

## METHOD 1315<sup>1</sup>

### MASS TRANSFER RATES OF CONSTITUENTS IN MONOLITHIC OR COMPACTED GRANULAR MATERIALS USING A SEMI-DYNAMIC TANK LEACHING 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.

#### Table of Contents

1.0	SCOPE AND APPLICATION.....	2
2.0	SUMMARY OF METHOD .....	3
3.0	DEFINITIONS .....	4
4.0	INTERFERENCES.....	5
5.0	SAFETY.....	5
6.0	EQUIPMENT AND SUPPLIES.....	5
7.0	REAGENTS AND STANDARDS.....	8
8.0	SAMPLE COLLECTION, PRESERVATION, AND STORAGE.....	8
9.0	QUALITY CONTROL .....	8
10.0	CALIBRATION AND STANDARDIZATION .....	9
11.0	PROCEDURE .....	9
12.0	DATA ANALYSIS AND CALCULATIONS.....	15
13.0	METHOD PERFORMANCE .....	20
14.0	POLLUTION PREVENTION.....	20
15.0	WASTE MANAGEMENT .....	20
16.0	REFERENCES.....	21
17.0	TABLES, DIAGRAMS, FLOWCHARTS, AND VALIDATION DATA.....	22

---

<sup>1</sup> This method has been derived from the MT001 and MT002 procedures (Ref. 12). The method is analogous to the monolithic mass transfer methods NEN 7345 (Ref. 9) developed under Dutch regulation and CEN/TS 15863 (Ref. 13) developed for the Comité Européen de Normalisation (CEN).

## 1.0 SCOPE AND APPLICATION

1.1 This method is designed to provide the mass transfer rates (release rates) of inorganic analytes contained in a monolithic or compacted granular material, under diffusion-controlled release conditions, as a function of leaching time. Observed diffusivity and tortuosity may be estimated through analysis of the resulting leaching test data.

1.2 This method is suitable to a wide range of solid materials which may be in monolithic form (e.g., cements, solidified wastes) or may be compacted granular materials (e.g., soils, sediments and stacked granular wastes) which behave as a monolith, in that the predominant water flow is around the material and release is controlled by diffusion to the boundary. 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 leaching characterization method provides intrinsic material parameters for release of inorganic species under mass transfer controlled leaching conditions. This test method is intended as a means for obtaining a series of eluents which may be used to estimate the diffusivity of constituents and physical retention parameters of the solid material under specified laboratory conditions.

1.4 This method is not applicable to characterize the release of organic analytes with the exception of general dissolved organic carbon.

1.5 This method is a characterization method and does not provide a solution considered to be representative of eluate under field conditions. This method is similar in structure and use to predecessor methods such as MT001.1 (see Ref. 12), NEN 7345 (see Ref. 9), ANSI/ANS 16.1 (see Ref. 15), and ASTM C1308 (see Ref. 11). However, this method differs from previous methods in that: 1) leaching intervals are modified to improve QC; 2) sample preparation accounts for mass transfer from compacted granular samples; and, 3) mass transfer may be interpreted by more complex release models that account for physical retention of the porous medium and chemical retention at the pore wall through geochemical speciation modeling.

1.6 The geometry of monolithic samples may be rectangular (e.g., bricks or tiles), cubes, wafers or cylinders. Samples may also have a variety of faces exposed to eluent, forming anything from 1-dimensional (1-D) through 3-dimensional (3-D) mass transfer cases. In all cases, a minimum sample size of 5 cm in the direction of mass transfer must be employed and the liquid-surface-area ratio (L/A) must be maintained at  $9 \pm 1$  mL/cm<sup>2</sup>.

Monolithic samples should be suspended or held in the leaching fluid such that at least 98% of the entire sample surface area is exposed to eluent and the bulk of the eluent (e.g., a minimum of 2 cm between any exposed surface and the vessel wall) is in contact with the exposed sample surface. [Figure 1](#) provides examples of appropriate sample holders and leaching configurations for 3-D and 1-D cases.

1.7 Compacted granular materials are granular solids, screened to pass through a 2-mm sieve, compacted following a modified Proctor compaction effort (see Ref. 10). The sample geometry

must be open-faced cylinders due to limitations of mechanical packing. However, the diameter and height of the sample holder may be altered to correspond appropriately with the diameter and volume of the leaching vessel. In all cases, the sample size of at least 5 cm in the direction of mass transfer must be employed and the L/A must be maintained at  $9 \pm 1$  mL/cm<sup>2</sup>.

The sample should be positioned at the bottom of the leaching vessel with a minimum of 5 cm of distance between the solid-liquid interface and the top of the vessel. The distance between the non-leaching faces (i.e., outside of the mold surfaces) and the leaching vessel wall should be minimized to < 0.5 cm, such that the majority of the eluent volume is on top of the sample. [Figure 2](#) shows an example of a holder and leaching configuration for a compacted granular sample.

1.8 The solvent system used in this characterization method is reagent water. Other systems (e.g., groundwater, seawater, and simulated liquids) may be used to infer material performance under specific environmental conditions. However, interaction between the eluent and the solid matrix may result in precipitation and pore blocking, which may interfere with characterization or complicate data interpretation.

1.9 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 for the intended application.

1.10 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 comprises leaching of continuously water-saturated monolithic or compacted granular material in an eluent-filled tank with periodic renewal of the leaching solution. The vessel and sample dimensions are chosen such that the sample is fully immersed in the leaching solution. Monolithic samples may be cylinders or parallelepipeds, while granular materials are compacted into cylindrical molds at optimum moisture content using modified Proctor compaction methods (see Ref. 10). In either case, the exposure of a regular geometric area to the eluent is recommended. Samples are contacted with reagent water at a specified L/A. The leaching solution is exchanged with fresh reagent water at nine pre-determined intervals (see NOTE below). The sample is freely drained and the mass is recorded to monitor the amount of eluent absorbed into the solid matrix at the end of each leaching interval. The eluate pH and specific conductance is measured for each time interval and

analytical samples are collected and preserved accordingly based on the determinative methods to be performed. Eluate concentrations are plotted as a function of time, as a mean interval flux, and as a cumulative release as a function of time. These data are used to estimate mass transfer parameters (i.e., observed diffusivity) for each constituent of potential concern (COPC). A flowchart for performing this method is shown in [Figure 3](#).

**NOTE:** The leaching schedule may be extended for additional exchanges with individual intervals of 14 days to provide more information about longer-term release.

### 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-to-surface area ratio (L/A) – The ratio representing the total liquid volume used in the leaching interval to the external geometric surface area of the solid material. L/A is typically expressed in units of mL of eluent/cm<sup>2</sup> of exposed surface area.

3.4 Observed mass diffusivity – The apparent, macroscopic rate of release due to mass transfer from a solid into a liquid as measured using a leaching test under conditions where mass transfer controls release. The observed diffusivity accounts for all physical and chemical retention factors influencing mass transfer and is typically expressed in units of cm<sup>2</sup>/s.

3.5 Effective mass diffusivity – The intrinsic rate of mass transfer in a porous medium accounting for physical retention. The effective mass diffusivity is typically expressed in units of cm<sup>2</sup>/s.

3.6 Physical retention factor – A mass transfer rate term that describes the retardation of diffusion due to intrinsic physical properties of a porous medium (e.g., effective porosity, tortuosity).

3.7 Chemical retention factor – A mass transfer rate term that describes the chemical processes (e.g., dissolution/precipitation, adsorption/desorption, complexation) occurring at the pore water interface with the solid mineral phases within the porous structure of the solid material.

3.8 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.

3.9 Eluate – The solution collected as an extract from a leaching test that contains the eluent plus constituents leached from the solid phase.

3.10 Refer to [Chapter One](#) and [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 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 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 The reaction of atmospheric gases can influence the measured concentrations of constituents in eluates. For example, reaction of carbon dioxide with eluents from highly alkaline or strongly reducing materials will result in neutralization of eluate pH and precipitation of carbonates. Leaching vessels, especially those used when testing highly alkaline materials, should be designed to be airtight in order to minimize the reaction of samples with atmospheric gases.

4.3 Use of certain solvent systems may lead to precipitation at the material surface boundary, which may reduce mass transport rates. For example, exposure of cement-based materials to seawater leads to sealing of the porous block (see Ref. 8).

## 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 Sample holder

## 6.1.1 Monolithic samples

6.1.1.1 A mesh or structured holder constructed of an inert material such as high density polyethylene (HDPE) or other material resistant to high and low pH is recommended.

6.1.1.2 The holder should be designed such that at least 98% of the external surface area of the sample may be exposed to eluent.

6.1.1.3 The holder should be designed to match the geometry of the mass transfer such that the bulk of the eluent may be in contact with the sample and the exposed surfaces of the sample centered within the leaching fluid.

**NOTE:** In the case of 1-D mass transfer from the axial face of a cylindrical sample, the outer diameter (OD) of the holder should be matched as closely as possible to the inner diameter (ID) of the leaching vessel so that the majority of the eluent is above the sample (e.g., in contact with the exposed material surface), while allowing for easy placement and removal of the holder in the leaching vessel (see [Figure 1](#)).

## 6.1.2 Compacted granular samples

6.1.2.1 A cylindrical mold constructed of an inert material such as HDPE or other material resistant to high and low pH is recommended.

6.1.2.2 The holder should be capable of withstanding the compaction force required to prepare the sample (see [Sec. 11.3](#)) without breaking or distorting.

**NOTE:** The outer diameter of the holder for a compacted granular sample should be matched as closely as possible to the inner diameter of the leaching vessel so that the majority of the eluent is above the sample (e.g., in contact with the exposed material surface) while allowing for easy placement and removal of the holder in the leaching vessel.

## 6.2 Leaching vessel

6.2.1 A straight-sided container constructed of a material resistant to high and low pH is recommended. Jars or buckets composed of HDPE, polycarbonate (PC), polypropylene (PP), or polyvinyl chloride (PVC) are recommended when evaluating the mobility of inorganic species.

6.2.2 The leaching vessel should have an airtight seal that can sustain long periods of standing without gas exchange with the atmosphere.

6.2.3 The container must be of sufficient volume to accommodate both the solid sample and an eluent volume based on an L/A of  $9 \pm 1$  mL /cm<sup>2</sup> sample surface area. Ideally, the vessel should be sized such that the headspace is minimized within the tolerance of the L/A.

