



Designation: ~~D3302/D3302M—19~~ D3302/D3302M – 22

## Standard Test Method for Total Moisture in Coal<sup>1</sup>

This standard is issued under the fixed designation D3302/D3302M; 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 reappraisal. A superscript epsilon ( $\epsilon$ ) indicates an editorial change since the last revision or reappraisal.

### 1. Scope

1.1 This test method covers the measurement of the total moisture in coal as it exists at the site, at the time, and under the conditions it is sampled. It is applicable to coals as mined, processed, shipped, or used in normal commercial pursuits. It is not applicable to coal-water slurries, sludges, or pulverized products under 0.5 mm-diameter sieve size. It is applicable to coals of all ranks within the recognized limitations imposed by oxidation and decomposition characteristics of lower rank coals. Because of its empirical nature, strict adherence to basic principles and permissive procedures are required for valid results (see [Appendix X1](#)). This complete standard is available to producers, sellers, and consumers as a total moisture method when other procedures or modifications are not mutually agreed on.

1.2 Since coal can vary from extremely wet (water-saturated) to completely dry, special emphasis must be placed on the sampling, sample preparation, and the moisture determination itself to ensure total reliability of measurement. Therefore, this standard entails collection of the gross sample, sample preparation, and the method of determination.

1.3 While it is recognized that such a standard may be unwieldy for routine usage in commercial operations, it can provide a common base for agreement in cases of dispute or arbitration. The complete standard is referred to as the referee method. Embodied in the standard is the commercial method starting with the crushed and divided sample when the gross sample is not too wet to crush and divide. See Practice [D2961/D2961M](#) and Test Method [D3173](#) for other moisture methods.

<https://standards.iteh.ai/catalog/standards/sist/d038784b-656f-4abd-ae77-e99ae0ced0e6/astm-d3302-d3302m-22>

1.4 *Units*—The values stated in either SI units or inch-pound units are to be regarded separately as standard. The values stated in each system are not necessarily exact equivalents; therefore, to ensure conformance with the standard, each system shall be used independently of the other, and values from the two systems shall not be combined.

1.5 *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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.*

1.6 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

### 2. Referenced Documents

#### 2.1 *ASTM Standards:*<sup>2</sup>

[D121 Terminology of Coal and Coke](#)

<sup>1</sup> This test method is under the jurisdiction of ASTM Committee [D05](#) on Coal and Coke and is the direct responsibility of Subcommittee [D05.21](#) on Methods of Analysis. Current edition approved Oct 1, 2019; Sept. 1, 2022. Published October 2019; September 2022. Originally approved in 1974. Last previous edition approved in 2017; 2019 as ~~D3302/D3302M—17~~; ~~D3302/D3302M – 19~~. DOI: ~~10.1520/D3302\_D3302M-19~~; 10.1520/D3302\_D3302M-22.

<sup>2</sup> For referenced ASTM standards, visit the ASTM website, [www.astm.org](http://www.astm.org), or contact ASTM Customer Service at [service@astm.org](mailto:service@astm.org). For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.



D388 Classification of Coals by Rank

D2013 Practice for Preparing Coal Samples for Analysis

D2234/D2234M Practice for Collection of a Gross Sample of Coal

D2961/D2961M Practice for Single-Stage Total Moisture Less than 15 % in Coal Reduced to 2.36 mm [No. 8 Sieve] Topsize

D3173 Test Method for Moisture in the Analysis Sample of Coal and Coke

D5865 Test Method for Gross Calorific Value of Coal and Coke

D7430 Practice for Mechanical Sampling of Coal

2.2 ISO Standard:<sup>3</sup>

ISO 13909-4 Hard Coal and Coke - Mechanical Sampling - Part 4: Coal - Preparation of Test Samples

### 3. Terminology

3.1 *Definitions*—For additional definitions of terms used in this test method, refer to Terminology D121.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *air drying, n*—a process of partial drying of a coal sample to bring it to near equilibrium with the atmosphere in the room in which further reduction/division of the sample is to take place.

3.2.2 *air-dry loss, n*—the loss in mass, expressed as a percent, resulting from each air-drying operation.

3.2.3 *easily oxidized coals, n*—low-rank coals such as subbituminous or lignitic coals.

3.2.4 *equilibrium, n*—condition reached in air drying when the change in mass of the sample, under conditions of ambient temperature and humidity, is no more than 0.1 %/h or 0.05 %/½ h.

