

## METHOD 1316<sup>1</sup>

### LIQUID-SOLID PARTITIONING AS A FUNCTION OF LIQUID-TO-SOLID RATIO IN SOLID MATERIALS USING A PARALLEL BATCH PROCEDURE

SW-846 is not intended to be an analytical training manual. Therefore, method procedures are written based on the assumption that they will be performed by analysts who are formally trained in at least the basic principles of chemical analysis and in the use of the subject technology.

In addition, SW-846 methods, with the exception of required methods used for the analysis of method-defined parameters, are intended to be guidance methods that contain general information on how to perform an analytical procedure or technique, which a laboratory can use as a basic starting point for generating its own detailed standard operating procedure (SOP), either for its own general use or for a specific project application. Performance data included in this method are for guidance purposes only and must not be used as absolute quality control (QC) acceptance criteria for purposes of laboratory QC or accreditation.

#### 1.0 SCOPE AND APPLICATION

1.1 This method is designed to provide the liquid-solid partitioning (LSP) of inorganic constituents (e.g., metals, radionuclides) and non-volatile organic constituents (e.g., polycyclic aromatic hydrocarbons (PAHs)), dissolved organic carbon) at the natural pH of the solid material as a function of liquid-to-solid ratio (L/S) under conditions that approach liquid-solid chemical equilibrium. Table 1 shows the range of target L/S values tested under this method.

1.2 The eluate concentrations at a low L/S provide insight into pore solution composition either in a granular bed (e.g., soil column) or in the pore space of low-permeability material (e.g., solidified monolithic or compacted granular fill). In addition, analysis of eluates for dissolved organic carbon and of the solid phase for total organic carbon allow for evaluation of the impact of organic carbon release and the influence of dissolved organic carbon on the LSP of inorganic constituents.

1.3 This method is intended to be used as part of environmental leaching assessment for the evaluation of disposal, beneficial use, treatment effectiveness and site remediation. The method is not required by federal regulations to determine whether waste passes or fails the toxicity characteristic as defined at 40 CFR 261.24.

1.4 This method is suitable for assessing the leaching potential of a wide range of solid materials. Examples of solid materials include: industrial wastes, soils, sludges, combustion residues, sediments, stabilized materials, construction materials, and mining wastes.

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<sup>1</sup> This method has been derived from the SR003 procedure (Ref. 1) using Environmental Protection Agency-reviewed and accepted methodologies (see Refs. 3 - 5). The method is analogous to batch L/S-dependence method EN12457 (Ref. 7) developed for the Comité Européen de Normalisation (CEN).

1.5 This method is a leaching characterization method used to provide intrinsic material parameters that control leaching of inorganic species under equilibrium conditions. This test method is intended as a means for obtaining an extract (i.e., the eluate) of a solid material which may be used to estimate the solubility and release of inorganic constituents under the laboratory conditions described in this method. Extract concentrations may be used in conjunction with information regarding environmental management scenarios to estimate anticipated leaching concentrations, and release rate and extent for individual material constituents in the management scenarios evaluated. Extract concentrations may also be used along with geochemical speciation modeling to infer the mineral phases that control the LSP in the pore structure of the solid material.

1.6 This method is not applicable to characterize the release of volatile organic analytes (e.g., benzene, toluene and xylenes).

1.7 This method provides solutions that are considered to be indicative of leachate under field conditions only where the field leaching pH and L/S ranges are encompassed by the laboratory extract final conditions and the LSP is controlled by aqueous-phase saturation of the constituent of interest. When LSP is controlled by the amount of the constituent present in the solid that may dissolve (i.e., for highly soluble species), the mass released (mg/kg), rather than the concentration, is indicative of field conditions.

1.8 The solvent used in this method is reagent water.

1.9 Analysts are advised to take reasonable measures to ensure that the sample is homogenized to the extent practical prior to employment of this method. Particle size reduction may provide additional assurance of sample homogenization. Table 2 designates a minimum dry equivalent mass of sample to be added to each extraction vessel and the associated extraction contact time as a function maximum particle diameter. If the heterogeneity of the sample is suspected as the cause of unacceptable levels of precision in replicate test results or is considered significant based on professional judgment, the sample mass used in the test procedure may be increased to a greater minimum dry equivalent mass than shown in Table 1 with the amount of extractant increased proportionately to maintain the designated L/S.

1.10 In the preparation of solid materials for use in this method, particle size reduction of samples with large grain size is used to enhance the approach towards liquid-solid equilibrium under the designated contact time interval of the extract process. The extract contact time for samples reduced to a finer maximum particle size will be shorter.

1.11 Prior to employing this method, analysts are advised to consult the base method for each type of procedure that may be employed in the overall analysis (e.g., Methods 9040, 9045, and 9050) for additional information on QC procedures, development of QC acceptance criteria, calculations, and general guidance. Analysts also should consult the disclaimer statement at the front of the manual and the information in Chapter Two for: 1) guidance on the intended flexibility in the choice of methods, apparatus, materials, reagents, and supplies, and 2) the responsibilities of the analyst for demonstrating that the techniques employed are appropriate for the analytes of interest, in the matrix of interest, and at the levels of concern.

In addition, analysts and data users are advised that, except where explicitly specified in a regulation, the use of SW-846 methods is not mandatory in response to federal testing requirements. The information contained in this method is provided by the Environmental

Protection Agency (EPA or the Agency) as guidance to be used by the analyst and the regulated community in making judgments necessary to generate results that meet the data quality objectives (DQOs) for the intended application.

1.12 This method is restricted to use by, or under supervision of, properly experienced and trained personnel. Each analyst must demonstrate the ability to generate acceptable results with this method.