## 6.3 Leaching setup

Example photos of three possible leaching equipment arrangements for monolithic and compacted granular samples are shown in Figures [1](#) and [2](#), respectively. The equipment used in the each of these cases is described below.

6.3.1 [Figure 1a](#) shows a monolithic sample 3-D configuration with the following accessories:

- Sample holder – PP sink washers, 43-mm OD, 37-mm ID, 6-mm high, with four holes drilled at the quadrants to accept 2-mm OD nylon string knotted at the top
- Sample stand – PVC pipe, 47-mm OD, 51-mm high, cut to have four legs approximately 8-mm wide and 30-mm high
- Leaching Vessel – PP bucket, 140-mm ID at top, 120-mm ID at bottom, 200-mm high (Berry Plastics #T51386CP3, VWR Scientific, or equivalent)

6.3.2 [Figure 1b](#) shows a monolithic sample 1-D configuration with the following accessories:

- Sample holder – Polyethylene (PE) mold, 54-mm OD, 100-mm high (MA Industries, Peach Tree City, GA, or equivalent), with the test sample cured in mold and cut to 51-mm high
- Leaching vessel – 250-mL PC jar, 60-mm ID, 100-mm high (Nalgene #2116-0250, Fisher Scientific, or equivalent)

6.3.3 [Figure 2](#) shows a compacted granular sample 1-D Configuration with the following accessories:

- Sample holder – PE mold, 100-mm OD, 200-mm high, (MA Industries, Peach Tree City, GA, or equivalent) cut to 63-mm high with three tabs drilled for 0.7-mm fishing line knotted at the top
- Leaching vessel – 1000-mL PC jar, 110-mm ID at top, 130-mm high (Nalgene #2116-1000, Fisher Scientific, or equivalent)
- Glass beads, borosilicate – 2-mm diameter

6.4 Filtration apparatus – Pressure or vacuum filtration apparatus composed of appropriate materials to maximize the collection of extracts and minimize the loss of COPCs (Nalgene #300-4000 or equivalent)

6.5 Filtration membranes – Composed of hydrophilic polypropylene or equivalent material with an effective pore size of 0.45  $\mu\text{m}$  (e.g., Andwin Scientific GH Polypro 28143-288 or equivalent)

6.6 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

6.7 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 Proctor compactor (for compacted granular samples only) – Equipped with a slide hammer capable of dropping a 4.5-kg weight over a 0.46-m interval (see Ref. 10 for further details)

## 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 its 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 Other reagents may be used in place of reagent water on a case-specific basis.

## 8.0 SAMPLE COLLECTION, PRESERVATION, AND STORAGE

8.1 See [Chapter Three, "Inorganic Analytes,"](#) and [Chapter 4, "Organic Analytes,"](#) for sample collection and preservation instructions.

8.2 Both plastic and glass containers are suitable for the collection of samples. All sample containers must be prewashed with a metal-free detergent and triple-rinsed with nitric acid and reagent water, depending on the history of the container. For further information, see [Chapter Three](#).

## 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, method blanks should be tested for each leaching interval. Refer to [Chapter One](#) for specific QC procedures.

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 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 this method is presented in [Figure 3](#). Microsoft Excel® data templates are available to aid in collecting and archiving of laboratory and analytical data.<sup>2</sup>

#### 11.1 Preparatory Procedures – Determination of solids and moisture content

The moisture and solids content of the sample material are used to relate leaching results to dry-material masses. When preparing compacted granular samples for testing, the moisture content or solid content is used to determine the optimum moisture content following the modified Proctor test. This method calculates moisture content 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.

---

<sup>2</sup> 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>.

11.1.1 Place 5 - 10 g of solid sample 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.

11.1.2 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)

11.1.3 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 ( $\text{g}_{\text{H}_2\text{O}}/\text{g}$ )

$M_{\text{dry}}$  = 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 Preparation of monolithic samples

11.2.1 If the material to be tested is granular, disregard this section and proceed to [Sec. 11.3](#).

11.2.2 A representative sample of monolithic material should be obtained by molding material components in place (e.g., cementitious media) or by coring or cutting a sample from a larger existing specimen.

11.2.3 The geometry of monolithic samples may be rectangular (e.g., bricks or tiles), cubes, wafers, or cylinders. Samples may also have a variety of faces exposed to eluent forming 1-, 2-, or 3-D mass transfer cases. Examples of monolithic sample leaching setups are shown in [Figure 1](#).

11.2.4 A minimum sample size of 5 cm in the direction of mass transfer must be employed and the L/A must be maintained at  $9 \pm 1$  mL/cm<sup>2</sup>.

**NOTE:** Since the sample holder and leaching vessel must correspond to the specifications in [Sec. 6.1](#), it is often easier to modify the sample size and geometry rather than the holder and vessel dimensions.

11.2.5 Proceed to [Sec. 11.4](#).

## 11.3 Preparation of compacted granular samples

Compacted granular materials, in most cases, must be open-faced cylinders due to the limitations of mechanical packing. However, the diameter and height of the sample holder may be altered to work appropriately with the diameter and volume of the leaching vessel. In all cases, a minimum sample size of 5 cm in the direction of mass transfer must be employed and the L/A must be maintained at  $9 \pm 1 \text{ mL/cm}^2$ .

Granular samples are compacted into the sample holder using a variation on the modified Proctor compaction (see Ref. 10) to include the use of 6-cm high-test molds. Shorter or taller molds (or packing depths) may be used as long as the compaction effort of  $56,000 \text{ ft}\cdot\text{lb}_f/\text{ft}^3$  is achievable. The number of packing layers should be maintained at the five layers specified in Ref. 10. However, the number of blows per layer in a 4-in diameter mold may be changed according to the follow formula:

$$\frac{56,000 \text{ ft}\cdot\text{lb}_f}{\text{ft}^3} \Bigg| \frac{\text{blow}}{1.5 \text{ ft} \times 10 \text{ lb}_f} \Bigg| \frac{\pi \left( \frac{0.3}{2} \text{ ft} \right)^2 \times h \text{ ft}}{5 \text{ layer}} = \frac{65.2 \times h \text{ blow}}{\text{layer}}$$

Where:  $h$  is the measured height of the sample mold (ft).

Thus, for the mold height of 4.584 in (0.382 ft) specified in the ASTM procedure, 25 blows per each of 5 layers are required. When a 6-cm (0.196 ft) mold height is used (as suggested in this method), 13 blows per each of 5 layers are required to obtain the same compaction effort.

The granular sample should be compacted at a moisture content corresponding to 90% of the modified Proctor optimum packing density in order to provide a uniform approach to obtaining a sample density that approximates field conditions. Optimum moisture content refers to the amount of moisture or fractional mass of water ( $\text{g}_{\text{H}_2\text{O}}/\text{g}$  material) in the granular sample that is present at the optimum packing density ( $\text{g-dry material}/\text{cm}^3$ ). Optimum packing density is defined in Ref. 10. The optimum moisture content of the test material is determined from a pre-test that measures the packing density of granular materials compacted at different levels of moisture content.

### 11.3.1 Pre-test to determine optimum moisture content

The pre-test is conducted as a series of five batch-wise packing trials with consecutive increases in moisture content until the maximum packing density has been surpassed. The optimum moisture content is determined as the maximum of a third-order polynomial fit through the graph of dry-packing density as a function of moisture content (wet basis).

11.3.1.1 Place 1500 g of "as received" material into a pail or bowl and mix well by hand to homogenize. As an alternative to hand mixing, a mechanical paddle mixer may be used.

**NOTE:** The pre-test may be conducted from a bulk supply of solid material (e.g., 10 kg total for five batches) as long as the starting mass for each trial is recorded and incremental water additions are used.

11.3.1.2 Mix a known amount of tap water with the bulk material in the pail or bowl until homogenized based on visual inspection. For the first point in the pre-test, no water needs to be added.

**NOTE:** The amount of water added should be enough to increase the moisture content in approximately 3 - 5% increments. Smaller additions may be needed in order to provide finer resolution of the packing density as a function of the moisture content curve.

11.3.1.3 Calculate the new moisture content (wet basis) for the trial as follows:

$$MC_{(wet)}^i = \frac{M_{test} \times MC_{wet} + W_{added}}{M_{test} + W_{added}}$$

Where:  $MC_{(wet)}^i$  = moisture content on a wet basis of the pre-test trial ( $g_{H_2O}/g$ )

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

$MC_{(wet)}$  = moisture content on a wet basis of the "as-tested" material ( $g_{H_2O}/g$ )

$W_{added}$  = mass of water added to the "as-tested" material ( $g_{H_2O}/g$ )

11.3.1.4 Compact approximately 1000 g of material into a tared 10-cm diameter mold into three consecutive layers of material. The compacted mass should have a level, flat surface as a top face.

11.3.1.5 Measure and record the height, diameter, and mass of the resulting compacted material.

11.3.1.6 Calculate and record the packing density (dry basis) as follows:

$$\rho_{pack} = \frac{m \times SC}{\pi \times h} \left( \frac{2}{d} \right)^2$$

Where:  $\rho_{pack}$  = packing density (dry basis) ( $g\text{-dry}/cm^3$ )

$m$  = mass of the compacted sample (g)

$SC$  = solids content of "as-tested" granular material ( $g\text{-dry}/g$ )

$d$  = measured diameter of the compacted sample (cm)

$h$  = measured height of the compacted sample (cm)

11.3.1.7 Repeat [Sec. 11.3.1.1](#) - [11.3.1.6](#) for four subsequent trials until the value of the calculated packing density decreases.

11.3.1.8 Plot the packing density as a function of moisture content. [Figure 4](#) shows an example of a packing density curve.

11.3.1.9 Determine the optimum moisture content at the maximum of the packing density curve. This value may be read directly from the graph or determined by the maximum of a third-order polynomial fit through the five pre-test data points (see the Microsoft Excel® spreadsheet template available at <http://www.vanderbilt.edu/leaching/downloads/test-methods/>).