3.2.5 *residual moisture, n*—that moisture remaining in the sample after air drying.

3.2.6 *total moisture, n*—see Terminology D121.

### 4. Summary of Test Method (See Fig. 1)

4.1 This test method is based on the loss in mass of a coal sample in an air atmosphere under rigidly controlled conditions of temperature, time, and airflow.

4.2 *Alternative Methods:*

4.2.1 *Referee Method*, which may be used in cases of dispute or arbitration. The gross moisture sample is air dried to equilibrate it with the atmosphere at each stage of division and reduction. No air drying is necessary if the sample is already at equilibrium with the atmosphere as indicated by stable mass.

4.2.2 *Commercial Method*, which may be used in routine commercial practice or when the concerned parties agree upon this method. The crushed and divided moisture sample is air dried to equilibrate it with the atmosphere in which further division and reduction are to occur.

4.2.3 Residual moisture determination is made in a heated forced-air circulation oven under rigidly defined conditions.

4.3 Total moisture is calculated from loss (or gains) in air drying and the residual moisture.

### 5. Significance and Use

5.1 The collection and treatment of the sample as specified for the referee method is intended for the express purpose of determining the total moisture in coal. The standard is available to producers, sellers, and consumers as a method of determination when other techniques or modifications are not mutually agreed upon.

<sup>3</sup> Available from International Organization for Standardization (ISO), ISO Central Secretariat, BIBC II, Chemin de Blandonnet 8, CP 401, 1214 Vernier, Geneva, Switzerland, <http://www.iso.org>.