## 2.0 SUMMARY OF METHOD

This method consists of five parallel extractions of a particle size-reduced solid material in reagent water over a range of L/S values from 0.5 to 10 mL eluent/g dry material (see Table 1). In addition to the five test extractions, a method blank without solid sample is carried through the procedure in order to verify that analyte interferences are not introduced as a consequence of reagent impurities or equipment contamination. If multiple materials or replicate tests are carried out in parallel, only one set of method blanks is necessary. In total, six bottles (i.e., five test positions and one method blank) are tumbled in an end-over-end fashion for a specified contact time based on the maximum particle size of the solid (see Table 2). At the end of the contact interval, the liquid and solid phases are roughly separated via settling or centrifugation. Extract pH and specific conductance measurements are then taken on an aliquot of the liquid phase. The bulk of the eluate is clarified by pressure or vacuum filtration in preparation for constituent analysis. Analytical aliquots of the extracts are collected and preserved accordingly based on the determinative methods to be performed. The eluate constituent concentrations are plotted as a function of L/S and compared to QC and assessment limits.

## 3.0 DEFINITIONS

3.1 Constituent of potential concern (COPC) – A chemical species of interest, which may or may not be regulated, but may be characteristic of release-controlling properties of the sample geochemistry.

3.2 Release – The dissolution or partitioning of a COPC from the solid phase to the aqueous phase during laboratory testing (or under field conditions). In this method, mass release is expressed in units of mg COPC/kg dry solid material.

3.3 Liquid-solid partitioning (LSP) – The distribution of COPCs between the solid and liquid phases at the conclusion of the extraction.

3.4 Liquid-to-solid ratio (L/S) – The fraction of the total liquid volume (including the moisture contained in the "as used" solid sample) to the dry mass equivalent of the solid material. L/S is typically expressed in volume units of liquid per dry mass of solid material (mL/g-dry).

3.5 "As-tested" sample – The solid sample at the conditions (e.g., moisture content and particle-size distribution) present at the time of the start of the test procedure. The "as-tested" conditions will differ from the "as-received" sample conditions if particle-size reduction and drying were necessarily performed.

3.6 Dry-mass equivalent – The mass of "as-tested" (i.e., "wet") sample that equates to the mass of dry solids plus associated moisture, based on the moisture content of the "as-tested" material. The dry-mass equivalent is typically expressed in mass units of the "as-tested" sample (g).

3.7 Eluent – The solution used to contact the solid material in a leaching test. The eluent is usually free of COPCs but may contain other species used to control the test conditions of the extraction.

3.8 Eluate – The solution collected as an extract from a leaching test that contains the eluent plus constituents leached from the solid phase.<sup>2</sup>

3.9 Refer to Chapter One, Chapter Three, and the manufacturers' instructions for definitions that may be relevant to this procedure.

#### 4.0 INTERFERENCES

Solvents, reagents, glassware, and other sample processing hardware may yield artifacts and/or interferences to sample analysis. All of these materials must be demonstrated to be free from interferences under the conditions of the analysis by analyzing method blanks. Specific selection of reagents may be necessary. Refer to each method to be used for specific guidance on QC procedures and to Chapters Three and Four for general guidance on glassware cleaning. Also refer to Methods 9040, 9045, and 9050 for a discussion of interferences.

#### 5.0 SAFETY

This method does not address all safety issues associated with its use. The laboratory is responsible for maintaining a safe work environment and a current awareness file of Occupational Safety and Health Administration (OSHA) regulations regarding the safe handling of the chemicals specified in this method. A reference file of material safety data sheets (MSDSs) should be available to all personnel involved in these analyses.

#### 6.0 EQUIPMENT AND SUPPLIES

The mention of trade names or commercial products in this manual is for illustrative purposes only, and does not constitute an EPA endorsement or exclusive recommendation for use. The products and instrument settings cited in SW-846 methods represent those products and settings used during the method development or subsequently evaluated by the Agency. Glassware, reagents, supplies, equipment, and settings other than those listed in this manual may be employed provided that method performance appropriate for the intended application has been demonstrated and documented.

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<sup>2</sup> The definition of "eluate" is used in this method to differentiate the output solution of a leaching test from the "leachate" solution collected from, or measured in, the field. The distinction between terms is made to minimize confusion when comparing laboratory results to field data (Ref. 7).

This section does not list all common laboratory glassware (e.g., beakers and flasks) that might be used.

## 6.1 Extraction vessels

6.1.1 Six wide-mouth bottles (i.e., five for test positions plus one for a method blank) constructed of inert material, resistant to high and low pH conditions and interaction with the constituents of interest, as described in the following sections.

6.1.1.1 For the evaluation of inorganic COPCs, bottles made of high density polyethylene (HDPE) (e.g., Nalgene #3140-0250 or equivalent), polypropylene (PP), or polyvinyl chloride (PVC) are recommended.

6.1.1.2 For the evaluation of non-volatile organic and mixed organic/inorganic COPCs, bottles made of glass or Type-316 stainless steel are recommended. Polytetrafluoroethene (PTFE) is not recommended for non-volatile organics, due to the sorption of species with high hydrophobicity (e.g., PAHs). Borosilicate glass is recommended over other types of glass, especially when inorganic analytes are of concern.

6.1.2. The extraction vessels must be of sufficient volume to accommodate both the solid sample and an extractant volume based on the schedule of L/S values shown in Table 1. For example, a 500-mL bottle is recommended when 100 g dry equivalent mass is contacted with 200 mL of eluent (see T03 in Table 1).

6.1.3 The vessels must have a leak-proof seal that can sustain end-over-end tumbling for the duration of the designated contact time.

