Designation: D 5744 – 96 (Reapproved 2001)

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

This standard is issued under the fixed designation D 5744; 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.

1. Scope

1.1 This test method covers a procedure that accelerates the natural weathering rate of a solid material sample so that diagnostic weathering products can be produced, collected, and quantified. Soluble weathering products are mobilized by a fixed-volume aqueous leach that is performed, collected, and analyzed weekly. When conducted in accordance with the following protocol, this laboratory test method has accelerated metal-mine waste-rock weathering rates by at least an order of magnitude greater than observed field rates **(1)**.[2]

1.1.1 This test method is intended for use to meet kinetic testing regulatory requirements for mining waste and ores.

1.2 This test method is a modification of an accelerated weathering test method developed originally for mining wastes **(2-4)**. 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 1000-g solid material sample, with water of a specified purity, and the collection and chemical characterization of the resulting leachate over a minimum period of 20 weeks.

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.

1.8 This test method is intended to describe the procedure for performing the accelerated weathering of solid materials to generate leachates. 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 the standard. 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]

D 75 Practices for Sampling Aggregates
D 276 Test Methods for Identification of Fibers in Textiles
D 420 Guide to Site Characterization for Engineering, Design and Construction Purposes
D 653 Terminology Relating to Soil, Rock, and Contained Fluids
D 737 Test Method for Air Permeability of Textile Fabrics[4]
D 1067 Test Methods for Acidity or Alkalinity of Water[4]
D 1125 Test Methods for Electrical Conductivity and Resistivity of Water
D 1193 Specification for Reagent Water
D 1293 Test Methods for pH of Water
D 1498 Practice for Oxidation-Reduction Potential of Water
D 2234 Test Methods for Collection of a Gross Sample of Coal
D 3370 Practices for Sampling Water
E 276 Test Method for Particle Size or Screen Analysis at No. 4 (4.75-mm) Sieve and Finer for Metal-Bearing Ores and Related Materials
E 877 Practice for Sampling and Sample Preparation of Iron Ores and Related Materials

3. Terminology

3.1 *Definitions:*

---

[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.

Current edition approved March 10, 1996. Published May 1996.

[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] Withdrawn.

Copyright © ASTM International, 100 Barr Harbor Drive, PO Box C700, West Conshohocken, PA 19428-2959, United States.

EXHIBIT
27

Copyright by ASTM Int'l (all rights reserved);
Reproduction authorized per License Agreement with ANSI; Thu Feb 2 19:46:01 EST 2006

3.1.1 *acid producing potential (AP)*, *n*—the potential for a solid material sample to produce acidic effluent, based on the percent of sulfide contained in that sample as iron-sulfide mineral (for example, pyrite or pyrrhotite) (**3**). The AP is commonly converted to the amount of calcium carbonate required to neutralize the resulting amount of acidic effluent produced by the oxidation of contained iron sulfide minerals; it is expressed as the equivalent tons of calcium carbonate per 1000 tons of solid material (**4**). The AP is therefore calculated by multiplying the percent of sulfide contained in the material by a stoichiometric factor of 31.25 (**5**).

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

3.1.3 *leach*, *n*—a 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*—the product of the weekly concentration for a constituent of interest and the weight of solution collected that may be interpreted for water quality impacts.

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

3.1.6 *neutralizing potential (NP)*, *n*—the potential for a solid material sample to neutralize acidic effluent produced from the oxidation of iron-sulfide minerals, based on the amount of carbonate present in the sample. The NP is also presented in terms of tons of calcium carbonate equivalent per 1000 tons of solid material (**4**). It is calculated by digesting the solid material with an excess of standardized acid and back-titrating with a standardized base to measure and convert the acid consumption to calcium carbonate equivalents (**3**, **6**).

3.1.6.1 *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.

3.1.7 *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 whose economic mineral content is less than a specified economic cutoff value.

4. Summary of Test Method

4.1 This accelerated weathering test method is designed to increase the geological-chemical-weathering rate for selected 1000-g solid material samples and produce a weekly effluent that can be characterized for solubilized 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 different solid material samples. The test procedure calls for weekly cycles comprised 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. A test duration of 20 weeks is recommended (**3**, **4**).

5. Significance and Use

5.1 The purpose of this accelerated weathering procedure is to determine the following: (*1*) whether a solid material will produce an acidic, alkaline, or neutral effluent, (*2*) whether that effluent will contain diagnostic cations (including trace metals) and anions that represent solubilized weathering products formed during a specified period of time, and (*3*) the rate at which these diagnostic cations and anions will be released (from the solids in the effluent) under the closely controlled conditions of the test.

NOTE 1—Examples of products that can be produced from the test include the following: (*1*) weekly effluent acidity and alkalinity determined by titration and (*2*) weekly aqueous concentrations of cations and anions converted to their respective release rates (for example, the average release of µg sulfate ion/g of solid material sample/week, over a 20-week period). In acid drainage studies, for example, the average weekly rates of acid production (measured as µg/g/wk of sulfate released) determined from accelerated weathering tests of mine waste samples are compared with the AP present in each sample. The number of years of acidic effluent expected to be produced under laboratory accelerated weathering conditions can then be estimated from this comparison. The years of accelerated weathering required to deplete a mine waste sample's NP are calculated similarly by determining the average weekly calcium and magnesium release rates and dividing the sample's NP by the sum of those rates (**7**).

5.2 The principle of the accelerated weathering test method is to promote more rapid oxidation of solid material constituents than can be accomplished in nature and maximize the loadings 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 temperature, volume, and application rate of water and oxygen. Specifically, an excess amount of air pumped up through the sample during the dry- and wet-air portions of the weekly cycle ensures that oxidation reactions are not limited by low oxygen concentrations. Weekly leaches with low ionic strength water ensure the removal of leachable oxidation 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 water that remains in the pores of the sample after the weekly leach. Evaporation 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 accelerate the dissolution of additional sample constituents. Precipitation occurs as evaporation continues, and the remaining water becomes over-saturated. Some of these precipitated salts are potential sources of acidity when re-solubilized (for example, melanterite, $FeSO_4 \cdot 7H_2O$; and jarosite, $K_2Fe_6(OH)_{12}(SO_4)_4$). 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 to accelerate the abiotic oxidation of such constituents as iron sulfide. The wet (saturated)-air portion of the weekly cycle enhances the bacteria-catalyzed oxidation of solid material sample constituents (for example, iron sulfide) by providing a moist micro-environment throughout the available surface area of the 1000-g sample. This

Copyright by ASTM Int'l (all rights reserved);
Reproduction authorized per License Agreement with ANSI; Thu Feb 2 19:46:01 EST 2006

micro-environment promotes the diffusion of weathering products (for example, resolubilized precipitation products) and metabolic byproducts (for example, ferric iron) between the microbes and the substrate without saturating the sample and affecting oxygen diffusion adversely.

