Designation: D5744 – 07$^{\varepsilon 1}$

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—Editorial corrections made in April 2010.

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 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]

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
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

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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.

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
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

3. Terminology

3.1 *Definitions:*

3.1.1 *acid producing potential (AP)*, *n*—the maximum potential for a solid material sample to produce acidic effluent can be determined based on the total sulfur present in the sample. 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 nonferrous 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 acidic effluent produced by the oxidation of contained iron sulfide minerals; 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*—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 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 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 an acidic effluent based on the amount of carbonate present in the sample. The NP is also expressed in terms of tonnes of calcium carbonate equivalent per 1000 tonnes of solid material (**3**). 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 residual acid to calcium carbonate equivalents (**2,6**). The residual acid is subtracted from the acid added to determine the acid consumption or acidity present.

3.1.6.1 *Discussion*—It should be noted that NP tests generally overestimate the capacity of mine waste samples to neutralize acid while maintaining drainage pH $\geq$ 6.0; the calcium plus magnesium carbonate content of the sample provides a more accurate NP quantification (**7**).

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.

3.1.7 *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 ranges from 0.5 : 1 to 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 NP accessible in a mine-rock sample to neutralize acid and maintain 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, $FeSO_4·7H_2O$; 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 subsequently 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 mg sulfate ion/g of solid material sample/week). The dissolution time required for NP depletion 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-mm (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 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 6.20. 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

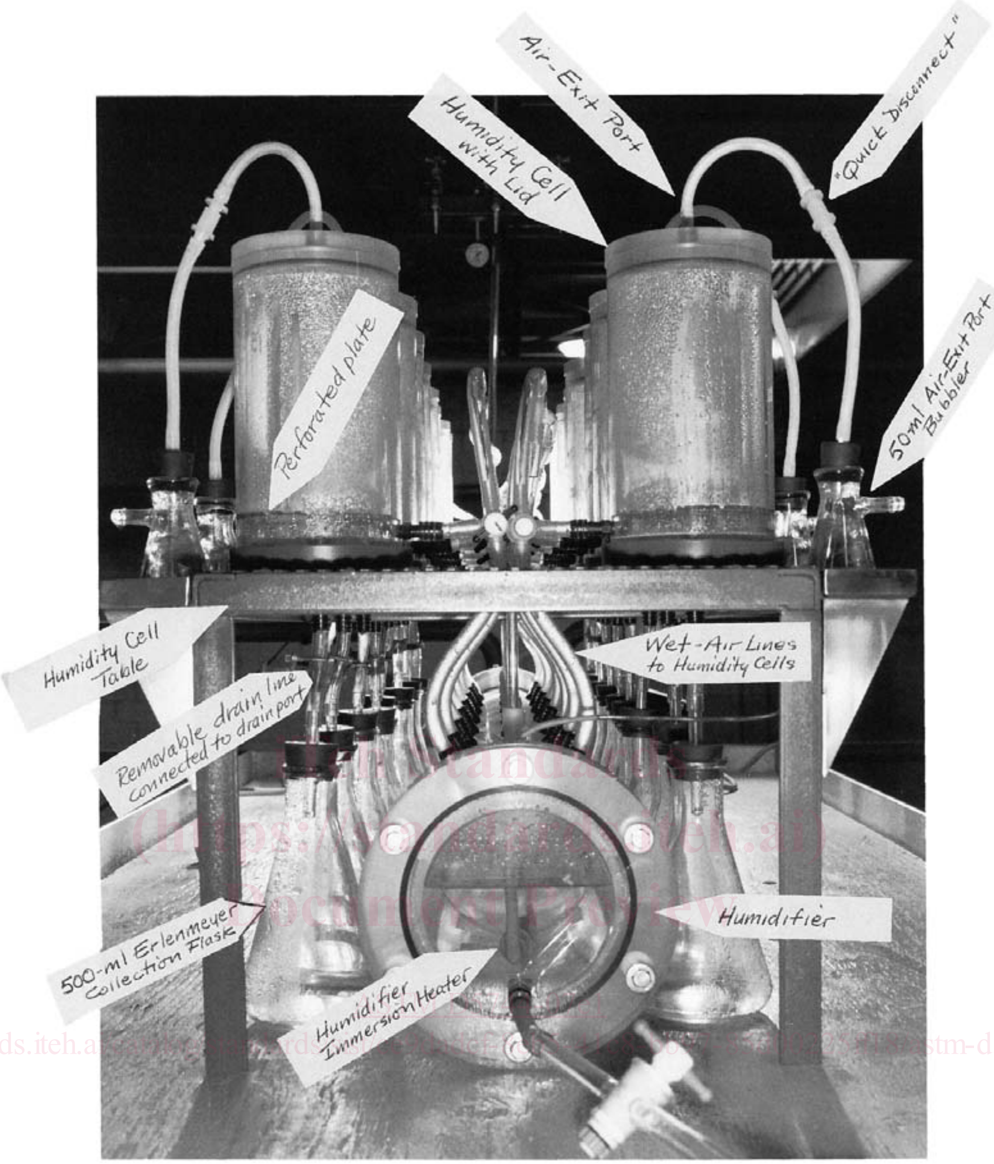

FIG. 1 Side View of 16-Cell Array (Option A)

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).[4]

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.[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 D1193. 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 in advance of the flow meter.

8. Sampling

8.1 Collect the samples 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.

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

[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. Pharmacopeial Convention, Inc. (USPC), Rockville, MD.