6.1.4 If centrifugation is anticipated to be beneficial for initial phase separation, the extraction vessels should be capable of withstanding centrifugation at  $4000 \pm 100$  rpm for a minimum of  $10 \pm 2$  min. Alternately, samples may be extracted in bottles that do not meet this centrifugation specification (e.g., Nalgene I-Chem #311-0250 or equivalent) and the solid-liquid slurries transferred into appropriate centrifugation vessels for phase separation as needed.

6.2 Balance – Capable of 0.01 g resolution for masses less than 500 g

6.3 Rotary tumbler – Capable of rotating the extraction vessels end-over-end at a constant speed of  $28 \pm 2$  rpm (e.g., Environmental Express, Charleston, SC or equivalent)

NOTE: The holding capacity of tumblers may vary and modifications (e.g., packing or bottle-holding inserts) may be necessary to accommodate the extraction vessels.

6.4 Filtration apparatus – Pressure or vacuum filtration apparatus composed of appropriate materials to maximize collection of extracts and minimize loss of COPCs (e.g., Nalgene #300-4000 or equivalent) (see Sec. 6.1)

6.5 Filtration membranes – Composed of polypropylene or equivalent material with an effective pore size of  $0.45 \mu\text{m}$  (e.g., Gelman Sciences GH Polypro #66548 from Fisher Scientific or equivalent)

6.6 pH meter – Laboratory model capable of temperature compensation (e.g., Accumet 20, Fisher Scientific or equivalent) with a minimum resolution of 0.1 pH units

6.7 pH combination electrode – Composed of chemically resistant materials

6.8 Conductivity meter – Laboratory model (e.g., Accumet 20, Fisher Scientific or equivalent), with a minimum resolution of 5% of the measured value

6.9 Conductivity electrodes – Composed of chemically resistant materials

6.10 Adjustable-volume pipettor – Oxford Benchmate series or equivalent. The necessary delivery range will depend on the buffering capacity of the solid material and acid/base strength used in the test.

6.11 Disposable pipettor tips

6.12 Centrifuge (recommended) – Capable of centrifuging the extraction vessels at a rate of  $4000 \pm 100$  rpm for  $10 \pm 2$  min

## 7.0 REAGENTS AND STANDARDS

7.1 Reagent-grade chemicals, at a minimum, should be used in all tests. Unless otherwise indicated, all reagents should conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society (ACS), where such specifications are available. Other grades may be used, provided the reagents are of sufficiently high purity to permit use without lessening the accuracy of the determination. Inorganic reagents and extracts should be stored in plastic to prevent interaction of constituents from glass containers. Organic reagents should be stored in glass to prevent leaching of contaminants from plastic containers.

7.2 Reagent water must be interference free. All references to water in this method refer to reagent water unless otherwise specified.

7.3 Consult Methods 9040 and 9050 for additional information regarding the preparation of reagents required for pH and specific conductance measurements.

## 8.0 SAMPLE COLLECTION, PRESERVATION, AND STORAGE

8.1 See Chapter Three, "Inorganic Analytes," and Chapter Four, "Organic Analytes," for sample collection and preservation information.

8.2 All solid samples should be collected using an appropriate sampling plan.

8.3 All containers should be composed of materials that minimize interaction with solution COPCs. For further information, see Chapters Three and Four.

8.4 Preservatives should not be added to samples before extraction.

8.5 Samples can be refrigerated, unless refrigeration results in an irreversible physical change to the sample.

8.6 Analytical extracts or leachates should be preserved according to the guidance given in the individual determinative methods for the COPCs.

8.7 Extract holding times should be consistent with the holding times specified in the individual determinative methods for the COPCs.

## 9.0 QUALITY CONTROL

9.1 Refer to Chapter One for guidance on quality assurance (QA) and quality control (QC) protocols. When inconsistencies exist between QC guidelines, method-specific QC criteria take precedence over both technique-specific criteria and Chapter One criteria, and technique-specific QC criteria take precedence over Chapter One criteria. Any effort involving the collection of analytical data should include development of a structured and systematic planning document, such as a quality assurance project plan (QAPP) or a sampling and analysis plan (SAP), which translates project objectives and specifications into directions for those who will implement the project and assess the results.

Each laboratory should maintain a formal QA program. The laboratory should also maintain records to document the quality of the data generated. Development of in-house QC limits for each method is encouraged. Use of instrument-specific QC limits is encouraged, provided such limits will generate data appropriate for use in the intended application. All data sheets and QC data should be maintained for reference or inspection.

9.2 In order to demonstrate the purity of reagents and sample contact surfaces, a method blank (e.g., a bottle without solid material but with eluent carried through the extraction, filtration and analytical sample preparation process) should be tested.

9.3 The analysis of extracts should follow appropriate QC procedures, as specified in the determinative methods for the COPCs. Refer to Chapter One for specific QC procedures.

9.4 Solid materials should be tested within one month of receipt unless the project requires that the "as-received" samples are tested sooner (e.g., the material is part of a time-dependent study or the material may change during storage due to oxidation or carbonation).

### 9.5 Initial demonstration of proficiency (IDP)

Leachate methods are not amenable to typical IDPs when reference materials with known values are not available. However, prior to using this method an analyst should have documented proficiency in the skills required for successful implementation of the method. For example, skill should be demonstrated in the use of an analytical balance, the determination of pH using methods 9040 and 9045 and the determination of conductance using method 9050.

## 10.0 CALIBRATION AND STANDARDIZATION

10.1 The balance should be calibrated and certified, at a minimum, annually or in accordance with laboratory policy.

10.2 Prior to measurement of eluate pH, the pH meter should be calibrated using a minimum of two standards that bracket the range of pH measurements. Refer to Methods 9040 and 9045 for additional guidance.

10.3 Prior to measurement of eluate conductivity, the meter should be calibrated using at least one standard at a value greater than the range of conductivity measurements. Refer to Method 9050 for additional guidance.