NOTE 2—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 $cm^2 \cdot s^{-1}$ versus 2.5×10^{-5} $cm^2 \cdot s^{-1}$ at 0 and 25°C, respectively) (**8**). However, in the humidity cell setting, corresponding oxygen diffusion rates in porous media are also functions of solid phase porosity and attendant tortuosity. Actual diffusion rates will therefore be somewhat slower than five orders of magnitude.

5.3 This test method has been tested on both coal and metal mine wastes to classify their respective tendencies to produce acidic, alkaline, or neutral effluent, and to subsequently measure the concentrations of selected inorganic components leached from the waste (**2-4, 7**). The following are examples of parameters for which the weekly effluent may be analyzed:

5.3.1 pH, Eh (oxidation/reduction potential), and conductivity (see Test Methods D 1293, Practice D 1498, and Test Methods D 1125, respectively, for guidance);

5.3.2 Dissolved gaseous oxygen and carbon dioxide;

5.3.3 Alkalinity/acidity values (see Test Methods D 1067 for guidance);

5.3.4 Cation and anion concentrations; and

5.3.5 Metals and trace metals concentrations.

NOTE 3—Sulfate and iron concentrations in the weekly leachates from solid material containing iron-sulfide minerals should be monitored because their release rates are critical measurements of iron-sulfide mineral oxidation rates. Acidic effluent or acid drainage is a consequence of iron-sulfide mineral oxidation and the subsequent aqueous transport of resulting hydrogen ion and oxidation/dissolution products to the receiving environment (for example, surface and ground waters).

5.4 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 buffering capacity of the solid material under specified laboratory conditions.

5.5 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 preservation, storage, and handling to prevent possible contamination of the samples or alteration of the concentrations of constituents through sorption or precipitation.

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

6. Apparatus

6.1 *Humidity Cell*—A modified column constructed of materials suitable to the nature of the analyses to be performed (see Practices D 3370 for guidance). Multiple humidity cells can be arranged in an array to accommodate the simultaneous accelerated 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 (¼ 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 (examples would be processed mill tailings or fly ash).

6.1.3 A perforated disk (comprised of materials suitable to the nature of analyses to be performed), approximately 3.15-mm (⅛-in.) thick, with an outside diameter (OD) suitable to the suggested vessel ID (6.1.1 and 6.1.2) is elevated approximately 12.5 mm (½ in.) above the cell bottom to support the solid material sample (see Fig. 1).

NOTE 4—The cell and particle size dimensions described above are those used commonly for assessing the potential of waste-rock and mill-tailings samples associated with coal and metal mining operations to produce acidic effluent. A "shoe box"-shaped cell design with similar dimensions is preferred by some researchers (**6**).

6.2 *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:

6.2.1 A thermostatically controlled heating element to maintain the water temperature at 30°C during the wet-air cycle.

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

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

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

6.5 *BK Bacteria Filter Tube*, for inclusion in the feed-air line, which must be capable of retaining 99.99 % of 0.1-µm particles.

6.6 *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.

6.7 *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.

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

6.9 *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.

6.10 *Dry Air Manifold*—A line of plastic tubing exiting the desiccant column and containing multiple regularly spaced "tee" connectors to supply air to each humidity cell.

6.11 *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 D 276 and D 737 for guidance).

Copyright by ASTM Int'l (all rights reserved);
Reproduction authorized per License Agreement with ANSI; Thu Feb 2 19:46:01 EST 2006

