



Designation: D3302/D3302M – 17

Standard Test Method for Total Moisture in Coal¹

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 reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

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 Test Methods [D2961](#) and [D3173](#) for other moisture methods.

1.4 The values stated in either SI units or inch-pound units are to be regarded separately as standard. The values stated in each system may not be exact equivalents; therefore, each system shall be used independently of the other. Combining values from the two systems may result in non-conformance with the standard.

¹ 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.

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

2. Referenced Documents

2.1 *ASTM Standards:*²

[D121 Terminology of Coal and Coke](#)

[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 Test Method 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](#)

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*—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*—the loss in weight, expressed as a percent, resulting from each air-drying operation.

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

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

² 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.

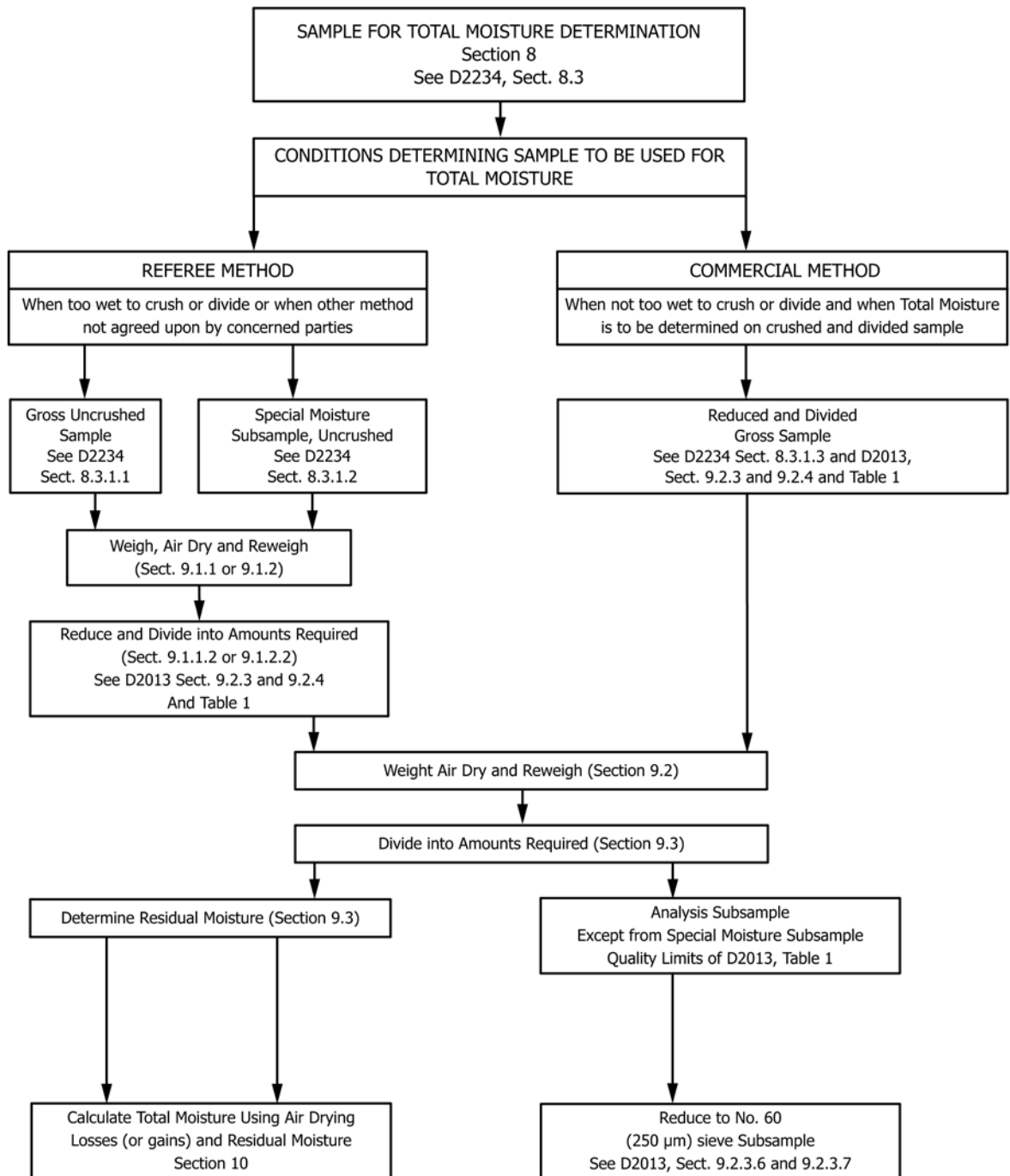


FIG. 1 Total Moisture Determination on Gross Sample, Special Moisture Subsample, or on Crushed and Divided Sample

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

3.2.6 *total moisture*—see Terminology D121.

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

4.1 This test method is based on the loss in weight 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 weight.

