Designation: D5744 – $13^{\varepsilon 1}$

Standard Test Method for
Laboratory Weathering of Solid Materials Using a Humidity Cell[1]

This standard is issued under the fixed designation D5744; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

ε^1 NOTE—Editorially updated Research Report footnote information in Section 13 in December 2013.

1. Scope

1.1 This kinetic test method covers a laboratory weathering procedure that (*1*) enhances reaction-product transport in the aqueous leach of a solid material sample of specified mass, and (*2*) measures rates of weathering-product mass release. Soluble weathering products are mobilized by a fixed-volume aqueous leach that is performed and collected weekly. Leachate samples are analyzed for pH, alkalinity/acidity, specific conductance, sulfate, and other selected analytes.

1.1.1 This test method is intended for use to meet kinetic testing regulatory requirements for mining waste rock and ores sized to pass a 6.3-mm (0.25-in.) Tyler screen.

1.1.2 Interlaboratory testing of this method has been confined to mine waste rock. Application of this test method to metallurgical-processing waste (for example, mill tailings) is outside the scope of the test method.

1.2 This test method is a modification of a laboratory weathering procedure developed originally for mining wastes(**1-3**).[2] However, it may have useful application wherever gaseous oxidation coupled with aqueous leaching are important mechanisms for contaminant mobility.

1.3 This test method calls for the weekly leaching of a well-characterized solid material sample (weighing at least 1000-g), with water of specified purity, and the collection and chemical characterization of the resulting leachate. Test duration is determined by the user's objectives of the test.

1.4 As described, this test method may not be suitable for some materials containing plastics, polymers, or refined metals. These materials may be resistant to traditional particle size reduction methods.

1.5 Additionally, this test method has not been tested for applicability to organic substances and volatile matter.

1.6 This test method is not intended to provide leachates that are identical to the actual leachate produced from a solid material in the field or to produce leachates to be used as the sole basis of engineering design.

1.7 This test method is not intended to simulate site-specific leaching conditions. It has not been demonstrated to simulate actual disposal site leaching conditions. Furthermore, the test is not designed to produce effluents that are in chemical equilibrium with the solid phase sample.

1.8 This test method is intended to describe the procedure for performing the laboratory weathering of solid materials. It does not describe all types of sampling and analytical requirements that may be associated with its application.

1.9 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.9.1 *Exception*—The values given in parentheses are for information only.

1.10 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:*[3]

D75 Practice for Sampling Aggregates
D276 Test Methods for Identification of Fibers in Textiles
D420 Guide to Site Characterization for Engineering Design and Construction Purposes (Withdrawn 2011)[4]

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[1] This test method is under the jurisdiction of ASTM Committee D34 on Waste Management and is the direct responsibility of Subcommittee D34.01.04 on Waste Leaching Techniques.

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[2] The boldface numbers in parentheses refer to the list of references at the end of this standard.

[3] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

[4] The last approved version of this historical standard is referenced on www.astm.org.

D653 Terminology Relating to Soil, Rock, and Contained Fluids
D737 Test Method for Air Permeability of Textile Fabrics
D1067 Test Methods for Acidity or Alkalinity of Water
D1125 Test Methods for Electrical Conductivity and Resistivity of Water
D1193 Specification for Reagent Water
D1293 Test Methods for pH of Water
D1498 Test Method for Oxidation-Reduction Potential of Water
D2234/D2234M Practice for Collection of a Gross Sample of Coal
D3370 Practices for Sampling Water from Closed Conduits
E276 Test Method for Particle Size or Screen Analysis at No. 4 (4.75-mm) Sieve and Finer for Metal-Bearing Ores and Related Materials
E877 Practice for Sampling and Sample Preparation of Iron Ores and Related Materials for Determination of Chemical Composition and Physical Properties
E1915 Test Methods for Analysis of Metal Bearing Ores and Related Materials for Carbon, Sulfur, and Acid-Base Characteristics
E2242 Test Method for Column Percolation Extraction of Mine Rock by the Meteoric Water Mobility Procedure
D5744 Test Method for Laboratory Weathering of Solid Materials Using a Humidity Cell
E691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method

3. Terminology

3.1 *Definitions:*

3.1.1 *acid-producing potential, AP, n*—maximum potential for a solid material sample to produce acidic effluent can be determined based on the total sulfur present in the sample.

3.1.1.1 *Discussion*—It is assumed that this sulfur is present as iron sulfides (for example, pyrite) **4**). This assumption leads to overestimation of the acid-producing potential of samples containing non-ferrous sulfide minerals such as galena (PbS) or non-acid-producing sulfur-bearing minerals such as gypsum ($CaSO_4$). The AP is commonly converted to the amount of calcium carbonate required to neutralize the resulting amount of the acidic effluent produced by the oxidation of contained iron sulfide minterals; it is expressed as the equivalent tonnes of calcium carbonate per 1000 tonnes of solid material (**3**). The AP is, therefore, calculated by multiplying the percent of sulfur contained in the material by a stoichiometric factor of 31.2 (**5**).

3.1.2 *interstitial water, n*—residual water remaining in the sample pore spaces at the completion of the fixed-volume weekly leach.

3.1.3 *leach, n*—weekly addition of water to solid material that is performed either dropwise or by flooding for a specified time period.

3.1.4 *loading, n*—mass of a chemical species, which is the product of the species concentration and the mass of the weekly leachate collected.

3.1.5 *mill tailings, n*—finely ground ore processing waste (commonly passing a 150-µm [100-mesh screen]) resulting from the mill processing of ore.

3.1.6 *neutralizing potential, NP, n*—capacity of a solid material sample to neutralize an acidic effluent while maintaining a drainage pH of at least 6.0. NP is expressed in terms of tonnes of calcium carbonate equivalent per 1000 tonnes of solid material (**3**).

3.1.6.1 *Discussion*—NP can be estimated using several techniques, including the following (*1*) determining the amount of calcium and magnesium carbonate in the sample; (*2*) digesting the solid material with an excess of standardized acid and back titrating with a standardized base to measure and convert the residual acid to calcium carbonate equivalents (**2, 6**); and (*3*) determining the carbonate carbon content in the sample (for example Test Method E1915 acid base classification).

3.1.6.2 *Discussion*—The AP and NP are specifically applicable to the determination of AP from mining wastes comprised of iron-sulfide and carbonate minerals. These terms may be applicable to any solid material containing iron-sulfide and carbonate minerals.

3.1.6.3 *Discussion*—Calcium plus magnesium carbonate determination generally provides a reasonably accurate NP quantification for samples in which carbonate minerals are present. Digestion and back-titration techniques generally overestimate the capacity of mine waste samples to neutralize acid while maintaining drainage pH $\geq$ 6.0. These techniques can yield negative values if there is excess soluble acidity on the sample. Carbonate-carbon determinations will overestimate the capacity of mine-waste samples to neutralize acid if they contain metal carbonate minerals that are not net neutralizing (for example, iron carbonates such as siderite [$FeCO_3$] (**7**).

3.1.6.4 *Discussion*—AP and NP comprise most acid-base classifications and these two components have historically been determined by several different analytical methods (**7**). However, only one acid-base classification is currently an ASTM standard, Test Method E1915. Test Method E1915 uses either pyrolysis or chemical treatment of the mine-waste sample to speciate and quantify sulfide-sulfur and carbonate-carbon concentrations, which are expressed as acid-generating potential (AGP) and acid neutralizing potential (ANP), respectively. Through this speciation, it provides a better estimate of acid generation than historic AP determinations in which non-ferrous and non-acid-generating sulfur minerals are present (for example, galena [$PbSO_4$] and gypsum [$CaSO_4$], respectively.

3.1.7 *run-of-mine, adj*—usage in this test method refers to ore and waste rock produced by excavation (with attendant variable particle sizes) from open pit or underground mining operations.

3.1.8 *waste rock, n*—rock produced by excavation from open pit or underground mining operations that has an economic mineral content less than a specified economic cutoff value for metallurgical processing.

4. Summary of Test Method

4.1 This laboratory-weathering procedure is designed to enhance the mass release of acidity/alkalinity, metals, and other pertinent analytes from a sample of solid material weighing at least 1000 g. This is done by providing conditions conducive to sample oxidation and then leaching the sample with a fixed-volume aqueous leach. Ratio of leach volume to sample mass is 0.5 : 1 or 1 : 1 depending upon the efficiency of sample wetting and amount of effluent required for chemical analyses. The weekly effluent produced is characterized for dissolved weathering products. This test method is performed on each sample in a cylindrical cell. Multiple cells can be arranged in parallel. This configuration permits the simultaneous testing of multiple splits of the same solid material sample, or of solid material samples each characterized by different compositions.

4.2 Two protocol options (Options A and B) comprise the test procedure, and these options differ only in the way that the oxygen is supplied to samples in the individual humidity cells. Option A protocol calls for weekly cycles composed of three days of dry air (less than 10 % relative humidity) and three days of water-saturated air (approximately 95 % relative humidity) pumped up through the sample, followed by a leach with water on Day 7. Option B protocol differs from Option A in that each cell is stored for six days under conditions of controlled and relatively constant temperature and humidity, and oxygen is supplied to the sample by diffusion (and possibly advection) of ambient air rather than by pumping. Although a test duration as short as 20 weeks may be suitable for some samples, more recent research indicates that a test duration well beyond 20 weeks may be required depending upon the objectives of the test (**8**,**9**).

5. Significance and Use

5.1 The laboratory weathering procedure will generate data that can be used to: (*1*) determine whether a solid material will produce an acidic, alkaline, or neutral effluent, (*2*) identify solutes in the effluent that represent dissolved weathering products formed during a specified period of time, (*3*) determine the mass of solute release, and (*4*) determine the rate at which solutes are released (from the solids into the effluent) under the closely controlled conditions of the test.

5.2 Data generated by the laboratory weathering procedure can be used to address the following objectives: (*1*) determine the variation of drainage quality as a function of compositional variations (for example, iron sulfide and calcium+magnesium carbonate contents) within individual mine-rock lithologies, (*2*) determine the amount of acid that can be neutralized by the sample while maintaining drainage pH ≥ 6.0 under the conditions of the test, (*3*) estimate mine-rock weathering rates to aid in predicting the environmental behavior of mine rock, and (*4*) determine mine-rock weathering rates to aid in experimental design of site-specific kinetic tests.

5.3 The laboratory-weathering procedure provides conditions conducive to oxidation of solid material constituents and enhances the transport of weathering reaction products contained in the resulting weekly effluent. This is accomplished by controlling the exposure of the solid material sample to such environmental parameters as reaction environment temperature and application rate of water and oxygen.

5.4 Because efficient removal of reaction products is vital to track mineral dissolution rates during the procedure, laboratory leach volumes are large per unit mass of rock to promote the rinsing of weathering-reaction products from the mine-rock sample. A comparison of laboratory kinetic tests with field tests has shown that more reaction products from mineral dissolution are consistently released per unit weight and unit time in laboratory weathering tests (**9**). For example, sulfate release rates observed in laboratory tests of metal-mine rock have been reported to be 3 to 8 times those for small-scale field test piles of Duluth Complex rock (**10**), and from 2 to 20 times those for small-scale field test piles of Archean greenstone rock (**11**). A greater increase is anticipated when laboratory rates are compared with field rates measured from operational waste-rock piles.

5.5 Fundamental assumptions governing Options A and B of the procedure:

5.5.1 **Option A**—An excess amount of air pumped up through the sample during the dry- and wet-air portions of the weekly cycle reduces the potential for oxidation reaction rates being limited by low-oxygen concentrations. Weekly leaches with low ionic strength water promote the removal of leachable mineral dissolution products produced from the previous week's weathering cycle. The purpose of the three-day dry-air portion of the weekly cycle is to evaporate some of the water that remains in the pores of the sample after the weekly leach without totally drying out the sample. Consequently, sample saturation is reduced and air flow is enhanced. During the dry-air portion of the cycle, the oxygen diffusion rate through the sample may increase several orders of magnitude as compared to its diffusion rate under more saturated conditions of the leach. This increase in the diffusion rate under near-dryness conditions helps promote the oxidation of such constituents as iron sulfide. Additionally, evaporation from the three days of dry air increases pore water cation/anion concentrations and may also cause increased acidity (for example, by increasing the concentration of hydrogen ion generated from previously oxidized iron sulfide). Increased acid generation will enhance the dissolution of additional sample constituents. As evaporation continues, the remaining water may become over-saturated with respect to some mineral phases, consequently causing them to precipitate. Some precipitated minerals are potential sources of acidity when re-dissolved (for example, melanterite, $FeSO_4 \cdot 7H_2O$; and jarosite, $K_2Fe_6(OH)_{12}(SO_4)_4$). Compared to the three days of dry air where the pore-water mass decreases over time, the wet (saturated)-air portion of the weekly cycle helps maintain a relatively constant mass of pore water in the sample (**12**). This may help promote some diffusion of weathering products (for example, re-dissolved precipitation products) in the remaining pore water without totally saturating the sample and adversely affecting oxygen diffusion.

NOTE 1—Under idealized conditions (that is, infinite dilution in air and water), published oxygen diffusion rates in air are five orders of magnitude greater than in water ($0.178\ cm^2\ s^{-1}$ versus $2.5 \times 10^{-5}\ cm^2 \cdot s^{-1}$ at 0 and 25°C, respectively) (**13**).

