

METHOD 1314¹

LIQUID-SOLID PARTITIONING AS A FUNCTION OF LIQUID-SOLID RATIO FOR CONSTITUENTS IN SOLID MATERIALS USING AN UP-FLOW PERCOLATION COLUMN 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.

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¹ This method has been derived from the CT001 procedure (Ref. 4) and is analogous to column percolation method CEN/TS 14405 (Ref. 5) developed for the Comité Européen de Normalisation (CEN).

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, etc.) in a granular solid material as a function of liquid-to-solid ratio (L/S) under percolation conditions. The first eluates of the column test may provide insight into the composition of pore solution 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). Analyses of eluates for dissolved organic carbon and of the solid phase for total organic carbon afford evaluation of the impact of organic carbon release and the influence of dissolved organic carbon on the LSP of inorganic constituents.

1.2 This method is intended to be used as part of an 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.

1.3 This method is suitable to a wide range of granular solid materials. Example materials include industrial wastes, soils, sludges, combustion residues, sediments, construction materials, and mining wastes. This method is not suitable to monolithic materials (e.g., cement-based and stabilized materials) without particle size reduction prior to testing.

1.4 This test method is intended as a means for obtaining a series of extracts (i.e., the eluates) of a granular solid material that may be used to show eluate concentrations and/or cumulative release as a function of L/S, which can be related to a time scale when data on mean infiltration rate, density and height of application are available.

1.5 This method provides options for the preparation of analytical samples that provide flexibility based on the level of detail required. For example, when the purpose of characterization is for comparison to previous testing, compositing of eluates may be possible to create a reduced set of analytical samples. [Table 1](#) outlines the eluate fractions and collection options, based on whether concentration or cumulative release is to be reported. The collection schemes are described below.

1.5.1 Complete characterization

For complete characterization of eluate concentration and cumulative release as a function of L/S, nine discrete eluate collections and analyses are required (see [Table 1](#), Option A). No compositing of eluate fractions is performed for complete characterization, and all eluate fractions are analyzed.

Eluate concentrations from complete characterization may be used in conjunction with information regarding environmental management scenarios to estimate anticipated leaching concentrations, release rates, and extents of release for individual material constituents in the management scenarios evaluated. Eluate 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.

1.5.2 Limited analysis

Under a limited analysis approach, nine eluate collections and analysis of six analytical samples are required. If evaluation is based on eluate concentrations, six discrete eluate fractions are chemically analyzed (see [Table 1](#), Option B). If evaluation is based on cumulative release, some eluate fractions are composited by volume-weighted averaging to create a set of six analytical samples (see [Table 1](#), Option C). The concentrations of composite analytical samples cannot be interpreted along with eluate fractions on the basis of concentration.

1.5.3 Index testing

For the determination of consistency between the subject material and previously characterized materials, nine eluate collections and analysis of three analytical samples are required. If consistency is to be determined by eluate concentrations, three discrete eluate fractions are chemically analyzed (see [Table 1](#), Option D). If consistency is to be determined by cumulative release, some eluate fractions are composited by volume-weighted averaging to create a set of three analytical samples (see [Table 1](#), Option E). The concentrations of composited analytical samples cannot be interpreted along with eluate fractions on the basis of concentration.

1.6 This method is not applicable to characterize the release of volatile organic analytes.

1.7 This method provides eluate solutions considered indicative of leachate under field conditions only where the field leaching pH is controlled by the alkalinity or acidity of the solid material and the field leachate is not subject to dilution or other attenuation mechanisms. The cumulative mass of constituent released over a L/S range may be considered an estimate of the maximum mass of that constituent to be leached under field leaching over intermediate timeframes (e.g., up to 100 years) and the domain of laboratory test pH.

1.8 Prior to employing this method, analysts are advised to take reasonable measures to ensure that the granular sample is homogenized to the most practical extent. Particle size reduction may provide additional assurance of sample homogenization.

1.9 In preparation of solid materials for use in this method, particle size reduction or exclusion of samples with large grain size is used to enhance the approach towards liquid-solid equilibrium over the residence time of eluent in the column.

1.10 The structure and use of this method is similar to that of NEN 7343 (see Ref. 3) and CEN/TS 14405 (see Ref. 5).

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](#), and the determinative methods for the target analytes) 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. Guidance on defining DQOs can be obtained at <http://www.epa.gov/QUALITY/qs-docs/g4-final.pdf>.

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

Eluent is introduced into a column of moderately packed granular material in an up-flow pumping mode, with eluate collection performed as a function of the cumulative L/S. Up-flow pumping is used to minimize air entrainment and flow channeling. The default eluent for most materials is reagent water. However, a solution of 1.0 mM calcium chloride in reagent water is used when testing materials with either a high clay content (i.e., to prevent deflocculation of clay layers) or high organic matter (i.e., to moderate mobilization of dissolved organic carbon). The flow rate is maintained between 0.5-1.0 L/S per day to increase the likelihood of local equilibrium between the solid and liquid phases, due to residence times longer than one day. Eluate volumes are chemically analyzed for a combination of inorganic and non-volatile organic analytes depending on the constituents of potential concern (COPC). For the purposes of chemical speciation modeling, the entire eluent volume up to 10 mL/g dry sample (g-dry) is collected in nine specific aliquots of varying volume. A limited subset of eluent volumes within the same L/S range may be collected and analyzed for regulatory and compliance purposes. A flowchart for performing this method is shown in [Figure 1](#).

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 performed.

3.6 Dry-mass equivalent – The mass of an "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.

3.9 Refer to [Chapter One](#), [Chapter Three](#), and the manufacturer's instructions for definitions that may be relevant to this procedure.

4.0 INTERFERENCES

4.1 Solvents, reagents, glassware, and other sample processing hardware may yield artifacts and/or interferences during 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 and purification of solvents by distillation in all-glass systems 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](#) and the determinative methods to be used for information regarding potential interferences.

4.2 When the test method is applied to solid materials with a clay content greater than 10% or an organic matter content greater than 1%, a solution of 1.0 mM calcium chloride in reagent water is recommended to minimize deflocculation of clay minerals. However, the use of calcium chloride solution will interfere with the determination of actual calcium and chloride release.

4.3 When this method is applied to fine-grained granular materials, tamping during column preparation may result in flow problems due to a low-permeability sample bed. This problem can be resolved by incorporating 20 - 50% inert material (e.g., 20-30-mesh normal sand or 2-mm borosilicate glass beads) into the solid sample. Alternatively, mass release from low-permeability materials may be measured using [Method 1315](#).

5.0 SAFETY

5.1 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 safety data sheets (SDSs) should be available to all personnel involved in these analyses.

5.2 During preparation and processing of extracts and/or eluents/eluates, some waste materials may generate heat or evolve potentially harmful gases when contacted with acids and bases. Adequate prior knowledge of the material being tested should be used to establish appropriate personal protection and workspace ventilation.

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.

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

6.1 Column apparatus

This method recommends the use of a specific column apparatus (see [Figure 2](#)). Equipment with equivalent specifications may be substituted. The apparatus should have valves and quick connectors (e.g., Luer lock fittings) such that the column with end caps can be removed for packing with test material and mass measurements.