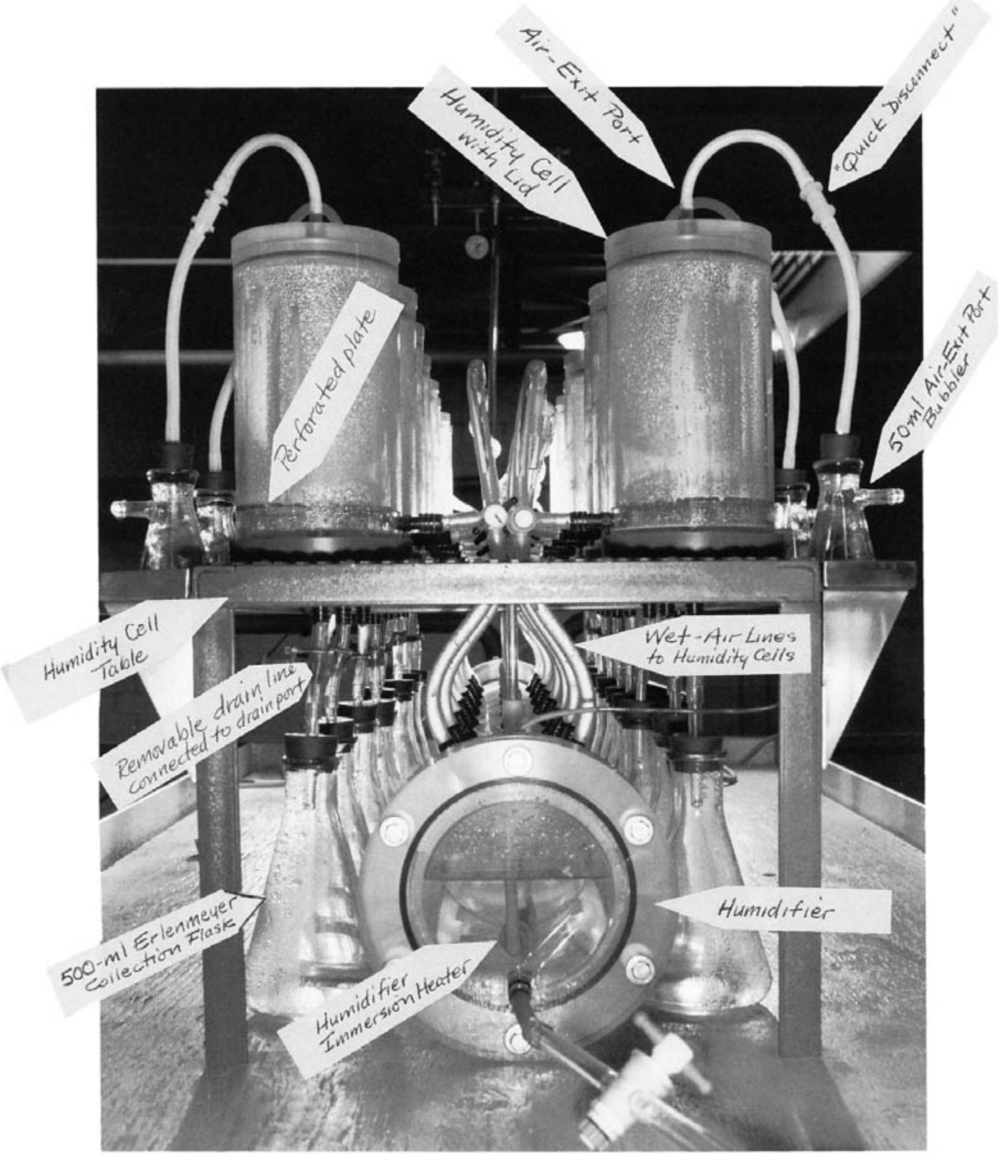

FIG. 1 Side View of 16-Cell Array

NOTE 5—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. In addition, pyrex (borosilicate) can contribute boron if this is a constituent of interest.

6.12 *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.

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

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

6.15 *Screen*, 6.3 mm (¼ in.).

6.16 *Screen*, 150 µm (100 mesh).

6.17 *Drying Oven*—Any thermostatically controlled drying oven capable of maintaining a steady temperature of 50 ± 2°C.

6.18 *pH Meter*—Any pH meter with a 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.

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

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

6.21 *Erlenmeyer Flask*, 500 mL or 1 L, one per each humidity cell.

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

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

7. Reagents

7.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that

Copyright by ASTM Int'l (all rights reserved);
Reproduction authorized per License Agreement with ANSI; Thu Feb 2 19:46:01 EST 2006

all reagents conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available.[5]

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 D 1193. The method by which the water is prepared, that is, distillation, 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 and a grade BK bacteria filter tube in advance of the flowmeter.

8. Sampling

8.1 Collect the samples using available sample methods developed for the specific industry (see Practices D 75 and E 877, Guide D 420, Terminology D 653, and Test Methods D 2234).

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 to be sent to the laboratory should be sufficient to provide 8 to 10 kg of bulk sample for splitting and testing (see 9.3).

NOTE 6—Additional information on theory and methods for obtaining representative samples is contained in Pitard (**9**).

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 D 420 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 D 3370 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 50 ± 2°C for 24 h, or until a constant weight is reached.

9.1.1 If exploration-generated or run-of-mine solid material samples are not readily available, archived dried and crushed samples from geological exploratory or development drilling programs may be used for preliminary evaluations of ore and waste rock from new operations; this is provided that the available solid material samples are not significantly finer than 95 % passing a No. 12 (1.7-mm) sieve. Document the sample drying and preparation procedures used during the drill sampling program in order to interpret the results properly. Evaluate the effects of drying temperature on metals volatilization (for example, mercury in cinnabar vaporizes at temperatures exceeding 80 to 90°C) and mineral morphology and chemistry modifications (for example, on heating at temperatures exceeding 100°C, chalcocite changes crystal form and is oxidized subsequently from Cu_2S to CuO, $CuSO_4$, and SO_2). Especially ensure that the effects of particle size distribution changes resulting from the more finely crushed sample are considered in the interpretation (that is, the potential for increased liberation of acid-producing and acid-consuming minerals with an attendant increase in mineral surface area).

9.1.2 In mining waste evaluations, the particle size for mill tailings will be significantly finer (commonly less than 150 µm/100 mesh) than the particle size distributions from ore and waste rock. Pilot plant tailings should be used if mill tailings are not available.

9.2 Screen the air-dried bulk samples through a 6.3-mm (¼-in.) screen in accordance with Test Method E 276. Crush any oversize material so that 100 % passes the screen.

NOTE 7—**Caution:** Recent accelerated weathering studies of run-of mine waste rock from metal mines demonstrate that crushing a bulk sample so it passes a 6.3-mm (¼-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 (¼-in.) material. A suggestion for avoiding this problem is to segregate the – 6.3-mm (¼-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 (¼-in.) material can be tested separately (for example, Brodie, et al (**10**) describe a large-scale humidity cell test that would accommodate – 75-mm material). Samples from the drill core and cuttings also present material sizing problems, which must be considered when interpreting drill core and cuttings accelerated weathering data. The drill core must be crushed to – 6.3-mm (¼-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 (¼-in.) due to the rotary-percussive nature of obtaining the sample.

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. Divide the test unit into eight nominal 1-kg test specimens. Seal each test specimen in a moisture-barrier bag.

NOTE 8—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.

9.4 Select one test specimen at random, and determine the moisture content by weighing and drying to constant weight at 80 ± 5°C.

9.4.1 Crush the dried test specimen so that at least 95 % passes a 1.7-mm (10-mesh) screen, in accordance with Test Method E 276.

9.4.2 Divide the crushed test specimen in half twice, using a riffle splitter with 6.35-mm (¼-in.) chutes, and select a ¼ subsample at random.

9.4.3 Transfer the selected subsample to a ring and puck grinding mill and grind to a nominal 95 % passing a 150-µm

---

[5] *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. Pharmaceutical Convention, Inc. (USPC), Rockville, MD.