11.3.2 Compacted granular test sample preparation

11.3.2.1 Using the optimum moisture content determined in [Sec. 11.3.1.9](#), calculate the amount of "as-received" material that is required to pack the sample holder to within 3 mm of the rim of the holder.

$$M_{\text{test}} = \frac{\rho_{\text{opt}} \times \pi \times (h - 0.3)}{\text{SC}} \left( \frac{d}{2} \right)^2$$

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

$\rho_{\text{opt}}$  = optimal packing density (dry basis) (g-dry/cm<sup>3</sup>) – determined in [Sec. 11.3.1.9](#)

h = measured height of the sample mold (cm)

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

d = measured diameter of the sample mold (cm)

11.3.2.2 Adjust the moisture content of the "as-received" material to the optimum moisture content using reagent water and mix until homogenized.

11.3.2.3 Pack the sample material into the sample holder using the modified Proctor compaction as described in Ref. 10.

11.3.2.4 Place a monolayer of borosilicate glass beads ([Sec. 6.3.3](#)) on the exposed sample surface to minimize scouring and mass loss during testing.

11.3.2.5 Begin the leach test procedure promptly or cover the sample with plastic wrap to minimize moisture loss to the atmosphere.

#### 11.4 Leaching procedure

This protocol is a semi-dynamic, tank-leaching procedure (see schematic in [Figure 5](#)) where the sample is exposed to eluate for a series of leaching intervals interspersed with eluent exchanges. The chemical composition of each eluate is determined and mass transfer from the bulk solid is determined as a function of cumulative leaching time. The schedule of leaching intervals for this method is shown in [Table 1](#).

11.4.1 Pre-test measurements – For the surface area calculation, measure and record the dimensions of the test specimen. This should include the diameter and height for a cylinder; length, width, and depth for a parallelepiped; or diameter of exposed surface for a compacted granular sample.

11.4.2 Measure and record the mass of the specimen. This value should be monitored for each eluent exchange.

11.4.3 If a holder is used, place the specimen in the monolith holder.

11.4.4 Measure and record the mass of the specimen and holder, if applicable.

11.4.5 The recommended temperature for conducting this method is room temperature (20 ± 2 °C). When conducted at temperature readings or variations other than those recommended, record the ambient temperature at each eluent renewal.

## 11.5 Eluent exchange

11.5.1 Fill a clean leaching vessel with the required volume of reagent water based on an L/A of  $9 \pm 1$  mL/cm<sup>2</sup>. Record the amount of eluent used.

11.5.2 Carefully place the specimen or the specimen and holder in the leaching vessel ([Figure 6a](#)) so that the sample is centered in the eluent (see [Figure 6b](#)). Submersion should be gentle enough so that the physical integrity of the monolith is maintained and scouring of the solid is minimized.

11.5.3 Cover the leaching vessel with the airtight lid and place in a safe location until the end of the leaching interval. [Table 1](#) shows the schedule of leaching intervals and cumulative release times for this method.

**NOTE:** Eluates of alkaline materials may be susceptible to neutralization through reaction with carbon dioxide. Precautions (e.g., ensuring airtight vessels or purging headspace) should be taken to minimize the effect of carbonation on eluates that may sit stationary for more than one week.

11.5.4 Prior to the end of the leaching interval, repeat [Sec. 11.5.1](#) in order to prepare a vessel for the next leaching interval.

11.5.5 At the end of the leaching interval (see [Table 1](#)), carefully remove the specimen or the specimen and holder from the vessel ([Figure 6c](#)). Drain the liquid from the surface of the specimen into the eluate for approximately 20 sec.

11.5.6 Measure and record the mass of the specimen or the mass of the specimen and holder ([Figure 6d](#)).

**NOTE:** The change in sample mass between intervals is an indication of the potential absorption of eluent by the matrix (mass gain) or erosion of the matrix (mass loss). In the case where a holder is used, moisture may condense on the holder during the leaching interval and sample absorption may not be evident.

**NOTE:** Mass gain may also be indicative of carbonate precipitation if the vessel is not tightly sealed and carbon dioxide is absorbed from the atmosphere.

11.5.7 Place the specimen or the specimen and holder into the clean leaching vessel filled with new eluent as prepared in [Sec. 11.5.4](#).

11.5.8 Cover the new leaching vessel with the airtight lid and place in a safe location until the end of the leaching interval.

## 11.6 Eluate processing

11.6.1 Measure and record the pH, specific conductivity, and oxidation reduction potential (ORP) of the eluate of the decanted eluate from the previous leaching interval (see Methods [9040](#), [9045](#), and [9050](#)).

**NOTE:** Measurement of pH, conductivity, and ORP should be taken within 15 minutes of eluent exchange ([Sec. 11.5](#)) 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 chemistry of COPCs.

11.6.2 Filter the remaining eluate through a 0.45- $\mu\text{m}$  membrane ([Sec. 6.5](#)).

11.6.3 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.

11.6.4 Collect all subsequent eluate by repeating the eluent exchange and eluate processing procedures in Secs. [11.5](#) and [11.6](#).

## 12.0 DATA ANALYSIS AND CALCULATIONS

### 12.1 Data reporting

12.1.1 [Figure 7](#) shows an example of a data sheet which 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) Material description (including monolithic or compacted granular)
- f) Moisture content of material used ( $\text{g}_{\text{H}_2\text{O}}/\text{g}$ )
- g) Dimensions (cm) and geometry of sample used
- h) Mass of solid material used (g)
- i) Mass of sample and holder at start of test (g)
- j) Eluate type (e.g., reagent water)
- k) 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 eluent exchange date and time
- c) Actual eluent exchange date and time
- d) Volume of eluent used (mL)
- e) Mass of sample and holder (g)
- f) Measured eluate pH

- g) Measured eluate conductivity (mS/cm)
- h) Measured ORP (mV) (optional)
- i) Concentration of all COPCs
- j) Analytical QC qualifiers as appropriate

## 12.2 Data presentation

### 12.2.1 Interval concentrations

12.2.1.1 At the conclusion of the schedule of leaching intervals (see [Table 1](#)), the concentration of COPCs in each eluate may be plotted as a function of cumulative leaching time. An example of this is shown in [Figure 8](#) for mass transport from a monolithic field sample of fixated scrubber sludge and lime.

12.2.1.2 If data is available from [Method 1313](#), interval concentrations and Method 1313 data may be plotted on the same graph as a function of eluate pH. This QC step is conducted in order to determine whether the concentration of COPCs approached equilibrium in any leaching interval (i.e., the driving force for mass transport from the matrix may not be constant, which is a common assumption of dynamic-tank leach testing). [Figure 9](#) shows this type of graph for the release from a field sample of fixated scrubber sludge and lime.

### 12.2.2 Interval mass release

At the conclusion of the schedule of leaching intervals (see [Table 1](#)), the interval mass released can be calculated for each leaching interval as follows:

$$M_{t_i} = \frac{C_i \times V_i}{A}$$

Where:  $M_{t_i}$  = mass released during the current leaching interval,  $i$  (mg/m<sup>2</sup>)

$C_i$  = constituent concentration in the eluate for interval  $i$  (mg/L)

$V_i$  = eluate volume in interval  $i$  (L)

$A$  = specimen external geometric surface area exposed to the eluent (m<sup>2</sup>)

### 12.2.3 Mean interval flux

The flux of a COPC in an interval may be plotted as a function of the generalized mean of the square root of cumulative leaching time ( $\sqrt{t}$ ). An example of a flux graph is shown in [Figure 10](#) for the release from a field sample of fixated scrubber sludge with lime. This graph may be used to interpret the mechanism of release (see Ref. 5 for further details).

12.2.3.1 The flux across the exposed surface of the sample can be calculated by dividing the interval mass release by the interval duration as follows:

$$F_i = \frac{M_i}{t_i - t_{i-1}}$$

Where:  $F_i$  = flux for interval,  $i$  ( $\text{mg}/\text{m}^2\cdot\text{s}$ )

$M_i$  = mass released during the current leaching interval,  $i$  ( $\text{mg}/\text{m}^2$ )

$t_i$  = cumulative time at the end of the current leaching interval,  $i$  (s)

$t_{i-1}$  = cumulative time at the end of the previous leaching interval,  $i-1$  (s)

12.2.3.2 The time used to plot each interval mass is the generalized mean of the square root of the cumulative leaching time using the cumulative time at the end of the  $i^{\text{th}}$  interval,  $t_i$ , and the cumulative time at the end of the previous interval,  $t_{i-1}$ .

$$\bar{t}_i = \left( \frac{\sqrt{t_i} + \sqrt{t_{i-1}}}{2} \right)^2$$

Where:  $\bar{t}_i$  = generalized mean leaching time for the current interval,  $i$  (s)

$t_i$  = cumulative time at the end of the current leaching interval,  $i$  (s)

$t_{i-1}$  = cumulative time at the end of the previous leaching interval,  $i-1$  (s)

**NOTE:** If the concentrations of a COPC in the eluates approach that shown in [Method 1313](#) for liquid-solid equilibrium, the flux curve will show the pattern in [Figure 10](#) with intervals of the same duration having the same flux value. When the eluate concentration approaches saturation, the driving force for mass transfer approaches zero, interval flux is limited, and intervals with like durations will display similar flux limitations.

#### 12.2.4 Cumulative release

12.2.4.1 The interval release calculated in 12.2.2 can be summed to provide the cumulative mass release as a function of leaching time. [Figure 11](#) shows the cumulative release curves for a field sample of fixated scrubber sludge with lime.

12.2.4.2 Interpretation of the cumulative release of constituents is illustrated using the analytical solution for simple radial diffusion from a cylinder into an infinite bath presented by Crank (see Ref. 8).

$$M_t = 2\rho C_o \left( \frac{D^{\text{obs}} t}{\pi} \right)^{1/2}$$

Where:  $M_t$  = cumulative mass released during leaching interval  $i$  ( $\text{mg}/\text{m}^2$ )

$\rho$  = density of the "as-tested" sample ( $\text{kg}/\text{m}^3$ )

$C_o$  = concentration of available COPC in the solid matrix ( $\text{mg}/\text{kg}$ )

$D^{\text{obs}}$  = observed diffusivity ( $\text{m}^2/\text{s}$ )

$t$  = leaching time (s)

When transformed to a log-log scale, the analytical solution presented by Crank becomes linear with the square root of time.

$$\log[M_t] = \log \left[ 2\rho C_0 \left( \frac{D^{obs}}{\pi} \right)^{1/2} \right] + \frac{1}{2}t$$

Thus, under the assumptions of the analytical solution presented by Crank, the mass release should be proportional to the square root of time. A line showing the square root of time is plotted in [Figure 11](#) along with the data. Since flux is the derivative of release, a similar treatment of flux as a function of leaching time using the simple diffusion model would be proportional to the negative square root of time as shown in [Figure 10](#).