6.1.1 A 30-cm, straight cylindrical column with an inner diameter (ID) of 5 cm and constructed of inert material, resistant to high and low pH conditions and interaction with constituents of interest.

6.1.1.1 For the evaluation of inorganic COPC mobility, equipment composed of borosilicate glass (e.g., Kimble-Kontes CHROMAFLEX #420830-3020 or equivalent), polytetra-fluoroethylene (PTFE), high density polyethylene (HDPE), polypropylene (PP), or polyvinyl chloride (PVC) is recommended.

6.1.1.2 For the evaluation of non-volatile organic and mixed organic/inorganic COPCs, equipment composed of glass or Type 316 stainless steel is recommended. PTFE is not recommended for non-volatile organics, due to 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 column must be of sufficient volume to accommodate a minimum of 300 g dry material plus a 1-cm layer of silica sand (20-30 mesh) used at the bottom of the column to distribute eluent flow and at the top of the column to form a coarse filter for eluate particulates.

6.1.3 The column must have end cap materials that form a leak-proof seal and that can withstand pressures, such as encountered when pumping eluent through the column.

6.2 Eluent feed stock container – Resealable bottle or other container, constructed of inert material, capable of withstanding extreme pH conditions and interaction with any constituents of interest (see guidance in [Sec. 6.1.2](#))

6.3 Eluent feed tubing – 2-mm or similarly small ID tubing composed of chemically inert material such as PVC or equivalent

NOTE: Larger ID tubing may be required as a feed to the pump and manifold if a single eluent stock container is used to feed multiple column set-ups.

6.4 Eluate collection bottles – Capable of assembly with column apparatus using simple water locks in order to prevent the intrusion of air (see [Figure 2](#))

6.5 20-30 mesh normal washed quartz sand

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

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

6.8 Filtration membranes – Composed of hydrophilic polypropylene or equivalent material with an effective pore size of 0.45 μm (e.g., Gelman Sciences GH Polypro #66548 from Fisher Scientific or equivalent)

6.9 pH meter – Laboratory model with the capability for temperature compensation (e.g., Accumet 20, Fisher Scientific or equivalent) and a minimum resolution of 0.1 pH units

6.10 pH combination electrode – Composed of chemically resistant materials

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

6.12 Conductivity electrodes – Composed of chemically resistant materials

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 reagent is 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.

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

7.3 Calcium chloride (1.0 mM), CaCl_2 – Prepared by dissolving 0.11 g of ACS grade (or better) solid calcium chloride in 1 L of reagent water

8.0 SAMPLE COLLECTION, PRESERVATION, AND STORAGE

8.1 See [Chapter Three, "Inorganic Analytes,"](#) and [Chapter Four, "Organic Analytes,"](#) for sample collection and preservation instructions.

8.2 All 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 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 aqueous sample holding times specified in the 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, at least one eluent blank should be tested. If multiple batches of eluent are employed, one eluent blank from each batch should be analyzed.

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 Unless the "as-received" samples are part of a time-dependent (e.g., aging) study, solid materials should be processed and tested within one month of their receipt.

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 for the method procedure is presented in [Figure 1](#). Microsoft Excel® data templates are available to aid in collecting and archiving of laboratory and analytical data.²

11.1 Preparatory procedures – Particle size reduction (if required)

11.1.1 In this method, particle size reduction is used to prepare large-grained samples for the column test so that the approach toward liquid-solid equilibrium is enhanced and fluid channeling along column walls is minimized. The maximum particle size of the solid should \leq 1/20 of the column diameter. For the column recommended in this method, a maximum particle size of 2.5 mm is acceptable. Therefore, 85% of the test material should pass through a 2.38-mm (U.S. No. 8) sieve. If less than 15% of the solid material is larger than the maximum acceptable particle size, this fraction of the solid may be excluded from the material tested, rather than particle size-reduced. The mass and nature of the discarded fraction should be documented.

11.1.2 Particle size reduction of an "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 the 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 the

² These Excel® 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>.

preparation of test samples due to the potential for mineral alteration and volatility loss. 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:

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

Where: M_{sieved} = mass of sample passing the sieve (g)

M_{total} = mass of total sample (g) (e.g., M_{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 ± 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 gram.

NOTE: The oven-dried sample is not used for the extraction and should be properly disposed 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_{dry} = mass of dry material specified in the method (g-dry)

M_{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_{wet} = moisture content on a wet basis ($\text{g}_{\text{H}_2\text{O}}/\text{g}$)

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

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

11.3 Apparatus preparation

11.3.1 Prepare the column test apparatus as depicted in [Figure 2](#). Eluent feed should be directed through the lower end cap and upwards into the column to minimize air retention in the packed bed and fluid channeling along the column walls.

NOTE: When solid samples may be affected by dissolved oxygen in the feed stock, an inert gas (e.g., nitrogen or argon) may be bubbled through the feed solution to displace oxygen or used to purge the headspace above the feed solution.

NOTE: When alkaline or other air-sensitive eluates are expected, the vapor space of empty collection bottles may be purged with an inert gas (e.g., nitrogen or argon) prior to eluate collection.

11.4 Column packing

11.4.1 The column is packed with test material surrounded by layers of quartz sand at the top and bottom of the column to provide flow pattern regulation and coarse filtration.

NOTE: The following procedure describes the packing of the column from starting at the outflow (top) of the column and ending with the inflow (bottom) of the column. This is done to allow for a wider layer of quartz sand on the inflow side in cases where less than a full column of test material is available. The column is inverted prior to assembly into the leaching apparatus and initial wetting.

11.4.2 Record the mass of the empty column with end caps and any tubing leads or valves that would be needed to completely separate the column, which may include both solid material and water, from the entire apparatus.

11.4.3 Secure one end cap to the outflow side of the column and invert the column and end cap so that the outflow side of the column is facing downward.

11.4.4 Place an approximately 1-cm thick layer of quartz sand ([Sec. 6.5](#)) on the outflow side of the column using a small scoop or spoon. Record the mass of the column and sand layer. Level the sand layer by tapping the sides of the column.

11.4.5 Pack the main body of the column with a minimum 300-g dry-mass equivalent of "as-tested" sample in approximately five layers with light tamping with a glass or plastic rod to level the material between layers. When enough test material is available to produce a full column, the top of the packed sample should be approximately 1 cm from the level of the column interface with the inflow end cap (the end facing upwards). Record the mass of the column, lower sand layer, and "as-tested" sample.

11.4.6 Place a layer of sand to fill the remaining gap between the sample packing and the interface between the column and inflow end cap. When enough test material is available to pack a full column, the sand layer at the inflow end of the column should be approximately 1 cm. This gap may be larger if less test material is used. Record the total mass of the completely packed column.

11.4.7 Secure the inflow end cap. Invert the column so that the inflow end of the column is downward prior to inserting the column into the leaching apparatus.

11.4.8 Calculate the "as-tested" mass of the sample packing by subtracting the mass of the column and lower sand layer ([Sec. 11.4.4](#)) from the mass of the column, sand layer and packing ([Sec. 11.4.5](#)).