Copyright by ASTM Int'l (all rights reserved);
Reproduction authorized per License Agreement with ANSI; Thu Feb 2 19:46:01 EST 2006

(100-mesh) screen, in accordance with Test Method E 276. Use the subsample for chemical and mineralogical characterization of the test unit.

9.5 Select one test specimen at random, and determine the particle size distribution in accordance with Test Method E 276.

9.6 Select one test specimen at random for use in the accelerated 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 vapor-barrier bags until it is time to load the humidity cells.

9.7 Reserve the remaining test specimens for replicated testing or to resolve disputed results.

10. Apparatus Assembly

10.1 The humidity cells are table-mounted at a height sufficient to accommodate the placement of both the humidifier and one Erlenmeyer 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 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 and 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). Attach a water-bubbling vessel to each humidity cell lid air exit port to prevent the short circuiting of air through cells containing more permeable solid material samples (Fig. 1). A separatory funnel rack is mounted on the table that holds the cells if the weekly water leach is applied dropwise (drip trickle). Multiple separatory funnels (one for each cell) are held in the rack during the drip trickle leach that is performed on the seventh day of each weekly cycle (Fig. 2). 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

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 (Erlenmeyer flask).

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 6.11) to the humidity cell's inside diameter dimensions so that it fits snugly yet lies flat on the perforated support.

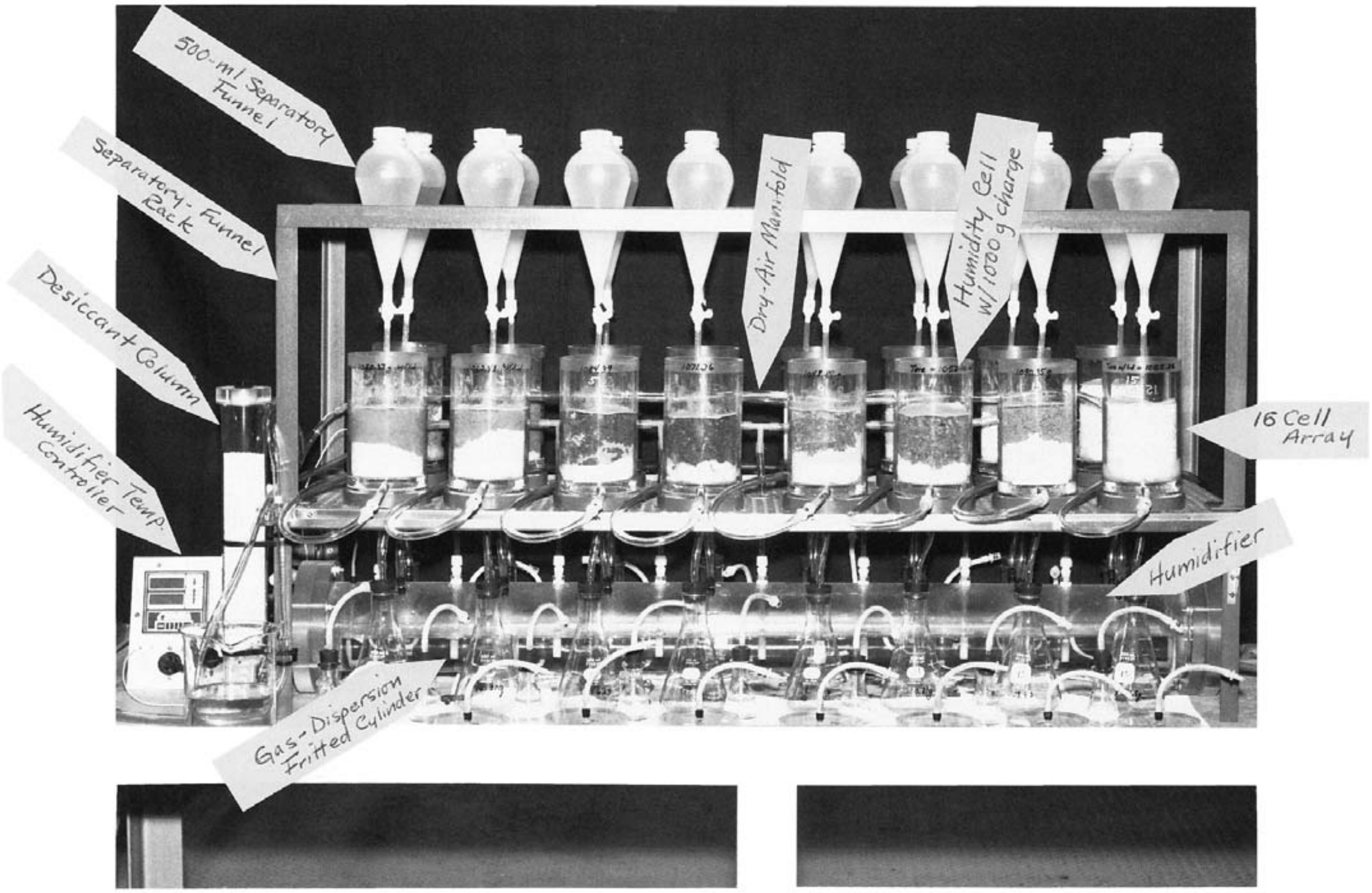

FIG. 2 Front View of 16-Cell Array with Separatory Funnel Rack

Copyright by ASTM Int'l (all rights reserved);
Reproduction authorized per License Agreement with ANSI; Thu Feb 2 19:46:01 EST 2006