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.

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.

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 to 15°C (18 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, weight-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 to 75 mm [2 to 3 in.] high.

6.3.2 *Pans for Crushed and Divided Sample*, noncorroding, weight-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-lbs] capacity and sensitive to 23 g [0.05 lbs] in 45 kg [100 lbs].

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

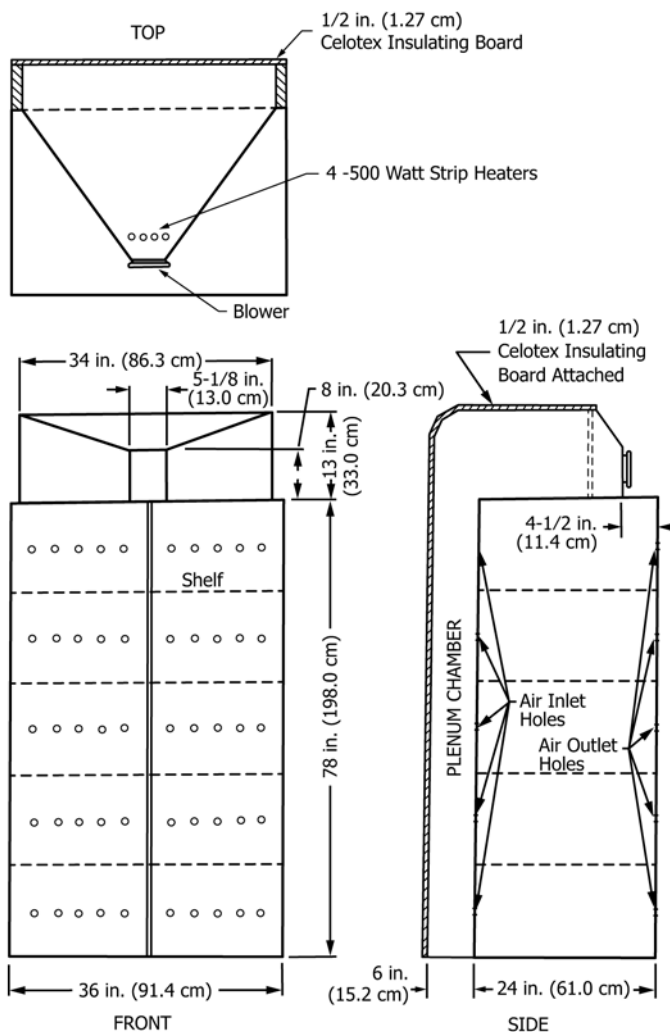


FIG. 2 Air-Drying Oven

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-µm (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-µm (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 weight 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 weight 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 weight 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 above ambient temperature. In no case shall the air drying be done at a temperature over 40°C. 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 weight 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 weight 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 weight losses.

8.2.3 *Weight of Increments*—The minimum weight 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 weight 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 weight 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- to 2-h intervals until the weight 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 weight 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 weight of the air dry sample. Avoid excess drying.