11.4.9 Calculate the dry mass equivalent packed of "as-tested" sample into the column using the solids content as follows:

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

Where: M_{dry} = dry-mass equivalent of sample in column (g-dry)

SC = solids content (g-dry/g)

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

11.5 Pump setup

11.5.1 Prior to the start of the test, set the flow rate of the pump to a value that will provide an eluate production rate of 0.75 ± 0.25 L/S per day. For example, given a dry-mass equivalent of 300 g-dry, an L/S of 0.75 mL/g-dry would translate to a volume of 225 mL/g-dry, in which case the pump should be set to a flow rate of 225 mL (0.75 L/S) per day.

11.5.2 Prime the tubing with eluent

11.5.2.1 Detach the inlet tubing from the bottom of the column and place the open end into a waste container.

11.5.2.2 Turn on the pump and allow the inlet tubing to fill with eluent. Remove any air bubbles trapped in the inlet tubing.

11.5.2.3 When the inlet tubing is full with eluent, stop the pump and reconnect the tubing to the bottom of the column.

11.6 Eluent collection schedule

11.6.1 [Table 2](#) provides a schedule of fraction end-point L/S, interval L/S, and eluate fraction volumes for collection, assuming a dry-mass equivalent of 300 g-dry. The minimum volume of each collection bottle should be scaled so as to capture the entire eluate fraction.

11.6.2 Using the assumed pump rate and the dry mass equivalent of the sample, estimate the durations of column testing required to reach the target eluate collection L/S shown in [Table 2](#) as follows:

$$T_i = \frac{M_{\text{dry}} - \sum L/S_i}{R_i}$$

Where: T_i = target time from start for collection of eluent fraction, i (day)

M_{dry} = dry-mass equivalent of sample in column (g-dry)

$\sum L/S$ = target cumulative L/S for interval i from [Table 1](#) (mL/g-dry)

R_i = pump rate assumed for interval, i (mL/day)

Alternatively, use the Microsoft Excel[®] spreadsheet template available at <http://www.vanderbilt.edu/leaching/downloads/test-methods/> to develop the schedule of target collection times.

NOTE: The schedule of predicted collection times is for reference purposes only. Typically, the eluate collection rate is slower than predicted initially, due in part to pump inefficiency, back pressure, and dead-volume lag times. The decision to switch collection bottles should be made based on the volume of eluate collected with time. The schedule may be revised with each eluate fraction collected, so that the prediction of future collections may be more accurate. Pump flow rate adjustment may be necessary.

11.6.3 After each eluate collection, calculate the time required to reach the next collection time using the equation in 11.6.2 and the pump rate calculated from the previous collection interval.

NOTE: If eluate volumes other than those calculated for the fraction volume (i.e., T01, T02, etc.) are collected (e.g., manual collection will have inherent errors in the collection volumes), the cumulative L/S may quickly become out of alignment with the tolerances shown in [Table 2](#). If this happens or appears to be likely, adjustments to the collection volumes for the two large collection fractions (i.e., T06 and T08) can be made in order to bring the cumulative L/S for subsequent collection intervals back in line with tolerances. The objective is to maintain the cumulative L/S target values for fractions T07 and T09. The Excel[®] data template available with LeachXS Lite™ can be helpful in maintaining target L/S ratios if updated after each collection interval. However, throughout the test, the user should ensure that the minimum eluate volume required for the chosen chemical analyses is collected.

11.7 Column test procedure – Column/eluent equilibration

11.7.1 Turn on the pump and allow the column to fill with eluent, thus wetting the column packing.

11.7.2 When the column packing is completely wetted and the eluent level is even with the top of the column (or just beginning to be seen through the effluent tubing at the top of the column apparatus), stop the pump and allow the column to equilibrate for 21 ± 3 hours.

11.8 Column test

11.8.1 Following equilibration, begin the column test by starting the pump and recording the date and time.

NOTE: The eluate production rate should be monitored frequently during the column test and the pump rate adjusted, such that the eluate production rate is maintained at approximately 0.75 ± 0.25 L/S per day.

11.8.2 When the eluate fraction has reached the target volume according to the predicted collection schedule, release the Luer lock connecting the active collection bottle and attach the eluent tubing to a new collection bottle.

NOTE: Alkaline eluate solutions may be susceptible to neutralization due to carbon dioxide uptake. When materials with alkaline pH are tested, precautions (e.g., purging collection bottles with inert gas) should be taken to prevent contact of the eluate with air.

11.9 Eluate processing

11.9.1 Decant a minimum volume (approximately 5 mL) of the eluate fraction from the collection vessel in order to measure the solution characteristics.

11.9.2 Measure and record the pH, specific conductivity, and oxidation-reduction potential (ORP) of the eluate (see Methods [9040](#), [9045](#) and [9050](#)).

NOTE: Measurement of pH, conductivity, and ORP should be taken within 15 minutes of eluate processing ([Sec. 11.9.1](#)) 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.

11.9.3 Separate any suspended particulates from the remaining liquid in the collection bottle by pressure or vacuum filtration through a 0.45- μm filtration membrane ([Sec. 6.8](#)).

NOTE: If either low volatility organic species or mercury is a COPC, pressure filtration is recommended over vacuum filtration in order to minimize volatility losses.

11.9.4 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.

11.10 Reiterate Secs. [11.8.2](#) - [11.9.4](#) until nine eluate fractions are collected up to an L/S of 10 ± 0.2 mL/g-dry.

NOTE: The complete method requires that all nine eluate fractions be collected from the column. However, for purposes of limiting chemical analysis or index testing where interpretation may be based on cumulative release from the column, eluate fractions may be composited by volume-weighted averaging to create a single analytical sample from multiple eluate fractions (see [Sec 11.11](#)).

11.11 Analytical sample preparation options

This method allows for options in the preparation of analytical samples based on the detail of characterization required (e.g., complete, limited or index) and the basis for data reporting (e.g., concentration or cumulative release). However, the complete set of nine eluate fractions must be collected in all cases.

11.11.1 [Table 1](#) shows the analytical preparation scheme for Options A-E described in the following sections. Each composite sample may be created either by combining the total eluate volumes and preserving the total sample for analysis; or combining aliquots of two eluate fractions using volume-weighted averages. However, it is recommended that composite analytical samples be prepared using aliquots of eluate fractions whenever possible, rather than whole eluate fractions. This approach allows for potential analysis of discrete eluate fractions, if desired, at a later date.

11.11.1.1 Option A – This sample preparation option is used for complete characterization and includes analysis of all eluate fractions. Since the entire cumulative release curve is captured in nine discrete fractions, reported data may be based on either eluate concentrations or cumulative release.

11.11.1.2 Option B – This sample preparation option is used only for limited analyses based on eluate concentration. Six discrete eluate fractions are analyzed. Data obtained using this option cannot be used for cumulative release since there are sections of the cumulative release curve not analyzed.

11.11.1.3 Option C – This sample preparation option is used only for limited analysis based on cumulative release. Six analytical samples are created from three discrete eluate fractions and three composite samples. In the scheme shown in [Table 1](#), the following fractions are composited:

- T04 and T05
- T06 and T07
- T08 and T09

11.11.1.4 Option D – This sample preparation option is used only for index testing based on eluate concentration. Three discrete eluate fractions are analyzed. Data obtained using this option cannot be used for cumulative release since there are sections of the cumulative release curve not analyzed.