## 11.0 PROCEDURE

A flowchart of the method is presented in Figure 1. Microsoft Excel<sup>®</sup> data templates are available to aid in collecting and archiving of laboratory and analytical data.<sup>3</sup>

### 11.1 Particle size reduction (if required)

11.1.1 In this method, particle size reduction is used to prepare large-grained samples for extraction so that the approach toward liquid-solid equilibrium is enhanced and mass transport through large particles is minimized. A longer extract contact time is required for larger maximum particle size designations. This method designates three maximum particle sizes and associated contact times (see Table 2). The selection of an appropriate maximum particle size from this table should be based on professional judgment regarding the practical effort required to size reduce the solid material.

11.1.2 Particle size reduction of "as received" sample may be achieved through crushing, milling, or grinding with equipment made from chemically inert materials. During the reduction process, care should be taken to minimize loss of sample and potentially volatile constituents in the sample.

11.1.3 If the moisture content of the "as-received" material is greater than 15% (wet basis), air drying or desiccation may be necessary. Oven drying is not recommended for preparation of test samples due to the potential for mineral alteration. In all cases, the moisture content of the "as received" material should be recorded.

**NOTE:** If the solid material is susceptible to interaction with the atmosphere (e.g., carbonation, oxidation), drying should be conducted in an inert environment.

11.1.4 When the material seems to be of a relatively uniform particle size, calculate the percentage less than the sieve size as follows:

$$\% \text{ Passing} = \frac{M_{\text{sieved}}}{M_{\text{total}}} \times 100 \%$$

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<sup>3</sup> These Excel<sup>®</sup> templates form the basis for uploading method data into the data management program, LeachXS Lite™. Both the data templates and LeachXS Lite™ are available at <http://vanderbilt.edu/leaching>.



Where:

$M_{\text{sieved}}$  = mass of sample passing the sieve (g)

$M_{\text{total}}$  = mass of total sample (g) (e.g.,  $M_{\text{sieved}}$  + mass not passing sieve)

11.1.5 The fraction retained by the sieve should be recycled for further particle size reduction until at least 85% of the initial mass has been reduced below the designated maximum particle size. Calculate and record the final percentage passing the sieve and the designated maximum particle size. For the uncrushable fraction of the "as received" material, record the fraction mass and nature (e.g., rock, metal or glass shards, etc).

11.1.6 Store the size-reduced material in an airtight container in order to prevent contamination via gas exchange with the atmosphere. Store the container in a cool, dark and dry place prior to use.

## 11.2 Determination of solids and moisture content

11.2.1 In order to provide the dry mass equivalent of the "as-tested" material, the solids content of the subject material should be determined. Often, the moisture content of the solid sample is recorded. In this method, the moisture content is determined and recorded on the basis of the "wet" or "as-tested" sample.

**WARNING:** The drying oven should be contained in a hood or otherwise properly ventilated. Significant laboratory contamination or inhalation hazards may result when drying heavily contaminated samples. Consult the laboratory safety officer for proper handling procedures prior to drying samples that may contain volatile, hazardous, flammable or explosive materials.

11.2.2 Place a 5 to 10-g sample of solid material into a tared dish or crucible. Dry the sample to a constant mass at  $105 \pm 2$  °C. Check for constant mass by returning the dish to the drying oven for 24 hours, cooling to room temperature in a desiccator and re-weighing. The two mass readings should agree within the larger of 0.2% or 0.02 g.

**NOTE:** The oven-dried sample is not used for the extraction and should be properly disposed of once the dry mass is determined.

11.2.3 Calculate and report the solids content as follows:

$$SC = \frac{M_{\text{dry}}}{M_{\text{test}}}$$

Where:

SC = solids content of "as-tested" material (g-dry/g)

$M_{\text{dry}}$  = mass of dry material specified in the method (g-dry)

$M_{\text{test}}$  = mass of "as-tested" solid equivalent to the dry-material mass (g)

11.2.4 Calculate and report the moisture content (wet basis) as follows:

$$MC_{\text{wet}} = \frac{M_{\text{test}} - M_{\text{dry}}}{M_{\text{test}}}$$

Where:

$MC_{(\text{wet})}$  = moisture content on a wet basis (g<sub>H<sub>2</sub>O</sub>/g)

$M_{\text{dry}}$  = mass of dry material specified in the method (g-dry)

$M_{\text{test}}$  = mass of "as-tested" solid equivalent to the dry-material mass (g)

### 11.3 Extraction setup schedule (Microsoft Excel® template provided)

An Excel® template accompanies this method and may be used to set up the extraction schedule. If using the provided template, disregard Sec. 11.3 and proceed to the extraction procedure Sec. 11.4.

11.3.1 Using the schedule shown in Table 1 as a guide, set up five test extractions and one method blank. The mass of solids in an extraction may be scaled to minimize headspace in each extraction vessel. However, the volume of eluent should always be based on the target L/S in Column B of Table 1.