Models other than the simple diffusion model presented by Crank may also be used to interpret mass release. For example, the Shrinking Unreacted Core Model (see Ref. 2) and the Coupled Dissolution-Diffusion Model (see Ref. 7) incorporate chemical release parameters (e.g., as derived from [Method 1313](#) data) into the model to better estimate release mechanisms and predictions (see Ref. 5 for further details).

### 12.2.5 Observed diffusivity

An observed diffusivity for each COPC can be determined using the logarithm of the cumulative release plotted versus the logarithm of time. In the case of a diffusion-controlled mechanism, this plot is expected to be a straight line with a slope of 0.5. An observed diffusivity can then be determined for each leaching interval where the slope is  $0.50 \pm 0.15$  (see Ref. 1 and Ref. 14) by the following:

$$D_i^{obs} = \pi \left[ \frac{M_{t_i}}{2\rho C_0 (\sqrt{t_i} - \sqrt{t_{i-1}})} \right]^2$$

Where:  $D_i^{obs}$  = observed diffusivity of a COPC for leaching interval  $i$  ( $m^2/s$ )

$M_{t_i}$  = mass released during leaching interval  $i$  ( $mg/m^2$ )

$t_i$  = cumulative contact time at the end of the current leaching interval,  $i$  (s)

$t_{i-1}$  = cumulative contact time at the end of the previous leaching interval,  $i-1$  (s)

$\rho$  = sample density (dry basis) ( $kg\text{-dry}/m^3$ )

$C_0$  = initial leachable content (i.e., available release potential) ( $mg/kg$ )

The mean observed diffusivity for each COPC is then determined by taking the average of the interval observed diffusivities. It should be reported with the computed uncertainty (i.e., standard deviation).

**NOTE:** Since the analysis presented above assumes a diffusion process, only those interval mass transfer coefficients corresponding to leaching intervals with slopes of  $0.50 \pm 0.15$  are included in the overall average mass-transfer coefficient.

### 12.3 Data representation by constituent

A concise representation of all relevant data for a single constituent may be presented as shown in [Figure 12](#) for arsenic from a field core of fixated scrubber sludge with lime (FSSL) material. The data shows eluate pH generation as a function of leaching time ([Figure 12a](#)), comparison between eluate

concentrations and Method 1313 data as a function of eluate pH ([Figure 12b](#)), constituent flux as a function of generalized mean cumulative leaching time ([Figure 12c](#)), and constituent release as a function of cumulative leaching time ([Figure 12d](#)).

#### 12.4 Interpolation/extrapolation to target time values

The collected time dependence data may be interpolated or extrapolated to the nearest target cumulative time ( $\Sigma t$ ) 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 ( $\log_{10}[\text{mg/L}]$ )

$c_i$  = the concentration measured at test position i (mg/L)

##### 12.4.2 Linear interpolation/extrapolation

Given a set of coordinate data sorted by increasing order according to  $\Sigma t$  value (e.g.,  $\Sigma t_1 < \Sigma t_2 < \dots < \Sigma t_n$ ), an interpolated/extrapolated  $\log_{10}$ -transformed concentration at a known  $\Sigma t$  target is calculated as:

$$C_T = a_T + b_T \Sigma t_T$$

Where:  $C_T$  = the concentration at target  $\Sigma t$  value,  $\Sigma t_T$  ( $\log_{10}[\text{s}]$ )

$a_T$  and  $b_T$  are coefficients of the linear interpolation/extrapolation equation

$\Sigma t_T$  = a target cumulative time value

Depending on the values of observed  $\Sigma t$  values relative to target  $\Sigma t$  values, the calculations of the coefficients  $a_T$  and  $b_T$  in the equation may differ according to the following algorithm:

- If  $\Sigma t_T < \Sigma t_1$ , then  $b_T = (C_2 - C_1) / (\Sigma t_2 - \Sigma t_1)$  and  $a_T = C_2 - b_T \cdot \Sigma t_2$  (extrapolation from the two points with closest  $\Sigma t$  values)
- If  $\Sigma t_T \geq \Sigma t_n$ , then  $b_T = (C_n - C_{n-1}) / (\Sigma t_n - \Sigma t_{n-1})$  and  $a_T = C_n - b_T \cdot \Sigma t_n$  (extrapolation from the two points with closest  $\Sigma t$  values)
- If  $\Sigma t_{j-1} \leq \Sigma t_T < \Sigma t_j$ , then  $b_T = (C_j - C_{j-1}) / (\Sigma t_j - \Sigma t_{j-1})$  and  $a_T = C_{j-1} - b_T \cdot \Sigma t_{j-1}$  (interpolation from the two closest points surrounding  $\Sigma t_T$ )

**NOTE:** Interpolation or extrapolation of data should only be conducted within a distance of  $\pm 20\%$  of the target  $\Sigma t$  value. Since the allowable L/S tolerance about a target L/S value is variable (see [Table 1](#)), interpolation/extrapolation should not create data at a target  $\Sigma t$  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 laboratory QC or accreditation.

13.2 Interlaboratory validation of this method was conducted using a solidified waste analog (material code SWA) and a contaminated smelter site soil (material code CFS). Repeatability and reproducibility was determined for mean interval flux excluding the first wash-off interval (see [Table 2](#)) and for cumulative mass released after 63 days of leaching (see [Table 3](#)). More details on the interlaboratory validation may be found in Ref. 4.

13.3 Ref. 5 and Ref. 12 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. International Ash Working Group (IAWG), *Municipal Solid Waste Incinerator Residues*, Amsterdam, the Netherlands, 1997.
2. Crank, *Mathematics of Diffusion*, Oxford University Press,, London, 1986.
3. A.C. Garrabrants, and D.S. Kosson, "Leaching Processes and Evaluation Tests for Inorganic Constituents Release from Cement-Based Matrices," in *Solidification/Stabilization of Hazardous, Radioactive and Mixed Wastes*, R. Spence, and C. Shi (eds), CRC Press, 2005.
4. 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.
5. 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.
6. J. de Groot, and H. A. van der Sloot, "Determination of Leaching Characteristics of Waste Materials Leading to Environmental Product Certification," in *Solidification and Stabilization of Hazardous, Radioactive, and Mixed Wastes*, 2nd Volume, ASTM STP 1123, T. M. Gilliam, and C. C. Wiles (eds), American Society for Testing and Materials, Philadelphia, PA, 1992.
7. Hinsenveld, and P.L. Bishop, "Use of the shrinking core/exposure model to describe the leachability from cement stabilized wastes," in *Stabilization and Solidification of Hazardous, Radioactive, and Mixed Wastes*, 3rd Volume, ASTM STP 1240, T. M. Gilliam, and C. C. Wiles (eds), American Society for Testing and Materials,, Philadelphia, PA, 1996.
8. D.E. Hockley, and H.A. van der Sloot, "Long-term Processes in a Stabilized Coal Waste Block Exposed to Seawater," *Environmental Science and Technology*, 25(8) 1408-1414, 1991.
9. NEN 7345, "Leaching Characteristics of Solid Earth and Stony Materials - Leaching Tests - Determination of the Leaching of Inorganic Constituents from Molded and Monolithic Materials with the Diffusion Test," *Dutch Standardization Institute*, Delft, The Netherlands, 1995.
10. ASTM D1557-07, "Standard Method for Laboratory Compaction Characteristics of Soil Using Modified Effort (56,000 ft-lbf/ft<sup>3</sup> (2,700 kN-m/m<sup>3</sup>)), " *ASTM International*, West Conshohocken, PA,
11. ASTM D1308-95, "Standard Method for Accelerated Leach Test for Diffusive Releases from Solidified Waste and a Computer Program to Model Diffusive, Fractional Leaching from Cylindrical Waste Forms," *ASTM International*, West Conshohocken, PA, 2001.
12. 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.
13. CEN/TS 15863, "Characterization of waste – Leaching Behaviour Tests – Dynamic Monolithic Leaching Test with Periodic Leachant Renewal," *Comité Européen de Normalisation*, Brussels, Belgium, 2009.

14. Sanchez, "Étude de la lixiviation de milieux poreux contenant des espèces solubles: Application au cas des déchets solidifiés par liants hydrauliques," doctoral thesis, Institut National des Sciences Appliquées de Lyon, Lyon, France, 1996.

15. ANSI/ANS 16.1, "American National Standard Measurement of the Leachability of Solidified Low-Level Radioactive Wastes by a Short-term Test Procedure," *American Nuclear Society*, La Grange Park, IL, 1986.

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

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

TABLE 1  
SCHEDULE OF ELUATE RENEWALS

Interval Label	Interval Duration (h)	Interval Duration (d)	Cumulative Leaching Time (d)
T01	2.0 ± 0.25	–	0.08
T02	23.0 ± 0.5	–	1.0
T03	23.0 ± 0.5	–	2.0
T04	–	5.0 ± 0.1	7.0
T05	–	7.0 ± 0.1	14.0
T06	–	14.0 ± 0.1	28.0
T07	–	14.0 ± 0.1	42.0
T08	–	7.0 ± 0.1	49.0
T09	–	14.0 ± 0.1	63.0

NOTE: This schedule may be extended for additional 14-day contact intervals to provide more information regarding longer-term release.

TABLE 2

METHOD PRECISION FOR MEAN INTERVAL FLUX (2<sup>nd</sup> – 9<sup>th</sup> Intervals)

Analyte	Symbol	Reapeatability – SWA %RSD <sub>r</sub>	Repeatability – CFS %RSD <sub>r</sub>	Reproducibility – SWA %RSD <sub>R</sub>	Reproducibility – CFS %RSD <sub>R</sub>
Aluminum	Al	7.3	13.3	25.3	25.3
Antimony	Sb	9.2	14.8	21.8	23.8
Arsenic	As	19.9	-	31.1	-
Barium	Ba	13.2	7.5	44.8	18.3
Boron	B	10.8	7.2	27.3	27.1
Cadmium	Cd	-	7.6	-	23.2
Calcium	Ca	8.1	6.6	28.7	26.0
Chromium	Cr	10.2	-	23.8	-
Lead	Pb	-	4.3	-	19.8
Potassium	K	12.4	10.8	28.8	40.1
Selenium	Se	10.9	13.3	30.8	32.4
Vanadium	V	8.5	11.3	22.3	30.6

Material	Reapeatability – SWA %RSD <sub>r</sub>	Repeatability – CFS %RSD <sub>r</sub>	Repeatability – Overall	Reproducibility – SWA %RSD <sub>R</sub>	Reproducibility – CFS %RSD <sub>R</sub>	Reproducibility – Overall
Mean	11%	10%	11%	29%	27%	28%

NOTE: First interval is removed from mean interval flux because of variances associated with wash-off of surface contaminants that do not pertain to the method precision.