11.11.1.5 Option E – This sample preparation option is used only for index testing based on cumulative release. Three analytical samples are created from one discrete eluate fraction and two composite samples. In the scheme shown in [Table 1](#), the following fractions are composited:

- T02, T03, T04, and T05
- T06, T07, T08, and T09

11.11.2 Volume-weighted composites

11.11.2.1 The volume of aliquots of eluate fractions for composite analytical samples may be calculated using the Excel template provided or the following formula:

$$V_i = \frac{F_i}{\sum_i^n F_i} \times V_{\text{sample}}$$

Where: V_i = the volume of an aliquot from eluate fraction, i (mL)
 F_i = the collected volume of eluate fraction, i (mL)
 V_{sample} = the total volume of the analytical sample (mL)
 n = total number of eluate fractions to be composited

As an illustration of volume-weighted averaging, eluate fraction aliquots are calculated as required to create an analytical sample by compositing eluate fractions T06 through T09 for index testing based on cumulative release. The calculation follows the example volumes shown in [Table 2](#) and assumes that an analytical sample volume of 100 mL is required.

$$\sum_i^n F_i = F_{T06} + F_{T07} + F_{T08} + F_{T09} = 450 \text{ mL} + 150 \text{ mL} + 1350 \text{ mL} + 150 \text{ mL} = 2100 \text{ mL}$$

$$V_{T06} = \frac{F_{T06}}{\sum_i^n F_i} \times V_{\text{sample}} = \frac{450 \text{ mL}}{2100 \text{ mL}} \times 100 \text{ mL} = 21.5 \text{ mL}$$

$$V_{T07} = \frac{F_{T07}}{\sum_i^n F_i} \times V_{\text{sample}} = \frac{150 \text{ mL}}{2100 \text{ mL}} \times 100 \text{ mL} = 7.1 \text{ mL}$$

$$V_{T08} = \frac{F_{T08}}{\sum_i^n F_i} \times V_{\text{sample}} = \frac{1350 \text{ mL}}{2100 \text{ mL}} \times 100 \text{ mL} = 64.3 \text{ mL}$$

$$V_{T09} = \frac{F_{T09}}{\sum_i^n F_i} \times V_{\text{sample}} = \frac{150 \text{ mL}}{2100 \text{ mL}} \times 100 \text{ mL} = 7.1 \text{ mL}$$

$$V_{\text{sample}} = V_{T06} + V_{T07} + V_{T08} + V_{T09} = 21.5 \text{ mL} + 7.1 \text{ mL} + 64.3 \text{ mL} + 7.1 \text{ mL} = 100.0 \text{ mL}$$

NOTE: The above illustration uses example eluate fraction volumes based on interval L/S ratios and an assumed test material mass. When calculating the aliquots of collected eluate fractions for composite samples, the actual collected fraction volumes should be used.

12.0 DATA ANALYSIS AND CALCULATIONS (Excel® template³)

12.1 Data reporting

12.1.1 [Figure 3](#) shows an example of a data sheet that may be used to report the concentration results of this method. At a minimum, the basic test report should include the following:

- 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) Packed bed dimensions (column ID and bed depth (cm))
- g) Mass of solid material in column packing
- h) Moisture content of solid material packed in column (g_{H₂O}/g)
- i) 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) Eluate collection date and time
- c) Amount of eluate collected (mass or volume)
- d) Measured eluate pH
- e) Measured eluate conductivity (mS/cm)
- f) Measured ORP (mV) (optional)
- g) Concentration of all COPCs
- h) Analytical QC qualifiers as appropriate

12.2 Data Interpretation (optional)

12.2.1 Concentration as a function of L/S

³ Excel® data templates are provided to aid in collection and archiving of laboratory and analytical data. These 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>.

12.2.1.1 A curve of the eluate concentration as a function of L/S 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 the cumulative collected L/S ratio. The curve indicates the nominal equilibrium concentration of the constituent of interest as a function of L/S from 0 to 10 mL/g-dry at natural pH. An example curve is provided in [Figure 4](#).

12.2.1.2 The lower limit of quantitation (LLOQ) of the determinative method 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.

12.3 Cumulative release as a function of L/S

12.3.1 The cumulative mass release of a COPC per unit solid material may be calculated as follows:

$$\Sigma M_i = \sum_{i=1}^9 [C_i \times (\Sigma L/S_i - \Sigma L/S_{i-1})]$$

Where: ΣM_i = the cumulative mass release through interval i (mg/kg-dry)

C_i = the concentration of the COPC in the eluent collected during interval i (mg/L)

$\Sigma L/S_i$ = the cumulative L/S of eluate collected through interval i (L/kg-dry)

$\Sigma L/S_{i-1}$ = the cumulative L/S of eluate collected through interval i-1 (L/kg-dry)

12.3.2 Prepare a curve of the cumulative mass release generated for each COPC by plotting the cumulative mass release calculated in [Sec. 12.3.1](#) as a function of the cumulative collected L/S. This curve provides an interpretation of the cumulative mass expected to be leached from a column of material as a function of L/S percolating through the column.

12.3.3 A comparison of the slope of the mass release curve to a unity slope, which is indicative of solubility-controlled release, may be made by plotting the cumulative mass release calculated in [Sec. 12.3.1](#) versus the logarithm of the cumulative collected L/S. An example is provided in [Figure 5](#).

12.4 Interpolation/extrapolation to target L/S values

The collected L/S dependence data may be interpolated or extrapolated to the nearest target L/S value for purposes of comparing different data sets (e.g., test replicates of the same or different materials). The most transparent and straightforward method is linear interpolation/extrapolation of data after \log_{10} transformation.

12.4.1 \log_{10} transformation

Collected concentration values are transformed by taking the \log_{10} of the measured concentration at each test position, i:

$$C_i = \log_{10}(c_i)$$

Where: C_i = \log_{10} -transformed concentration at test position i (\log_{10} [mL/g-dry])
 c_i = the concentration measured at test position i (mg/L)

12.4.2 Linear interpolation/extrapolation

Given a set of coordinate data $\{(\Sigma L/S_i, C_i) : i = 1, \dots, n\}$ sorted by increasing order according to $\Sigma L/S$ value (e.g., $\Sigma L/S_1 < \Sigma L/S_2 < \dots < \Sigma L/S_n$), an interpolated/extrapolated \log_{10} -transformed concentration at a known L/S target is calculated as:

$$C_T = a_T + b_T \cdot \Sigma L/S_T$$

Where: C_T = the concentration at target $\Sigma L/S$ value, $\Sigma L/S_T$ (\log_{10} [mg/L])
 a_T and b_T are coefficients of the linear interpolation/extrapolation equation
 $\Sigma L/S_T$ = a target pH value

Depending on the values of observed L/S values relative to target L/S values, the calculations of the coefficients a_T and b_T in the equation may differ according to the following algorithm:

If $\Sigma L/S_T < \Sigma L/S_1$, then $b_T = (C_2 - C_1) / (\Sigma L/S_2 - \Sigma L/S_1)$ and $a_T = C_2 - b_T \cdot \Sigma L/S_2$ (extrapolation from the two points with closest L/S values);

If $\Sigma L/S_T \geq \Sigma L/S_n$, then $b_T = (C_n - C_{n-1}) / (\Sigma L/S_n - \Sigma L/S_{n-1})$ and $a_T = C_n - b_T \cdot \Sigma L/S_n$ (extrapolation from the two points with closest L/S values);

If $\Sigma L/S_{j-1} \leq \Sigma L/S_T < \Sigma L/S_j$, then $b_T = (C_j - C_{j-1}) / (\Sigma L/S_j - \Sigma L/S_{j-1})$ and $a_T = C_j - b_T \cdot \Sigma L/S_j$ (interpolation from the two closest points surrounding $\Sigma L/S_T$).