11.3.2 Calculate and record the amount of "as-tested" material equivalent to the dry mass in Column D of Table 1 as follows:

$$M_{\text{test}} = \frac{M_{\text{dry}}}{SC}$$

Where:

$M_{\text{test}}$  = mass of "as-tested" solid equivalent to the dry-material mass (g)

$M_{\text{dry}}$  = mass of dry material specified in the method (g-dry)

SC = solids content of "as-tested" material (g-dry/g)

11.3.3 Calculate and record the volume of moisture contained in the "as-tested" sample in Column E of Table 1 as follows:

$$V_{W,\text{sample}} = \frac{M_{\text{test}} \times (1 - SC)}{\rho_w}$$

Where:

$V_{W,\text{sample}}$  = volume of water in the "as tested" sample (mL)

$M_{\text{test}}$  = mass of "as-tested" solid equivalent to the dry-material mass (g)

SC = solids content of the "as tested" sample (g-dry/g)

$\rho_w$  = density of water (1.0 g/mL at room temperature)

11.3.4 Calculate and record the volume of reagent water required to bring each extraction to the target L/S in Column F of Table 1 as follows:

$$V_{RW} = M_{\text{dry}} \times LS - V_{W,\text{sample}}$$

Where:

$V_{RW}$  = volume of reagent water required to complete L/S (mL)

$M_{dry}$  = mass of dry material specified in the method (g-dry)

LS = liquid-to-dry-solid ratio (10 mL/g)

$V_{W,sample}$  = volume of water in "as tested" sample (mL)

The size of the extraction bottle should be sufficient to contain the combined volume of solid material and eluent, ideally with a minimum amount of headspace.

#### 11.4 Extraction procedure

11.4.1 Label five bottles with test position numbers and an additional bottle as a method blank according to Column A in Table 1.

11.4.2 Place the dry-mass equivalent ( $\pm 0.1$  g) of "as-tested" sample as shown in Column D in Table 1 into each of the five test position extraction vessels.

**NOTE:** Do not put solid material in the method blank extraction vessel.

11.4.3 Add the appropriate volume ( $\pm 0.5$  mL) of reagent water to both the test position and method blank extraction vessels as specified in Column F of Table 1.

11.4.4 Tighten the leak-proof lid on each bottle and tumble all extractions (i.e., test positions and method blanks) in an end-over-end fashion at a speed of  $28 \pm 2$  rpm at room temperature ( $20 \pm 2$  °C). The contact time for this method will vary depending on the maximum particle size as shown in Table 2.

**NOTE:** The length of the contact time is designed to enhance the approach toward liquid-solid equilibrium. Longer contact times are required for larger particles to compensate for the effects of intra-particle diffusion. See Table 2 for required contact times based on the maximum particle size.

11.4.5 Remove the extraction vessels from the rotary tumbler and clarify the extracts by allowing the bottles to stand for  $15 \pm 5$  min. Alternately, centrifuge the extraction vessels at  $4000 \pm 100$  rpm for  $10 \pm 2$  min.

**NOTE:** If clarification is significantly incomplete after settling or centrifugation, eluate measurements for pH, conductivity, and oxidization-reduction potential (ORP) may be taken on filtered samples. In this case, perform the filtration in Sec. 11.4.8 prior to eluate measurement in Sec. 11.4.6 and note the deviation from the written procedure.

**CAUTION:** Following separation from the solid phase, eluate samples lack the buffering provided by the solid phase and therefore may be susceptible to pH change resulting from interaction with air.

11.4.6 For each extraction vessel, decant a minimum volume (approximately 5 mL) of clear, unpreserved supernatant into a clean container.

11.4.7 Measure and record the pH, specific conductivity, and ORP of the extracts (see Methods 9040, 9045, and 9050).

NOTE: Measurement of pH, conductivity, and ORP should be taken within 15 minutes of eluate processing (Sec. 11.4.8) to avoid neutralization of the solution due to exposure to carbon dioxide, especially when alkaline materials are tested.

NOTE: The measurement of ORP is optional, but strongly recommended, especially when testing materials where oxidation is likely to change the LSP of COPCs.

11.4.8 Separate the solid from the remaining liquid in each extraction vessel by pressure or vacuum filtration through a clean 0.45- $\mu\text{m}$  pore size membrane (Sec. 6.5). The filtration apparatus may be exchanged for a clean apparatus as often as necessary until all liquid has been filtered.

11.4.9 Immediately, preserve and store the volume(s) of eluate required for chemical analysis. Preserve all analytical samples in a manner that is consistent with the determinative chemical analyses to be performed.

## 12.0 DATA ANALYSIS AND CALCULATIONS (EXCEL<sup>®</sup> TEMPLATE PROVIDED)

### 12.1 Data reporting

12.1.1 Figure 2 shows an example of a data sheet that may be used to report the concentration results of this method. This example is included in the Excel<sup>®</sup> template. At a minimum, the basic test report should include:

- a) Name of the laboratory
- b) Laboratory technical contact information
- c) Date and time at the start of the test
- d) Name or code of the solid material
- e) Particle size (85 wt% less than designated particle size)
- f) Ambient temperature during extraction ( $^{\circ}\text{C}$ )
- g) Extraction contact time (h)
- h) Eluate-specific information (see Sec. 12.1.2 below)

12.1.2 The minimum set of data that should be reported for each eluate includes:

- a) Eluate sample ID
- b) Target L/S (mL/g-dry)
- c) Mass of "as tested" solid material used (g)
- d) Moisture content of material used ( $\text{g}_{\text{H}_2\text{O}}/\text{g}$ )
- e) Volume of eluent used (mL)
- f) Measured final eluate pH
- g) Measured eluate conductivity (mS/cm)
- h) Measured ORP (mV) (optional)
- i) Concentrations of all COPCs
- j) Analytical QC qualifiers as appropriate

### 12.2 Data interpretation and presentation (optional)

### 12.2.1 LSP curve

12.2.1.1 A constituent LSP curve can be generated for each COPC after chemical analysis of all extracts by plotting the constituent concentration in the liquid phase as a function of L/S used for each extraction. The curve indicates the equilibrium concentration of the COPC as a function of L/S at the natural pH.

12.2.1.2 The lower limit of quantitation (LLOQ) for the analytical technique for each COPC may be shown as a horizontal line. COPC concentrations below this line indicate negligible or non-quantitative concentrations.

NOTE: The LLOQ is highly matrix-dependent and should be determined as part of a QA/QC plan.