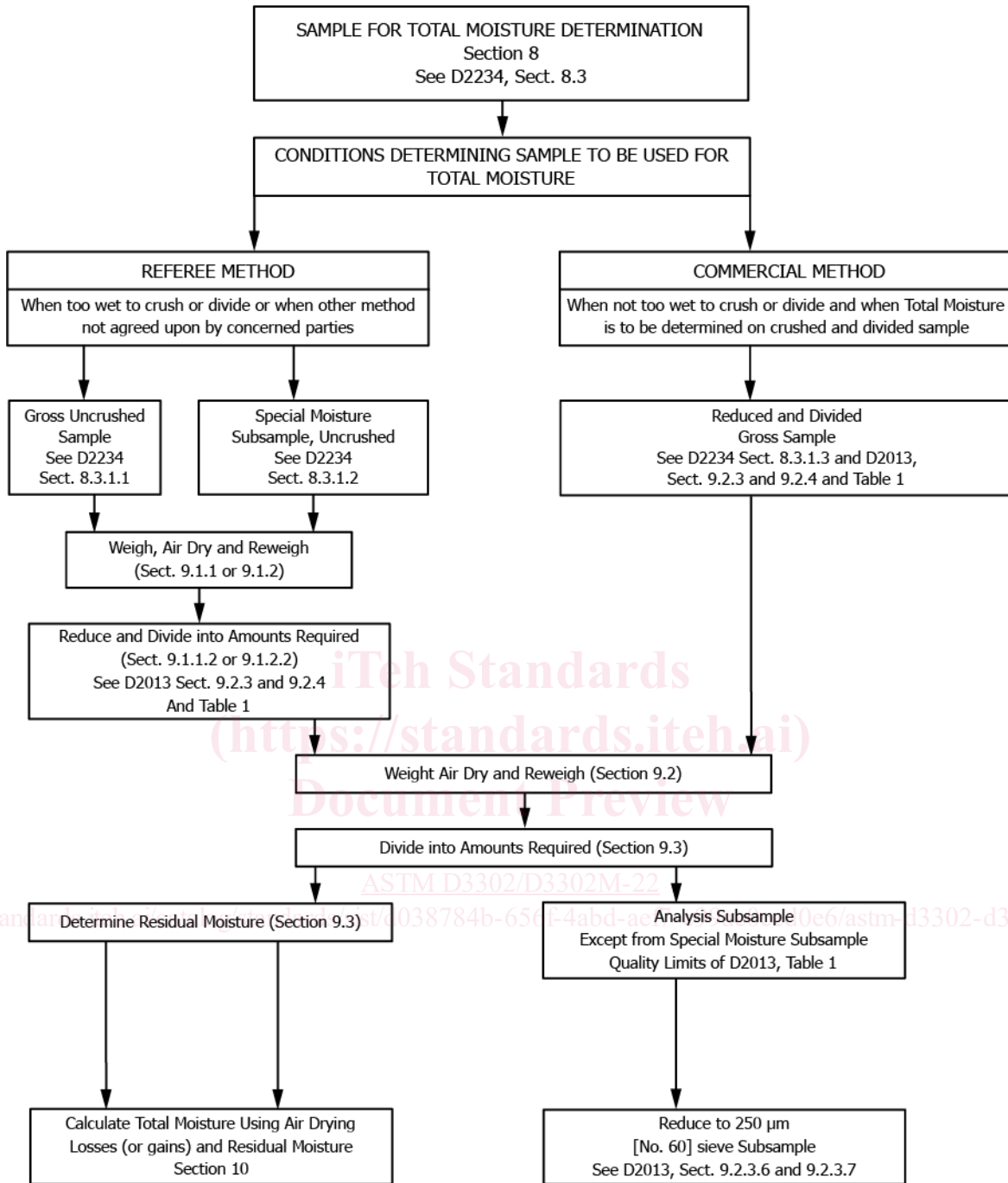


FIG. 1 Total Moisture Determination on Gross Sample, Special Moisture Subsample, or on Crushed and Divided Sample

5.2 The commercial method, which determines total moisture content of the crushed and divided sample, is designated as the method for total moisture for routine commercial practice.

## 6. Apparatus

6.1 *Drying Floor*—A smooth clean floor area in a room free of contamination by dust or other material and that permits air circulation without excessive heat or air currents. Conditions for an air-drying floor should approach those established for oven drying as much as possible.

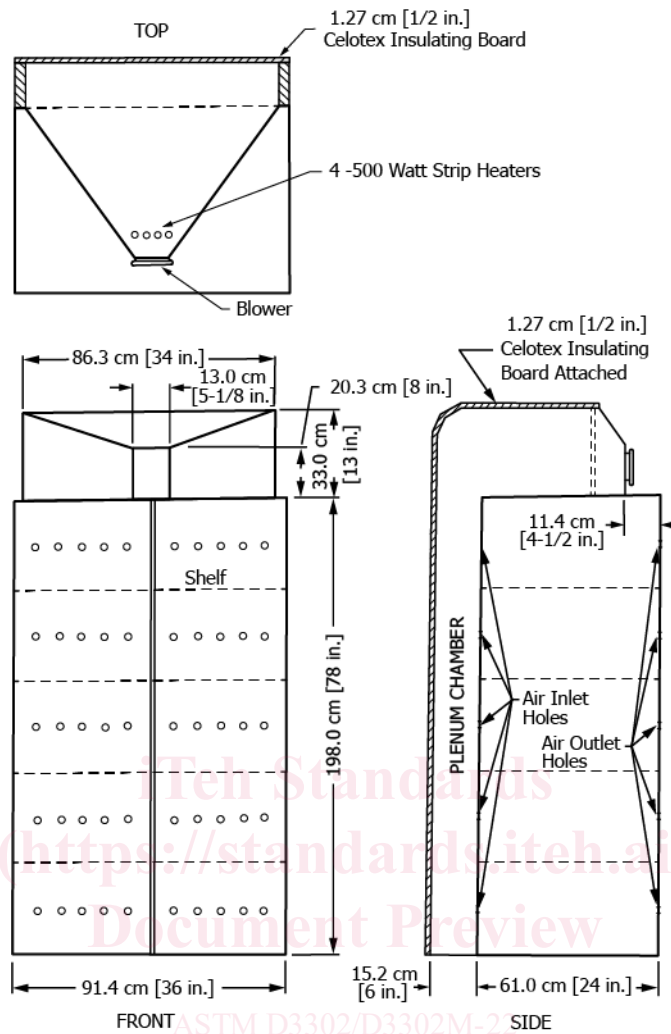


FIG. 2 Air-Drying Oven

6.2 *Air-Drying Oven*—A device for passing slightly heated air over the sample. The oven should be capable of maintaining a temperature of 10 °C to 15 °C [18 °F to 27 °F] above ambient temperature with a maximum oven temperature of 40 °C [104 °F], unless ambient temperature is above 40 °C [104 °F], in which case ambient temperature shall be used. In the case of easily oxidized coals, the temperature should not be more than 10 °C [18 °F] above ambient temperature. Air changes shall be at the rate of one to four per minute. A typical oven is shown in Fig. 2.