5.5.2 **Option B**—In contrast to Option A, Option B protocol does not include dry air or wet air introduction to the humidity cells during the weekly cycle. Instead, Option B requires that temperature and relative humidity be maintained within a constant range by storing the cells in an environmentally controlled enclosure during the 6 days following the weekly 500- or 1000-mL leach. Consequently, oxygen is delivered to the cells by diffusion (and possibly advection) of ambient air, rather than by pumping. Because it lacks a dry-air cycle, more interstitial water is retained in the Option B sample than in the Option A sample during the weekly cycle. Furthermore, the interstitial water content for Option B is more constant than that in Option A during the weekly dry-air cycle. In addition, the interstitial water content for Option B is less variable over the course of testing than that in Option A (**14**).

5.6 This test method has been conducted on metal-mine wastes to classify their tendencies to produce acidic, alkaline, or neutral effluent, and to measure the concentrations of selected inorganic components leached from the waste (**2-3, 14-16**).

NOTE 2—Interlaboratory testing of this method to date has been confined to mine waste rock. The method has not been tested for applicability to metallurgical-processing waste. Although the method has been applied by some practitioners to finely ground metallurgical-processing wastes such as mill tailings, those materials were not included in the interlaboratory testing of the method. Consequently, modifications of this method might be necessary to deal with problems associated with finely ground materials, which would make this method as written, inappropriate for kinetic testing of finely ground materials. For kinetic testing of finely ground materials, please refer to the biological acid production potential method in the appendix of Test Methods E1915 or other kinetic methods accepted by the regulatory jurisdiction.

5.7 The following are examples of parameters for which the scheduled weekly, semi-monthly, or monthly collected effluent may be analyzed (see 11.5.2 for suggested effluent collection frequency):

5.7.1 pH, Eh (oxidation/reduction potential), and conductivity (see Test Methods D1293, Practice D1498, and Test Methods D1125, respectively, for guidance);

5.7.2 Alkalinity/acidity values (see Test Methods D1067 for guidance);

5.7.3 Cation and anion concentrations;

5.7.4 Metals and trace metals concentrations.

5.8 An assumption used in this test method is that the pH of each of the leachates reflects the progressive interaction of the interstitial water with the acid-generating or acid-neutralizing capacity, or both, of the solid material under specified laboratory conditions.

5.9 This test method produces leachates that are amenable to the determination of both major and minor constituents. It is important that precautions be taken in sample collection, filtration, preservation, storage, and handling to prevent possible contamination of the samples or alteration of the concentrations of constituents through sorption or precipitation.

5.10 The leaching technique, rate of leach water addition, liquid-to-solid ratio, and apparatus size may not be suitable for all types of solid material.

5.11 Notable differences have been observed between Option A and Option B protocols:

5.11.1 Water retention in the solid-material sample between weekly leaches is more variable for Option A than in Option B; for Option A, standard deviations from the mean water retention can range from 20 to 60 % of the mean value; comparable values for Option B have been reported at less than 9 % (**14**).

5.11.2 Greater water retention in Option B cells may favor dissolution of, and consequent acid neutralization by, magnesium-bearing minerals; increased retention may facilitate transport of acidic reaction products from iron-sulfide minerals to magnesium-bearing minerals (**14**).

5.11.3 Comparisons of sulfate mass release from the same sample subjected to Option A and Option B protocols indicate no significant difference in sulfate concentration as a result of water-retention variation between protocols (**14**). This suggests the increased water retention of Option B does not limit oxygen diffusion to the extent that sulfide mineral oxidation rates are reduced (**14**). However, samples containing greater than 7 % sulfur have not as yet been subjected to comparable Option A and Option B protocol studies.

NOTE 3—Examples of products from the test include the following: (*1*) effluent pH, acidity/alkalinity, and specific conductance; (*2*) cumulative mass release of individual solutes; and (*3*) release rates for individual solutes (for example, the average release of µg sulfate/g of solid material sample/week). The dissolution time required for depletion of estimated NP and the subsequent duration of acid generation can be estimated using the values generated in items (*2*) and (*3*) above (**15**).

6. Apparatus

Options A and B:

6.1 *Humidity Cell*—A modified column constructed of materials suitable to the nature of the analyses to be performed (see Practices D3370 for guidance). Multiple humidity cells can be arranged in an array to accommodate the simultaneous laboratory weathering of different solid material types (Fig. 1). Two different sets of humidity cell dimensions are used to accommodate particle size differences present in the solid material:

6.1.1 Cells having suggested dimensions of 10.2-cm (4.0-in.) inside diameter (ID) by 20.3-cm (8.0-in.) height can be used to accommodate coarse solid material samples that have been either screened or crushed to 100 % passing 6.3 mm (0.25 in.).

6.1.2 Cells with suggested dimensions of 20.3-cm (8.0-in.) ID by 10.2-cm (4.0-in.) height can be used to accommodate solid material samples that pass a 150-µm (100-mesh) screen.

NOTE 4—Some coarse solid material samples may break down into finer-grained weathering products that could inhibit airflow and result in material being ejected from the cell during Option A's dry-air cycle. Consequently, use of the 20.3-cm ID cell rather than the 10.2-cm ID cell may be more appropriate (**9**). It should be noted that there are no published ruggedness testing results for this cell.

NOTE 5—For Option A, if samples are to be tested in the 20.3-cm ID cell, the air-entry port to the 20.3 cm ID cell needs to be moved from beneath the sample to just slightly above the sample so that air flow is directed across the sample surface rather than attempting to infiltrate the sample up through its bottom surface. The air-exit port is centered in the lid.

6.1.3 For cell wall thicknesses, 0.635-cm (0.25-in.) and 0.318-cm (0.125-in) cm thickness have been used for Options A and B, respectively.

6.1.4 A perforated disk (constructed of materials suitable to the nature of analyses to be performed), approximately

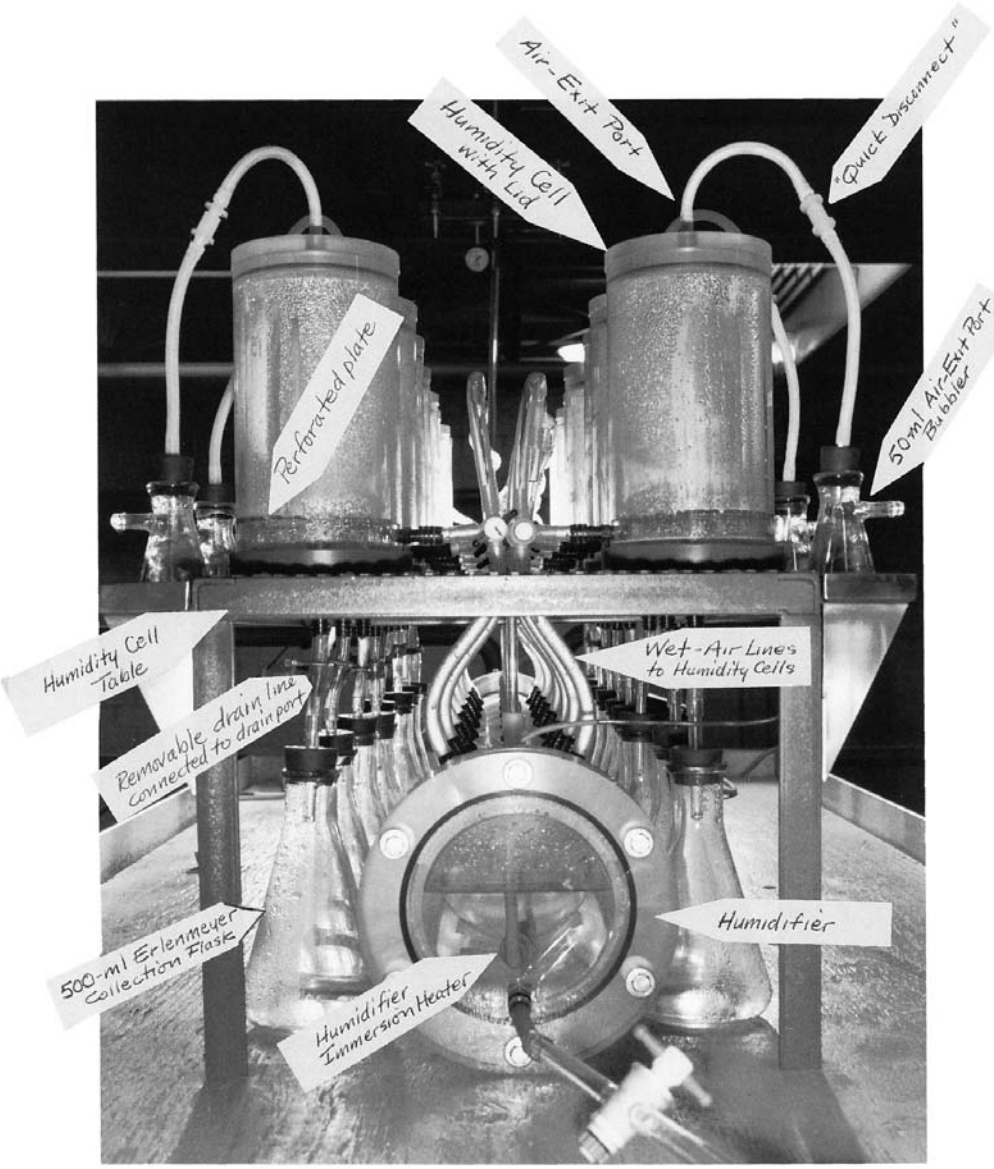

FIG. 1 Side View of 16-Cell Array (Option A)

0.315-cm (0.125-in.) thick, with an outside diameter (OD) suitable to the suggested vessel ID (6.1.1 and 6.1.2) is elevated approximately 1.25 cm (0.5 in.) above the cell bottom to support the solid material sample (see Fig. 1).

6.1.5 For Option A, the cell lid and base are 1.27 cm (0.5 in.) thick and machined so they each include a lip and plug; the plug portion fits into the ID of the humidity-cell top/bottom, and the lip fits over the rim of the cell opening. A hole is drilled in the center of the lid and base and tapped to accommodate a barbed NPT fitting for attachment to flexible tubing. The tubing from the lid leads to the air-exit port bubbler described in 6.19 and 6.20. The tubing from the base drains into a collection vessel.

NOTE 6—Lids for Option A can have an "O"-ring seal installed (machined into the plug surface) if air leakage makes it difficult to maintain constant airflow among individual cells. Both the "O"-ring seal and the air-exit port bubbler (described in 6.20) have been helpful in maintaining airflow through individual cells of a multiple cell array during the dry- and wet-air portions of the weekly cycle. However, flow rates may still differ somewhat from cell to cell because of porosity differences between samples of differing particle-size distribution.

6.1.6 Lids for Option B do not require a barbed NPT fitting. The centered hole in the Option B lid is left open to allow for exchange of ambient air during the six-day portion of the weekly cycle. A hole is drilled in the center of the base and tapped to accommodate a barbed NPT fitting. Leachate from the cell drains directly through this fitting into a collection vessel.

NOTE 7—The cell and particle size dimensions described above are those used commonly for assessing the potential of waste-rock samples associated with metal-mining operations to produce acidic effluent. A "shoe box"-shaped cell design with similar dimensions is preferred by some researchers (**6**).

6.2 *Separatory-Funnel Rack,* capable of holding 500-mL or 1-L separatory funnels above the humidity cells.

6.3 *Filter Media,* such as a 12-oz/yd^2 polypropylene felt characterized by 22-µm (0.009-in.) diameter filaments. The media should be able to transmit dry air at a rate of 20 to 30 cfm (see Test Methods D276 and D737 for guidance).

NOTE 8—Caution must be used in the selection of filter media materials since they may affect the effluent pH and chemistry adversely. Both pyrex wool and quartz wool retain as much as 10 to 15 g of water per g of wool (retained water tends to re-humidify the dry-air cycle to as much as 85 % relative humidity). Additionally, pyrex wool causes the neutral effluent pH to be raised by as much as 2 pH units due to leaching of the wool (**11**). In addition, pyrex (borosilicate) can contribute boron if this is a constituent of interest.

6.4 *Two Riffle Splitters,* with 0.63-cm (0.25-in.) and 2.5-cm (1.0-in.) wide riffles, respectively; the riffle splitter is a commonly used device for obtaining representative splits of dry, free-flowing granular materials.

6.5 *Laboratory Balance,* capable of weighing to 0.1 g.

6.6 *Analytical Balance,* capable of weighing to 1.0 mg.

6.7 *Screen,* 6.3 mm (0.25 in.).

6.8 *Screen,* 150 mm (100 mesh).

6.9 *Drying Oven*—Any thermostatically controlled drying oven capable of maintaining a steady temperature of 40 ± 2°C.

6.10 *pH Meter*—Any pH meter with readability of 0.01 units and an accuracy of ±0.05 units at 25°C; two-channel operation (that is, pH and Eh) is desirable.

6.11 *Conductivity Meter,* capable of reading in micromhos (microseimens); calibrate at 25°C.

6.12 *Separatory Funnel,* 500 mL or 1 L, one per each humidity cell.

6.13 *Collection Vessel (vessel such as an Erlenmeyer flask or Nalgene bottle),* 500 mL or 1 L, one per each humidity cell.

6.14 *Volumetric Flask,* 500 mL or 1 L.