Data taken from Ref. 4.

TABLE 3

## METHOD PRECISION FOR CUMULATIVE RELEASE AFTER 63 DAYS

Analyte	Symbol	Repeatability – SWA %RSD <sub>r</sub>	Repeatability – CFS %RSD <sub>r</sub>	Reproducibility – SWA %RSD <sub>R</sub>	Reproducibility – CFS %RSD <sub>R</sub>
Aluminum	Al	5.4	5.3	23.6	22.9
Antimony	Sb	6.9	5.9	19.7	14.4
Arsenic	As	15.9	-	31.0	-
Barium	Ba	7.5	3.9	35.6	16.5
Boron	B	8.4	3.7	22.6	25.7
Cadmium	Cd	-	4.8	-	18.4
Calcium	Ca	4.6	3.2	23.9	24.6
Chromium	Cr	7.7	-	17.7	-
Lead	Pb	-	1.6	-	12.0
Potassium	K	10.8	6.3	24.8	44.4
Selenium	Se	8.7	3.6	26.7	20.5
Vanadium	V	5.7	4.2	21.1	22.8

Material	Repeatability – SWA %RSD <sub>r</sub>	Repeatability – CFS %RSD <sub>r</sub>	Repeatability – Overall	Reproducibility – SWA %RSD <sub>R</sub>	Reproducibility – CFS %RSD <sub>R</sub>	Reproducibility – Overall
Mean	8%	4%	6%	25%	22%	23%

Data taken from Ref. 4.