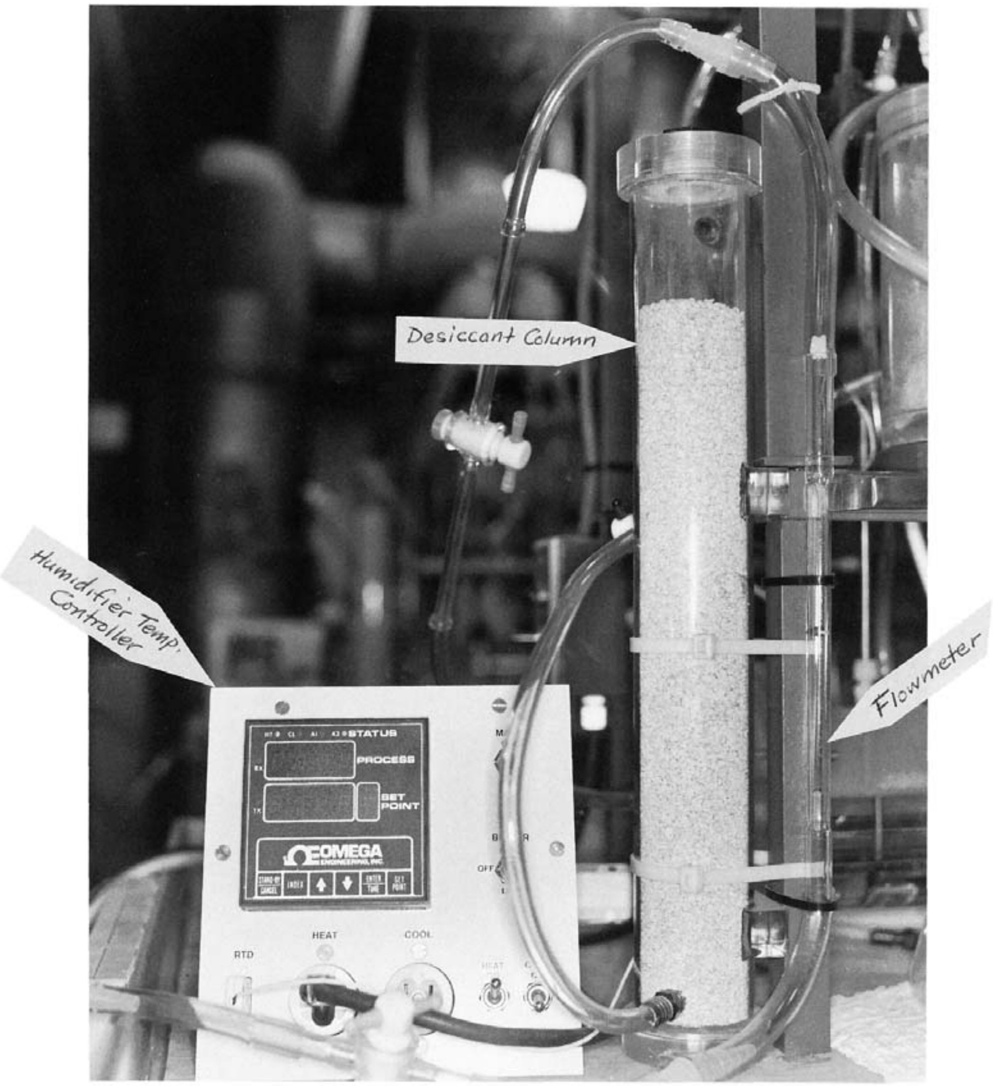

FIG. 3 Detail of Desiccant Column and Flowmeter

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 (9.6) into the humidity cell (see Fig. 4). Prior to the transfer, mix the contents of each bag by gentle rolling to eliminate possible stratification that may have occurred during sample storage.

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 Leach*:

11.2.1 The first leach (whether drip trickle or flooded), designated as the Week 0 leach, initiates the 20-week long humidity cell test and establishes the starting or initial characteristics of the leachate. Either a 500-mL or 1-L volume of water may be used for the weekly leaches, depending on the weekly pore volume desired or the quantity of solution required for analytical purposes; however, once a weekly volume has been selected, that weekly volume must remain constant throughout the 20-week testing period. A centrifuged cell culture of *Thiobacillus ferrooxidans* may be used in the first leach in order to ensure that optimum conditions for accelerated weathering are present at the beginning of the test (see Appendix X1 for the preparation of a washed cell suspension of *Thiobacillus ferrooxidans*).

NOTE 9—In the testing of mining wastes, cation (including metals and trace metals) and anion loadings are commonly high in the Week 0 leachate due to the dissolution of pre-existing soluble oxidation salts present in the sample prior to sample collection. The average number of weekly accelerated weathering cycles required to flush these pre-existing salts ranges from 3 to 5 weeks. Oxidation products observed during these 3 to 5 weeks are principally from the pre-existing salts, while those products observed after this period are considered to be solely a function of the accelerated weathering procedure. A method for estimating the amount of pre-existing oxidation salts present in a solid material sample is described by Sobek, et al (**6**). A comparison of estimated salt storage data obtained using this method with the first three weeks of humidity cell

Copyright by ASTM Int'l (all rights reserved);
Reproduction authorized per License Agreement with ANSI; Thu Feb 2 19:46:01 EST 2006