Option A:

6.15 *Digital Hygrometer/Thermometer,* with a relative humidity range of 5 to 95 %, and temperature range of -40 to 104°C (-40 to 220°F).

6.16 *Cylindrical Humidifier,* with suggested dimensions of 12.1-cm (4.75-in.) ID by 134.6-cm (53.0-in.) length. The following associated equipment are needed to provide saturated air for the three-day wet-air portion of the weekly cycle:

6.16.1 A thermostatically controlled heating element to maintain the water temperature at 25°C during the wet-air cycle.

6.16.2 An aeration stone (similar to aquarium-aeration equipment) or commercially available gas dispersion fritted cylinders or disks to bubble air into the humidifier water.

6.17 *Flow meter,* capable of delivering air to each humidity cell at a rate of approximately 1 to 10 L/min/cell.

6.18 *Oil/Water Trap,* 0.01-µm, for inclusion in the feed-air line.

6.19 *Air-Exit Port Bubbler*—A 50-mL Erlenmeyer flask with a rubber stopper containing a vent and air-inlet tube (Fig. 1). The bubbler is connected to the air exit port in the humidity cell lid with flexible tubing. This helps maintain similar positive air pressure throughout all of the humidity cells.

6.20 *Flexible-Tubing Quick Disconnect*—A fitted, two-piece connection placed in the middle of the air-exit port flexible tubing so that the bubbler can be disconnected from the humidity cell to facilitate the measurement of air flow and relative humidity.

6.21 *Desiccant Column,* 5.1-cm (2-in.) ID by 50.8-cm (20-in.) length, plastic or glass cylinder capped on both ends (one cap should be removable for desiccant replacement), with an air inlet port on the bottom and an air exit port on the top.

6.22 *Dry Air Manifold*—A cylindrical manifold constructed from 2.25-in. ID schedule 40 acrylic plastic tubing, 28 in. long and fitted with 16 NPT barbed fittings. The airline exiting the desiccant column is routed directly to the cylinder, which then supplies dry air to each cell through an airline attached to its corresponding NPT barbed fitting. The cylindrical manifold fits atop the separatory-funnel rack.

Option B:

6.23 *Environmentally-Controlled Enclosure*—Any enclosure suitably sized to accommodate the number of samples being tested and associated equipment, and capable of maintaining consistent humidity (±10 %) and temperature (±2°C).[5]

6.23.1 *Temperature Control*—Any commercially-available heater capable of maintaining consistent temperature within the enclosure.

6.23.2 *Humidity Control*—Any commercially-available humidifier and dehumidifier capable of maintaining consistent humidity within the enclosure.

6.23.3 *Instruments to Measure Temperature and Humidity*—Any commercially-available manual or digital hygrometer/thermometer (see 6.15). Temperature should be readable to at least 1°C and relative humidity to 1 %.

6.23.4 *Fan*—Any commercially-available fan to provide air circulation within the enclosure.

7. Reagents

7.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available.[6]

7.2 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean reagent water as defined by Type III at 18 to 27°C conforming to Specification D1193. The method by which the water is prepared, that is, distillation,

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[5] The tolerance ranges for humidity and temperature are the range of differences of maximum and minimum values from the mean of the respective data.

[6] *Reagent Chemicals, American Chemical Society Specifications*, American Chemical Society, Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see*Analar Standards for Laboratory Chemicals*, BDH Ltd., Poole, Dorset, U.K., and the *United States Pharmacopeia and National Formulary* , U.S. Pharmacopeial Convention, Inc. (USPC), Rockville, MD

ion exchange, reverse osmosis, electrodialysis, or a combination thereof, should remain constant throughout testing.

7.3 *Purity of Air*—The feed air line shall contain a 0.01-µm oil/water trap in advance of the flow meter.

8. Sampling

8.1 Collect the samples to be tested using available sample methods developed for the specific industry (see Practices D75 and E877, Guide D420, Terminology D653, and Test Methods D2234/D2234M).

8.2 The sampling methodology for materials of similar physical form shall be used where no specific methods are available.

8.3 The amount of material recommended to be sent to the laboratory should be sufficient to provide 8 to 10 kg of bulk sample for splitting, analysis, and testing (see 9.3).

NOTE 9—Additional information on theory and methods for obtaining representative samples is contained in Pitard (**16**).

8.4 To prevent sample contamination or constituent loss prior to testing, store the samples in closed containers that are appropriate to the sample type and desired analyses (see Guide D420 for guidance).

8.5 The time elapsed between sample collection and subsequent humidity cell testing should be minimized to reduce the amount of sample pre-oxidation (see Practices D3370 for guidance). Report the length of time between sample collection and testing.

9. Sample Preparation

9.1 Air dry as-received bulk samples of solid material to prevent the additional oxidation of reactive minerals or compounds. If air-drying is not practicable, oven dry the solid material at a maximum temperature of 40°C for 24 h, or until a constant weight is reached.

NOTE 10—Oven drying at temperatures above 40°C may introduce chemical and physical changes in certain mineral species comprising the sample (**9**). These potential changes should be evaluated and accounted for in the analysis of the test data.

9.2 After reserving any coarse material needed for Method E2242-02 (Meteoric Water Mobility Procedure) or other possible testing and analyses, screen the air-dried bulk samples through a 6.3-mm (0.25-in.) screen in accordance with Test Method E276. Crush any oversize material so that 100 % passes the screen. For particles finer than 15.2 cm (6 in.), the generation of excessive fines can be limited by stage crushing the oversize material in three steps: *(1)* large jaw crusher set at 1.92 cm, *(2)* small jaw crusher set at 0.95 cm, and *(3)* roll crusher set at 0.64 cm. After each of the first two crushing steps, the -0.64 cm fraction is collected and the oversize is passed to the next crushing phase.

9.2.1 **Caution**: Crushing a bulk sample so it passes a 6.3-mm (0.25-in.) screen may change the character of the sample by artificially increasing liberation and consequent surface areas of acid-producing and acid-consuming minerals contained in the + 6.3-mm (0.25-in.) material. A suggestion for avoiding this problem is to segregate the − 6.3-mm (0.25-in.) fraction by screening rather than crushing, and to test that fraction according to the protocol and equipment described in this test method. The + 6.3-mm (0.25-in.) material can either be stage-crushed (as in 9.2), or tested separately. For example, column testing could be conducted, although no standard protocol has been established for this testing. Samples from the drill core and cuttings also present material sizing problems, which must be considered when interpreting drill core and cuttings laboratory weathering data. The drill core must be crushed to − 6.3-mm (0.25-in.) to fit the cell described in this test method. The resulting size distribution from crushing will differ from that of run-of-mine due to differences in fracture patterns inherent to blasting practices that produce run-of-mine material. By contrast, drill cuttings size fractions are commonly less than 6.3-mm (0.25-in.) due to the rotary-percussive nature of obtaining the sample. The effects of particle size distribution changes resulting from the more finely crushed sample or from rotary-drill cuttings should be considered in the interpretation of data. In particular, particle size reduction will increase specific surface area of acid-generating and acid-neutralizing minerals and is likely to increase liberation of these minerals. Both of these effects will tend to increase the surface area of these minerals available for reaction. If this increase is biased towards either acid-generating or acid-neutralizing minerals, the balance of acid-generating and acid-neutralizing reactions will shift.

9.3 Mix and divide the bulk sample to obtain a representative test unit with a weight in the range of 8 to 10 kg, using a riffle splitter with 1-in. (2.54-cm) chutes (perform in accordance with Test Method E877, Sampling and Preparation Procedure-Riffling). Divide the test unit into eight nominal 1-kg test specimens. Store each test specimen in a resealable plastic bag (to prevent continued atmospheric oxidation of sulfide mineral-bearing samples, samples could be vacuum sealed or refrigerated).

NOTE 11—The dried sample should be mixed through the riffle splitter at least once before making any splits; recombine the splits resulting from the sample mixing exercise by pouring individual splits either over each other or through the splitter again. Once the actual split is made, it is wise to re-mix it (according to the above procedure) prior to making the next split. Mixing the sample through the riffle splitter may still result in segregation of the sample. If segregation persists, use of a rotary sample divider is advised.

9.4 *Head-Sample Analysis*—Select one 1-kg test specimen at random, and crush the dried test specimen so that at least 95 % passes a 1.7-mm (10-mesh) screen, in accordance with Test Method E276.

9.4.1 Divide the crushed test specimen in half twice, using a riffle splitter with 6.35-mm (0.25-in.) chutes (in accordance with Test Method E877, Sampling and Preparation Procedure-Riffling), and select a 250-g subsample at random for head sample analysis.

9.4.2 Transfer the selected 250-g subsample to a ring and puck grinding mill and grind to a nominal 95 % passing a 150-µm (100-mesh) screen, in accordance with Test Method E276. Use the pulverized subsample to perform the following determinations: (*1*) total sulfur, sulfide, and carbonate analyses in accordance with Test Method E1915, (*2*) whole rock and trace-element chemistry analyses, and (*3*) mineral characterization to identify and quantify the different mineral species comprising the sample.

NOTE 12—Because accurate estimation of a sample's capacity to neutralize acid (NP) requires identification of carbonate mineral speciation (that is, calcite, dolomite, ferroan dolomite, siderite, and so forth), and quantification of calcium and magnesium content in iron-bearing minerals (that is, ankerite, ferroan dolomite, and siderite, and so forth), these determinations are strongly recommended. It is also recommended that sample whole-rock chemistry and mineralogy be compared to ensure that chemistry is consistent with mineralogy and vice versa. Additionally, leach extraction testing of the pre-test sample, compared with leach extraction testing of post-test sample, may be beneficial in determining the extent of solutes released by mineral dissolution and subsequently sequestered in secondary solid phases during testing **(17)**.

9.5 *Screen-Fraction Analysis*—Select one 1-kg test specimen at random, and determine the particle size distribution in accordance with Test Method E276. Sieve openings of 6, 10, 28, 35, 48, 100, 200, and 270 Tyler mesh are suggested.

9.6 The following analyses are recommended:

9.6.1 Determine the total sulfur, sulfide, and carbonate contents of individual size fractions in accordance with Test Method E1915. Whole rock or trace element chemical analyses, or both, may also be performed on these fractions.

9.6.2 Determine the extent of acid-generating (for example, iron sulfide, iron sulfate) and acid-neutralizing (for example, calcium carbonate, magnesium carbonate) mineral liberation of the individual size fractions.

NOTE 13—Paragraphs 9.6.1 and 9.6.2 are recommended as best practice to quantify the amount of sulfide and carbonate minerals present in each fraction and their degree of liberation within these fractions. The objective of the analyses described in 9.6.1 and 9.6.2 is to aid the user in correlating drainage quality with solid-phase composition by providing more detailed description of the exposed surface areas of acid-producing and acid-neutralizing minerals. These surface areas strongly affect the rates of acid production and neutralization. With regard to the applications of testing presented in 5.1 and 5.2, the analyses described will help: (*1*) identify whether the material being tested will produce acidic, alkaline, or neutral effluent; (*2*) determine the variation of drainage quality as a function of solid-phase composition; (*3*) measure the amount of estimated NP accessible in the solid; and (*4*) in general, aid in predicting the environmental behavior of the solid (see Refs 8, 18, and 19). To perform all of the determinations described in 9.6.1 and 9.6.2 a larger mass of sample material may be required than the stated 1 Kg **(18)**.

9.7 Select one 1-kg test specimen at random for use in the laboratory weathering test method. Divide the test specimen into four nominal 250-g subsamples using the riffle splitter with 25.4-mm (1-in.) chutes, and label and store in resealable plastic bags until it is time to load the humidity cells.

NOTE 14—If the leach volume from the 1 kg sample mass does not provide sufficient effluent to meet analytical needs, a 2-kg test specimen could be used as long as the 0.5 : 1 or 1 : 1 leach-volume to sample-mass ratio is maintained. Split the sample between two cells. Record and then combine recovered volumes of the weekly effluent from both cells. Also record the combined volume.

9.8 Reserve the remaining test specimens for replicated testing or to resolve disputed results (recommend sample preservation by vacuum seal or refrigerated storage).

10. Apparatus Assembly

10.1 **Option A**—The humidity cells are table-mounted at a height sufficient to accommodate the placement of both the humidifier and one collection flask for effluent collection from the bottom of each cell (Fig. 1). During the water-saturated and dry-air portions of each weekly cycle, feed air is metered to the bottom of each cell (or midway up the side in cells designed for minus 100 mesh material) at the selected rate (1 to 10 L/min). Feed air for the three-day dry-air portion is routed first through a desiccant column and then to each of the cells through a dry-air manifold (Fig. 2, Fig. 3). Feed air for the water-saturated air portion is routed through a water-filled humidifier by means of aeration stones or gas dispersion fritted cylinders/disks, and then to each humidity cell (Fig. 2). If necessary, a water-bubbling vessel can be attached to the air-exit port of each humidity cell lid to maintain constant airflow among the individual cells (Fig. 1).

10.2 **Option B**—The humidity cells are mounted on a rack of sufficient height to accommodate placement of vessels for collection of effluent from the bottom of each cell. The upper portion of the rack doubles as a separatory-funnel rack, and is of sufficient height to accommodate placement of the funnel spigot above the humidity-cell lid. A simple rack of wood construction is shown in Fig. 4. Note that holes are drilled in the humidity-cell shelf to accommodate the barbed fitting (drain) that is centered at the bottom of each cell. Unlike the Option A apparatus, no air plumbing is required. Unlike Option A, Option B cells are stored in an enclosure in which temperature and humidity are controlled during the 6 days following the leach. Shelves for cell storage and space for temperature- and humidity-control equipment are required in the enclosure.