FIGURE 1

EXAMPLES OF MONOLITHIC SAMPLE HOLDERS

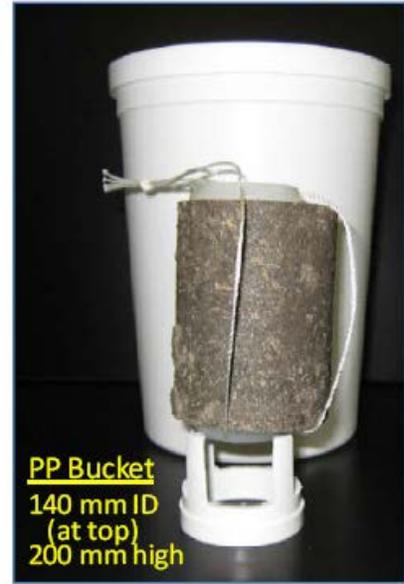
3-D Configuration



Sample Holder



Sample, Holder and Stand



3-D Leaching Setup

1-D Configuration



Empty Sample Holder



Full Sample Holder



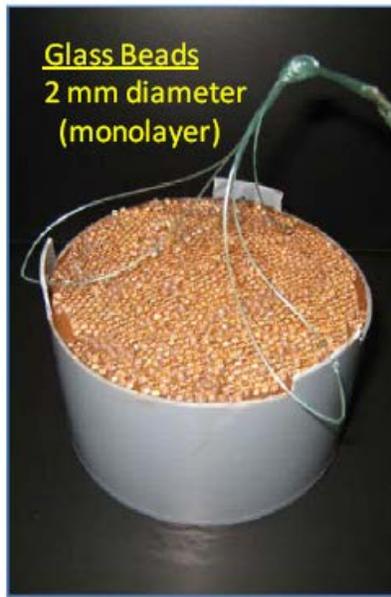
1-D Leaching Setup

FIGURE 2

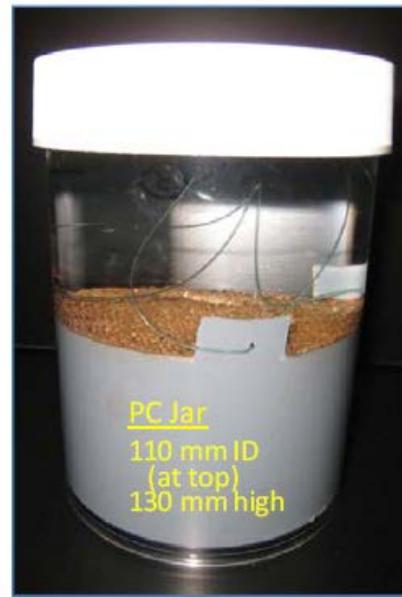
EXAMPLE COMPACTED GRANULAR SAMPLE HOLDER AND SETUP



Empty Sample Holder



Compacted Sample



1-D Leaching Setup

FIGURE 3  
METHOD FLOWCHART

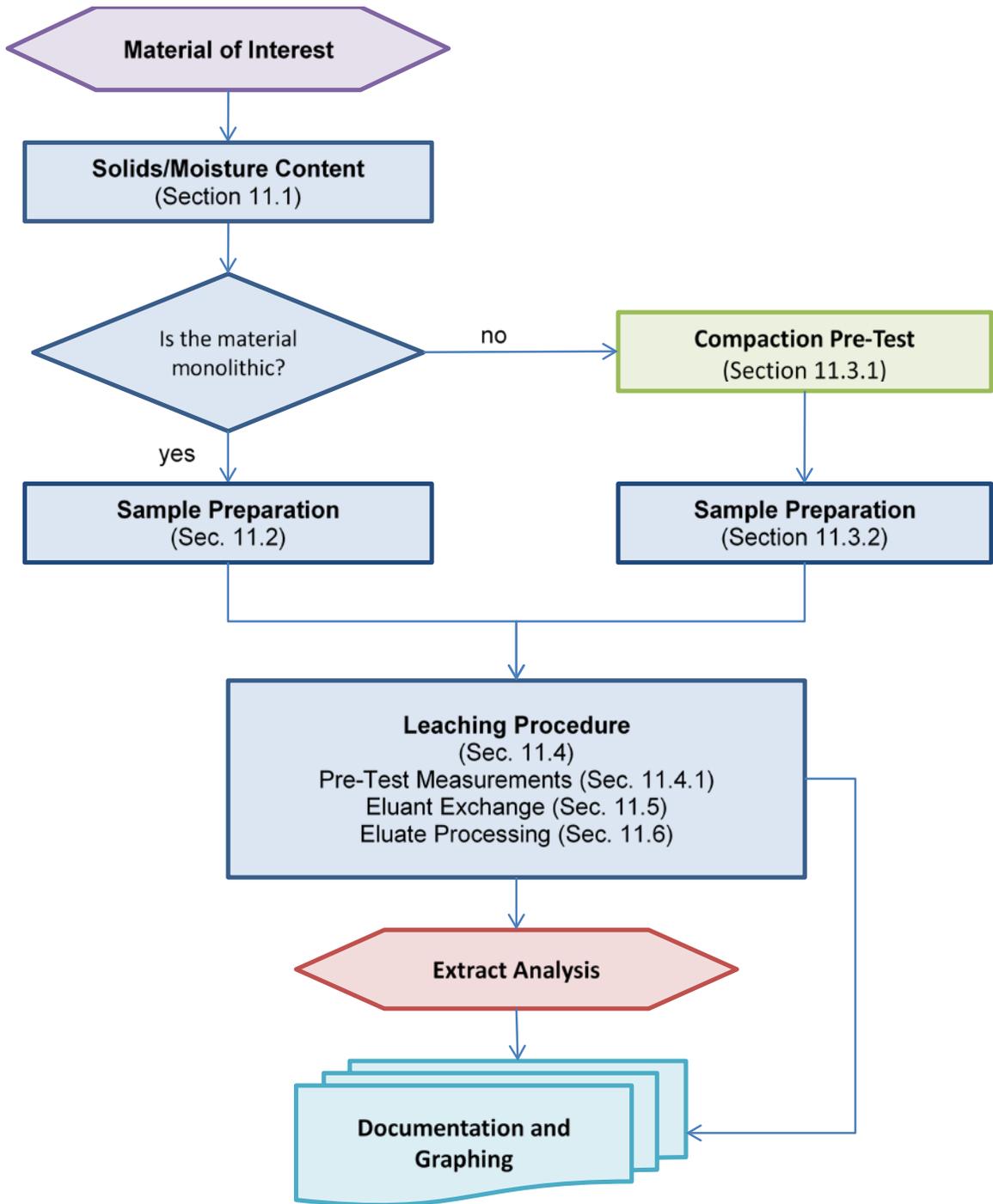


FIGURE 4

EXAMPLE CURVE OF PACKING DENSITY AS A FUNCTION OF MOISTURE CONTENT

$$y = 55.975x^3 - 65.036x^2 + 1.8352$$

$$r^2 = 0.983$$

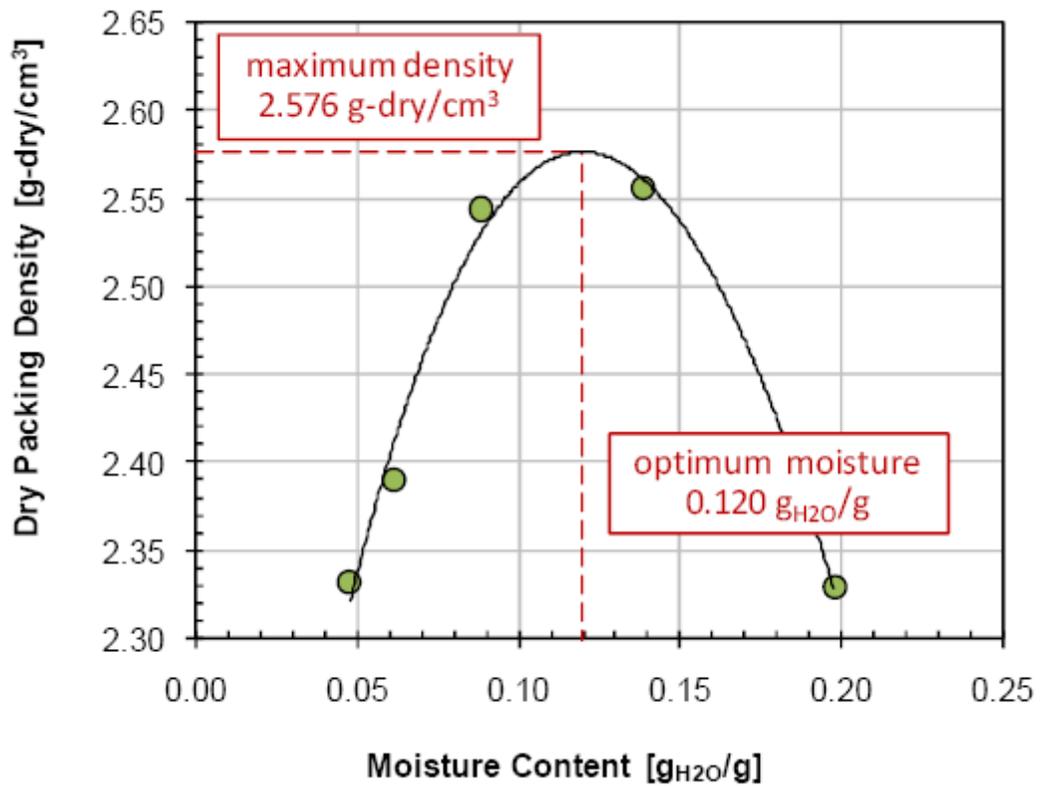


FIGURE 5

SCHEMATIC OF SEMI-DYNAMIC MASS TRANSFER TEST PROCESS

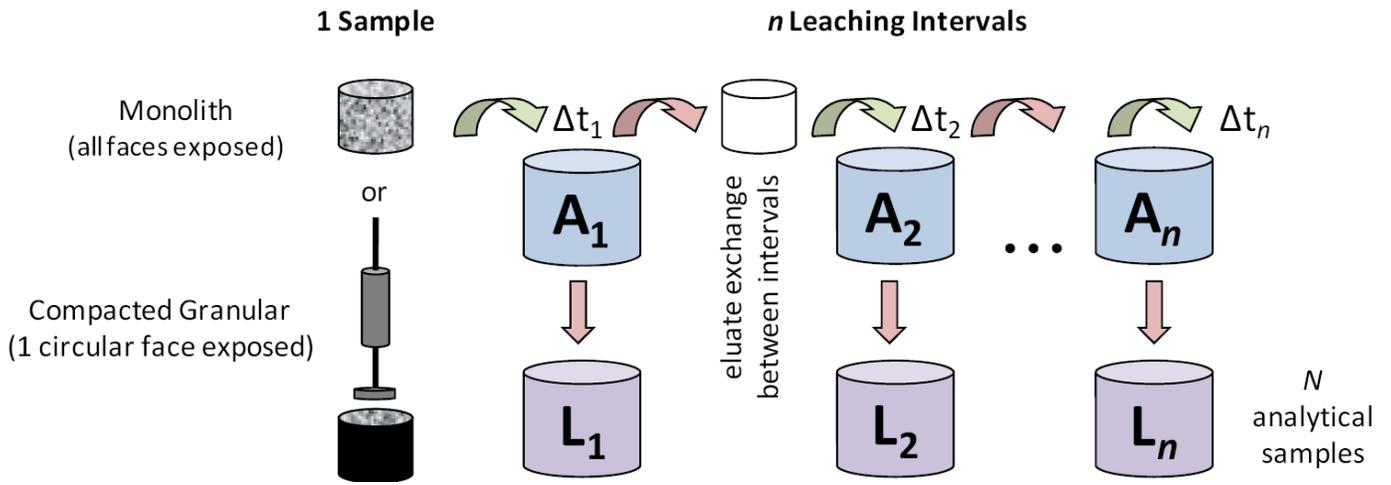


Figure obtained and modified from Ref. 1.

FIGURE 6

EXAMPLE LEACHING PROCEDURE STEPS



Start of Leaching Interval



Sample Centered in Eluant (top view)



Removing Sample for Exchange



Mass of Sample and Holder

FIGURE 7

EXAMPLE DATA REPORTING FORMAT

<b>ABC Laboratories</b> 123 Main Street Anytown, USA Contact: John Smith (555) 111-1111	<b>EPA METHOD 1315</b> <b>Report of Analysis</b>	Client Contact: Susan Jones (555) 222-2222
Material Code: <b>XYZ</b> Material Type: Coal Combustion Fly Ash Date Received: 10/1/20xx Test Start Date: 11/1/20xx Report Date: 12/1/20xx  Test Type: Compacted Granular Eluent: ASTM Type II Water	Particle Size: 88% passing 2-mm sieve Mass used in Column: 860 g Moisture Content: 0.002 g <sub>H<sub>2</sub>O</sub> /g Sample Geometry: Cylinder Sample Diameter: 10.0 cm Sample Depth: 60.3 cm Mass of Sample & Holder: 1020 g Lab Temperature: 21 ± 2 °C	

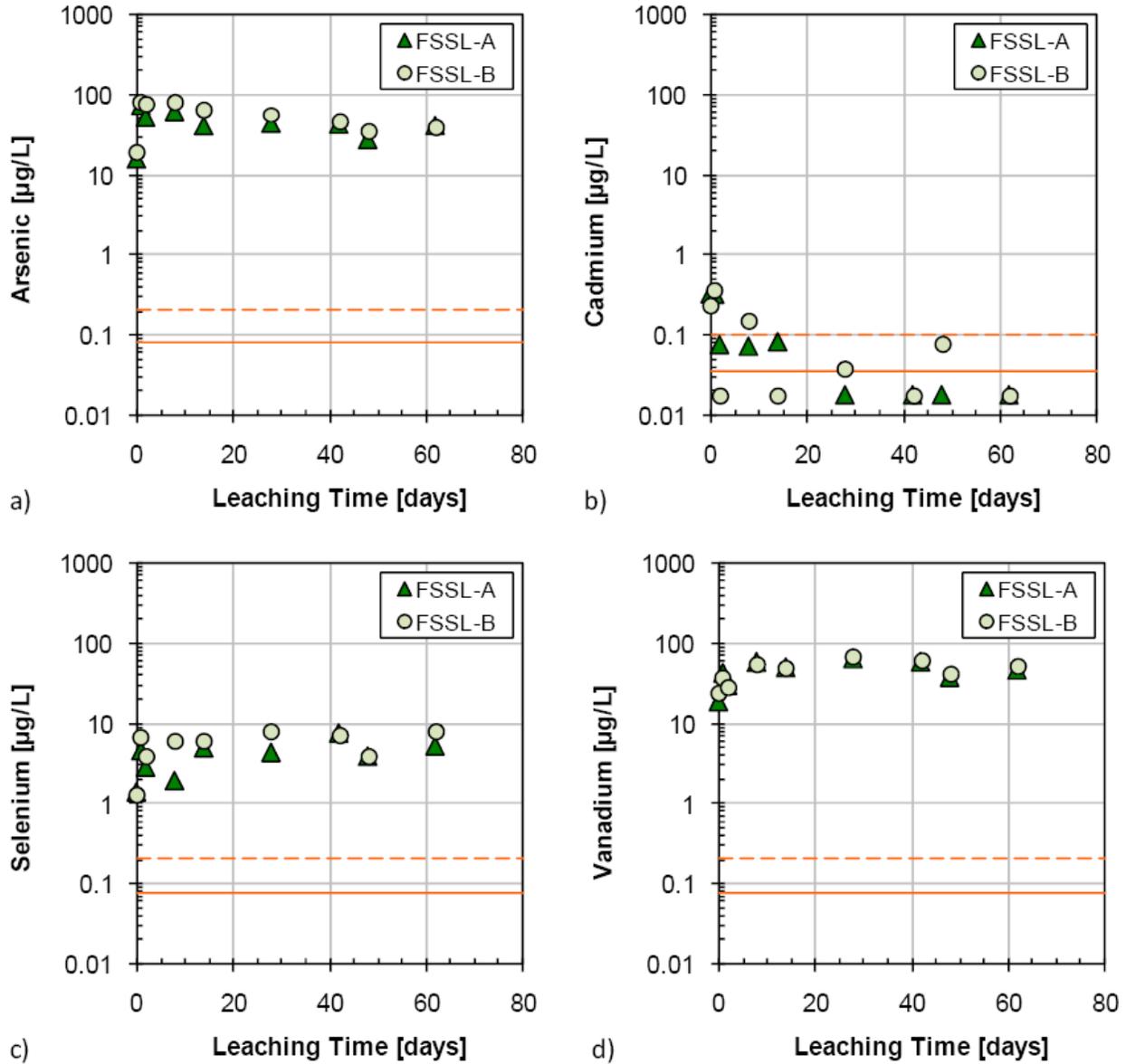
Test Position	Replicate	Value	Units	Method	Note		
T01	A						
	Eluate Sample ID	XYZ-1315-T01-A					
	Exchange Date	11/1/20xx					
	Target Exchange Time	12:00	PM				
	Actual Exchange Time	12:15	PM				
	Mass of Sample & Holder	1026	g				
	Eluate Mass	730.4	g				
	Eluate pH	8.82	-	EPA 9040			
	Eluate Conductivity	5.4	mS/c	EPA 9050			
	Eluate ORP	NA	mv				
	<b>Chemical Analysis</b>	<b>Value</b>	<b>Units</b>	<b>QC Flag</b>	<b>Method</b>	<b>Date</b>	<b>Dilution Factor</b>
	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-1315-T02-A					
	Exchange Date	11/1/20xx					
	Target Exchange Time	12:00	PM				
	Actual Exchange Time	12:18	PM				
	Mass of Sample & Holder	1027	g				
	Eluate Mass	725.0	g				
	Eluate pH	9.15	-	EPA 9040			
	Eluate Conductivity	2.8	mS/c	EPA 9050			
	Eluate ORP	NA	mv				
	<b>Chemical Analysis</b>	<b>Value</b>	<b>Units</b>	<b>QC Flag</b>	<b>Method</b>	<b>Date</b>	<b>Dilution Factor</b>
	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 8