9.1.1.2 Proceed with sample reduction and division in accordance with Practice **D2013**, Section 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). 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 weight. Return the pans with sample to the oven and continue the operation. Calculate the percent weight loss. Repeat the drying and weighing process at 1- to 2-h intervals until the weight loss is less than 0.1 %/h. Allow the sample to reach equilibrium with ambient temperature and humidity before the final air dry weight is recorded. Avoid excess drying.

9.1.2.2 Proceed with sample reduction and division in accordance with Practice **D2013**, Section 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.

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.

9.2.2 The minimum weight of the crushed and divided sample is specified in Practice **D2013**, Table 1. 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 weight 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 1**), recording to the nearest 0.1 g. Spread the sample evenly to a depth of no more than 25 mm [1.0 in.], preferably less for shorter drying time, unless the coal is subbituminous C or lignite rank (Classification **D388**), or the rank is not known, and the nominal top size is 2.36 mm (No.8) in which case, spread the sample evenly (using a strike plate) to a depth less than 19.1 mm [0.75 in.] but no less than 12.7 mm [0.50 in.] (**1**).³ Place both the pan containing the sample and emptied sample container in the 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. Ambient air can be used with no heating. Air changes in the oven must be at the rate of one to four per minute. (The sample may be stirred gently from time to time to promote uniform and quicker drying.) Continue drying until the coal surfaces appear to be dry. Remove the pan and container from the oven and weigh together. Carefully transfer residual coal from the dried sample container to the sample in the pan. Weigh the empty container and subtract its weight from the combined weight of pan, sample, and container recorded both before and after the first air-drying period. Calculate the percent weight loss of the sample. Return the pan or pans with sample to the oven and continue drying. Weigh at 1- to 2-h intervals until the weight loss drops to a level near the 0.1 %/h target (about 0.2 to 0.3 %/h). Complete the air drying at ambient temperature, allowing the sample to reach equilibrium (0.1 % loss per hour or 0.05 % loss per half hour) with both ambient temperature and humidity before the final air-dry weight is recorded (**Note 2**). When this point is reached, record the final air-dry weight. Calculate the percent air-drying loss. Strictly observe the cautions of Section 7.

NOTE 1—If the moisture determination is to be made without delay in the immediate vicinity of sample preparation, the sample need not be placed in a container, but can be weighed directly in a tared drying pan.

NOTE 2—This final air-drying phase may be accomplished by leaving the sample in the oven, cutting off the heat, but continuing the air circulation at ambient temperature, or the sample may be removed from the oven for the ambient temperature-drying phase. Required time is reduced by using a heater timer adjusted to cut off the heat 2 h or more before the sample is to be weighed finally, leaving the circulating fan running. This makes overnight drying more practical and reliable. (The time required after the drying at elevated temperature to bring the weight

³ The boldface numbers in parentheses refer to a list of references at the end of this standard.

loss rate to 0.1 %/h at ambient temperature may be as much as 1½ to 3 h, perhaps more.)

9.2.4 Calculate the rate of weight change at the end of air-drying, in percent per hour, as follows:

9.2.4.1 Ensure that the sample and air-drying pan(s) are at room temperature.

9.2.4.2 Weigh the sample and pan(s) to the nearest 0.1 gram. Record this weight (W_0) and the exact time of day (T_0) to the nearest minute. Also, obtain the tare weight of the air-drying pan(s), and record as W_t .

NOTE 3—The tare weight for this calculation is best obtained from records created at the start of the air-drying period, but can also be obtained at the end of air-drying. If the latter is the case, an estimate of W_t will need to be used for this calculation, with the actual W_t determined later and the calculation repeated.

9.2.4.3 Allow ambient air to circulate over the pan(s) for a period of at least twenty minutes.

9.2.4.4 Re-weigh the sample and pan(s) to the nearest 0.1 gram. Record this weight (W_1) and the exact time of day (T_1) to the nearest minute. Calculate the rate of weight change per hour according to the following:

$$\text{Rate of weight change per hour} = \frac{(W_1 - W_0)(6000)}{(W_0 - W_t)(T_1 - T_0)} = \%/\text{hour} \quad (1)$$

The factor of 6000 in the above calculation converts the ($T_1 - T_0$) minutes difference to hours, and expresses the quotient as a percent. A negative result to the calculation indicates a rate of weight loss; a positive result indicates a rate of weight gain. In calculating the percent air-dry loss later in this Method, use W_1 (rather than W_0) in computing L , the loss of weight during air-drying.