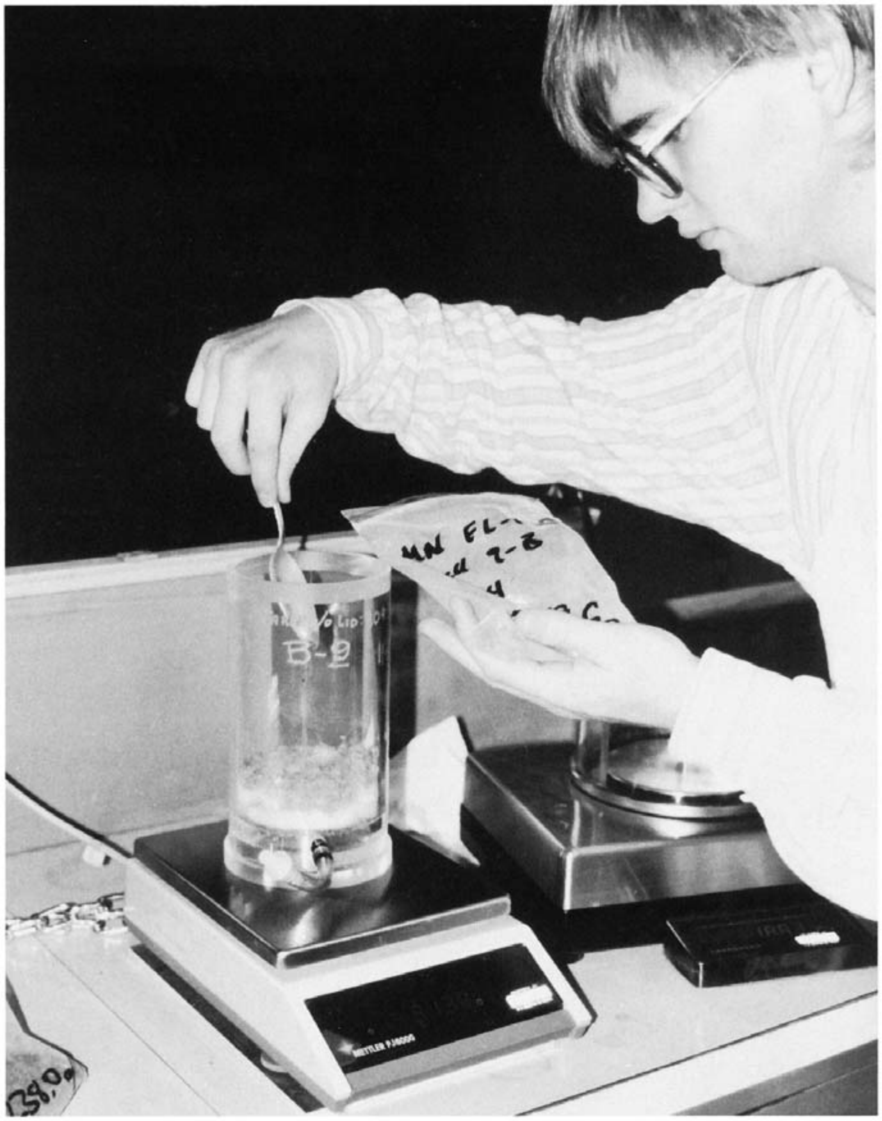

FIG. 4 Loading Humidity Cell with Filter Media and 1000-g Sample Charge

effluent loadings from three different samples is described by White and Jeffers (7).

11.2.2 Fill a separatory funnel for each cell with de-ionized water using a volumetric flask. If the leach is to be performed using the drip trickle method, set each separatory funnel above its corresponding cell, and adjust the drip rate (approximately 3 to 4 mL/min) so that the solid material sample is wetted thoroughly but not flooded.

11.2.3 A minimum of 2 to 3 h is commonly required to complete the drip trickle leach.

11.2.4 If the leach is to be performed by flooding, the separatory funnel can be used to meter 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.

11.2.4.1 Allow the flooded cell to sit for a period of 1 h before draining the leachate into the Erlenmeyer collection flask. The 1-h 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.).

11.2.5 The following is performed once the leaching process has been completed: to reduce the effects of evaporation, and to prevent the contamination of each cell by airborne contaminants, 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.

Copyright by ASTM Int'l (all rights reserved);
Reproduction authorized per License Agreement with ANSI; Thu Feb 2 19:46:01 EST 2006

11.2.6 Disconnect the cells on the day following the leach, and weigh and record the weight of each cell and Erlenmeyer collection flask. Set each filled collection flask aside for leachate analyses. (Measurements of pH and Eh and sample preservation procedures must be performed as soon as possible after leachate collection.) Return each cell, replace the filled collection flasks with clean, tared Erlenmeyer flasks, hook up all connections, and begin the dry-air cycle.

11.3 *Dry-Air Cycle*:

11.3.1 The commencement of the three-day dry-air period marks the beginning of each new weekly cycle of the accelerated 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.

11.3.2 To perform the dry-air cycle, feed air is metered to the humidity cell array with a flowmeter (see 6.3) set at a target rate in the range of 1 to 10 L/min per cell, depending on the objectives of the testing. The air flow rate must be checked daily and adjusted to the target value ±0.5 L/min.

11.3.3 Feed air from the flowmeter 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 6.23).

11.3.4 To maintain similar positive air pressure through the cells, attach a water-bubbling vessel 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).

11.3.5 The dry air is passed through each humidity cell for three days. Air flow rates from each of the cells should be checked each day, recorded, and adjusted, if necessary. See also Note 10.

11.4 *Wet-Air Cycle*:

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

11.4.2 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).

11.4.3 The water temperature in the humidifier is maintained at 30 ± 2°C to ensure that the sparged air maintains a relative humidity of approximately 95 % as measured with a hygrometer (see 6.23) from one of the humidifier exit lines (see Fig. 1). Air flow rates to each of the cells should be checked each day, recorded, and adjusted, if necessary.

NOTE 10—It is good practice to measure the air flow rates and relative humidity of the air exiting each humidity cell during each day of the three-day dry- and wet-air periods; the measurements should be taken at the same time each 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).

NOTE 11—Coals spoils in eastern states are commonly saturated; Caruccio (**11**) has suggested the following geographic control alternative to the dry-air versus saturated-air scheduling:

(*1*) *Eastern States Samples*—Six days of saturated air (versus three days dry/three days wet); and

(*2*) *Western States Samples*—Three days dry/three days wet.

11.5 *Subsequent Weekly Leaches*:

11.5.1 A second leach with water is performed on the day following the end of the three-day wet-air period (that is, 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.

11.5.2 Subsequent 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 11.2.2-11.2.5. Weekly weighing of the test cells is optional.

11.6 It is recommended that the weekly accelerated weathering cycles described in 11.2 11.311.4 11.5 be performed for a minimum of 20 weeks.

NOTE 12—Additional weeks of accelerated weathering may be required to demonstrate the nature of the material, depending on the chemical composition of the solid material. For some metal mining wastes, researchers have shown that as much as 60 to 120 weeks of accelerated weathering data may be required to demonstrate the complete weathering characteristics of a particular sample (**7, 12**). The criteria for ending the testing may be site specific and should be agreed upon before initiating the testing.

11.7 *Leachate Analyses*:

11.7.1 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. Suggested minimum weekly analyses should include pH, Eh, conductivity, and sulfate-ion concentration; acidity, alkalinity, and selected metals could be analyzed less frequently (for example, at Weeks 0, 1, 2, 4, 8, 12, 16, and 20), especially if changes in leachate chemistry are slow. 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.

