Designation: D 3302 – 02a

Standard Test Method for Total Moisture in Coal[1]

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 D 2961 and D 3173 for other moisture methods.

1.4 The values stated in SI units are to be regarded as the standard. The values given in parentheses are for information only.

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:*

D 121 Terminology of Coal and Coke[2]

D 2013 Practice for Preparing Coal Samples for Analysis[2]

D 2234 Practice for Collection of a Gross Sample of Coal[2]

D 2961 Test Method for Single-Stage Total Moisture Less than 15% in Coal Reduced to 2.36-mm (No. 8 Sieve) Topsize[2]

D 3173 Test Method for Moisture in the Analysis Sample of Coal and Coke[2]

D 5865 Test Method for Gross Calorific Value of Coal and Coke[2]

3. Terminology

3.1 *Definitions*—For additional definitions of terms used in this test method, refer to Terminology D 121.

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.

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

3.2.6 *total moisture*—see Terminology D 121.

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

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[1] This test method is under the jurisdiction of ASTM Committee D5 on Coal and Coke and is the direct responsibility of Subcommittee D05.21 on Methods of Analysis.

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[2] *Annual Book of ASTM Standards*, Vol 05.06.

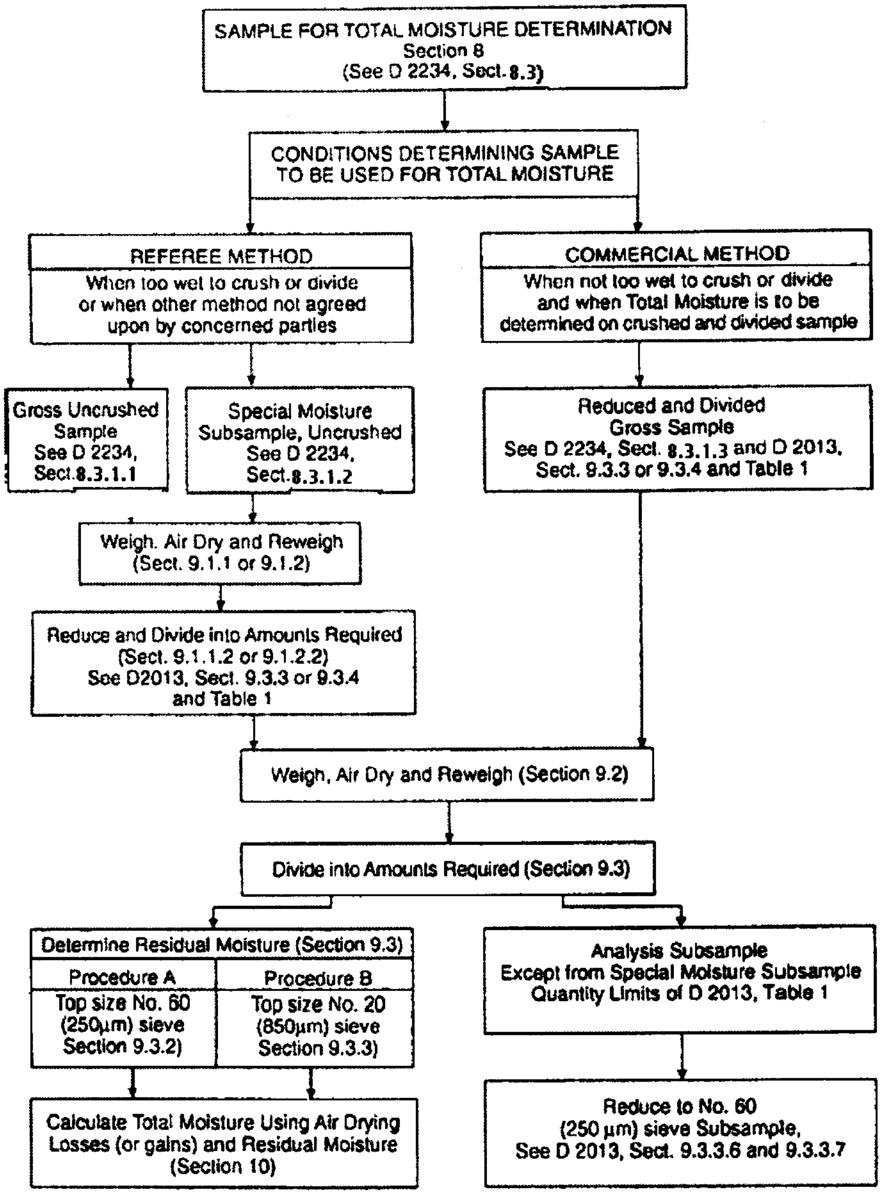

FIG. 1 Total Moisture Determination on Gross Sample, Special Moisture Subsample, or on Crushed and Divided Sample

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 should 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.

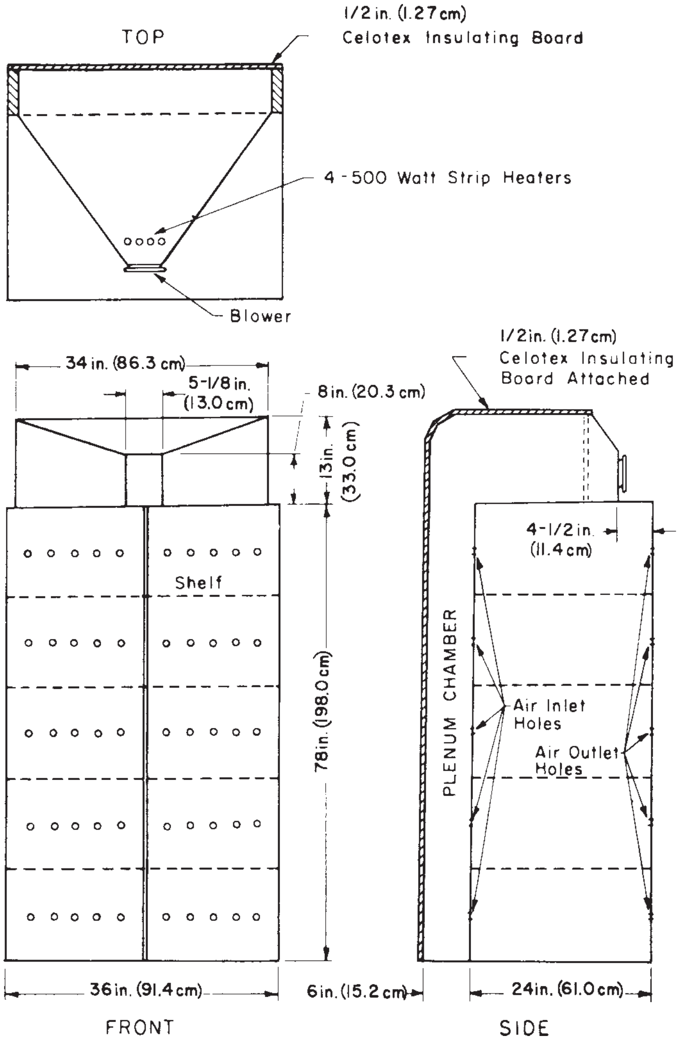

FIG. 2 Air-Drying Oven

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-µm (No. 60) sieve by 0 sample)—This oven is described in Test Method D 3173 and can be of the form illustrated in Fig. 2 in Test Method D 3173.

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 D 3173.

7. Precautions

7.1 In collecting, handling, reducing, and dividing the gross 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 D 2234 shall apply to the collection of the total moisture sample. Particular attention is directed to Section 8. The increments as established in Table 2 of Practice D 2234 for mechanically cleaned coal are deemed adequate for general purpose sampling for total moisture.

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 (Note 1). Record the weight of the air dry sample. Avoid excess drying.

NOTE 1—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.