### 6.3 *Drying Pans:*

6.3.1 *Pans for Gross Sample*, noncorroding, mass-stable at temperature used, of sufficient size so that the sample can be spread to a depth of not more than twice the diameter of the largest particles if larger than 13 mm [0.5 in.] or not more than 25 mm [1.0 in.] depth for smaller coal, with pan sides about 50 mm to 75 mm [2 in. to 3 in.] high.

6.3.2 *Pans for Crushed and Divided Sample*, noncorroding, mass-stable at temperature used, of sufficient size so that the sample can be spread to a depth of not more than 25 mm [1.0 in.] with sides not more than 38 mm [1.5 in.] high.

6.4 *Scale (Gross Sample)*—A scale of at least 45 kg [100 lb] capacity and sensitive to 23 g [0.05 lb] in 45 kg [100 lb].

6.5 *Balance (Crushed Sample)*, sensitive to 0.1 g with a capacity sufficient to weigh pan, sample, and container.

6.6 *Laboratory Sample Containers*—Heavy vapor-impervious bags, properly sealed, or noncorroding cans such as those with an

airtight, friction top or screw top sealed with a rubber gasket and pressure-sensitive tape for use in storage and transport of the laboratory sample. Glass containers, sealed with rubber gaskets, can be used, but care must be taken to avoid breakage in transport.

6.7 *Drying Oven* (for residual moisture on 250  $\mu\text{m}$  [No. 60] sieve by 0 sample)—This oven is described in Test Method **D3173** and can be of the form illustrated in Fig. 1 in Test Method **D3173**.

6.8 *Analytical Balance*, sensitive to 0.1 mg (for residual moisture on 250  $\mu\text{m}$  [No. 60] by 0 sample).

6.9 *Capsules*, with covers, described in Test Method **D3173**.

## 7. Precautions

7.1 In collecting, handling, reducing, and dividing any moisture sample, all operations shall be done rapidly and in as few operations as possible, since moisture loss depends on several factors other than total moisture content, such as time required for crushing, atmospheric temperature and humidity, and type of crushing equipment.

7.2 While awaiting preparation, the uncrushed gross moisture sample shall be sealed in appropriate containers in order that it be protected from moisture change as a result of exposure to ambient air, rain, snow, wind, and sun, or contact with absorbent materials.

7.3 If the gross sample requires air drying, then the initial mass of the original gross moisture sample and container shall be recorded, and the moisture loss or gain of sample and containers shall be determined before the sample is reduced.

7.4 Whenever a distinct change of humidity occurs during the course of preparation of an air-dried sample, the subsample should be weighed and equilibrated with the new atmosphere and the mass loss or gain used in the calculation of total moisture content.

7.5 Whenever subsamples are stored or transported and moisture condenses on the container, then the container and subsample shall be weighed, equilibrated to the new atmosphere by air drying, and the mass loss or gain shall be used in the calculation of total moisture content.

7.6 Since most coals have a tendency to oxidize on exposure to air, the air-drying procedure should not be prolonged past the time necessary to bring the sample to equilibrium with the temperature and humidity of the air in the room in which further reduction and division are to be made. Easily oxidized coals must not be air dried at a temperature exceeding 10 °C [18 °F] above ambient temperature. In no case shall the air drying be done at a temperature over 40 °C [104 °F]. Air drying of low-rank coals should not exceed 18 h because of oxidation. In the case of lignite, the goal of reaching equilibrium should be weighed against the possibility of oxidation.

7.7 Protect crushed, divided, pulverized, or pulverizing samples from atmospheric changes affecting surface moisture or otherwise affecting sample integrity.

7.7.1 Procedures useful in maintaining uniform temperature and humidity conditions and minimum airflow in moisture determination and sample preparation area include the following: (1) closed dust control system, recycling filtered air; (2) hood over dust-producing equipment to minimize airflow required to remove dust; and (3) pulling makeup air from within the building to replace exhausted air or using tempered or conditioned makeup air.

7.7.2 Avoid heatup of pulverizer by: (1) using pulverizer large enough to process sample quickly and (2) allowing time for pulverizer to come to room temperature before reuse.