10.3 *Option A and B*—A separatory funnel rack is mounted on the table that holds the cells if the weekly water leach is applied dropwise (drip leach). Multiple separatory funnels (one for each cell) are held in the rack during the drip leach that is performed on the seventh day of each weekly cycle (Fig. 2, Fig. 4). The separatory funnel can be used to meter the required water volume slowly down the sides of the cell wall until the sample is flooded if the weekly leach is to be a flooded leach.

11. Procedure

Options A and B:

11.1 *Cell Loading:*

11.1.1 If more than one humidity cell is used at one time, label each with a sequential number, and use the same number for the matching collection vessel.

11.1.2 Weigh each humidity cell (without its lid) and each collection vessel; record the tare weights of each to the nearest 0.1 g.

11.1.3 Cut the filter media (such as 12-oz/yd^2 polypropylene described in 6.3) to the humidity cell's inside diameter dimensions so that it fits snugly yet lies flat on the perforated support.

11.1.4 Re-weigh the humidity cell, and record the resulting tare to the nearest 0.1 g; the original cell tare (11.1.2) minus the new cell tare is the weight of the filter media.

11.1.5 Transfer the contents from each of the four bags containing the 250-g samples (9.7) into the humidity cell (Fig. 5). Prior to the transfer, mix the contents of each bag by gentle rolling to eliminate possible stratification that may have occurred during sample storage.

FIG. 2 Front View of 16-Cell Array (Option A) with Separatory Funnel Rack

11.1.6 Re-weigh the loaded cell, and record the weight to the nearest 0.1 g; the loaded cell weight minus the combined cell and filter-media tare weight is the weight of the sample charge.

11.2 *First Water Leach:*

11.2.1 The leach application method for the first water leach (and all subsequent weekly leaches) is the flood leach.

NOTE 15—Both drip and flood-leach alternatives were provided in the original D5744-96 method. However, a subsequent interlaboratory study (20) that compared drip with flood-leach alternatives determined that there was little difference in results from the drip and flood-leach applications. Results from the two laboratories participating in the study showed that the drainage pH values for the end of rate periods were within 0.2 units of the mean, and sulfate release rates were consistently within 10 % of the mean. Consequently, because there was little difference between results from the two leach alternatives, and to simplify the method protocol, the flood-leach alternative is designated as the preferred water-leach application method. This does not preclude use of the drip-leach application if the flood-leach application becomes impracticable.

11.2.2 The first flooded leach, designated as the Week 0 leach, initiates the humidity cell test and establishes the starting or initial characteristics of the leachate. Because the laboratory-weathering test is made up of seven-day cycles, the sample is always leached on the same day of the week (for example, Monday). On the scheduled leaching day for Week 0, if removal of pre-leaching oxidation products is identified as a necessary component of the test objective, perform three consecutive leaches (using either a 500-mL or 1-L volume per leach) (12, 14). Report the rationale for using the three consecutive leaches. Otherwise, use a single 500 mL or 1-L volume leach. Initial selection of either the 500-mL or 1-L volume of water is dependent upon the pore volume desired, quantity of solution required for analytical purposes, and the detection limits desired or available for analyses. However, after the Week 0 leach (that is, initial 500-mL or 1-L leaches), the selected weekly volume (500 mL or 1 L) may need to be changed because the volume originally selected is either determined to be insufficient to flush the reaction products from the sample, or rates of release may be too slow to produce detectable solutes of interest. Consequently, a weekly review of the drainage quality data from the selected weekly leach volume is recommended to ensure that the selected volume is adequate for the objectives of the test. Report any changes in the weekly leach volume and document the rationale for the change.

NOTE 16—Prior studies of humidity-cell tests on mining wastes showed that cation and anion loadings were commonly high in the leachate recovered from the Week 0 500-mL leach. These high loadings were due to the dissolution of pre-existing soluble oxidation salts present in the sample prior to sample collection. The first 3 to 5 weeks of weekly 500-mL leaches were required to flush these pre-existing salts from the samples being studied (15). The objective of performing three consecutive leaches during the Week 0 leach is to reduce the influence of pre-existing salts on succeeding weekly drainage quality, if this influence is identified

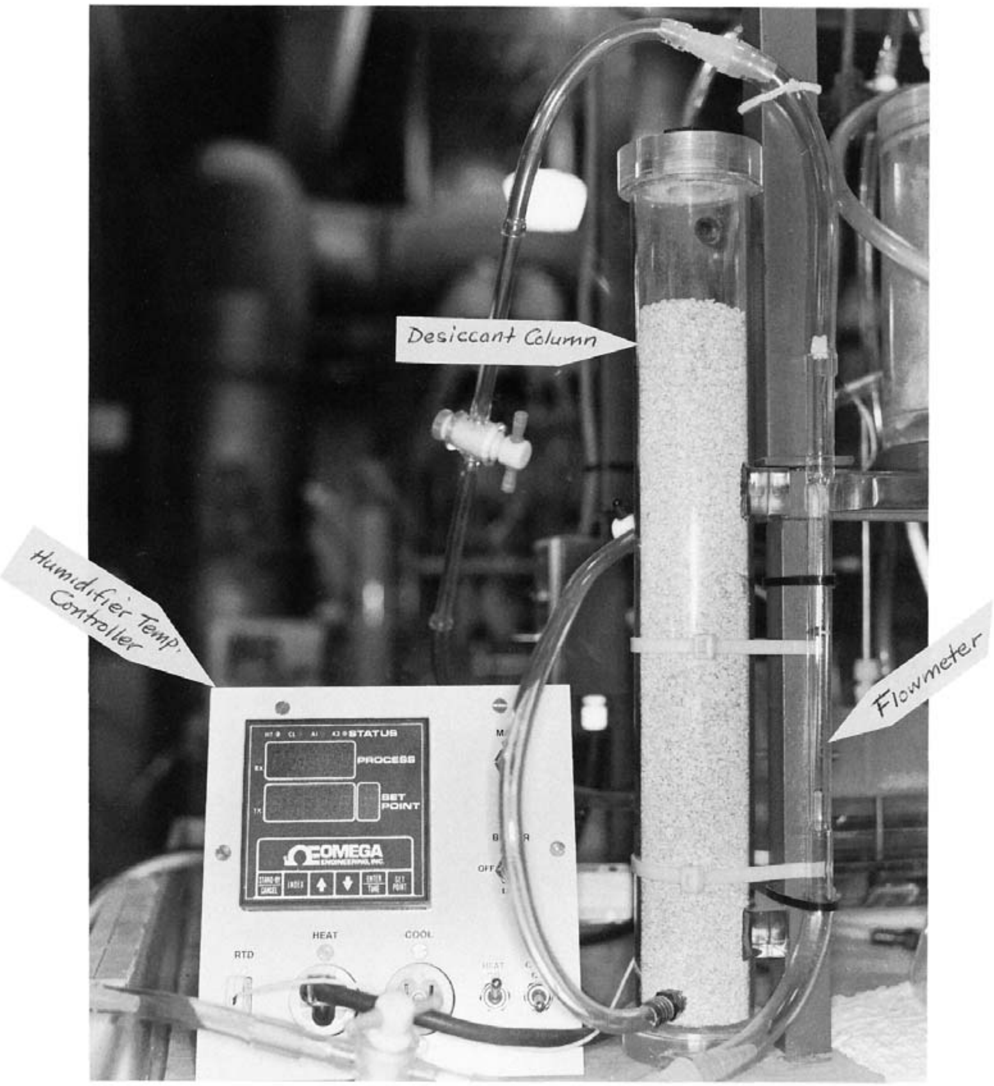

FIG. 3 Detail of Desiccant Column and Flow Meter

as a critical component of the test objective. It is possible that some pre-existing salts may continue to remain in the sample despite the three consecutive Week-0 leaches. Therefore, caution should be used in interpreting the rate of mass release observed during the first 3 to 5 weeks of the test.

NOTE 17—If objectives of the test require more effluent volume for chemical analyses than is provided by either the 500 or 1000 mL leach, run two cells of the same material as described in Note 14 while maintaining the leach-volume to sample-mass ratio as 0.5 : 1 or 1 : 1.

NOTE 18—Assuming a sample density of 2.6 to 3.0 g/cm^3 and a porosity of 40 to 45 %, a 500-mL leach represents approximately 1.5 to 2 pore volumes and a 1000-g sample would fill a 4-in. ID humidity cell to a height of approximately 2.5 to 3.5 in. above its perforated base.

11.2.3 Fill a separatory funnel for each cell with de-ionized water using a volumetric flask. Set each separatory funnel above its corresponding cell, place the collection vessel beneath its corresponding cell, and remove the cell lid. Plug the humidity cell air-entry and effluent-drainage ports with removable waterproof caps to prevent premature drainage of the flooded water leach (see Fig. 4). Using the separatory funnel, discharge the selected water volume slowly down the sides of the cell wall until the sample is flooded. This application method reduces hydraulic agitation of the sample surface commonly caused by pouring liquid from an open-mouthed vessel. Alternatively, flooding may be accomplished by any application apparatus (for example, a peristaltic pump) that supplies the selected volume of leachant at a reasonable rate without causing agitation and suspension of the finer fractions contained in the sample charge.

11.2.4 Allow the flooded cell to sit for a period of at least 1 h before draining the leachate into the collection vessel by removing the drainage-port plug. The leach time commences after all of the leachant has been placed in the cell. The solid material sample should be saturated and covered with leachant to a depth sufficient to maintain sample saturation. In testing mining wastes, the observed depth of leachant cover from a 500-mL flooded leach performed in 10.2-cm (4.0-in.) ID cells is approximately 2.5 cm (1.0 in.).

NOTE 19—Duration of the flooded leach reported by researchers and

NOTE 1—Cells are placed in this configuration only during the leach portion of the weekly cycle. Upon completion of the leach and subsequent effluent collection, cells are covered and placed in a separate room where temperature and humidity are controlled. Note: The side ports visible on the humidity-cell bases are plugged air entry ports (these cells were originally constructed for use in Option A – the drainage ports are centered in the bottom of each cell and are fitted with barbed NPT fittings that project through corresponding holes drilled in the cell-support base board).

FIG. 4 End View of 16-Cell Array (Option B) with Separatory Funnel Rack

practitioners is variable. Morin and Hutt (**9**) recommend 2 h for – 6.3 mm samples (for example, waste rock) and at least 4 h for – 150 mm samples (for example, tailings). Lapakko and Antonson (**18**) reported pyrite oxidation rates based on sulfate mass release from weekly 10-min duration flooded leaches of 12 Archean greenstone samples. These oxidation rates were in good agreement with published rates, suggesting that sufficient oxidation-product leaching occurred within the 10-min leach residence time.

11.2.5 The following is performed once the leaching process has been completed: to reduce the effects of evaporation, and to minimize potential airborne contamination of each cell, place the lids on their corresponding cells and let the cells complete the leachate draining process for the remainder of the leaching day and overnight.

11.2.6 Disconnect the cells on the day following the leach, and weigh and record the weight of each cell and collection vessel. Set each filled collection vessel aside for leachate analyses (remember that three vessels per sample may be required to collect leachant if the test objective requires three consecutive leaches for the Week 0 leach). Measurements of effluent parameters and sample preservation procedures must be performed as soon as possible after leachate collection. Return each cell, replace the filled collection vessels with clean, preweighed collection vessels, remove the air-entry port plug, hook up all connections (Option A), and begin the weekly cycle.

NOTE 20—Some samples become less permeable over time and require longer leachant collection times that extend 2 to 4 h into the second day of the weekly cycle. Consequently, 11.2.5 recommends that the leachant collection continue overnight and parameters be measured on day two of the cycle. However, to avoid introducing excessive variability in effluent parameter measurements such as pH and Eh, Minnesota Department of Natural Resources (John Folman, MN DNR, personal communication, October 17, 2006) uses a flow-through cell connected between the cell drainage port and the sample collection bottle. This enables MN DNR to measure pH and Eh from the draining leachate during the leaching day. Consequently, it is recommended that pH and Eh be measured by the end of the leach day (if an apparatus such as a flow-through cell is not used, volumes of leachant removed for these measurements need to be recorded and added to the tally of final leachant collection volume).