NOTE: Interpolation or extrapolation of data should only be conducted within a distance of $\pm 20\%$ of the target L/S value. Since the allowable L/S tolerance about a target L/S value is variable (see [Table 2](#)), interpolation/extrapolation should not create data at a target L/S value where collected data is missing.

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 contaminated smelter site soil (material code CFS) and a brass foundry sand (material code JaFS). Repeatability and reproducibility were determined at an L/S of 10 mL/g-dry for eluate concentration (see [Table 3](#)) and for cumulative mass released (see [Table 4](#)). More details on the interlaboratory validation may be found in Ref. 1.

13.3 Ref. 2 and Ref. 4 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, <https://www.acs.org/content/dam/acsorg/about/governance/committees/chemicalsafety/publications/less-is-better.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 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 at: <http://www.labsafetyinstitute.org/FreeDocs/WasteMgmt.pdf>.

16.0 REFERENCES

1. A.C. Garrabrants, D.S. Kosson, R. DeLapp, P. Kariher, P.F.A.B. Seignette, H.A. van der Sloot, L. Stefanski, and M. Baldwin, "Interlaboratory Validation of the Leaching Environmental Assessment (LEAF) Method 1314 and Method 1315," EPA 600/R-12/624, *U.S. Environmental Protection Agency*, Washington, DC, 2012.
2. A.C. Garrabrants, D.S. Kosson, H.A. van der Sloot, F. Sanchez, and O. Hjelmar, "Background Information for the Leaching Environmental Assessment Framework (LEAF) Test Methods," EPA/600/R-10-170, *U.S. Environmental Protection Agency*, Washington, DC, 2010.
3. NEN 7343, "Leaching Characteristics of Solid Earth and Stony Materials - Leaching Tests - Determination of the leaching of Inorganic Constituents from Powdery and Granular Materials with the Percolation Test," *Dutch Standardization Institute*, Delft, The Netherlands, 1995.
4. 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.

5. CEN/TS 14405, "Characterization of Waste - Leaching Behaviour Tests - Up-flow Percolation Test (Under Specified Conditions)," *Comité Européen de Normalisation*, Brussels, Belgium, 2004, 2004.

17.0 TABLES, DIAGRAMS, FLOWCHARTS, AND VALIDATION DATA

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

TABLE 1

SCHEDULE OF FRACTION COLLECTIONS AND ANALYTICAL SAMPLES

| Fraction Label | Σ LS (mL/g-dry) | Option A: Characterization – Conc. | Option A: Characterization – Σ Rel | Option B: Limited Analysis – Conc. | Option C: Limited Analysis – Σ Rel | Option D: Index Testing – Conc. | Option E: Index Testing – Σ Rel |
|----------------|------------------------|------------------------------------|---|------------------------------------|---|---------------------------------|--|
| T01 | 0.2 ± 0.1 | ✓ | ✓ | ✓ | ✓ | ✓ | ✓ |
| T02 | 0.5 ± 0.1 | ✓ | ✓ | ✓ | ✓ | X | ↓ |
| T03 | 1.0 ± 0.1 | ✓ | ✓ | ✓ | ✓ | X | ↓ |
| T04 | 1.5 ± 0.2 | ✓ | ✓ | X | ↓ | X | ↓ |
| T05 | 2.0 ± 0.2 | ✓ | ✓ | ✓ | T05c | ✓ | ✓T05c |
| T06 | 4.5 ± 0.2 | ✓ | ✓ | X | ↓ | X | ↓ |
| T07 | 5.0 ± 0.2 | ✓ | ✓ | ✓ | ✓T07c | X | ↓ |
| T08 | 9.5 ± 0.2 | ✓ | ✓ | X | ↓ | X | ↓ |
| T09 | 10.0 ± 0.2 | ✓ | ✓ | ✓ | ✓T09c | ✓ | ✓T09c |

NOTE: Σ Rel = Cumulative release

✓ = Collect eluate fraction (or pool of fractions) as analytical sample

↓ = Composite eluate fraction with next fraction to create analytical sample

X = No fraction to collect

TABLE 2

SCHEDULE OF ELUATE FRACTIONS FOR COLLECTION WITH EXAMPLE VOLUMES

| Interval Label | End Point Σ L/S (mL/g-dry) | Fraction L/S (mL/g-dry) | Example Fraction Volume (mL) |
|----------------|-----------------------------------|-------------------------|------------------------------|
| T01 | 0.2 \pm 0.1 | 0.2 | 60 |
| T02 | 0.5 \pm 0.1 | 0.3 | 90 |
| T03 | 1.0 \pm 0.1 | 0.5 | 150 |
| T04 | 1.5 \pm 0.2 | 0.5 | 150 |
| T05 | 2.0 \pm 0.2 | 0.5 | 150 |
| T06 | 4.5 \pm 0.3 | 2.5 | 750 |
| T07 | 5.0 \pm 0.2 | 0.5 | 150 |
| T08 | 9.5 \pm 0.3 | 4.5 | 1350 |
| T09 | 10.0 \pm 0.2 | 0.5 | 150 |
| B01 | Eluent | N/A | 100 |

NOTE: Example fraction volumes based on assumed packing mass of 300 g-dry

TABLE 3

METHOD PRECISION FOR ELUATE CONCENTRATION AT L/S=10 mL/g-dry

| Analyte | Symbol | Repeatability – CFS %RSD _r | Repeatability – JaFS %RSD _r | Reproducibility – CFS %RSD _R | Reproducibility – JaFS %RSD _R |
|------------|--------|--|---|--|---|
| Aluminum | Al | 8.6 | 12.7 | 16.6 | 28.2 |
| Barium | Ba | 6.9 | 13.9 | 23.0 | 24.2 |
| Boron | B | 11.9 | 14.4 | 14.3 | 23.9 |
| Cadmium | Cd | 9.2 | - | 14.6 | - |
| Calcium | Ca | 6.9 | 5.6 | 12.7 | 52.5 |
| Copper | Cu | 8.1 | 13.3 | 15.2 | 31.8 |
| Magnesium | Mg | - | 7.7 | - | 53.3 |
| Manganese | Mn | - | 22.0 | - | 26.6 |
| Molybdenum | Mo | 25.2 | 27.5 | 28.4 | 54.4 |
| Lead | Pb | 7.3 | 12.4 | 14.8 | 23.5 |
| Thallium | Tl | 9.6 | - | 19.2 | - |
| Zinc | Zn | 8.6 | 23.0 | 16.0 | 58.5 |

| Material | Repeatability – CFS %RSD _r | Repeatability – JaFS %RSD _r | Repeatability – Overall | Reproducibility – CFS %RSD _R | Reproducibility – JaFS %RSD _R | Reproducibility – Overall |
|----------|--|---|----------------------------|--|---|------------------------------|
| Mean | 10% | 15% | 13% | 18% | 38% | 28% |

Data taken from Ref. 1.

