Use this weight to determine the percent solids in step 10.2.14.
10.2.17 If the percent solids are < 0.5% the filtrate is defined as the TCLP extract. Proceed to section 10.5.14.
10.3 Preliminary Determine whether particle reduction is required.
10.3.1 Particle size reduction is not required if the solid phase will pass through a 9.5 mm standard sieve. If particle size reduction is required, grind the solid phase in a mortar and pestle until it will pass through a 9.5 mm standard sieve.
10.4 Preliminary Determination of appropriate extraction fluid.
10.4.1 This procedure applies only to the solid phase of the aliquot.
10.4.2 Weight out a small subsample of the solid phase of the aliquot, reduce the particle size to approximately 1mm in diameter (if needed) and transfer 5.0 grams to a 500 beaker.
10.4.3 Add 96.5 grams of water to the beaker, add the magnetic stir bar, cover with a watch glass and stir vigorously for 5 minutes.
10.4.4 Measure the pH, record the pH on the TCLP prep sheet.
10.4.5 If the pH is < 5.0 use extraction fluid 1.
10.4.6 If the pH is > 5.0 add 3.5 mL 1N hydrochloric acid, cover with a watch glass, heat to 50ºC and hold at 50ºC for 10 minutes.
10.4.7 Let the solution cool to room temperature and record the pH on the TCLP prep sheet. If the pH is <5.0 use extraction fluid 1, otherwise use extraction fluid 2.
10.5 TCLP Extraction Procedure
10.5.1 If the aliquot was 100% solid and at least 100 grams of the aliquot remains, the aliquot used for the preliminary determinations can be used for the TCLP extraction procedure.
10.5.2 If the aliquot was multiphasic, calculate the weight of appropriate extraction fluid to extract the retained solids. If the weight of extraction fluid required is at least 100 grams the retained solids from the preliminary determinations can be used for the TCLP extraction procedure. The liquid phase will be combined with the filtered extraction fluid from the solid phase after extraction.
10.5.3 If the weight of extraction fluid required is not at least 100 grams, filter an adequate amount of original sample so that enough solids are retained to require at least 100 grams of extraction fluid.
10.5.4 If another aliquot of sample needs to be filtered to obtain sufficient solids to perform the TCLP extraction, follow the procedure described in steps 10.2.3 to 10.2.13 using a large

enough initial sample size to require at least 100 grams of extraction fluid. The liquid phase will be combined with the filtered extraction fluid from the solid phase after extraction.

10.5.5 Calculate the weight of extraction fluid to use as follows:

Weight of extraction fluid = [20 * percent solids * weight of the waste filtered]/100

10.5.6 If another aliquot of the sample was filtered for the extraction procedure, determine whether particle size reduction is required by repeating steps 10.3 and 10.3.1.

10.5.7 Quantitatively transfer the retained solids and filter from the filtration apparatus to the extraction vessel.

10.5.8 Slowly add the calculated weight of the appropriate extraction fluid to the extraction vessel, cap the extraction vessel, and place in the TCLP agitation device.

10.5.9 Tumble the extraction vessel end-over-end for 18+/- 2 hours at 23ºC +/- 2ºC and 30 +/- 2 rpm. Record the start time, end time, rpm, start temperature, and end temperature on the TCLP prep sheet.

10.5.10 After the 18 +/- 2-hour extraction, filter all the material in the extraction vessel through the TCLP filtration apparatus using a new filter. This filter can be changed if it becomes clogged.

10.5.11 If the sample is multiphasic, combine the liquid filtrate from either step 10.2.8 or 10.5.4, whichever is appropriate, with the filtered extraction fluid.

10.5.12 If the sample is multiphasic but the liquid filtrate is not compatible with the extracted liquid, analyze each phase separately and combine the results mathematically using the following formula:

Final Analyte Concentration: $(V_1C_1 + V_2C_2)/(V_1+V_2)$

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)

10.5.13 Measure and record the pH of the filtered extract on the TCLP prep sheet.

10.5.14 Transfer an aliquot of the extract to a new HDPE screw cap bottle and add 5mls of 1:1 nitric acid per liter to preserve the sample. Measure and record the pH of the preserved aliquot on the TCLP prep sheet. The pH must be < 2. If not, add additional aliquots of 1:1 nitric acid until the pH is <2.

10.5.15 Digest a 50ml aliquot of the preserved TCLP extract by method SW846-3010B and analyze the digestate by either method SW846-6010B or SW846-6020.

10.6 Quality Control

10.6.1 A matrix blank is prepared for each extraction fluid used for each batch.

10.6.2 A matrix spike and matrix spike duplicate are prepared for every ten samples in a batch.

10.6.3 A duplicate sample is extracted for every batch for each extraction fluid.

10.6.4 An LCS and LCSD are prepared for each batch.

11 Evaluation of the Linearity of the Initial Calibration.

11.1 Not applicable

12 References

12.1 EPA Test Methods for evaluating solid waste, method SW846-1311, Revision 0, July 1992

13 Practical Quantitation Limits (PQLs), Precision and Accuracy Criteria, and Quality Control Approach

Not applicable