9.3 Residual Moisture on Prepared Sample:

9.3.1 Immediately after obtaining the final air-dry weight on the 2.36-mm (No. 8) sieve size sample, prepare the 250- μm (No. 60) sieve size sample in accordance with Practice D2013, Sections 9.2.3.6 and 9.2.3.7, placing the sample in a tightly sealed container.

9.3.2 The 250- μm (No. 60) sieve size sample for residual moisture determination shall have a minimum weight of 50 g.

9.3.3 Determine residual moisture on the 250- μm (No. 60) sieve size sample without prolonged delay after its preparation to avoid unmeasured moisture change and oxidation, especially for low-rank, easily oxidized coals.

NOTE 4—Oxidation after sampling of susceptible low-rank coal or lignite may result in a reduction of the calorific value. Unnecessary exposure of the sample to air from the time of sampling or delay in analysis shall be avoided.

9.3.4 Mix sample thoroughly with mixing wheel or otherwise before extracting portions.

9.3.5 (This procedure, as well as an acceptable alternative, may be found in Test Method D3173.) Heat an empty capsule under the conditions at which the sample is to be dried, place the cover on the capsule, cool over a desiccant for 15 to 30 min, and weigh. Dip out with a spoon or spatula from the sample approximately 1 g of the sample. Put this quickly into the

capsule, close, and weigh at once to ± 0.1 mg. Place the uncovered capsule in a preheated oven ($107 \pm 3^\circ\text{C}$) through which passes a current of air that has been dried by a suitable desiccant such as Drierite or magnesium perchlorate or other efficient air-drying procedure. Close the oven at once and heat for 1 h. Open the oven, remove and cover the capsule quickly, cool in a desiccator over desiccant, and weigh as soon as cooled to ambient temperature.

NOTE 5—The residual moisture content in analytical samples can change when the samples are exposed to an atmosphere in which the relative humidity differs from that prevailing during sample preparation. Consequently, for tests of certain properties (such as calorific value, see Test Method D5865, Section 12.3), in which a small error may have significant economic impact, it is advisable to measure residual moisture at the same time the economically significant property is determined.

10. Calculation

10.1 Calculate the percent total moisture, M , as follows when one stage of air drying has been used:

$$M = [R(100 - ADL)/100] + ADL \quad (2)$$

where:

M = total moisture, weight %;
 ADL = air dry loss, weight %; and
 R = residual moisture, weight %.

10.1.1 Calculate percent air-dry loss, ADL , as follows:

$$ADL = (L/G) \times 100 \quad (3)$$

where:

ADL = air-dry loss, weight %;
 L = loss in weight in air drying, g; and
 G = weight of gross sample, g.

10.1.2 Calculate percent residual moisture, R , as follows:

$$R = [(W - H)/W] \times 100 \quad (4)$$

where:

W = weight of sample used, g and
 H = weight of sample after heating, g.

10.2 Calculate the percent total moisture, M , as follows when two stages of air drying have been used (on uncrushed gross sample and on crushed and divided laboratory sample or on crushed and divided samples from two stages of crushing, for instance, to the 4.75- and 2.36-mm (Nos. 4 and 8) top sieve sizes):

$$M' = [R(100 - ADL')/100] + ADL' \quad (5)$$

$$M = [M'(100 - ADL)/100] + ADL \quad (6)$$

where:

M = total moisture;
 M' = moisture in crushed and divided laboratory sample;
 ADL = air-dry loss, uncrushed gross sample or first-stage crushed and divided sample;
 ADL' = air-dry loss, laboratory sample; and
 R = residual moisture.