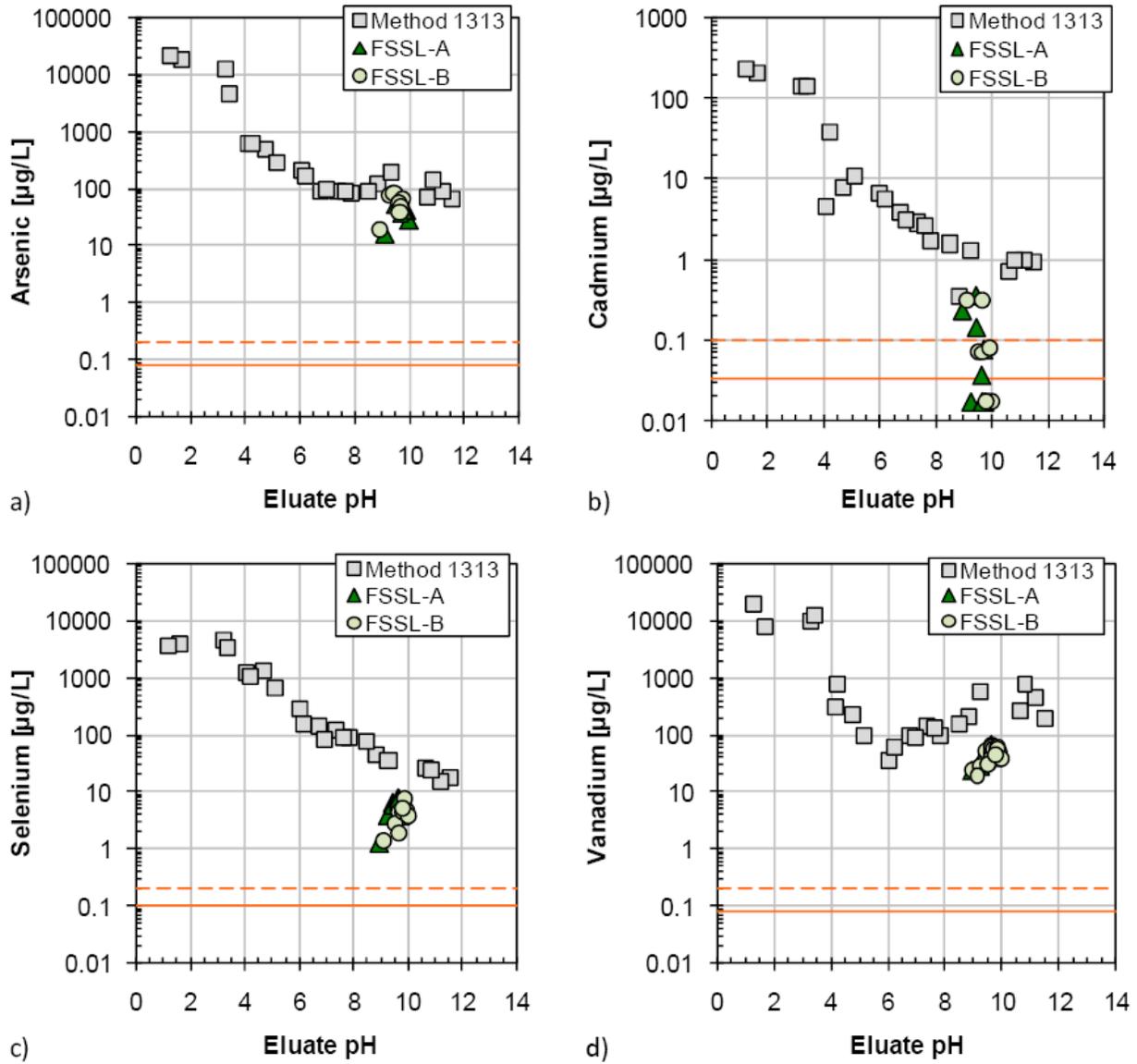
EXAMPLE INTERVAL CONCENTRATION GRAPHS



**NOTE:** Orange lines represent cumulative release if all eluate extracts were at the quantitation limit (dashed) and detection limit (solid). Chemical analyses below the detection limit are shown at ½ the detection limit value.

FIGURE 9

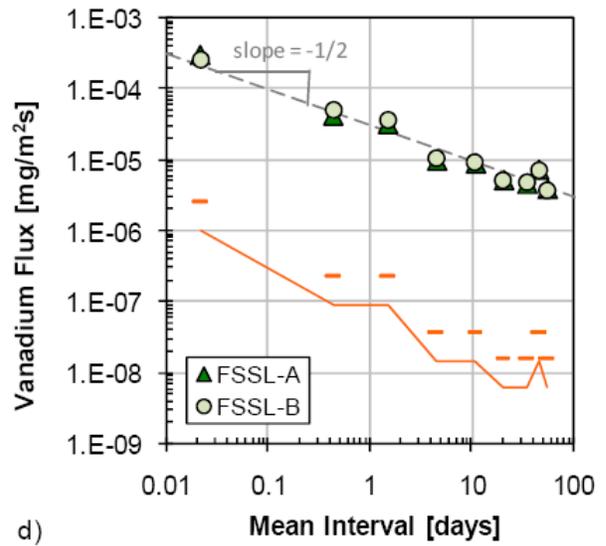
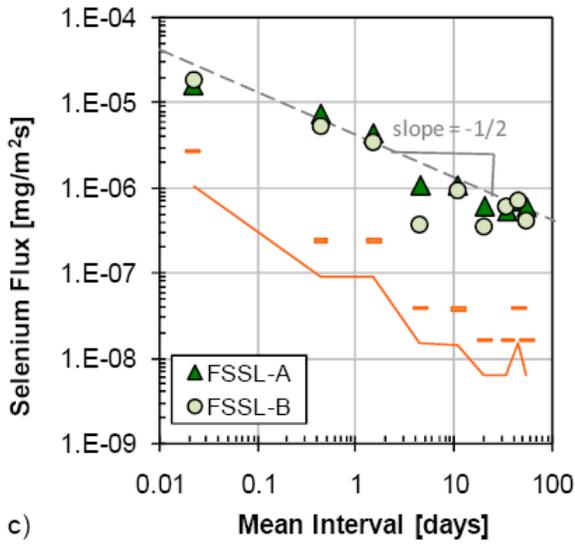
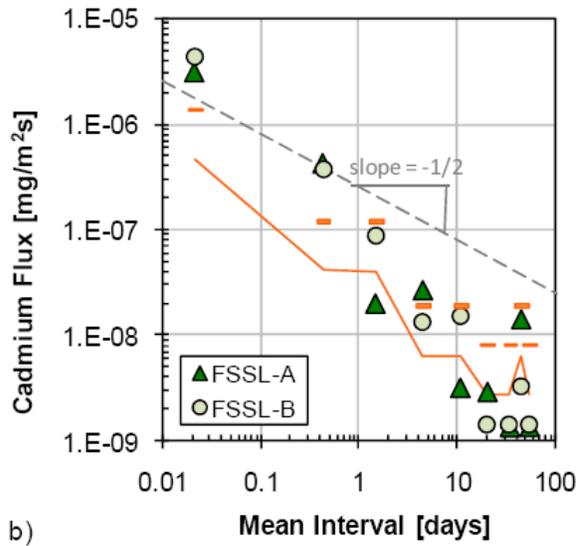
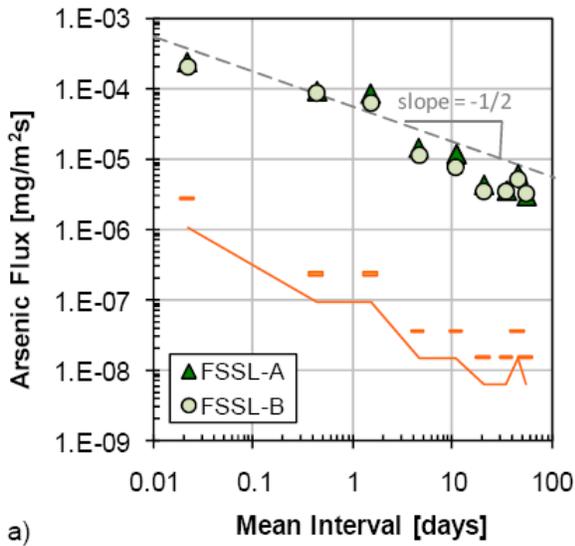
EXAMPLE OF SATURATION CHECK BETWEEN INTERVAL CONCENTRATIONS AND METHOD 1313 DATA



**NOTE:** Orange lines represent cumulative release if all eluate extracts were at the quantitation limit (dashed) and detection limit (solid). Chemical analyses below the detection limit are shown at  $1/2$  the detection limit value.

FIGURE 10

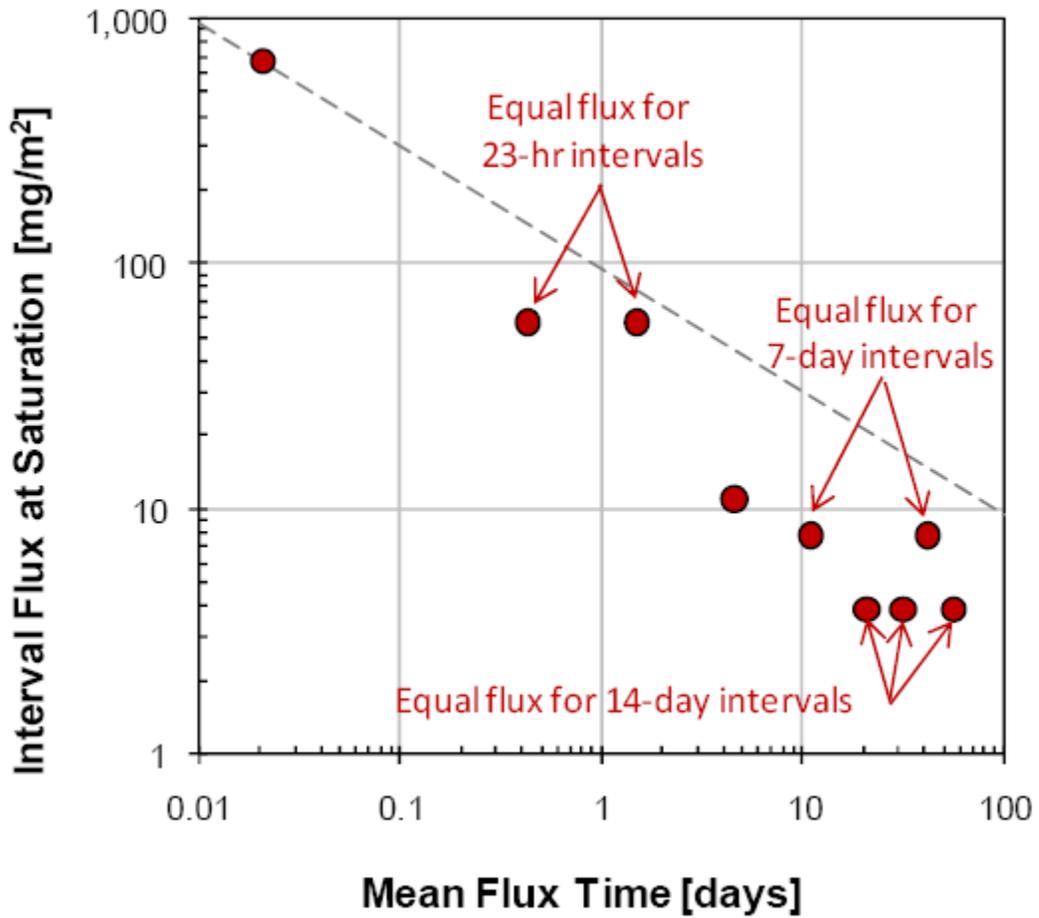
EXAMPLE INTERVAL FLUX GRAPHS



NOTE: Orange data represent cumulative release if all eluate extracts were at the quantitation limit (dashes) and detection limit (solid line).