NOTE 21—It is recommended that sulfate, calcium, magnesium, sodium, and potassium concentrations in the weekly leachates from mine-rock samples containing iron-sulfide minerals be monitored according to a practical schedule. Their respective concentrations are critical measurements of mass release from (*1*) iron-sulfide mineral oxidation, (*2*)

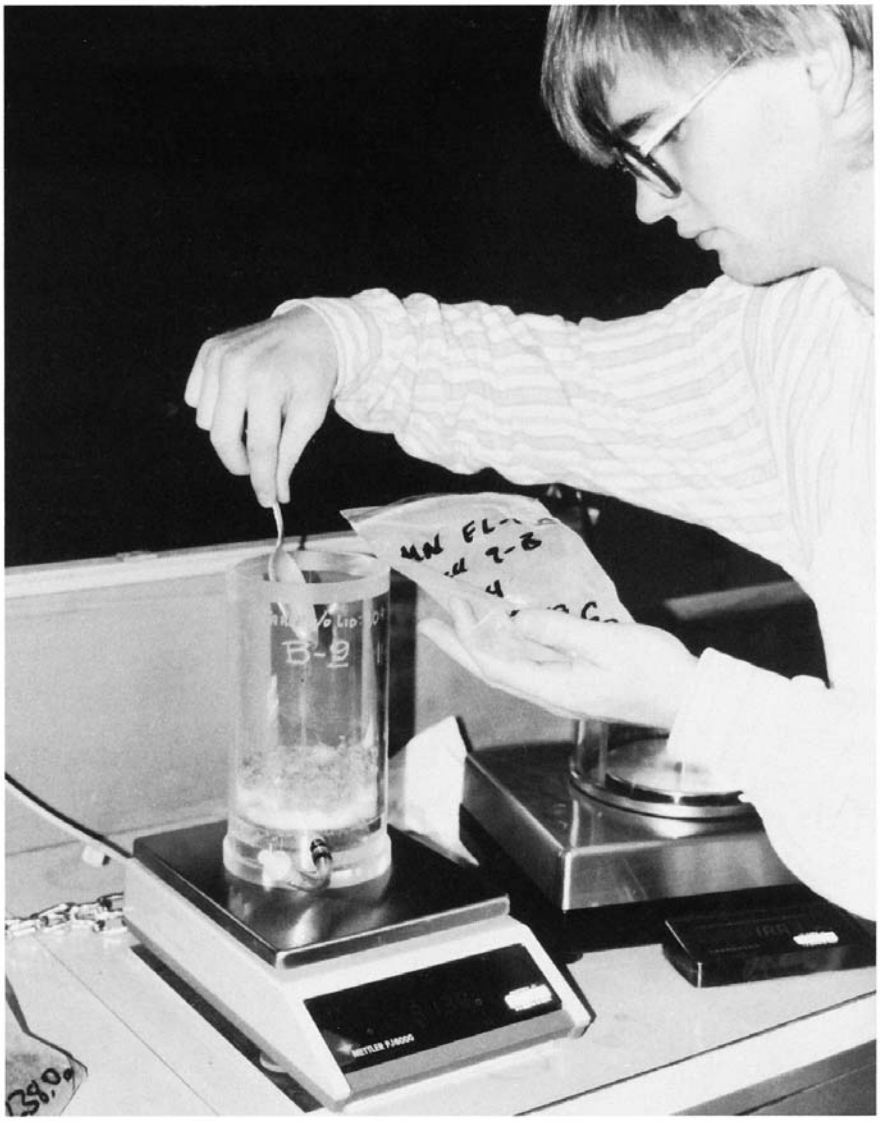

FIG. 5 Loading Humidity Cell with Filter Media and 1000-g Sample Charge

calcium- and magnesium-carbonate mineral dissolution, and (*3*) calcium, magnesium, sodium and potassium-silicate mineral dissolution. When leachate pH is ≤3.5, iron concentrations are also of interest. Additionally, trace metals present in the leachate should be monitored, and their presence could be screened by analysis (for example, ICP-MS) to identify potentially problematic leachable chemical species. A proposed monitoring schedule for parameter measurements and chemical species analyses is suggested in 11.5.1. Calculation of charge balance from the analyses of each leachant sample is also recommended to assess the accuracy of drainage-quality analyses.

11.3 *Subsequent Weekly Water Leaches:*

11.3.1 A second flooded leach (either 500 mL or 1 L) is performed on day seven of the first weekly cycle. This leach marks the end of the first weekly cycle and is designated as the Week 1 leach.

11.3.2 Subsequent flooded leaches are designated as Week 2, Week 3 ... Week *n*, and they mark the end of the weekly cycle for that numbered week. Perform each weekly leach as described in 11.2.2 – 11.2.6.

11.4 *Test Duration:*

11.4.1 The criteria for ending the testing will depend on the objective of the test, which may be site specific.

11.4.2 If the objective is to determine the relative reactivity (for example, drainage pH, rates of sulfate release) as a function of compositional variations (for example, iron sulfide and calcium+magnesium carbonate contents) within individual mine-rock lithologies for the purpose of segregating mine wastes, then a specific test duration is not prescribed. The test duration will depend on the composition of the solids and the intended application of the results. Drainage pH and rates of trace metal release are likely to vary with time, dependent on the solid composition. However, the following examples are presented as guidance to help determine a practical test duration for this objective:

11.4.2.1 If drainage pH is initially neutral, and calcium and magnesium carbonate minerals contents exceed a few tenths of one percent, their dissolution has potential to maintain drainage pH in the circumneutral range for tens of weeks despite presence of iron-sulfide minerals. For example, presence and dissolution of 1.4 weight percent calcium carbonate (calcite) maintained drainage pH from tailings, containing 6.6 weight percent pyrite, near 8 for 100 weeks. Drainage pH decreased below 6 after 122 weeks, at which time the calcite was depleted (**21**). Subsequent acid neutralization resulted from dissolution of silicate minerals. Drainage pH decreased below 4 after 136 weeks, below 3 after 313 weeks, and reached a minimum of roughly 2.3 after 320 weeks (MN DNR unpublished data). Consequently, if the minimum pH were needed to assess relative reactivity, test duration in excess of 320 weeks would be required. Further examples of determining the relative reactivity of sulfidic mine-waste lithologies in which silicate mineral dissolution accounts for the majority of acid neutralization are presented by Lapakko and Antonson (**22**).

11.4.2.2 If drainage pH is initially below 3.0 and test results are intended to: (*1*) identify mine wastes that produce highly acidic drainage; and (*2*) manage aforementioned mine wastes with such caution that applicable water quality standards are met, then the test should continue until consistent acidic drainage pH can be verified, if consistent pH parameters were defined in the test objectives. In this limited case the test could be of short duration. However, the following should be noted for this test objective: (*1*) quantification of "consistent pH" has not been technically established and would require definition as part of the test design with consideration of its objectives; (*2*) drainage pH and solute release rates might vary even after 60 weeks of reaction, and significant changes may take place even after several years (**9**); and (*3*) ultimate rates of solute release could deviate from those observed prior to test termination.

11.4.3 If the objective is to determine the amount of actually-available estimated NP present in the sample, then testing will need to continue until acidic effluent is produced or until the total calcium plus magnesium release indicates all calcium and magnesium carbonate minerals have been depleted (for example, low levels [1.9 to 4.2 % $CaCO_3$] of carbonate minerals in mine-rock samples subjected to humidity-cell testing caused a lag-to-acid breakthrough that ranged from 24 to 204 weeks - (**11**).

11.5 *Leachate Analyses:*

11.5.1 After pH, Eh, and conductivity measurements have been made (either through use of a flow-through cell or from subsamples of the leachate), filter the remaining leachate using a 0.45 µm filter. Transfer any solids remaining on the filter back into the humidity cell.

11.5.2 Analyze the leachates for specific constituents or properties, or use them for biological testing procedures as desired, using (*1*) appropriate ASTM test methods or (*2*) methods accepted for the site where disposal will occur. Where no appropriate ASTM test methods exist, other test methods may be used and recorded in the report, provided that they are sufficiently sensitive to assess potential water quality impacts at the proposed disposal site. The frequency of analyses must be consistent with test objectives, and interpretation and application of the results. The suggested minimum frequency for analysis of pH, alkalinity/acidity, and specific conductance is weekly through week 20, at least every two weeks through week 40, and at least every four weeks thereafter. Eh can also be determined as needed. It would be most rigorous to analyze concentrations of sulfate and other selected solutes weekly during the first 20 weeks. However, this may not be feasible. At a minimum, it is suggested that analyses be performed at weeks 0, 1, 2, 4, 6, 8, 10, 12, 14, 16, 18, 20. For comprehensive chemical characterization of analytes not needed on a weekly basis to meet objectives of the test, the minimum recommended frequency is 0, 1, 2, 4, 8, 12, 16, and 20 weeks. As test duration increases beyond 20 weeks, these analyses can be conducted at least every four weeks through week 40, and at least every eight weeks thereafter. Whether visible phase separation during storage of the leachates occurs or not, appropriate mixing should be used to ensure the homogeneity of the leachates prior to their use in such analyses or testing.

NOTE 22—When analytical frequency decreases (for example, from every two weeks to every four to eight weeks), estimation is necessary, and consequently error is introduced. The degree of error (for example, from interpolation) is likely to be smaller during periods when solute concentrations are relatively stable. This potential for error must be considered when planning a change in analytical frequency and in subsequent data analysis, interpretation, and application. If calculation of mass release using interpolated concentrations is deemed inadequate based on the objectives of the test, effluent samples may be composited over periods determined to be appropriate (for example, a volume-weighted composite from 4 to 5 weeks of weekly effluent). Compositing of weekly effluent has the advantage of reducing frequency and cost of chemical analysis. Potential disadvantages of compositing weekly samples include (*1*) possible solute precipitation during the composite period, which would require sample digestion; and (*2*) masking of exceptionally high or low concentrations in several weekly effluent samples by mixing with moderate concentration samples from the remainder of the composite period.

11.5.3 Table 1 is an example of a spreadsheet format used for recording 20 weeks of leachate analytical data.

11.5.4 Fig. 6 is an example of a method used to plot the temporal variation (by week) of leachate pH, sulfate load, and cumulative sulfate load from 21 weeks of laboratory weathering (see 12.9 for the calculation of cumulative load and release rates).

11.6 *Weathered Solid Material Analyses (recommended as best practice):*

11.6.1 Weigh the humidity cell after collection of the final effluent and completion of a three-day dry-air period.

11.6.2 Transfer the weathered residue and filter media to a clean drying pan, and dry to constant weight at 40°C. Record the final weight.

11.6.3 Perform any gross sample examination (for example, sample texture and weathering-product mineral characterization) desired for the weathered residues prior to size reduction. To facilitate such an examination, empty the humidity cell

TABLE 1 Example Format for Recording 20 Weeks of Humidity Cell Leach Data

Cell 6,8C											
Week>>>	0	1	2	3	4	5	6	7	8	9	10
Concentration (μg/g)^A											
*Cu	9.190	0.103	0.051	0.078	0.064	0.062	0.058	0.074	0.003	0.130	0.060
*Zn	8.30	0.42	0.22	0.62	0.20	0.12	0.13	0.11	0.10	0.19	0.34
SO_4	4361	2568	1737	1763	1616	1635	1843	1424	1790	1540	1200
Liquid weight (g)	387.7	454.8	424.7	399.4	413.0	391.1	423.0	398.5	434.3	403.9	394.5
Loads (μg × 10^{-3})^A											
*Cu	3.56	0.05	0.02	0.03	0.03	0.02	0.02	0.03	0.00	0.05	0.02
*Zn	3.22	0.19	0.09	0.25	0.08	0.05	0.05	0.04	0.04	0.08	0.13
SO_4	1691	1168	738	704	667	639	780	567	777	622	473
Cum (μg × 10^{-3})^A											
SO_4	1690.8	2858.7	3596.4	4300.5	4967.9	5607.4	6387.1	6954.5	7731.9	8353.9	8827.3
Condition	4960	3900	2500	2290	2420	2530	2380	2300	2810	1987	1822
pH	3.160	5.020	4.730	4.5	5.030	4.560	4.76	3.97	4.37	4.28	4.32
Eh	586.9	613	582.2	597.3	521	553.3	504.7	555.8	610.7	590.3	549.3

Week>>>^{A}	11	12	13	14	15	16	17	18	19	20
Concentration (μg/g)^A										
*Cu	0.146	0.108	0.174	0.240	0.250	0.260	0.450	0.640	0.830	1.020
*Zn	0.31	0.13	0.20	0.28	0.32	0.37	0.48	0.59	0.70	0.81
SO_4	1220	1239	1117.5	996	1040	1084	1175	1266	1357	1448
Liquid weight (g)	392.4	407.6	419.0	412.9	382.3	426.7	392.5	406.8	421.4	399.3
Loads (μg × 10^{-3})^A										
*Cu	0.06	0.0	0.1	0.10	0.1	0.11	0.2	0.3	0.3	0.41
*Zn	0.12	0.1	0.1	0.11	0.1	0.16	0.2	0.2	0.3	0.32
SO_4	478.5	505.0	468.2	411.2	397.6	462.5	461.2	515.0	571.8	578.2
Cum (μg × 10^{-3})^A										
SO_4	9306	9811	10 279	10 690	11 088	11 550	12 012	12 527	13 099	13 677
Condition	1872	1980	1823	1570	1905	2010	1636	2050	1751	2040
pH	3.84	4.05	4.23	3.81	3.47	4	3.4	3.37	3.21	3.02
Eh	552.2	551.3	570.1	561.6	556.1	579.8	567.5	565.2	583.1	578.2

^{A} μg × 10^{-3} is equivalent to mg. μg/g represents mass of solute per unit mass of recovered lechant sample; it is also equivalent to mg/L, assuming that the leachant sample is a dilute solution having a density of 1 g/mL.