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

11.7.3 Fig. 5 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 accelerated weathering (see 12.9 for the calculation of cumulative load and release rates).

11.8 *Weathered Solid Material Analyses*:

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

11.8.2 Transfer the weathered residue and filter media to a clean drying pan, and dry to constant weight at 50 ± 5°C. Record the final weight.

NOTE 13—Perform any gross sample examination (for example, sample texture and weathering product mineralogic characterization) desired for the weathered residues prior to pulverization. To facilitate such an examination, empty the humidity cell 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).

Copyright by ASTM Int'l (all rights reserved);
Reproduction authorized per License Agreement with ANSI; Thu Feb 2 19:46:01 EST 2006

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)											
*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^A – 3)											
*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^A – 3)											
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)										
*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^A – 3)										
*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^A – 3)										
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} Some of these blocks did not have data and were filled by linear interpolation, or these values were affected by the linear interpolation.

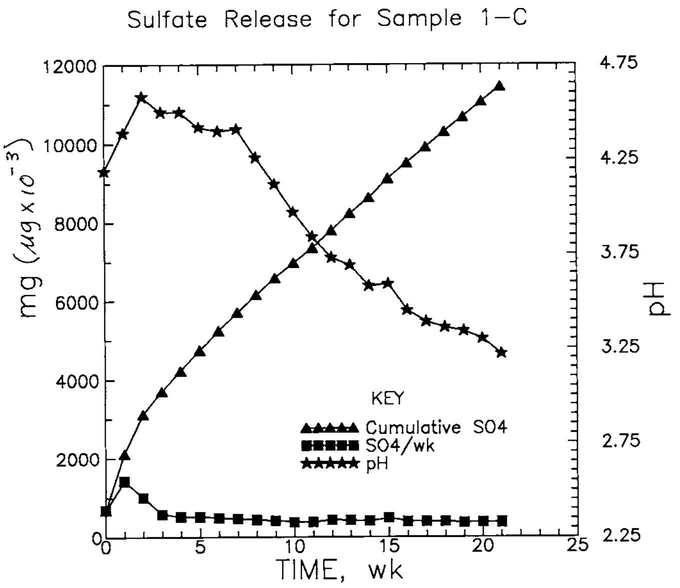

FIG. 5 Plot of Temporal Variation of pH, Sulfate Load, and Cumulative Sulfate Load from 21 Weeks

11.8.3 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 accelerated 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.

11.8.4 After drying to constant weight and prior to splitting, use an instrument such as a rolling pin to break up cemented lumps in the sample (if the cemented lumps cannot be sufficiently reduced to pass through the chutes of a riffle splitter, remove, record, and weigh separately):

11.8.4.1 Split the sample into halves using a riffle splitter with 2.54-cm (1-in.) chutes, and reserve one half to determine the particle size distribution in accordance with Test Method E 276.

11.8.4.2 Split the remaining half sample into two quarters using a riffle splitter with 2.54-cm (1-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 E 276.

11.8.5 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.

12. Calculation

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

$$M_f = M_{hf} - M_h \tag{1}$$

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:

Copyright by ASTM Int'l (all rights reserved);
Reproduction authorized per License Agreement with ANSI; Thu Feb 2 19:46:01 EST 2006

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

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 interstitial leachant contained in the solid material at the completion of the leach:

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

where:

M_i = mass of the residual interstitial leachant contained in the material, g,
M_{hfsw} = mass of the humidity cell, filter, and solid material 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 tared 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. Values below detection limits for the analytical method have zero loading for the affected week.

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 calculated head concentration of the constituents of interest:

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

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 Calculate the difference between the initial chemical analyses and the calculated head for the constituents of interest. It is recommended that if they differ by more than 10 %, the quality assurance procedures be checked, any deficiencies be subsequently corrected, and the analyses be repeated.

NOTE 14—Although agreement within 10 % is desirable, several assays must be summed to determine the total load that increases the potential for error. Moreover, determining the head concentration may be difficult since small volumes are typically analyzed. Normalization of the weekly loadings may be required based on the beginning and ending residue analyses.

NOTE 15—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.):

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

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.

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

Copyright by ASTM Int'l (all rights reserved);
Reproduction authorized per License Agreement with ANSI; Thu Feb 2 19:46:01 EST 2006

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, mohs	Eh, mV	pH	$CaCO_3$ equivalent, parts per thousand	
				Acidity	Alkalinity
Week 0					
Week 1					
Week 2					
Week ...					
Week 20					

[A] The flask number corresponds with the humidity cell number.

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. 5). The slope of the cumulative load plot between each inflection point is calculated and represents the release rate asμ g constituent/g sample/week for the weeks between and including the inflection points. Fig. 5 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:

TABLE 4 Calculated Release Rate for Weeks 0 to 2 and 2 to 21 from Cumulative Sulfate Plot, Fig. 5

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

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

where:

R_n = release rate of the constituent for n weeks between and including the inflection points,μ g/g/week,
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.

13. Precision and Bias

13.1 *Precision*—The precision of the procedure for measuring the rate of accelerated weathering is currently being determined using actual waste-rock samples from western United States metal mines. No accepted reference material is currently available. Within-laboratory precision performed by a single laboratory in duplicate cells is summarized in Table 5.

13.2 *Bias*—Bias has not been determined at this time because no accepted reference material is currently available for determining the bias present in the accelerated weathering procedure.

14. Keywords

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

Copyright by ASTM Int'l (all rights reserved);
Reproduction authorized per License Agreement with ANSI; Thu Feb 2 19:46:01 EST 2006

TABLE 5 Comparison of Sulfate Release Rates (in 20-Week Increments) Between Separate Humidity Cell Tests Conducted on Two Splits from Two Different Samples (Samples 1-B and 1-C)

Test Duration (20-Week Intervals)	Average Sulfate Release Rates,µ g/g/week			
	Sample 1-B		Sample 1-C	
	Cell 1A	Cell 3A	Cell 5A	Cell 7A
0–20	182.5	184.1	523.3	558.7
20–40	321.8	254.6	887.1	868.1
40–60	354.8	197.5	1985.6	1481.1
60–80	306.5	192.1	1834.5	1292.1
80–100	294.0	235.0	1783.4	1247.6
100–120	227.6	141.3	3165.0	1660.7

APPENDIX

(Nonmandatory Information)

X1. PREPARATION OF A WASHED CELL SUSPENSION OF *THIOBACILLUS FERROOXIDANS* FOR HUMIDITY CELL TESTING

X1.1 *Cell Suspension*—Prepare an iron-grown culture of *T. ferrooxidans* with a cell density of 10^6 to 10^7/mL.