10.2.1 Calculate air-dry losses ADL and ADL' as for ADL in 10.1.1.

TABLE 1 Concentration Range and Limits for Repeatability and Reproducibility for the Determination of Total Moisture in Coal

Coal	Range	Repeatability Limit, r	Reproducibility Limit, R
Bituminous	1.6–7.9%	0.14	0.62
Subbituminous and lignite	12.4–31.2%	0.42	0.70

10.2.2 Calculate percent residual moisture, R , as in 10.1.2.

11. Precision and Bias⁴

11.1 *Precision*—The precision of this test method for the determination of Total Moisture in Coal. The repeatability and reproducibility limits given in Table 1 apply only to coals without free (surface) moisture in the gross sample before preparation of No. 8 (2.36-mm) sieve-size subsamples. When duplicate No. 8 (2.36-mm) sieve-size subsamples are air dried, reduced to No. 60 (250- μ m) sieve size, and their residual

⁴ Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D05-1015.

moistures determined, the following repeatability and reproducibility limits apply to the resulting total moisture values.

11.1.1 *Repeatability*—the value below which the absolute difference between two test results of separate and consecutive test determinations, carried out on the same sample in the same laboratory by the same operator using the same apparatus on samples taken at random from a single quantity of homogeneous material, may be expected to occur with a probability of approximately 95%.

11.1.2 *Reproducibility*—the value below which the absolute difference between two test results carried out in different laboratories using samples taken at random from a single quantity of material that is as homogeneous as possible, may be expected to occur with a probability of approximately 95%.

11.2 *Bias*—See Appendix X1 on accuracy of determination of moisture in coal.

11.2.1 Since there is no acceptable reference materials for determining the bias for this test method, no statement on bias is being made.

12. Keywords

12.1 air-drying; air-drying loss; coal; residual moisture; total moisture

APPENDIXES

(Nonmandatory Information)

X1. ACCURACY OF DETERMINATION OF MOISTURE IN COAL

X1.1 The accurate determination of moisture in coal of various ranks has long been a subject of discussion and investigation. As has been pointed out in the referenced investigations, one of the major difficulties in assigning absolute merit to a particular procedure is the multiplicity of conditions under which water exists in coals and the difficulties involved in obtaining sharp separation and distinction among these conditions.

X1.2 As stated by Gauger (2), “Water recoverable from coal is obtained from the following sources: (1) decomposition of organic molecules (sometimes called combined water), (2) surface-adsorbed water, (3) capillary-condensed water, (4) dissolved water, and (5) water of hydration of inorganic constituents of the coal.”

X1.3 Brown (3) further refers to (1) “Free” or “adherent” moisture (essentially surface adsorbed) possessing the physical properties of ordinary water, (2) physically bound or “inherent” moisture of vapor pressure lowered by the small diameter of the pores of the coal structure in which it is absorbed, and (3) chemically bound water of hydration or “combined” water.

X1.4 It becomes apparent, then, that “total moisture” in principle represents a measurement of all of the water not chemically combined.

X1.5 Traditionally, thermal treatment has provided the most commonly used basis for attempting to separate the nonchemically bound water from coal, and the measurement is normally made by weight loss. The “absolute” separation of adsorbed moisture without loss of a portion of chemically bound water is most difficult, if not impossible. The separation is particularly difficult in geologically younger coals of lower rank. Investigators (4,5,6) have shown that the amount of water extracted is a function of both temperature and time. The problem is further compounded by the susceptibility of certain coals to oxidation.

X1.6 Because of such problems, investigators have proposed a number of schemes to satisfy their particular objectives in the measurement of water associated with coal. These include:

X1.6.1 Heating in air at varying temperatures and for varying time intervals.

X1.6.2 Heating in inert atmospheres (nitrogen, helium, argon, and so forth).

X1.6.3 Separation of water by distillation with benzene, toluene, xylene, kerosine, and so forth.

X1.6.4 Measurement of water by such chemical methods as Karl Fischer titrations.