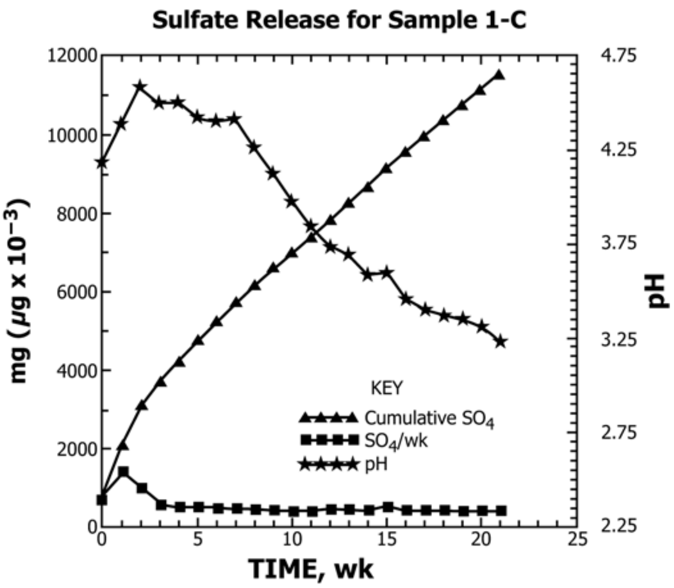

FIG. 6 Plot of Temporal Variation of pH, Sulfate Load, and Cumulative Sulfate Load from 21 Weeks

contents into a clean drying pan carefully by pushing gently on the bottom of the perforated plate with a wooden dowel until the sample exits the cell mouth. The perforated plate is accessed through the humidity cell drain port (see Fig. 1).

11.6.4 Identify and mark the top versus bottom portions of the sample for gross sampling purposes. Formations of cemented lumps of sample termed "ferricrete" that result from the laboratory weathering process are common in iron-sulfide-mineral rich samples. Depending on the sample mineralogy, the degree of "ferricrete" cementation may vary vertically within the sample, and the investigator may wish to segregate the sample into upper, middle, and lower thirds to document and characterize such changes.

11.6.5 After drying to constant weight, screen the sample through a series of Tyler screens sufficient to produce both plus and minus 10-mesh (1.7 mm) fractions; roll-crush the plus 10-mesh fractions to pass a 10 mesh screen; recombine the resulting minus 10-mesh materials for splitting:

11.6.5.1 Split the sample into halves using a riffle splitter with 6.35-mm (0.25-in.) chutes (in accordance with Test Method E877, Sampling and Preparation Procedure-Riffling), and reserve one half to perform a sequential extraction leach; the objective of the sequential extraction is to identify and quantify metal concentrations contained in secondary mineral precipitates that remain in the laboratory-weathered sample after the final weekly leach. Report the resulting metal concentrations from each step of the extraction protocol. Examples of sequential extraction protocols are listed in Note 23.

11.6.5.2 Split the remaining half sample into two quarters using a riffle splitter with 6.35-mm (0.25-in.) chutes, and submit one quarter for mineral characterization; pulverize the other quarter in either a ring-and-puck or disk-pulverizing machine to 95 % passing a 150-μm (100-mesh) screen in accordance with Test Method E276.

11.6.6 Mix the pulverized residue in a blender or on a rolling cloth. Use the prepared residue for chemical characterization and for comparison with the pre-weathered solid material sample.

NOTE 23—In metal-mine waste studies, goals of post-leaching sample characterization include: (*1*) measuring the mass of iron-sulfide and carbonate minerals (that is, the calcium plus magnesium fraction) remaining in the sample and assessing the availability of these minerals for additional reaction; (*2*) identifying presence of secondary mineral formation (for example, gypsum, iron oxyhydroxide or "ferricrete", and jarosite) and estimating its mass; and (*3*) determining occurrence of silicate dissolution in samples with high iron-sulfide mineral content. Carbon-sulfur contents of the remaining iron-sulfide and carbonate minerals are commonly measured by combustion-infrared spectrophotometer analyses of the pulverized sample (see 9.4.2). Traditional mineralogical techniques such as optical microscopy, X-ray diffraction (XRD), scanning electron microscopy (SEM) and electron-probe microanalysis (EPMA) **(23, 24)** may be required to speciate the carbonate mineral occurrence and to assess the availability of the iron sulfides and carbonates. Traditional mineralogical techniques also can be applied to identify mineral surface features indicative of mineral dissolution. The presence of secondary minerals can be assessed using traditional mineralogical techniques and leach extractions **(8, 17)**. Leach extractions are particularly useful in determining the presence of metals released from primary minerals (for example, iron sulfides) and subsequently sequestered in secondary phases (for example, adsorbed onto iron oxyhydroxides) that will readily release these metals if the pH of pore water decreases. Some secondary mineral precipitates may accumulate despite efforts to remove most or all of the reaction products during the weekly leach. Sequential extractions can be used to determine the extent of this accumulation, and some examples of sequential extraction protocols are described in Leinz and others **(25)** and Ribet and others **(26)**. However, solicitation of geochemical and mineralogical expertise is recommended to (*1*) identify the sequential extraction protocol most appropriate for the mine waste being tested, and (*2*) interpret the results generated.

Option A:

11.7 Temperature in the working area should be maintained at 25°C (±2°C). Minimum, maximum, mean, standard deviation, and number of temperature measurements are to be recorded. Occurrences of temperature values that fall outside the required range should be reported and differences in effluent quality during these excursions should be discussed.

11.8 *Dry-Air Cycle:*

11.8.1 The commencement of the three-day dry-air period marks the beginning of each new weekly cycle of the laboratory weathering humidity cell test; the first full-week cycle after the first leaching is designated Week 1; subsequent weeks (commencing with the second dry-air period) are designated as Week 2, Week 3 ... . Week *n*, etc.

11.8.2 To perform the dry-air cycle, feed air is metered to the humidity cell array with a flow meter (see 6.17) set at a target rate in the range of 1 to 10 L/min per cell, depending on the objectives of the testing. During the first weeks of the test, the airflow rate should be checked daily and adjusted to the target value ±0.5 L/min. This daily check should continue until the cell airflow rate has stabilized within the target value range. Subsequent to this stabilization, airflow rate measurements can be performed on a less frequent but routine schedule during the dry-air cycle. As indicated in 5.5.1, the sample should not be dried completely.

11.8.3 Feed air from the flow meter is routed first through a desiccant column and then to each of the cells through a dry-air manifold (Fig. 2). Air exiting the desiccant column should have a relative humidity of less than 10 % as measured with a hygrometer (see 6.15).

11.8.4 To maintain similar positive air pressure through the cells, it is recommended that a water-bubbling vessel be attached to each humidity cell air exit port coming out of the humidity cell lid; a 50-mL Erlenmeyer flask with a rubber stopper containing a vent and air inlet tube serves as a simple and efficient bubbler (Fig. 1).

11.8.5 The dry air is passed through each humidity cell for three days. Airflow rates from each of the cells should be checked routinely, recorded, and adjusted, if necessary. See 11.8.2 and Note 24.

11.8.6 Weigh cells at end of dry-air cycle and calculate the change in retained pore-water mass during the three-day dry-air cycle (see calculation described in 12.3).

11.9 *Wet-Air Cycle:*

11.9.1 The three-day wet-air period commences on the fourth day of each weekly cycle.

11.9.2 To perform the wet-air cycle of the method, feed air is routed through a water-filled humidifier via aeration stones or gas dispersion fritted cylinders/disks and then to each humidity cell (Fig. 2).

11.9.3 The water temperature in the humidifier is maintained at 25 ± 2°C to ensure that the sparged air maintains a relative humidity of approximately 95 % as measured with a hygrometer (see 6.15) from one of the humidifier exit lines (see Fig. 1). Recent intralaboratory testing showed that 99.9 % relative humidity was consistently achieved when water temperature in the humidifier was maintained within a range of 18.1 to 30.4°C **(27)**. Airflow rates to each of the cells should be checked according to 11.8.2, recorded, and adjusted, if necessary.

11.9.4 Weigh cells at end of wet-air cycle and calculate the change in retained pore-water mass as in 11.8.6.

NOTE 24—Substantial fluctuations have been reported in the interstitial water mass remaining from the weekly leach after the 3-day dry air portion of Option A weekly cycles **(14)**. Consequently, routine measurements of airflow rates, relative humidity, and pore-water mass are needed until airflow rates and post leach pore-water mass stabilize. After stabilization, these measurements can be performed on a less frequent but regular basis. Airflow and relative humidity measurements should be taken at the same time of day from the humidity cell air exit port; these measurements can be accomplished by installing a quick-disconnect fitting in the tubing that connects the air exit port to the bubbler (Fig. 1). Pore-water mass remaining after the dry and wet-air cycles is quantified by weighing the cell at the end of each cycle. The objective of these routine measurements is to prevent excessive drying or saturation of the sample. For example, sample pore water retained after the leach is reported to range from 70 to 150 mL, while pore-water loss after the three-day dry-air cycle is about 60 to 100 mL. Pore-water volumes typically change less than 3 mL during the wet-air cycle **(12)**. If both Option A and B protocols are used to evaluate splits of the same sample, it would useful to weigh Option B cells during the same portions of the weekly cycle as Option A for comparison of the data.

Option B:

11.10 *Store Cells in Environmentally-Controlled Enclosure*—On the first day following the weekly leach, remove the cells from the leach rack, weigh each and record the values (as in 11.2.6), return the covers to the respective cells, and then place the cells in the environmentally-controlled enclosure for the remaining six days of the weekly cycle. Weigh each cell again on the seventh day, prior to the next leach (which marks the end of the weekly cycle) and record the pre-leach weight. Calculate the change in retained pore-water mass **(27)** during the six days of storage in the environmentally-controlled enclosure by subtracting the pre-leach weight (at the end of the weekly cycle) from the post-leach weight (at the beginning of the weekly cycle) and report the results (see 12.3).

NOTE 25—Intralaboratory testing was conducted with covers on the Option B cells for the remaining six days of the weekly cycle. The mass of pore water retained in the covered Option B cells was consistently greater than that of Option A after the same time period. Drainage-quality results from the covered Option B cells produced higher rates of calcium and magnesium release than did Option A cells. Because the mass of pore water retained in the covered Option B cells was consistently greater than Option A, it is conceivable that the higher water contents favored dissolution of calcium and magnesium minerals by facilitating transport of acidic reaction products from the surfaces of iron sulfides to calcium/magnesium carbonates **(14)**. During a later study of the Option B protocol (MN DNR unpublished data), testing was conducted with the covers off for the remaining six days of the weekly cycle, and the amount of evaporation was relatively uniform over three consecutive weekly cycles (that is, 78, 81, and 86 %/week reduction in pore water).

11.10.1 Temperature in the enclosure should be maintained at 25°C (±2°C) **(8, 27)**. For temperature readings, weekly averages should be calculated. The minimum, maximum, mean, standard deviation and number of weekly average temperatures should be reported. All temperature data should be recorded and archived.

11.10.2 Enclosure should be maintained at a constant humidity (60 %, ±10 %) **(8, 27)**. For relative humidity readings, weekly averages should be calculated. The minimum, maximum, standard deviation and number of relative humidity measurements should be reported. All humidity data should be recorded and archived. Occurrences of temperature or humidity values that fall outside the required range should be reported as described in 11.7.

NOTE 26—For example, over the course of Option B testing reported in an interlaboratory study, temperature and relative humidity were measured three to four times weekly **(20)**.

12. Calculation

12.1 Calculate the mass, in g, of the dry filter media:

$$M_f = M_{hf} - M_h \quad (1)$$

where:

M_f = mass of the filter media, g,
M_{hf} = mass of the humidity cell and filter media, g, and
M_h = mass of the humidity cell, g.

12.2 Calculate the mass, in g, of the dry solid material contained in the humidity cell:

$$M_{sd} = M_{hfsd} - M_{hf} \quad (2)$$

where:

M_{sd} = mass of the dry solid material, g,
M_{hfsd} = mass of the humidity cell, filter, and solid material, g, and
M_{hf} = mass of the humidity cell and filter media, g.

12.3 Calculate the mass, in g, of residual pore water contained in the solid material:

$$M_i = M_{hfsw} - M_{hfsd} \quad (3)$$

where:

M_i = mass of the residual interstitial leachant contained in the material, g,
M_{hfsw} = mass of the humidity cell, filter, solid material, and pore water after leach, g, and
M_{hfsd} = mass of the humidity cell, filter, and dry solid material, g.

12.4 Calculate the mass, in g, of the weekly collected effluent:

$$M_e = M_{ef} - M_{et} \quad (4)$$

where:

M_e = mass of the collected effluent, g,
M_{ef} = mass of the collection flask and collected effluent, g, and
M_{et} = mass of the collection flask, g.

12.5 Calculate the weekly loading, in µg, of the constituents of interest:

$$L_e = C_e \times M_e \quad (5)$$

where:

L_e = loading of the constituent of interest in the effluent, µg,
C_e = concentration of the constituent in the effluent, µg/g, and
M_e = mass of the weekly collected effluent, g.

12.5.1 If an analyte is not measured during a particular week, it may be estimated by linear interpolation between data points (see Note 22). However, flag all estimated values so they are not confused with experimental data.

12.5.2 Weekly effluent analyses may produce values that are below quantitation or detection limits for some analytes. Four alternatives for assigning values to these analytes could be considered: (*1*) if values reported as less than the limit are important to the study, a more sensitive analytical method should be sought; (*2*) values reported as less than the limit could be approximated as half the limit; (*3*) values reported as less than the limit can be approximated as the limit. (a conservative estimate of analyte release that may have an adverse impact on water quality); or (*4*) values reported less than the limit could be considered as zero for all subsequent calculations. Record the alternative selected with calculation documentation. Options 2 and 3 may overestimate mass loadings when concentrations are actually very close to zero, while options 2 and 4 could underestimate mass loadings. Consequently, participating parties (for example, operators and regulating jurisdictions) should agree upon a mutually-acceptable approach prior to testing.