12.2.1.3 Figure 3 provides example LSP curves as a function of L/S for a coal combustion fly ash and a coal combustion flue gas desulfurization filter cake.

## 13.0 METHOD PERFORMANCE

13.1 Performance data and related information are provided in SW-846 methods only as examples and guidance. The data do not represent required performance criteria for users of the methods. Instead, performance criteria should be developed on a project-specific basis, and the laboratory should establish in-house QC performance criteria for the application of this method. Performance data must not be used as absolute QC acceptance criteria for purposes of laboratory QC or accreditation.

13.2 Interlaboratory validation of this method was conducted using a coal combustion fly ash (material code EaFA), a contaminated smelter site soil (material code CFS) and a solidified waste analog (material code SWA). The median values and inner quartile ranges (IQRs) for repeatability and reproducibility were determined for eluate concentration results across all study materials and pH target values (see Table 3). More details on the interlaboratory validation may be found in Ref. 6.

13.3 Refs. 1 and 2 may provide additional guidance and insight on the use, performance and application of this method.

## 14.0 POLLUTION PREVENTION

14.1 Pollution prevention encompasses any technique that reduces or eliminates the quantity and/or toxicity of waste at the point of generation. Numerous opportunities for pollution prevention exist in laboratory operations. The EPA has established a preferred hierarchy of environmental management techniques that places pollution prevention as the management option of first choice. Whenever feasible, laboratory personnel should use pollution prevention techniques to address their waste generation. When wastes cannot be feasibly reduced at the source, the Agency recommends recycling as the next best option.

14.2 For information about pollution prevention that may be applicable to laboratories and research institutions consult *Less is Better: Laboratory Chemical Management for Waste Reduction*, a free publication available from the ACS, Committee on Chemical Safety, [http://portal.acs.org/portal/fileFetch/C/WPCP\\_012290/pdf/WPCP\\_012290.pdf](http://portal.acs.org/portal/fileFetch/C/WPCP_012290/pdf/WPCP_012290.pdf).

## 15.0 WASTE MANAGEMENT

The EPA requires that laboratory waste management practices be conducted consistent with all applicable rules and regulations. Laboratories are urged to protect the air, water, and land by minimizing and controlling all releases from hoods and bench operations, complying with the letter and spirit of any sewer discharge permits and regulations, and by complying with all solid and hazardous waste regulations, particularly the hazardous waste identification rules and land disposal restrictions. For further information on waste management, consult *The Waste Management Manual for Laboratory Personnel* available from ACS at the web address listed in Sec. 14.2.

## 16.0 REFERENCES

1. D.S. Kosson, H.A. van der Sloot, F. Sanchez and A.C. Garrabrants, "An Integrated Framework for Evaluating Leaching in Waste Management and Utilization of Secondary Materials," *Environmental Engineering Science*, 19(3) 159-204, 2002.
2. A.C. Garrabrants, D.S. Kosson, H.A. van der Sloot, F. Sanchez and O. Hjelm, "Background Information for the Leaching Environmental Assessment Framework (LEAF) Test Methods," EPA/600/R-10-170, U.S. Environmental Protection Agency, Washington, DC, December 2010.
3. F. Sanchez, R. Keeney, D.S. Kosson and R. DeLapp, "Characterization of Mercury-Enriched Coal Combustion Residues from Electric Utilities Using Enhanced Sorbents for Mercury Control," EPA-600/R-06/008, U.S. Environmental Protection Agency, Washington DC, February 2006.
4. F. Sanchez, D.S. Kosson, R. Keeney, R. DeLapp, L. Turner and P. Kariher, "Characterization of Coal Combustion Residues from Electric Utilities Using Wet Scrubbers for Multi-Pollutant Control," EPA-600/R-08/077, U.S. Environmental Protection Agency, Washington DC, July 2008.
5. D.S. Kosson, F. Sanchez, P. Kariher, L.H. Turner, R. DeLapp and P. Seignette "Characterization of Coal Combustion Residues from Electric Utilities – Leaching and Characterization Data," EPA-600/R-09/151, U.S. Environmental Protection Agency, Washington DC, December 2009.
6. A.C. Garrabrants, D.S. Kosson, L. Stefanski, R. DeLapp, P.F.A.B. Seignette, H.A. van der Sloot, P. Kariher, and M. Baldwin, "Interlaboratory Validation of the Leaching Environmental Assessment (LEAF) Method 1313 and Method 1316," DRAFT, U.S. Environmental Protection Agency, Washington DC, April 2012.

7. EN12457 Characterization of Waste – Leaching – Compliance Test for Leaching of Granular Waste Materials and Sludges, Comité Européen de Normalisation, Brussels, Belgium, 2001.

#### 17.0 TABLES, DIAGRAMS, FLOWCHARTS, AND VALIDATION DATA

The following pages contain the tables and figures referenced by this method.

TABLE 1  
EXAMPLE SCHEDULE FOR EXTRACTION SETUP

| A             | B         | C                        | D                              | E                                   | F                            | G                            |
|---------------|-----------|--------------------------|--------------------------------|-------------------------------------|------------------------------|------------------------------|
| Test Position | Target LS | Minimum Dry Mass (g-dry) | Mass of "As-Tested" Sample (g) | Moisture in "As-Tested" Sample (mL) | Volume of Reagent Water (mL) | Recommended Bottle Size (mL) |
| T01           | 10.0      | 20                       | 22.2                           | 2.2                                 | 198                          | 250                          |
| T02           | 5.0       | 40                       | 44.4                           | 4.4                                 | 196                          | 250                          |
| T03           | 2.0       | 100                      | 111.1                          | 11.1                                | 189                          | 500                          |
| T04           | 1.0       | 200                      | 222.2                          | 22.2                                | 178                          | 500                          |
| T05           | 0.5       | 400                      | 444.4                          | 44.4                                | 156                          | 1000                         |
| B03           | QC        | –                        |                                |                                     | 200                          | 250                          |
| Total         |           | –                        | 844.4                          |                                     | 1120                         |                              |

**NOTE:** 1) This schedule assumes a target liquid volume of 200 mL.  
 2) This schedule is based on "as tested" solids content of 0.90 g-dry/g.  
 3) Test position marked B01 is a method blank of reagent water.