X1.2 *Culture Wash Procedure:*

X1.2.1 Divide 500 mL of iron-grown culture into two 250-mL samples, and place each into a separate centrifuge bottle. Balance the two filled centrifuge bottles and centrifuge at 12 000 r/min (relative centrifugal force (RCF) 19 140) for 15 min. The iron-grown culture should be separated into a concentrated cell pellet and a nutrient- and waste-laden aqueous phase (supernatant) after centrifuging. Carefully decant and discard the resulting supernatant. This step can be repeated to increase cell density.

X1.2.2 Suspend each cell pellet in 5 to 10 mL of deionized water that has been adjusted to pH 3.0 using H_2SO_4. Combine both suspended cell pellets in a single 50-mL centrifuge tube. Place the 50-mL centrifuge tube containing both suspended cell pellets in the centrifuge, and balance it with a matching 50-mL, water-filled blank tube. Centrifuge at 20 000 r/min (RCF 36 590) for 15 min. Carefully decant and discard the supernatant.

X1.2.3 Suspend the resulting cell pellet with 20 mL of deionized water that has been adjusted to pH 3.0, and agitate until a cell suspension results. Adjust the resulting 20-mL cell suspension to a final 100-mL volume using deionized water adjusted to pH 3.0.

X1.3 *Humidity Cell Inoculation*—Obtain a 10-mL aliquot from the 100-mL volume of washed cell suspension described in X1.2.3, and bring it up to either a 500 or 1000-mL volume (depending on the leach volume selected in 11.2.1) with either 490 or 990 mL, respectively, of deionized water. Use this volume immediately to inoculate the solid material sample during the first leach cycle.

NOTE X1.1—The preparation of a replicate inoculum, as described in X1.3, is recommended; designate it as a "blank inoculum," and analyze it for all parameters along with the first leach cycle leachate.

REFERENCES

(1) Lapakko, K., *Field Dissolution of Test Piles of Duluth Complex Rock*, Report to the U.S. Bureau of Mines, Cooperative Agreement No. C0219003, Minnesota Department of Natural Resources, St. Paul, MN, 1993.

(2) Caruccio, F. T., *Proceedings of the Second Symposium on Coal Mine Drainage Research*, Bituminous Coal Research, Monroeville, PA, 1968, pp. 107–151.

(3) Lawrence, R. W., *Mining and Mineral Processing Wastes*, F. M. Doyle, ed., *Proceedings of the Western Regional Symposium on Mining and Mineral Processing Wastes*, Berkeley, CA, AIME, Littleton, CO, 1990, pp. 115–121.

(4) Ferguson, K. D., and Morin, K. A., *Proceedings, of the Second International Conference on the Abatement of Acidic Drainage*, MEND, Ottawa, Ont., 1991, pp. 83–86.

(5) Erickson, P. M., and Hedin, R. S., *Proceedings, of the Mine Drainage and Surface Mine Reclamation*, U.S. Bureau of Mines, Info. Circ. 9183, 1988, pp. 11–19.

(6) Sobek, A. A., Schuller, W. A., Freeman, J. R., and Smith, R. M., *U.S. EPA Report EPA 600/2-78-054*, 1978.

(7) White, W. W., III, and Jeffers, T. H.," The Environmental Geochemistry of Sulfide Oxidation," C. N. Alpers and D. Blowes, eds., *Proceedings of the American Chemical Society Symposium Series 550*, American Chemical Society, Washington, DC, 1994, pp. 608–630.

(8) Perry, R. H., and C. H. Chilton, 1973, "Chemical Engineer's Handbook," Fifth Edition, McGraw-Hill Book Company, pp. 3–222, 3–223, Table 3-249.

(9) Pitard, F., *Pierre Gy's Sampling Theory and Sampling Practice*, Vols I and II, CRC Press, 1989.

Copyright by ASTM Int'l (all rights reserved);
Reproduction authorized per License Agreement with ANSI; Thu Feb 2 19:46:01 EST 2006

(10) Brodie, M. J., Broughton, L. M., and Robertson, A. M., A Conceptual Rock Classification System for Waste Management and a Laboratory Method for ARD Prediction from Rock Piles, *In Proceedings of the Second International Conference on the Abatement of Acidic Drainage*, Montreal, Canada, September 16–18, 1991, pp. 119–135.

(11) Caruccio, personal communications, May 1995.

(12) Lapakko, K., *Mining and Mineral Processing Wastes*, F. M. Doyle, ed., *Proceedings of the Western Regional Symposium on Mining and Mineral Processing Wastes*, Berkeley, CA, AIME, Littleton, CO, 1990, pp. 81–86.

ASTM International takes no position respecting the validity of any patent rights asserted in connection with any item mentioned in this standard. Users of this standard are expressly advised that determination of the validity of any such patent rights, and the risk of infringement of such rights, are entirely their own responsibility.

This standard is subject to revision at any time by the responsible technical committee and must be reviewed every five years and if not revised, either reapproved or withdrawn. Your comments are invited either for revision of this standard or for additional standards and should be addressed to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee, which you may attend. If you feel that your comments have not received a fair hearing you should make your views known to the ASTM Committee on Standards, at the address shown below.

This standard is copyrighted by ASTM International, 100 Barr Harbor Drive, PO Box C700, West Conshohocken, PA 19428-2959, United States. Individual reprints (single or multiple copies) of this standard may be obtained by contacting ASTM at the above address or at 610-832-9585 (phone), 610-832-9555 (fax), or service@astm.org (e-mail); or through the ASTM website (www.astm.org).

Copyright by ASTM Int'l (all rights reserved);
Reproduction authorized per License Agreement with ANSI; Thu Feb 2 19:46:01 EST 2006