12.6 Calculate the final residue loading, in µg, of the constituents of interest:

$$L_r = C_r \times M_r \quad (6)$$

where:

L_r = loading of constituent in the residue, µg,
C_r = concentration of the constituent in the residue, µg/g, and
M_r = mass of the dried weathered residue and filter media, g.

12.7 Calculate the head concentration of the constituents of interest:

$$C_h = (L_{e0}+L_{e1}+L_{e2} \ldots +L_{ef}+L_r)/M_{sd} \tag{7}$$

where:

L_{e0} = loading of the constituent for Week 0, µg,
L_{e1} = loading of the constituent for Week 1, µg,
L_{e2} = loading of the constituent for Week 2, µg,
L_{ef} = loading of the constituent for the final week, µg,
L_r = loading of the constituent in the residue, µg, and
M_{sd} = mass of the dry solid material at the start of the test, g.

12.8 To check the material balance, calculate the difference between the initial chemical analyses of the unleached solids and the calculated head for the constituents of interest. Tabulate and report the results.

NOTE 27—Table 2 and Table 3 are examples of recording formats used to record weekly humidity cell and collection flask data.

12.9 Release rates for constituents of interest (diagnostic cations and anions) are calculated in two steps:

12.9.1 Weekly loads are determined by multiplying the constituent concentrations (determined from weekly leachate analyses) by the mass of recovered leachate; cumulative constituent loads are then determined by summing the respective weekly loads (for example, the cumulative load for Week 1 is the sum of loads for Week 0 and Week 1, and the cumulative load for Week 3 is the sum of loads for Weeks 0, 1, 2, and 3, etc.):

$$L_n = \sum_{i=0}^{n} (C_i \times M_i) \tag{8}$$

where:

L_n = cumulative loading of the constituent for n weeks, µg,
n = total number of weeks,
i = i^{th} week,
C_i = effluent concentration for the i^{th} week, µg/g, and
M_i = effluent mass for the i^{th} week, g.

12.9.2 Cumulative loads are plotted versus the number of weeks comprising the test, and inflection points on the cumulative plot are identified (see Fig. 6). The slope of the cumulative load plot between each inflection point is calculated and represents the release rate as µg week-1 for the weeks between and including the inflection points. Fig. 6 shows that the first inflection point on the cumulative sulfate plot occurs at Week 2. Note that the release rates for Weeks 0 to 2 and Weeks 2 to 21 can be calculated using (Eq 9); the results are summarized in Table 4:

$$R_n = \frac{(L_{n2} - L_{n1})}{(n_2 - n_1)} \tag{9}$$

TABLE 2 Humidity Cell Data Sheet

Humidity Cell No.______: Dry mass, g (to nearest 0.1 g)

Empty humidity cell (M_h):
Humidity cell + filter media (M_{hf}):
Filter media (M_f)
Humidity cell + filter + sample (M_{hfsd}):
Sample charge (M_{sd}):
Erlenmeyer collection flask (M_{et}):

Humidity Cell No. ______: Weekly Mass, g (to nearest 0.1 g)

Week No.	Humidity Cell + Filter + Sample at:		
	End, 3-Day Dry	End, 3-Day Wet	End, Leach (M_{hfsw})
Week 0	N/A	N/A	X
Week 1	X	X	X
Week 2	X	X	X
Week ...	X	X	X
Week 20	X	X	X

TABLE 3 Collection Flask Data Sheet

Collection Flask No.^{A} ______: Weekly Mass, g (to nearest 0.1 g)

Week No.	Flask + Effluent (M_{ef})	Flask Tare (M_{et})	Effluent (M_e)
Week 0	X	X	X
Week 1	X	X	X
Week 2	X	X	X
Week ...	X	X	X
Week 20	X	X	X

Collection Flask No.^{A} ______: Weekly Effluent Parameters

Week No.	Conductivity, y, mohs	Eh, mV	pH	CaCO$_3$ equivalent, parts per thousand	
				Acidity	Alkalinity
Week 0					
Week 1					
Week 2					
Week ...					
Week 20					

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^{A} The flask number corresponds with the humidity cell number.

TABLE 4 Calculated Release Rate for Weeks 0 to 2 and 2 to 21 from Cumulative Sulfate Plot, Fig. 6

n Weeks	L_{n2}, µg × 10^{-3}	L_{n1}, µ g × 10^{-3}	n_2	n_1	R_n, µg/week
0 to 2	3122.4	688.7	2	0	1216.9
2 to 21	11 432.6	3122.4	21	2	437.4

where:

R_n = release rate of the constituent for n weeks between and including the inflection points, µg week^{-1},

L_{n2} = constituent cumulative load, the final week of n weeks between and including the inflection points, µg,

L_{n1} = constituent cumulative load, the initial week of n weeks between and including the inflection points, µg,

n_2 = final week of n weeks between and including the inflection points, and

n_1 = initial week of n weeks between and including the inflection points.

12.9.3 An alternative to calculating rates of release as in 12.9.2, is to perform linear regression on the data bracketed between each inflection point delineated by creating the cumulative load plot.

12.9.4 Rates of release can also be calculated by averaging weekly release rates. Weekly solute mass release can be calculated as the product of the observed concentration and the volume of drainage. The weekly release rate is calculated as this mass divided by a time of one week, in the desired units. Rates of solute release can then be determined by averaging the weekly rates for selected periods. The standard deviation can also be calculated to describe variability of rates over these periods.

13. Precision and Bias[7]

13.1 *Precision*—The precision of these test methods is based on an interlaboratory study (ILS) of Test Method D5744. Two of the three participating laboratories conducted their respective ILS testing from 1996 through 2002. Data from the third laboratory was generated from 1992 through 1995 and included with the other two laboratory's data to produce a three-laboratory ILS. Because Test Method D5744 protocol requires lengthy test durations (20 to 265 weeks in the present ILS) only three laboratories were available to participate in the ILS. Samples from six different rock types (lithologies) were subjected to two primary test conditions, which consisted of a drip and flood-leach alternative. Each leach alternative was performed under both Option A and Option B protocols. Drip versus flood-leach alternatives and Option A versus Option B protocol results were also compared and reported. Paragraphs 13.1.1 – 13.1.6 summarize the ILS tests and test comparisons. Although multiple lithologies and laboratories are listed under the "Repeatability" category, each lithology's repeatability was determined separately by a single laboratory. Similarly, under "Reproducibility," each lithology's reproducibility was individually determined by participating multiple laboratories.

13.1.1 *Option A Repeatability*—Average deviation within the single laboratories from their mean of duplicate results and percent relative average deviation values were determined under two test conditions (*1*) drip-leach alternative (two lithologies, three laboratories) and (*2*) flood-leach alternative (one lithology, two laboratories).

13.1.2 *Option B Repeatability*—Average deviation within the single laboratories from their mean of duplicate results and percent relative average deviation values were determined for the drip-leach alternative (four lithologies, one laboratory).

13.1.3 *Option A Reproducibility*—Average deviation between the multiple laboratories from their polled mean results and percent relative average deviation values were determined under two test conditions (*1*) drip-leach alternative (one lithology, three laboratories) and (*2*) flood-leach alternative (one lithology, two laboratories).

13.1.4 *Option A Intralaboratory Comparison of Alternatives*—Average deviation and percent relative average deviation were determined for drip versus flood-leach alternatives (one lithology, two laboratories).

13.1.5 *Option B Intralaboratory Comparison of Alternatives*—Average deviation and percent relative average deviation values were determined for drip versus flood-leach alternatives (one lithology, one laboratory).

13.1.6 *Options A and B Intralaboratory Comparison*—Average deviation percent relative average deviation values were determined for Option A drip versus Option B drip-leach alternative (five lithologies, one laboratory).

13.1.7 Every "test result" represents an individual determination, and contributing laboratories reported duplicate or triplicate test results from their selected test conditions. Test Method D5744-96 was followed for the design and analysis of the data; the details are given in ASTM RR: D34–1019.

13.1.8 Because assessments of variability typically involved comparisons of drainage quality from duplicate or triplicate samples of selected waste-rock lithologies, two and occasionally three sets of data resulted. Consequently, a simple data analysis approach was adopted for each of the test conditions applied to specific lithologies. Drainage pH values were compared using the average deviation and percent relative average deviation from the mean. Mean pH was calculated by converting observed pH values from duplicate or triplicate cells to hydrogen ion concentrations, averaging these concentrations, and determining the negative logarithm of the average. Release rates of sulfate, calcium, and magnesium for duplicate or triplicate cells were also compared using the average deviation and percent relative average deviation from the mean. This data-analysis approach was applied separately to each analyte (pH, sulfate, calcium, and magnesium) under each of the test conditions (see Tables E5 through E11, Annex D, ASTM RR: D34-1019).

13.1.9 Because of the large volume of data produced by duplicate or triplicate humidity-cell testing of selected waste-rock lithologies under the two different test conditions combined with Option A and B protocols, sulfate release rates from the gabbro lithology were selected to illustrate the repeatability and reproducibility of Test Method D5744 protocol. The ILS-generated data for repeatability, reproducibility, and comparison of alternatives (expressed as average deviation and percent relative average deviation from the mean) for sulfate release rates from gabbro are summarized in Tables 5-7, Tables 8-10, and Tables 11-13, respectively. Repeatability, reproducibility, and comparison of alternatives for pH, calcium, and magnesium from the other five waste-rock

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[7] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D34-1019. Contact ASTM Customer Service at service@astm.org.

TABLE 5 Repeatability of Sulfate in Duplicate Cells Under Repeatability in Single Laboratories: Option A 500-mL Drip-Leach Alternative

				Sulfate Release Rate[A]			Difference from Mean			
Material	% S	Lab	Week	Cell 1	Cell 2	Mean[B]	Δ Cell 1[C]	Δ Cell 2[C]	Average Deviation[D]	Relative Average Deviation, %[E]
Gabbro	0.56	1	20	207	168	188	19.5	-19.5	19.50	10.40
Gabbro	0.56	1	39	146	119	133	13.5	-13.5	13.50	10.19
Gabbro	0.56	1	59	102	95	99	3.5	-3.5	3.50	3.55
Gabbro	0.59	1	20	208	199	204	4.5	-4.5	4.50	2.21
Gabbro	0.59	1	59	124	134	129	-5.0	5.0	5.00	3.88
Gabbro	0.63	1	20	144	158	151	-7.0	7.0	7.00	4.64
Gabbro	0.63	1	39	99	98	99	0.5	-0.5	0.50	0.51
Gabbro	0.63	1	59	71	58	65	6.5	-6.5	6.50	10.08
Gabbro	0.68	1	20	203	192	198	5.5	-5.5	5.50	2.78
Gabbro	0.68	1	59	119	126	123	-3.5	3.5	3.50	2.86
Gabbro	0.71	1	10	284	367	326	-41.5	41.5	41.50	12.75
Gabbro	0.71	1	39	139	157	148	-9.0	9.0	9.00	6.08
Gabbro	0.71	1	59	154	165	160	-5.5	5.5	5.50	3.45
Gabbro	0.84	1	20	185	182	184	1.5	-1.5	1.50	0.82
Gabbro	0.84	1	39	89	95	92	-3.0	3.0	3.00	3.26
Gabbro	0.84	1	59	90	100	95	-5.0	5.0	5.00	5.26
Gabbro	0.99	1	10	366	395	381	-14.5	14.5	14.50	3.81
Gabbro	0.99	1	39	175	174	175	0.5	-0.5	0.50	0.29
Gabbro	0.99	1	59	198	210	204	-6.0	6.0	6.00	2.94
Gabbro	1.39	1	10	482	410	446	36.0	-36.0	36.00	8.07
Gabbro	1.39	1	59	234	251	243	-8.5	8.5	8.50	3.51
Gabbro	1.39	2	5	507	453	480	27.0	-27.0	27.00	5.63
Gabbro	1.39	2	20	250	253	252	-1.5	1.5	1.50	0.60

[A] μmol (kg rock · wk)$^{-1}$.

[B] Mean = (Cell 1 + Cell 2)/2

[C] Δ Cell = difference of Cell 1 or Cell 2 from the mean.

[D] Average deviation = (| Δ Cell 1 | + | Δ Cell 2 |) / 2.

[E] Percent relative average deviation = (Avg. Deviation/mean) × 100.

TABLE 6 Repeatability of Sulfate in Triplicate Cells Under Repeatability Condition in Single Laboratories: Option A 500-mL Drip-Leach Alternative

				Sulfate Release Rate[A]				Difference from Mean				
Material	% S	Lab	Week	Cell 1	Cell 2	Cell 3	Mean[B]	Δ Cell 1[C]	Δ Cell 2[C]	Δ Cell 3[C]	Average Deviation[D]	Relative Average Deviation, %[E]
Gabbro	1.39	3	10	296	206	233	245	51.00	-39.00	-12.00	34.00	13.88
Gabbro	1.39	3	59	255	191	211	209	16.00	-18.00	2.00	12.00	5.74
Gabbro	1.39	3	100	155	143	179	159	-4.00	-16.00	20.00	13.33	8.39
Gabbro	1.39	3	145	109	128	142	126	-17.33	1.67	15.67	11.56	9.15
Gabbro	1.39	3	172	288	425	362	358	-70.33	66.67	3.67	46.89	13.09
Gabbro	1.39	3	265	499	505	500	501	-2.33	3.67	-1.33	2.44	0.49

[A] μmol (kg rock · wk)$^{-1}$.