X2. CONSIDERATIONS ON THE POTENTIAL FOR UNMEASURED CHANGES IN MOISTURE WHEN PROCESSING BIAS TEST REFERENCE SAMPLES

X2.1 When working with reference samples from a bias test of a mechanical sampling system it is desirable to minimize unmeasured changes in moisture, either gains or losses. (See [Note X2.1](#).) A number of issues in the handling and preparation of such samples can combine to foster an unmeasured moisture change that appears as a moisture bias erroneously attributed to the sampler. (See [Note X2.2](#).) Therefore, in handling bias test reference samples it is wise to consider, at a minimum, the following factors that can contribute to unmeasured moisture changes.

NOTE X2.1—An unmeasured change in moisture, either a gain or loss, is typically important if it is greater than the largest tolerable bias as defined in D7430, but there could be other alternative criteria depending on the circumstances.

NOTE X2.2—An apparent moisture bias (either high or low) may not necessarily be due to unmeasured moisture changes in the reference samples, but may in fact be caused by the coal sampling system itself or by moisture loss from the fractured pore spaces found in final system samples. Investigation of such sampling system issues is beyond the scope of this Appendix.

X2.1.1 *Total Moisture Content*—High total moisture is typical of washed products and low rank coals. The former can often have visible moisture on particle surfaces, and the latter have large pore spaces filled with inherent moisture. In both cases, unmeasured moisture can easily be lost during handling and processing. High total moistures can also affect the overall ease and time required to floor-air-dry bulk samples, if that procedure is performed.

X2.1.2 *Dried Coal*—Although unmeasured moisture losses are far more common than gains, the latter can sometimes occur and deserves attention when working with coals that have been dried to some degree.

X2.1.3 *Oxidation*—Upon exposure to air, most coals oxidize to some extent, and that can affect certain analytical results (e.g., calorific value, sulfur forms, free swelling index and rheological properties). Low rank coals are more easily oxidized than coals of higher rank. This is an important consideration, especially if samples are to be air dried more than once before testing in the lab.

X2.1.4 *Size Consist*—Given the same mass of material, the total surface area of a sample will be greater when the particles are small rather than large. That, in turn, can facilitate relatively easy unmeasured moisture loss from the sample with smaller particles. This loss can also be an issue when floor-air-drying because the fines may tend to cluster together and at first resist loss of surface moisture. Later, some of that moisture may escape unmeasured as the particles separate during reduction and division and the previously-hidden surface moisture is exposed.

X2.1.5 *Environmental Factors*—Processing bulk samples in unfavorable environments, such as where temperatures are substantially above or below 23°C, relative humidity is significantly below 50%, under windy conditions, etc., can act on susceptible coals to remove some of the more easily lost

moisture before it can be measured. In addition, precipitation can add moisture that is obviously not part of the sample.

X2.1.6 *Equipment Capacities*—Processing reference samples with equipment not appropriate for the mass to be reduced and divided can lead to unmeasured moisture loss. Either reduction (crushing) or division (riffing) can be problematic.

X2.1.6.1 Small-mass samples processed with equipment having large internal volumes is one issue. Samples provide the necessary moisture to “wet” or “condition” the inside surfaces of crushers and riffles and maintain a moisture-stable internal environment. The moisture removed from small samples will be a larger percentage of the total moisture than is the case for samples of larger mass, and is more likely to precipitate an unmeasured moisture loss

X2.1.6.2 Large-mass samples processed with equipment having small internal volumes is another issue. Equipment that is too small in terms of allowed throughput per unit time can lead to longer processing times that promote moisture loss, or cause pluggage that requires opening the equipment to remove the blockage which disrupts the moisture-stable internal environment. Either of these situations can cause unmeasured moisture loss.

X2.1.6.3 Processing samples with equipment of appropriate capacity reduces the chance for unmeasured moisture loss, but may not eliminate it completely. That loss could still occur, but the small amount might not be detectable. Rapid processing can also help reduce the opportunity for unmeasured moisture loss to occur.

X2.1.7 *Combinations of the Above, Together with Other Unidentified Factors*—Known and unknown elements operating in concert can be surprisingly effective at causing unmeasured changes in moisture. Particular attention is suggested regarding factors that might not be readily apparent.