9.1.1.2 Proceed with sample reduction and division in accordance with Method D 2013, Section 9.3.3 or 9.3.4, observing precautions of Method D 2013, 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 at least one air exchange 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 (Note 1). 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 Method D 2013, Section 9.3.3 or 9.3.4, observing precautions of Method D 2013, 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 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 Method D 2013, 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 described in the following method, but 4.75-mm (No. 4) by 0 or 850-µm (No. 20) by 0 samples can be air dried by this method as stages in the determination of total moisture, using appropriate quantities.

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 2), 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. 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 3). 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 2—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 3—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 loss rate to 0.1 %/h at ambient temperature may be as much as 1½ to 3 h, perhaps more.)

9.3 *Residual Moisture on Prepared Sample*:

9.3.1 The procedure for determining the residual moisture on the air-dried sample is dependent on the top size to which the air-dried sample is crushed or pulverized. Procedure A is used on samples prepared to a top sieve size of 250 µm (No. 60) U.S.A Standard. Procedure B is used on samples prepared to a top sieve size of 850 µm (No. 20) U.S.A Standard.

9.3.2 *Procedure A, Top Sieve Size 250 µm (No. 60) U.S.A Standard*:

9.3.2.1 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 Method D 2013, Sections 9.3.3.6 and 9.3.3.8, placing the sample in a tightly sealed container.

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

9.3.2.3 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.

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

9.3.2.5 (This procedure, as well as an acceptable alternative, may be found in Test Method D 3173.) 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 spatule 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 ± 3°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 D 5865, Section 12.3), in which a small error may have significant economic impact, it is advisable to measure residual moisutre at the same time the economically significant property is determined.

9.3.3 *Procedure B, Top Sieve Size 850 µm (No. 20) U.S.A Standard*:

9.3.3.1 Immediately after obtaining the final air-dry weight on the 2.36-mm (No. 8) sieve size sample, reduce the sample to pass 95 % through an 850-µm (No. 20) sieve using suitable crushing equipment. Divide by riffling, using the small enclosed riffle, to not less than 250 g. Quickly place in a tightly sealed container. (For small riffle, see Method D 2013, Fig. 3.)

9.3.3.2 Thoroughly mix before extracting portions for residual moisture determination.

9.3.3.3 Proceed as in 9.3.2.5, except that the quantity weighed into the capsule shall be approximately 5 g and the drying time shall be 1½ h.

10. Calculation

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

$$M = [R\,(100 - ADL)/100] + ADL \tag{1}$$

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:

$$ADL = (L/G) \times 100 \tag{2}$$

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:

$$R = [(W - H)/W] \times 100 \tag{3}$$

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):

$$M' = [R\,(100 - ADL')/100] + ADL' \tag{4}$$

$$M = [M'\,(100 - ADL)/100] + ADL \tag{5}$$

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.

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

11. Precision and Bias [3]

11.1 *Precision*—The relative precision of this test method for the determination of total moisture covers the concentration range from 1.6 to 7.9 % for bituminous coals and 12.4 to 31.2 % for subbituminous and lignitic coals. The repeatability and reproducibility intervals given below 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-mm) sieve size, and their residual moistures determined, the following repeatability and reproducibility intervals apply to the resulting total moisture values.

11.1.1 *Repeatability*—The difference in absolute value between two consecutive test results, carried out on duplicate subsamples in the same laboratory, by the same operator using the same apparatus, should not exceed the repeatability interval for more than 5 % of such paired values (95 % confidence level). When such a difference is found to exceed the repeatability, there is reason to question one or more of the test results. The repeatability interval for this test method is:

Bituminous	0.14 %
Subbituminous and lignite	0.42 %

11.1.2 *Reproducibility*—The difference in absolute value of replicate determinations, carried out in different laboratories, on representative samples, should not exceed the reproducibility interval for more than 5 % of such paired values (95 % confidence level). When such a difference is found to exceed the reproducibility interval, there is reason to question one or more of the test results. The reproducibility interval for this test method is:

Bituminous	0.62 %
Subbituminous and lignite	0.70 %

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

APPENDIX

(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 (**1**), "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 (**2**) 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 (**3**,**4**,**5**) 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.

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[3] Supporting data are available from ASTM Headquarters. Request RR D05-1015.

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