## 8. Sampling

8.1 The principles, terms, organization, and collection as set forth in Practice **D2234/D2234M** and Practice **D7430** shall apply to the collection of the total moisture sample. Particular attention is directed to Section 8 of Practice **D2234/D2234M** and Section 7 of Practice **D7430**. The increments as established in Table 2 of Practice **D2234/D2234M** or Table 1 of Practice **D7430** for mechanically cleaned coal are deemed adequate for general purpose sampling for total moisture.

## 8.2 Sampling of Coal for Total Moisture Determinations:

8.2.1 *Types of Moisture Samples*—Moisture determinations as specified in the method to be used are to be made on the following kinds of samples.

8.2.1.1 *Entire Gross Sample*—For referee tests, air dry the entire gross sample and measure the mass loss from the entire gross sample during this drying. This procedure can be carried out on the entire gross sample as a single batch or on groups of primary increments or as separate operations on the individual primary increments; obtain, by one of these means, the total mass loss from the entire gross sample. After this air drying, the sample can be crushed or divided, or both, as required by the referee test for moisture.

8.2.1.2 *Special Moisture Subsample*—For moisture testing, a special subsample can be taken from a gross sample before any operations of air drying or crushing. Take this subsample from the gross sample in accordance with the requirements of 8.2 of Practice D2234/D2234M or 8.2 of Practice D7430.

8.2.1.3 *Other Subsamples for Moisture Testing*—For moisture testing, a subsample can be used that is collected after the initial crushing and dividing of a gross sample. The procedures for the crushing and dividing, and for this subsequent subsampling for moisture, are given in Practice D2013.

8.2.2 *Special Precautions*—Collect samples and subsamples for moisture in such a manner that there is no unmeasured loss of moisture of significant amount. Make adequate weighings before and after drying or other operations to measure all significant mass losses.

8.2.3 *Mass of Increments*—The minimum mass of each increment must be that which is sufficient as to be free of bias. This depends on the top size of the coal in the stream being sampled, the dimensions of the collection device, and other factors of the withdrawal of the increment. Since much of the moisture tends to be distributed uniformly across the surface, moisture bias is present when the size consist of the sample is not the same as the size consist of the lot sampled. In addition, when there is no knowledge of the sampling characteristics for moisture, each increment shall not weigh less than the values in Table 2 of Practice D2234/D2234M or Table 1 of Practice D7430.

8.2.4 *Number of Increments*—The number of increments required for a given degree of precision depends on the mass of the increments, the distribution of the moisture, and the total amount of moisture. The distribution of moisture, however, is not easily evaluated independent of total moisture; consequently, the combined effects can be measured by determining the sampling characteristics for moisture.

8.2.4.1 *Moisture Sampling Based Only on Size*—When there is no knowledge of the sampling characteristics for moisture, collect at least the number of increments from the lot of coal as those given in Table 2 of Practice D2234/D2234M or Table 1 of Practice D7430. When a special moisture subsample is taken from the gross sample before any drying or crushing operations, collect the number of increments for the subsample as specified in 8.2 of Practice D2234/D2234M or 8.2 of Practice D7430.

## 9. Procedure

### 9.1 Air-Drying Loss on Gross Sample—Referee Method:

9.1.1 *Procedure A, Drying Floor*—This procedure is particularly applicable if the gross moisture sample is too large an amount to ship reasonably or is too wet to handle or ship without loss of moisture.

9.1.1.1 Weigh and record the mass of the gross moisture sample. Spread the sample on the drying floor to a depth of not more than twice the top size of the coal. Mix or stir the coal from time to time, being careful not to lose any of the coal particles. Continue the air drying and mixing until the surface of the sample appears dry. Weigh the entire sample and redistribute over the floor for additional drying. Continue the drying and stirring, weighing at 1 h to 2 h intervals until the mass loss of the total sample becomes no more than 0.1 %/h. If the sample surface appears dry, and the time required for reduction and division is well established, air drying can be stopped when the mass loss is less than 0.1 % per twice the required time for processing. *Example:* If reduction and division of the sample is expected to require 20 min, the air-drying procedure can be stopped when the rate of moisture loss is less than 0.1 %/40 min. If this procedure is used, a second air drying is required to establish the 0.1 %/h rate before the final preparation of the laboratory sample. Record the mass of the air dry sample. Avoid excess drying.