X2.2 **Alternatives for Mitigation**—If important (see [Note X2.1](#)) unmeasured changes in moisture are considered likely, the following are offered as possible courses of action. Multiple approaches used together may provide a greater assurance of success.

X2.2.1 *Bias Test Design*—The test design can be changed to collect reference samples of different mass more appropriate to the situation. For example, larger samples contain an absolute amount of moisture that is greater than contained in smaller samples, and can be more tolerant of unmeasured changes in the absolute amount of moisture because the change in percentage moisture would be small. However, larger samples can take longer to process, providing more time for unmeasured moisture change to occur. Conversely, small samples, with less absolute moisture, might be more susceptible to changes in the absolute amount, giving rise to a larger percentage of unmeasured change. However, smaller samples might be processed faster, providing less time for unmeasured

moisture change to occur. The choice of larger or smaller mass reference samples requires careful consideration of these offsetting effects.

X2.2.2 Suitable Processing Equipment—Crushers, riffles, sample pans, etc., that have capacities appropriate for the sample mass to be processed can reduce the chance for unmeasured moisture change.

X2.2.3 Location of Processing—If the closest venue for processing is questionable because of low or high temperature, low humidity, high wind, rain or other factors, an alternative site might prove to be a better location. In that case, samples can be sealed in moisture-impervious containers and moved to the new site. Note that any moisture visible when opening such containers is part of the total moisture in the samples, and it may be necessary to air-dry the samples at that point.

X2.2.4 Subsample Before Air Drying or Crushing—A special moisture subsample (see section 8.2.1.2) could be extracted from each reference sample before air-drying or crushing. However, applying the cited standards that call for specific processes of mixing, dividing, subsampling and testing may not be feasible in all situations. The user may wish to consult the descriptions of equipment and other relevant information contained in ISO 13909 Part 4 on coal sample preparation that illustrates a number of alternative mechanical and manual procedures. When considering this course of action, it must be realized that subsampling a large mass sample that has not been air-dried may, in itself, cause unmeasured moisture change.

X2.2.5 Subsample After Air Drying but before Crushing, Following the Referee Method for Moisture, with a Separate Sample for Other Analyses.

X2.2.5.1 The most certain way of guarding against unmeasured moisture change is to follow the Referee Method contained in this standard (see section 9.1.) However, the procedure involves air-drying the entire quantity of each

reference sample to a rate of mass loss no greater than 0.1%/h. That can be an arduous task when sample masses are large (e.g., 50 to 100 kg or more) and the number of samples is great (e.g., 20 to 30 or more). In addition, the handling required could lead to contamination of samples or loss of particulate material, and the process could also be quite costly. Nevertheless, if that first air-drying is performed, the entire sample can then be reduced (e.g., to 2.36 mm (No 8) sieve size) and divided to produce two subsamples of convenient mass (e.g., about 1000 g each). One would be air dried a second time (at the risk of oxidizing low rank coals, but with little chance of unmeasured moisture change), then tested for residual moisture after further reduction (e.g., to 250 um (No. 60) sieve size) and division (e.g., to 50 g). The data from both air-dry losses and the residual moisture would be combined to determine the total moisture. This could be expected to produce very reliable results for moisture.

X2.2.5.2 The second subsample, actually not part of the Referee Method for moisture, would be tested for other bias test analytes without a second air-drying (so as to not risk oxidation, but with a greater chance of unmeasured moisture loss). This could be expected to produce reliable results for those other analytes on the dry basis that might have otherwise been affected by oxidation during a second air-drying, but the moisture result might not be correct. The results on this second subsample could either be used on the dry basis or calculated to the moisture-containing basis of the Referee sample, then used for bias test purposes.

X2.3 Additional Considerations—The above issues and alternatives are not intended as a complete exploration of all possibilities. Users confronted with the potential for important unmeasured moisture changes are encouraged to think critically about the problem and investigate other potential solutions.

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