TABLE 4

METHOD PRECISION FOR CUMULATIVE RELEASE AT L/S=10 mL/g-dry

| Analyte | Symbol | Repeatability – CFS %RSD _r | Repeatability – JaFS %RSD _r | Reproducibility – CFS %RSD _R | Reproducibility – JaFS %RSD _R |
|------------|--------|--|---|--|---|
| Aluminum | Al | 4.6 | 7.2 | 14.3 | 13.1 |
| Barium | Ba | 3.4 | 6.2 | 23.0 | 10.9 |
| Boron | B | 2.9 | 4.4 | 11.1 | 6.3 |
| Cadmium | Cd | 2.2 | - | 8.5 | - |
| Calcium | Ca | 2.1 | 3.6 | 8.2 | 19.9 |
| Copper | Cu | 3.9 | 4.6 | 15.3 | 21.4 |
| Magnesium | Mg | - | 4.2 | - | 17.2 |
| Manganese | Mn | - | 7.0 | - | 14.9 |
| Molybdenum | Mo | 4.4 | 4.5 | 12.3 | 11.1 |
| Lead | Pb | 1.8 | 9.5 | 10.8 | 15.1 |
| Thallium | Tl | 2.9 | - | 10.2 | - |
| Zinc | Zn | 4.0 | 7.3 | 11.3 | 18.6 |

| Material | Repeatability – CFS %RSD _r | Repeatability – JaFS %RSD _r | Repeatability – Overall | Reproducibility – CFS %RSD _R | Reproducibility – JaFS %RSD _R | Reproducibility – Overall |
|----------|--|---|----------------------------|--|---|------------------------------|
| Mean | 3% | 6% | 5% | 13% | 15% | 14% |

Data taken from Ref. 1.

FIGURE 1
METHOD FLOWCHART

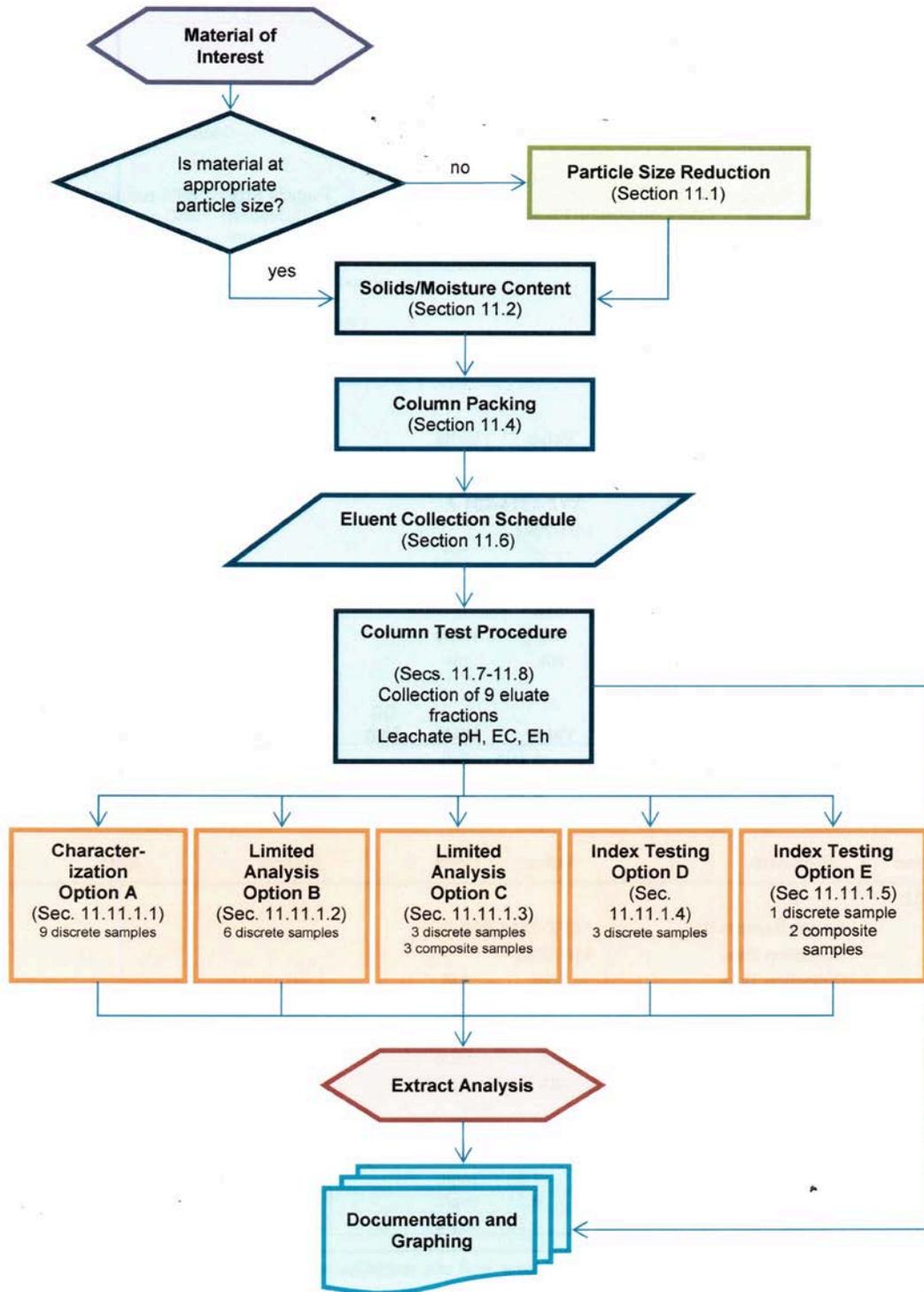
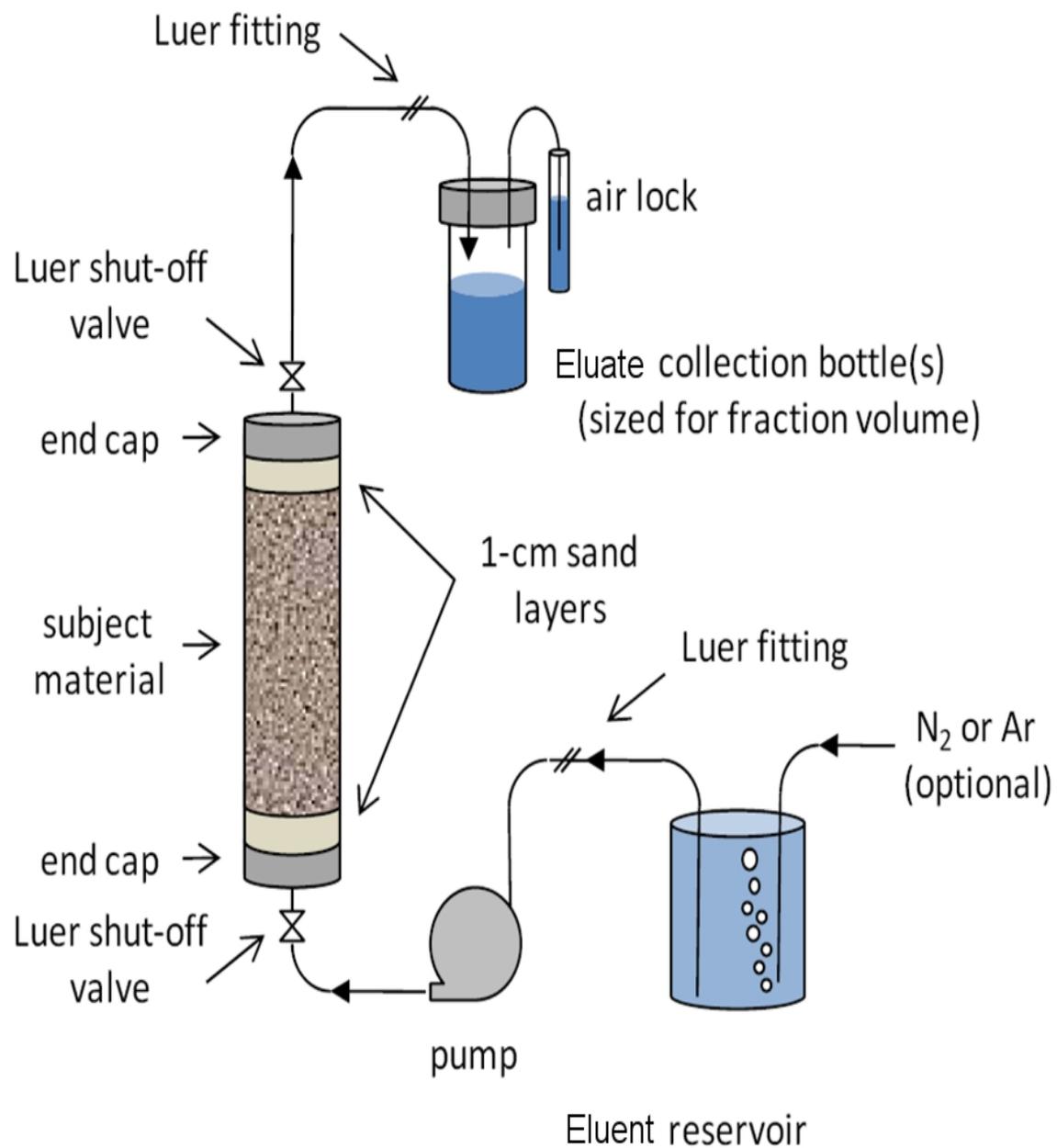