FIGURE 11

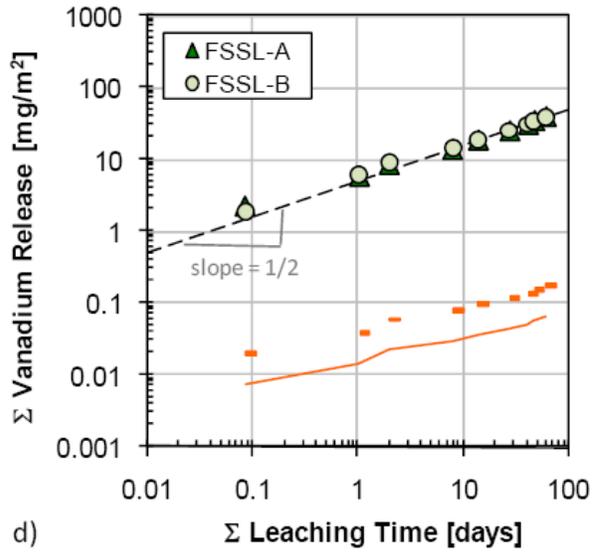
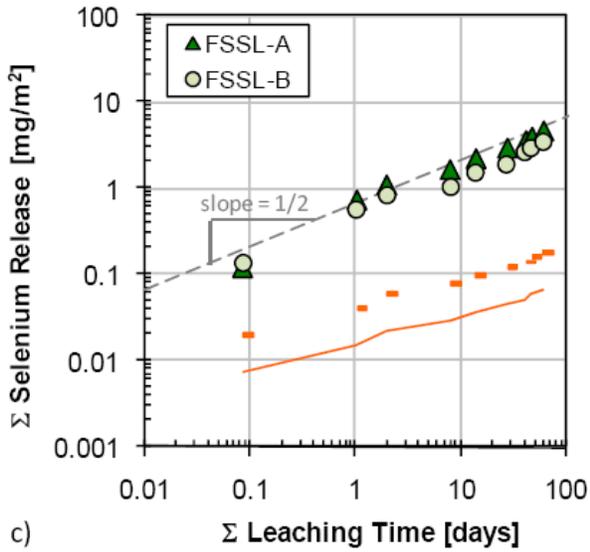
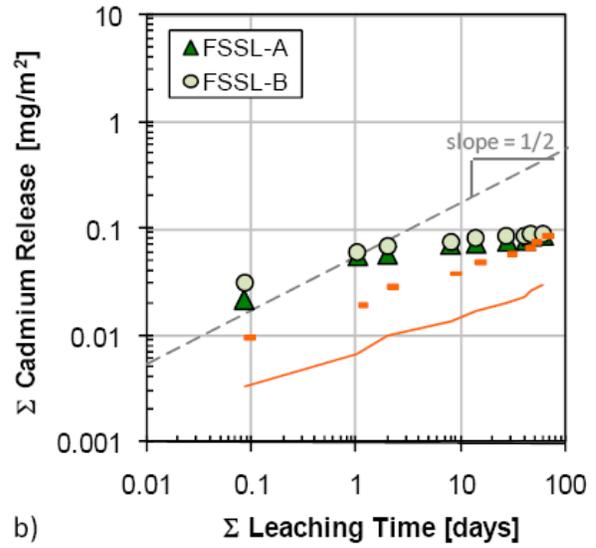
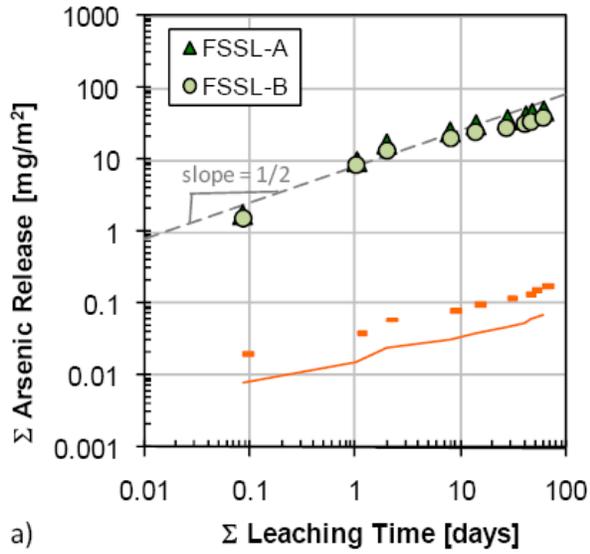
INTERVAL FLUX AT ELUATE SATURATION



**NOTE:** This figure assumes that the concentration in the eluate approaches saturation during the leaching interval (i.e., the driving force for diffusion approaches zero). When the leaching solution is saturated, the resulting mass release and interval flux is constant for intervals of the same duration.

FIGURE 12

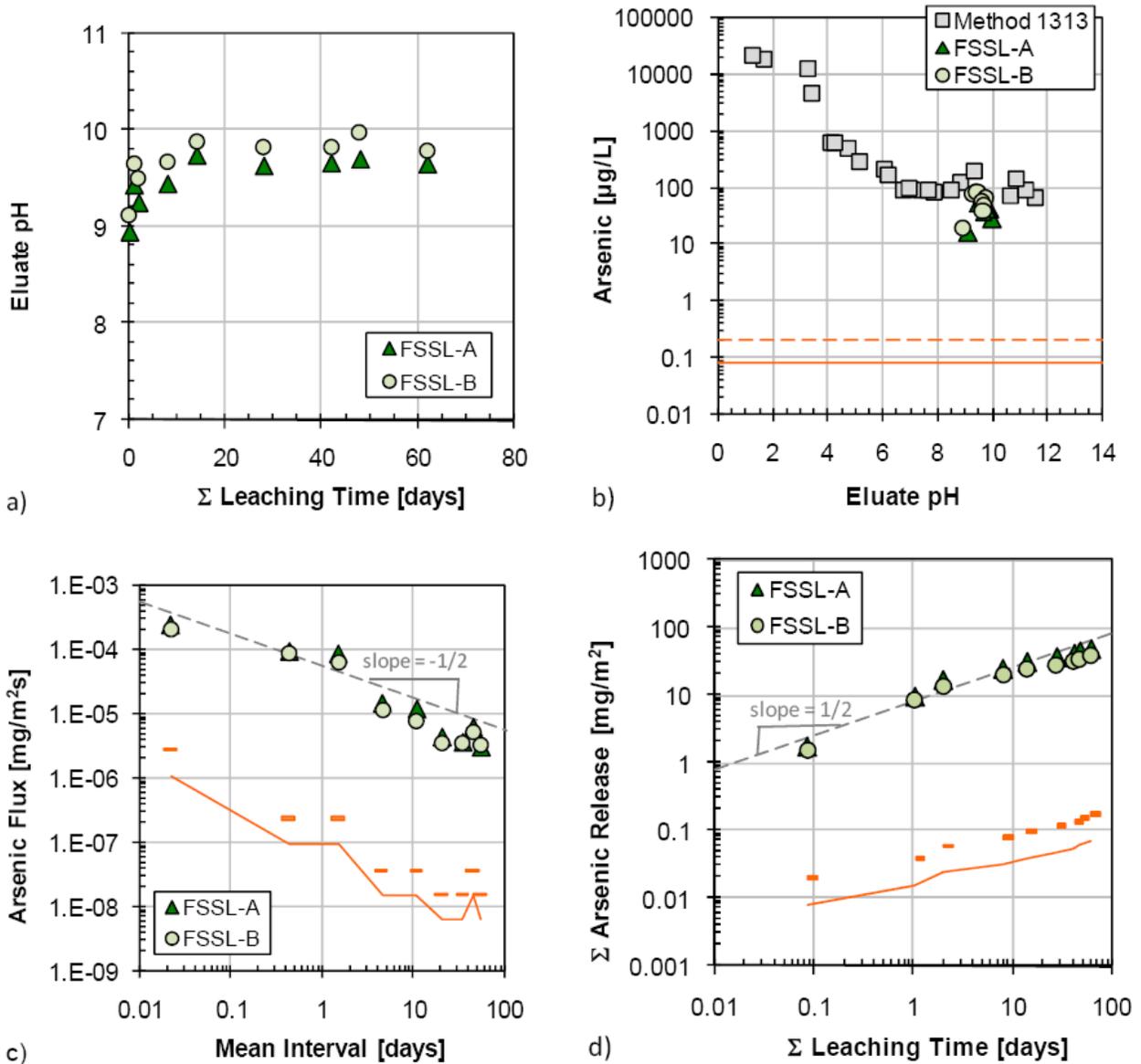
EXAMPLE CUMULATIVE RELEASE GRAPHS



**NOTE:** Orange data represent cumulative release if all eluate extracts were at the quantitation limit (dashes) and detection limit (solid line).

FIGURE 13

DATA REPRESENTATION BY CONSTITUENT (QUAD FORMAT)



NOTE: Orange data represent cumulative release if all eluate extracts were at the quantitation limit (dashes) and detection limit (solid line).