[B] Mean = (Cell 1 + Cell 2 + Cell 3)/3

[C] Δ Cell = difference of Cell 1, Cell 2, or Cell 3 from the mean.

[D] Average Deviation = (| Δ Cell 1 | + | Δ Cell 2 | + | Δ Cell 3 |) / 3.

[E] Percent Relative Avg. Deviation = (Avg. Deviation/mean) × 100.

lithologies are contained in Tables E5 through E11, Annex D, ASTM RR: D34-1019.

13.1.10 While percent relative average- deviation from the mean for each test category was generally within 10 %, occasional variation occurred beyond these limits. However, in no case was variation in pH or release rates of sulfate, calcium, and magnesium sufficient to misclassify sample acid-producing potential. Causes of elevated interlaboratory and intralaboratory variation include: (*1*) deviation based on temperature differences (that is, winter versus summer), (*2*) variation as test duration extended beyond three years, and (*3*) deviation in duplicated siltite-argillite samples from low sulfur largely as sulfate.

13.1.11 *Repeatability Limit (r)*—The task group determined use of average deviation from the single-laboratory mean was more appropriate than standard deviation. Consequently, *r* was not calculated in accordance with Practice E691.

13.1.11.1 Repeatability average deviation and percent relative average deviation for sulfate from gabbro rock samples are listed in Tables 5-7, respectively.

13.1.11.2 Average deviation and percent relative average deviation for comparisons of two leach alternatives and Option A versus Option B protocol for sulfate from gabbro rock samples are listed in Tables 11-13.

13.1.12 *Reproducibility Limit (R)*—The task group determined use of average deviation from the pooled mean of the

TABLE 7 Repeatability of Sulfate in Duplicate Cells Under Repeatability Condition in Single Laboratories: Option A 500-mL Flood-Leach Alternative

				Sulfate Release Rate[A]			Difference from Mean			
Material	% S	Lab	Week	Cell 1	Cell 2	Mean[B]	Δ Cell 1[C]	Δ Cell 2[C]	Average Deviation[D]	Relative Average Deviation, %[E]
Gabbro	1.39	3	10	273	235	254	19.0	-19.0	19.00	7.48
Gabbro	1.39	3	59	189	221	205	-16.0	16.0	16.00	7.80
Gabbro	1.39	3	100	173	181	177	-4.0	4.0	4.00	2.26
Gabbro	1.39	3	145	118	127	123	-4.5	4.5	4.50	3.67
Gabbro	1.39	3	172	416	340	378	38.0	38.0	38.00	10.05
Gabbro	1.39	3	212	731	701	716	15.0	-15.0	15.00	2.09
Gabbro	1.39	3	265	386	535	461	-74.5	74.5	74.50	16.18

[A] μmol (kg rock · wk)$^{-1}$.
[B] Mean = (Cell 1 + Cell 2)/2
[C] Δ Cell = difference of Cell 1 or Cell 2 from the mean.
[D] Avg. Deviation = (| Δ Cell 1 | + | Δ Cell 2 |) / 2.
[E] Percent Relative Avg. Deviation = (Avg. Deviation/mean) × 100.

TABLE 8 Reproducibility of Sulfate in Triplicate Cells Under Reproducibility Condition in Single Laboratories: Option A 500-mL Drip-Leach Alternative

				Sulfate Release Rate[A]				Difference from Mean				
Material	% S	Lab	Week	Lab 1	Lab 3	Lab 2	Mean[B]	Δ Lab 1[C]	Δ Lab 3[C]	Δ Lab 2[C]	Average Deviation[D]	Relative Average Deviation, %[E]
Gabbro	1.39	1, 2, 3	10	482	245	378.00	368	113.67	-123.33	9.67	82.22	22.32
Gabbro	1.39	1, 2, 3	59	234	209	214.00	219	15.00	-10.00	-5.00	10.00	4.57
Gabbro	1.39	1, 2, 3	100	184	159	172.00	172	12.33	-12.67	0.33	8.44	4.92
Gabbro	1.39	1, 2, 3	125	214	139	152.00	168	45.67	-29.33	-16.33	30.44	18.09

[A] μmol (kg rock · wk)$^{-1}$.
[B] Mean = (Lab 1 + Lab 2 + Lab 3)/3
[C] Δ Lab = respective differences of Lab 1, Lab 3, or Lab 2 Cells from the mean.
[D] Avg Deviation = (| Δ Lab 1 | + | Δ Lab 2 | + | Δ Lab 3 |) / 3.
[E] Percent Relative Avg. Deviation = (Avg. Deviation/mean) × 100.

TABLE 9 Reproducibility of Sulfate in Duplicate Cells Under Reproducibility Condition in Single Laboratories: Option A 500-mL Drip-Leach Alternative

				Sulfate Release Rate[A]			Difference from Mean			
Material	% S	Lab	Week	Lab 3	Lab 2	Mean[B]	Δ Lab 3[C]	Δ Lab 2[C]	Average Deviation[D]	Relative Average Deviation, %[E]
Gabbro	1.39	2, 3	145	142	116	129	13.00	-13.00	13.00	10.08
Gabbro	1.39	2, 3	172	358	151	255	103.50	-103.50	103.50	40.67
Gabbro	1.39	2, 3	212	521	120	321	200.50	-200.50	200.50	62.56

[A] μmol (kg rock · wk)$^{-1}$.
[B] Mean = (Lab 1 + Lab 2)/2
[C] Δ Lab= respective differences of Lab 1 or Lab 2 Cells from the mean.
[D] Avg. Deviation = (| Δ Lab 1 | + | Δ Lab 2 |) / 2.
[E] Percent Relative Avg. Deviation = (Avg. Deviation/mean) × 100.

laboratories was more appropriate than standard deviation. Consequently Reproducibility limit (R) was not calculated in accordance with Practice E691.

13.1.12.1 Reproducibility average deviation and percent relative average deviation for sulfate from gabbro rock samples are listed in Tables 8-10, respectively.

13.2 *Bias*—At the time of the study, there was no accepted reference material suitable for determining the bias for this test method; therefore, no statement on bias is being made.

13.3 The precision statement was determined through statistical examination of humidity-cell-test results from 68 samples that comprised 6 different waste-rock lithologies. Testing was conducted under two test conditions, which were each conducted under Option A and B protocols by the participating laboratories.

13.4 To judge the equivalency of two test results, it is recommended to choose the material most similar in characteristics to the test material.

14. Keywords

14.1 chemical weathering; humidity cell; laboratory weathering; mill tailings; ore; oxidation; solid material; waste rock

TABLE 10 Reproducibility of Sulfate in Duplicate Cells Under Repeatability Condition in Single Laboratories: Option A 500-mL Flood-Leach Alternative

				Sulfate Release Rate[A]			Difference from Mean			
Material	% S	Lab	Week	Lab 3	Lab 2	Mean[B]	Δ Lab 3[C]	Δ Lab 2[C]	Average Deviation[D]	Relative Average Deviation, %[E]
Gabbro	1.39	2, 3	0-10	254	406	330	-76.00	76.00	76.00	23.03
Gabbro	1.39	2, 3	10-59	205	241	223	-18.00	18.00	18.00	8.07
Gabbro	1.39	2, 3	59-100	177	199	188	-11.00	11.00	11.00	5.85
Gabbro	1.39	2, 3	100-145	122	167	145	-22.50	22.50	22.50	15.57
Gabbro	1.39	2, 3	145-172	378	157	268	110.50	-110.50	110.50	41.31
Gabbro	1.39	2, 3	172-212	716	156	436	280.00	-280.00	280.00	64.22

[A] μmol (kg rock · wk)$^{-1}$.
[B] Mean = (Lab 2 + Lab 3)/2
[C] Δ Cell = difference of Lab 2 or Lab 3 from the mean.
[D] Avg. Deviation = (| Δ Lab 2 | + | Δ Lab 3 |) / 2.
[E] Percent Relative Avg. Deviation = (Avg. Deviation/mean) × 100.

TABLE 11 Repeatability of Sulfate in Duplicate Cells Under Repeatability Condition in Single Laboratories: Comparison of Option A 500-mL Drip Versus Flood-Leach Alternative

				Sulfate Release Rate[A]			Difference from Mean			
Material	% S	Lab	Week	Drip Cell	Flood Cell	Mean[B]	Δ Drip Cell[C]	Δ Flood Cell[C]	Average Deviation[D]	Relative Average Deviation, %[E]
Gabbro	1.39	3	10	245	254	250	-4.5	4.5	4.50	1.80
Gabbro	1.39	3	59	209	205	207	2.0	-2.0	2.00	0397
Gabbro	1.39	3	100	159	177	168	-9.0	9.0	9.00	5.36
Gabbro	1.39	3	145	126	122	124	2.0	-2.0	2.00	1.61
Gabbro	1.39	3	172	358	378	368	-10.0	10.0	10.00	2.72
Gabbro	1.39	3	212	546	716	631	-85.0	85.0	85.00	13.47
Gabbro	1.39	3	265	412	460	436	-24.0	24.0	24.00	5.50
Gabbro	1.39	2	10	378	406	392	-14.0	14.0	14.00	3.57
Gabbro	1.39	2	59	214	241	228	-13.5	13.5	13.50	5.93
Gabbro	1.39	2	100	172	199	186	-13.5	13.5	13.50	7.28
Gabbro	1.39	2	145	139	167	153	-14.0	14.0	14.00	9.15
Gabbro	1.39	2	172	151	157	154	-3.0	3.0	3.00	1.95
Gabbro	1.39	2	212	120	156	138	-18.0	18.0	18.00	13.04

[A] μmol (kg rock · wk)$^{-1}$.
[B] Mean = (Drip Cell + Flood Cell)/2
[C] Δ Cell = difference of Drip Cell or Flood Cell from the mean.
[D] Avg. Deviation = (| Δ Drip Cell | + | Δ Flood Cell |) / 2.
[E] Percent Relative Avg. Deviation = (Avg. Deviation/mean) × 100.

TABLE 12 Repeatability of Sulfate in Duplicate Cells Under Repeatability Condition in Single Laboratories: Comparison of Option B 500-mL Drip Versus Flood-Leach Alternative

				Sulfate Release Rate[A]			Difference from Mean			
Material	% S	Lab	Week	Drip Cell	Flood Cell	Mean[B]	Δ Drip Cell[C]	Δ Flood Cell[C]	Average Deviation[D]	Relative Average Deviation, %[E]
Gabbro	1.39	2	10	470	640	555	-85.0	85.0	85.00	15.32
Gabbro	1.39	2	59	300	350	325	-25.0	25.0	25.00	7.69
Gabbro	1.39	2	100	210	250	230	-20.0	20.0	20.00	8.70
Gabbro	1.39	2	145	180	180	180	0.0	0.0	0.00	0.00
Gabbro	1.39	2	172	180	180	180	0.0	0.0	0.00	0.00
Gabbro	1.39	2	212	198	196	197	1.0	–1.0	1.00	0.51
Gabbro	1.39	2	265	215	234	225	-9.5	9.5	9.50	4.23

[A] μmol (kg rock · wk)$^{-1}$.
[B] Mean = (Drip Cell + Flood Cell)/2
[C] Δ Cell = difference of Drip Cell or Flood Cell from the mean.
[D] Avg. Deviation =(| Δ Drip Cell | + | Δ Flood Cell |) / 2.
[E] Percent Relative Avg. Deviation = (Avg. Deviation/mean) × 100.

TABLE 13 Repeatability of Sulfate in Duplicate Cells Under Repeatability Condition in Single Laboratories: Comparison of Option A Versus Option B 500-mL Drip-Leach Alternative

				Sulfate Release Rate[A]			Difference from Mean			
Material	% S	Lab	Week	Opt A Cell	Opt B Cell	Mean[B]	Δ Opt A[C]	Δ Opt B[C]	Average Deviation[D]	Relative Average Deviation, %[E]
Gabbro	1.30 % S_T, 1.24 % S^{-2}	2	59	214	305	260	-45.50	45.50	45.50	17.53
Gabbro	1.30 % S_T, 1.24 % S^{-2}	2	100	172	207	190	-17.50	17.50	17.50	9.23
Gabbro	1.30 % S_T, 1.24 % S^{-2}	2	145	140	180	160	-20.00	20.00	20.00	12.50
Gabbro	1.30 % S_T, 1.24 % S^{-2}	2	172	150	180	165	-15.00	15.00	15.00	9.09
Gabbro	1.30 % S_T, 1.24 % S^{-2}	2	212	120	198	159	-39.00	39.00	39.00	24.53

[A] μmol (kg rock · wk)$^{-1}$.

[B] Mean = (Opt A Cell + Opt B Cell)/2

[C] Δ Cell = difference of Opt A Cell or Opt B Cell from the mean.

[D] Avg. Deviation = (| Δ Opt A Cell | + | Δ Opt B Cell |) / 2.

[E] Percent Relative Avg. Deviation = (Avg. Deviation/mean) × 100.

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