FIGURE 2

SCHEMATIC OF COLUMN TEST APPARATUS



NOTE: Figure not drawn to scale

FIGURE 3

EXAMPLE DATA REPORT FORMAT

| | | | |
|---|--|--|---|
| ABC Laboratories 123 Main Street Anytown, USA Contact: John Smith (555) 111-1111 | | EPA METHOD 1314 Report of Analysis | Client Contact: Susan Jones (555) 222-2222 |
| Material Code: XYZ | Material Type: Coal Combustion Fly Ash | Particle Size: 88% passing 2-mm sieve | Mass used in Column: 360 g |
| Date Received: 10/1/20xx | Test Date: 11/1/20xx | Moisture Content: 0.002 g _{H₂O} /g | Column ID: 4.8 cm |
| Report Date: 12/1/20xx | | Packing Bed Depth: 28 cm | Eluent: ASTM Type II Water |
| | | Lab Temperature: 21 ± 2 °C | |

| Test Position | Replicate | Value | Units | Method | Note | | |
|---------------|--------------------------|----------------|--------------|----------------|------------------------|-----------|------|
| T01 | A | | | | | | |
| | Eluate Sample ID | XYZ-1314-T01-A | | | | | |
| | Collection Date | 11/1/20xx | | | | | |
| | Collection Time | 12:35 | PM | | | | |
| | Eluate Mass | 70.4 | g | | | | |
| | Eluate pH | 8.82 | - | EPA 9040 | | | |
| | Eluate Conductivity | 5.4 | mS/c | EPA 9050 | | | |
| | Eluate ORP | NA | mv | | | | |
| | Chemical Analysis | Value | Units | QC Flag | Method | | |
| | | | | | Date | | |
| | | | | | Dilution Factor | | |
| | Al | 4.72 | mg/L | | EPA 6020 | 11/7/20xx | 1000 |
| | As | 0.12 | mg/L | | EPA 6020 | 11/7/20xx | 10 |
| | Cl | 5.42 | mg/L | | EPA 9056 | 11/9/20xx | 1 |

| Test Position | Replicate | Value | Units | Method | Note | | |
|---------------|--------------------------|----------------|--------------|----------------|------------------------|-----------|------|
| T02 | A | | | | | | |
| | Eluate Sample ID | XYZ-1314-T02-A | | | | | |
| | Collection Date | 11/1/20xx | | | | | |
| | Collection Time | 9:15 | AM | | | | |
| | Eluate Mass | 105.1 | g | | | | |
| | Eluate pH | 9.15 | - | | | | |
| | Eluate Conductivity | 2.3 | mS/c | | | | |
| | Eluate ORP | NA | mv | | | | |
| | Chemical Analysis | Value | Units | QC Flag | Method | | |
| | | | | | Date | | |
| | | | | | Dilution Factor | | |
| | Al | 2.99 | mg/L | | EPA 6020 | 11/7/20xx | 1000 |
| | As | 0.21 | mg/L | | EPA 6020 | 11/7/20xx | 10 |
| | Cl | 4.20 | mg/L | U | EPA 9056 | 11/7/20xx | 1 |