Table data modified from Ref. 1.

TABLE 2  
EXTRACTION PARAMETERS AS FUNCTION OF MAXIMUM PARTICLE SIZE

| Particle Size (85% less than) (mm) | US Sieve Size | Minimum Dry Mass (g-dry) | Contact Time (h) | Recommended Vessel size (mL) |
|------------------------------------|---------------|--------------------------|------------------|------------------------------|
| 0.3                                | 50            | 20 ± 0.05                | 24 ± 2           | 250                          |
| 2.0                                | 10            | 40 ± 0.1                 | 48 ± 2           | 500                          |
| 5.0                                | 4             | 80 ± 0.1                 | 72 ± 2           | 1000                         |



TABLE 3

## MEDIAN METHOD PRECISION ACROSS STUDY MATERIALS

| Analyte           | Symbol | Repeatability               |                          | Reproducibility             |                           |
|-------------------|--------|-----------------------------|--------------------------|-----------------------------|---------------------------|
|                   |        | Median<br>%RSD <sub>r</sub> | IQR<br>%RSD <sub>r</sub> | Median<br>%RSD <sub>R</sub> | IQR<br>% RSD <sub>R</sub> |
| Antimony          | Sb     | 12%                         | 8-13%                    | 21%                         | 17-27%                    |
| Arsenic           | As     | 15%                         | 12-28%                   | 31%                         | 23-34%                    |
| Barium            | Ba     | 7%                          | 5-9%                     | 16%                         | 13-30%                    |
| Boron             | B      | 7%                          | 5-10%                    | 13%                         | 9-34%                     |
| Calcium           | Ca     | 6%                          | 3-7%                     | 8%                          | 7-19%                     |
| Molybdenum        | Mo     | 7%                          | 6-9%                     | 18%                         | 11-31%                    |
| Selenium          | Se     | 8%                          | 4-9%                     | 20%                         | 13-24%                    |
| Vanadium          | V      | 6%                          | 4-12%                    | 16%                         | 13-26%                    |
| Median of Medians |        | 7%                          | 3-28%                    | 17%                         | 7-34%                     |

Data taken from Ref. 6.