9.1.1.2 Proceed with sample reduction and division in accordance with Practice D2013, subsections 9.2.3 or 9.2.4, observing precautions of Practice D2013, Section 7. Use enclosed equipment where possible to minimize moisture change.

#### 9.1.2 Procedure B, Air Drying Oven:

9.1.2.1 Distribute the gross moisture sample over the required number of tared pans. Weigh each pan with sample as it is filled from the gross sample. Place in an air-drying oven that has been adjusted to maintain temperature no more than 10 °C [18 °F] above ambient temperature for easily oxidized coals or no more than 15 °C [27 °F] above ambient temperature for other coals (oven temperature not to exceed 40 °C [104 °F]). Ambient air may be used with no heating. Maintain air circulation through the oven at a rate of one to four air exchanges per minute, but in no case should it be sufficiently high to blow fine particles from the pans. Gently stir the sample from time to time to ensure uniform drying throughout the sample. Continue drying with intermittent stirring until the coal surfaces appear to be dry. Remove from oven, weigh, and record the mass. Return the pans with sample to the oven and continue the operation. Calculate the percent mass loss. Repeat the drying and weighing process at 1 h to 2 h intervals until the mass loss is less than 0.1 %/h. Allow the sample to reach equilibrium with ambient temperature and humidity before the final air dry mass is recorded. Avoid excess drying.

9.1.2.2 Proceed with sample reduction and division in accordance with Practice D2013, subsections 9.2.3 or 9.2.4, observing precautions of Practice D2013, Section 7. Use enclosed equipment where possible to minimize moisture change.

#### 9.2 Air-Drying Loss on Crushed and Divided Coal Sample, Referee and Commercial Method:

9.2.1 Proceed with determination of air-drying losses (or gains) without unnecessary delay under either of the following conditions:

9.2.1.1 When the air-drying loss has been determined on the gross sample (9.1) and it has been crushed and divided in accordance with 9.1.1.2 or 9.1.2.2. (See Note 1.)

9.2.1.2 When the gross sample is not too wet to crush and has been crushed and divided to 4.75 mm [No. 4] or 2.36 mm [No. 8] top sieve size. However, when the gross sample was collected by a mechanical coal-sampling system, and therefore the final accumulation of crushed and divided increments is a system sample, the requirement to proceed with initial air drying may not always be appropriate since the following must be given consideration.

(1) For cases where a system sample contains no visible surface moisture and has a mass substantially in excess of the minimum specified in Practice D2013, Table 1—for example, 20 kg of 4.75 mm [No. 4] sieve size—(Note 2), it is expected that reduction and division—for example, to 1000 g of 2.36 mm [No. 8] sieve size—of the entire sample using appropriately sized equipment can be performed prior to air drying without noticeably affecting the total moisture content (Note 3).

(2) For cases where a system sample does contain visible surface moisture or is close to or below the specified minimum mass, an initial air drying is advisable to best account for all the moisture.

(3) For cases where there is concern that initial air drying followed by reduction and division could facilitate moisture loss due to increased surface area or newly opened pore spaces in the coal, or both – for example, when 2.36 mm [No. 8] sieve size has been produced from some larger size—a second air drying could be performed. (See Note 1 regarding air drying of samples more than once.)

NOTE 1—Common laboratory work flow often involves testing samples for analytes in addition to moisture. Multiple air-drying periods can have deleterious effects on some test results for those additional analytes—for example, calorific value and rheological properties. Appendix X2 contains a workaround for such situations.

NOTE 2—For guidance on minimum sample mass when top sizes are greater than specified in Table 1 of Practice D2013, it is advisable to consult Table 1 in Part 4 of ISO 13909.

NOTE 3—Appendix X2 presents important considerations regarding unmeasured changes in moisture when processing bias test reference samples. Information contained there relating to sample mass and equipment capacities also applies to working with system samples.

9.2.2 The sample must remain in an airtight container with minimum unused volume until testing is started. Preparation of top-sieve size 2.36 mm [No. 8] by 0 samples is described in the following method, but 4.75 mm [No. 4] by 0 samples can be air dried by this method as a stage in the determination of total moisture, using the appropriate quantity.

9.2.3 Weigh and record the tare mass of the air-drying pan or pans. Quickly empty the sample from its airtight container into the tared pan or pans and weigh with container (Note 4), recording to the nearest 0.1 g. Spread the sample evenly to a depth of no more