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

FIGURE 4

EXAMPLE ELUATE CONCENTRATION CURVES FOR COMPLETE CHARACTERIZATION OF A COAL COMBUSTION FLY ASH

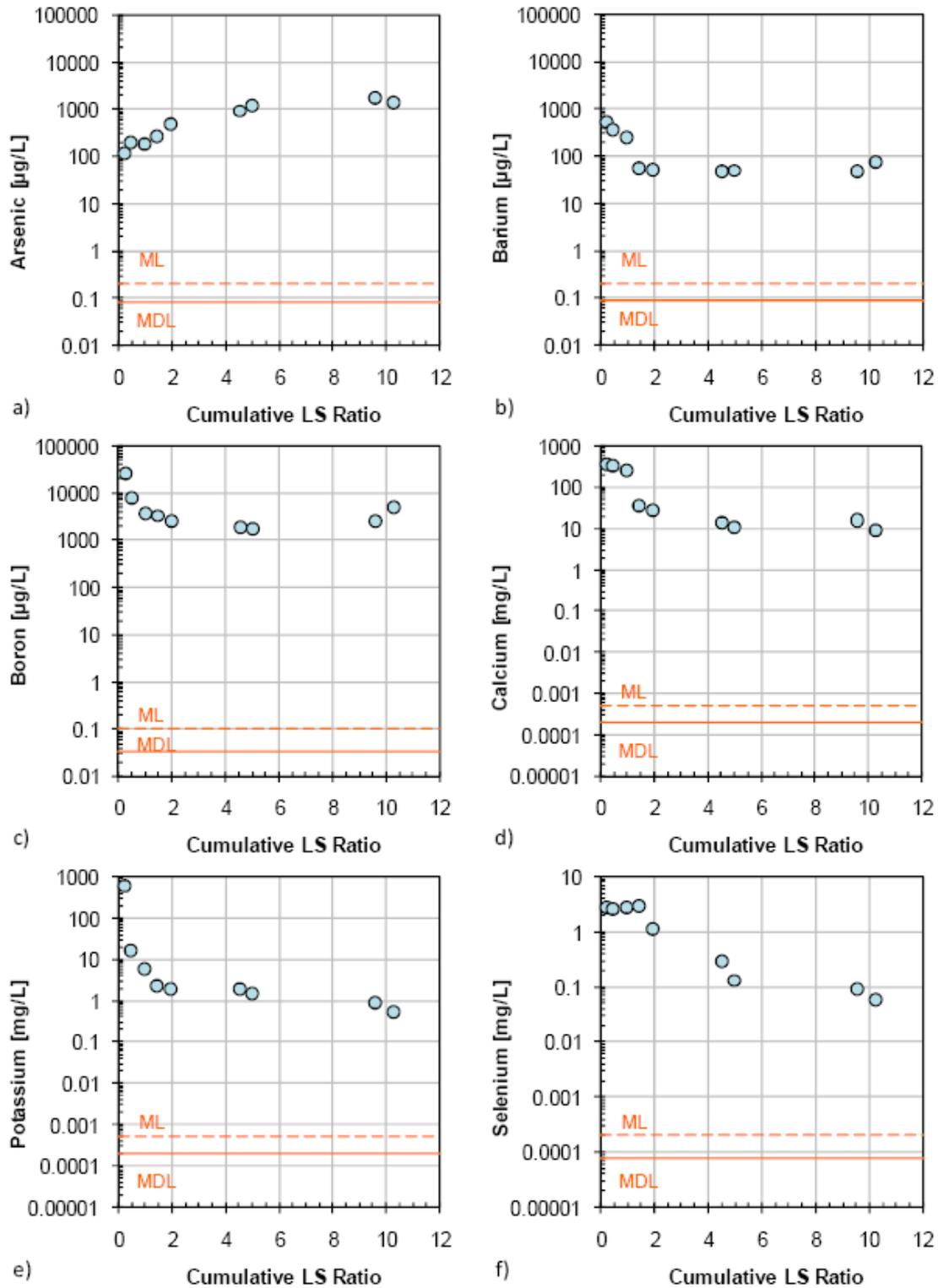
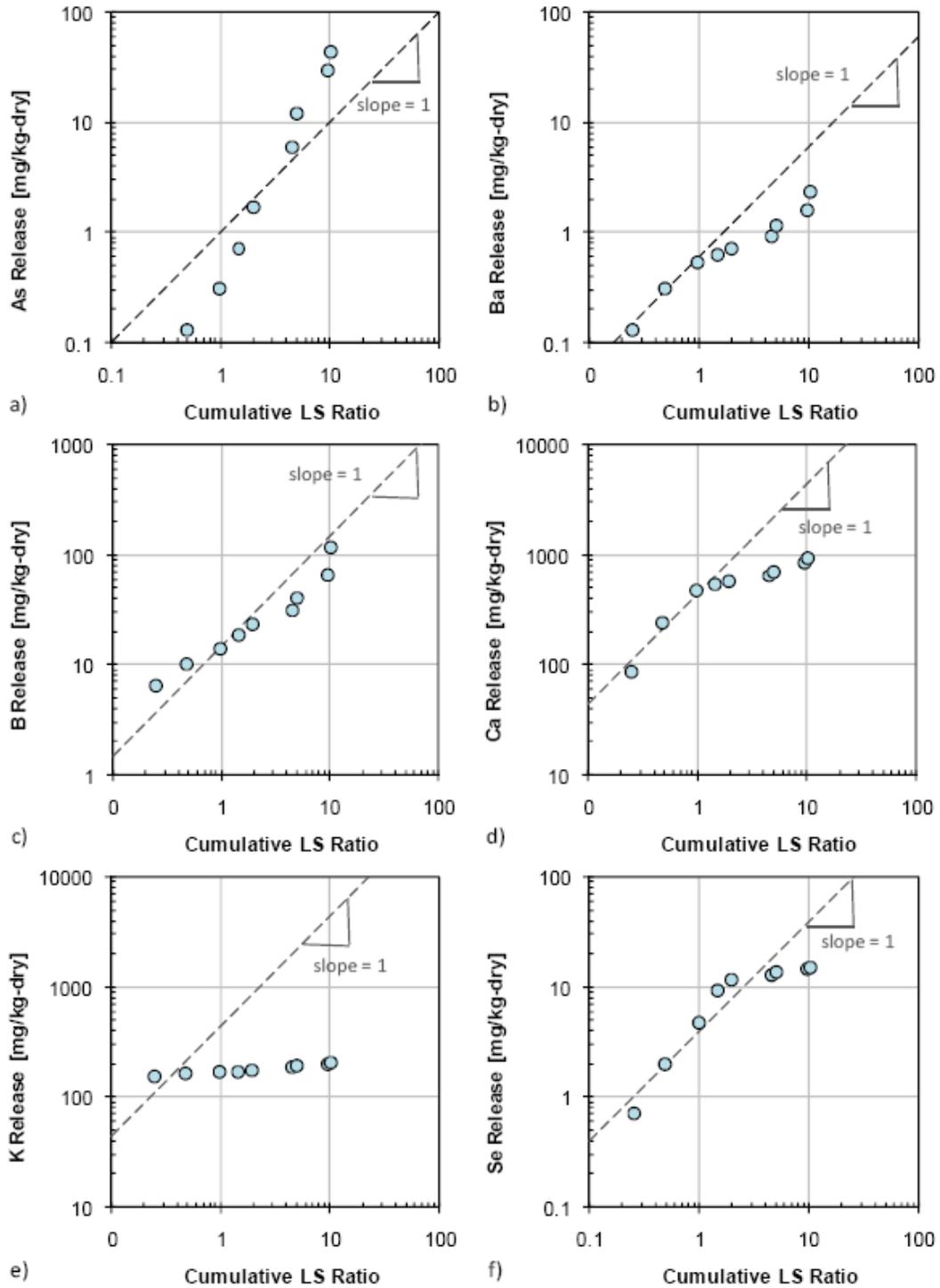


FIGURE 5

EXAMPLE CUMULATIVE RELEASE CURVES FOR COMPLETE CHARACTERIZATION OF A COAL COMBUSTION FLY ASH



NOTE: Dashed line represents solubility control (slope = 1).