FIGURE 1  
METHOD FLOWCHART

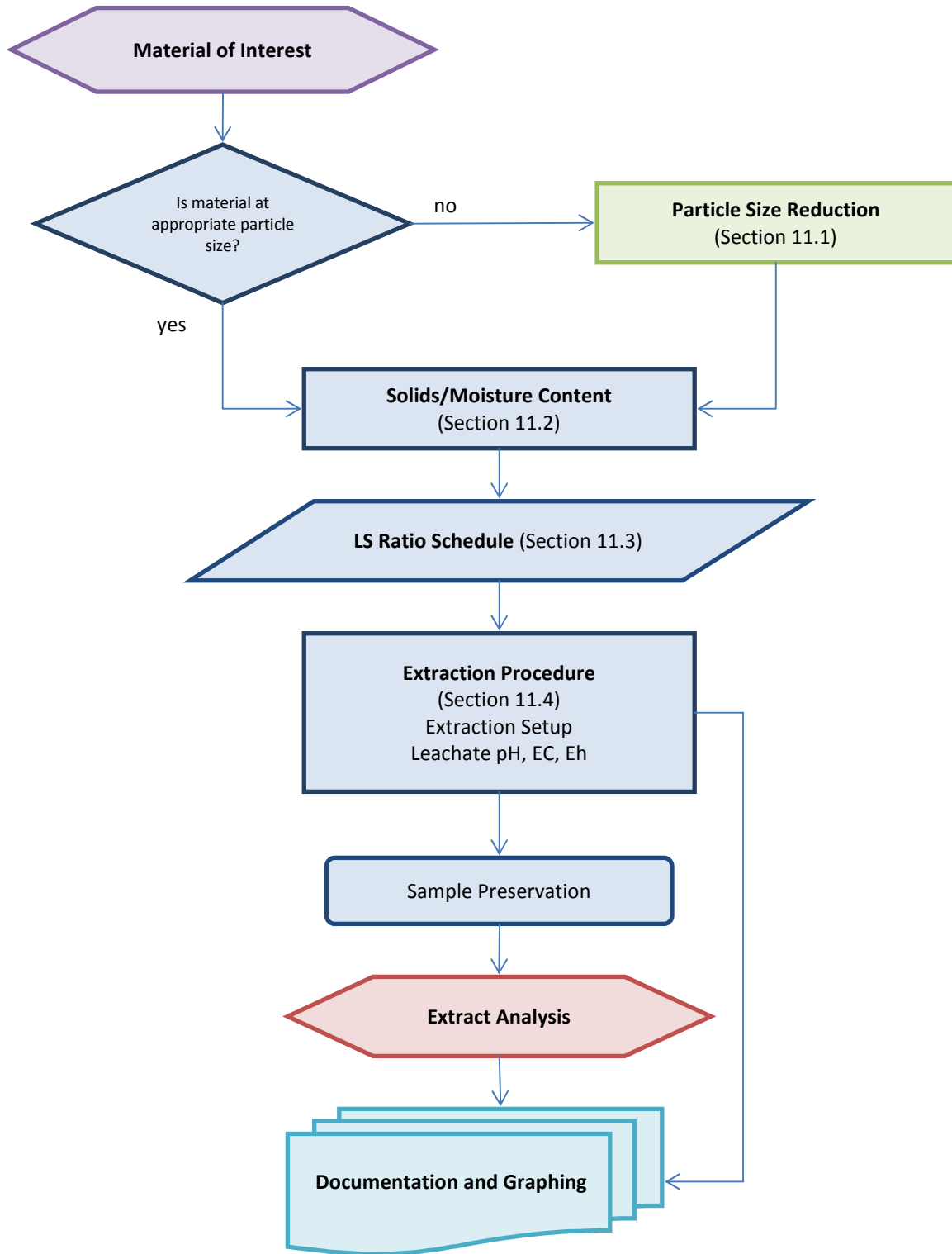


FIGURE 2

EXAMPLE DATA REPORTING FORMAT

| ABC Laboratories   |  | EPA METHOD 1316<br>Report of Analysis         |  |  |  |  |
|--|--|---|--|--|--|--|
| 123 Main Street<br>Anytown, USA<br>Contact: John Smith<br>(555) 111-1111 |  | Client Contact: Susan Jones<br>(555) 222-2222 |  |  |  |  |
| Material Code: <b>FAX</b>  |  | Particle Size: 88% passing 2-mm sieve         |  |  |  |  |
| Material Type: Coal Combustion Fly Ash                                   |  | Contact Time: 860 g                           |  |  |  |  |
| Date Received: 10/1/20xx   |  | Lab Temperature: 21 ± 2 °C                    |  |  |  |  |
| Test Start Date: 11/1/20xx   |  | Eluent Used: ASTM Type II Water               |  |  |  |  |
| Report Date: 12/1/20xx   |  |   |  |  |  |  |

| Test Position | Replicate                | Value                 | Units                          | Method         | Note                   |
|---------------|--------------------------|-----------------------|--------------------------------|----------------|------------------------|
| T01           | A                        |                       |                                |                |                        |
|               | <b>Eluate Sample ID</b>  | <b>XYZ-1316-T01-A</b> |                                |                |                        |
|               | Solid Material           | 40.0                  | g                              |                |                        |
|               | Moisture Content         | 0.01                  | g <sub>H<sub>2</sub>O</sub> /g |                |                        |
|               | Water Added              | 386.0                 | mL                             |                |                        |
|               | Target L/S               | 10.0                  | mL/g-dry                       |                |                        |
|               | Eluate pH                | 1.89                  | -                              | EPA 9040       |                        |
|               | Eluate Conductivity      | 12.6                  | mS/cm                          | EPA 9050       |                        |
|               | Eluate ORP               | 203                   | mv                             |                |                        |
|               | <b>Chemical Analysis</b> | <b>Value</b>          | <b>Units</b>                   | <b>QC Flag</b> | <b>Method</b>          |
|               | Al                       | 216.0                 | mg/L                           |                | EPA 6020               |
|               | As                       | 7.64                  | mg/L                           |                | EPA 6020               |
|               | Cl                       | < 4.13                | mg/L                           | U              | EPA 9056               |
|               |                          |                       |                                |                | <b>Date</b>            |
|               |                          |                       |                                |                | 11/7/20xx              |
|               |                          |                       |                                |                | 11/7/20xx              |
|               |                          |                       |                                |                | 11/9/20xx              |
|               |                          |                       |                                |                | <b>Dilution Factor</b> |
|               |                          |                       |                                |                | 1000                   |
|               |                          |                       |                                |                | 10                     |
|               |                          |                       |                                |                | 1                      |

| Test Position | Replicate                | Value                 | Units                          | Method         | Note                   |
|---------------|--------------------------|-----------------------|--------------------------------|----------------|------------------------|
| T02           | A                        |                       |                                |                |                        |
|               | <b>Eluate Sample ID</b>  | <b>XYZ-1316-T02-A</b> |                                |                |                        |
|               | Solid Material           | 20.0                  | g                              |                |                        |
|               | Moisture Content         | 0.01                  | g <sub>H<sub>2</sub>O</sub> /g |                |                        |
|               | Water Added              | 400.0                 | mL                             |                |                        |
|               | Target L/S               | 5.0                   | mL/g-                          |                |                        |
|               | Eluate pH                | 3.86                  | -                              | EPA 9040       |                        |
|               | Eluate Conductivity      | 0.99                  | mS/cm                          | EPA 9050       |                        |
|               | Eluate ORP               | 180                   | mv                             |                |                        |
|               | <b>Chemical Analysis</b> | <b>Value</b>          | <b>Units</b>                   | <b>QC Flag</b> | <b>Method</b>          |
|               | Al                       | 449.0                 | mg/L                           |                | EPA 6020               |
|               | As                       | 97.9                  | mg/L                           |                | EPA 6020               |
|               | Cl                       | < 4.13                | mg/L                           | U              | EPA 9056               |
|               |                          |                       |                                |                | <b>Date</b>            |
|               |                          |                       |                                |                | 11/7/20xx              |
|               |                          |                       |                                |                | 11/7/20xx              |
|               |                          |                       |                                |                | 11/7/20xx              |
|               |                          |                       |                                |                | <b>Dilution Factor</b> |
|               |                          |                       |                                |                | 1000                   |
|               |                          |                       |                                |                | 10                     |
|               |                          |                       |                                |                | 1                      |

QC Flag Key: U Value below lower limit of quantitation as reported (<"LLOQ")

FIGURE 3

EXAMPLE CONCENTRATION RESULTS FROM A COAL COMBUSTION FLY ASH AND FLUE-GAS DESULFURIZATION FILTER CAKE

