Designation: D 3302 – 07

Standard Test Method for Total Moisture in Coal[1]

This standard is issued under the fixed designation D 3302; 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 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:* [2]

D 121 Terminology of Coal and Coke

D 2013 Practice for Preparing Coal Samples for Analysis

D 2234/D 2234M Practice for Collection of a Gross Sample of Coal

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

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

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

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.

[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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[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.

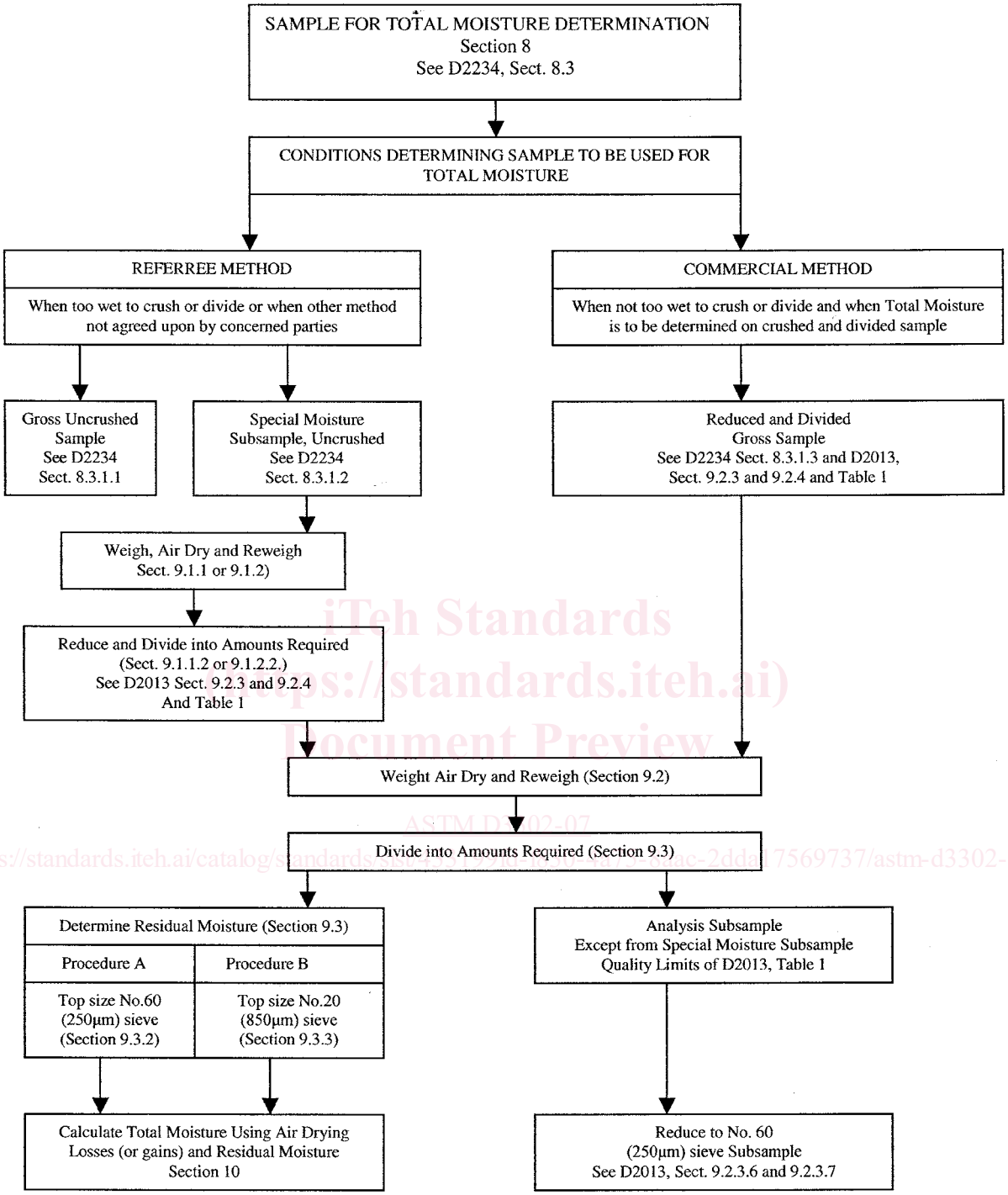

FIG. 1 Total Moisture Determination on Gross Sample, Special Moisture Subsample, or on Crushed and Divided Sample

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 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. 1 in Test Method D 3173.

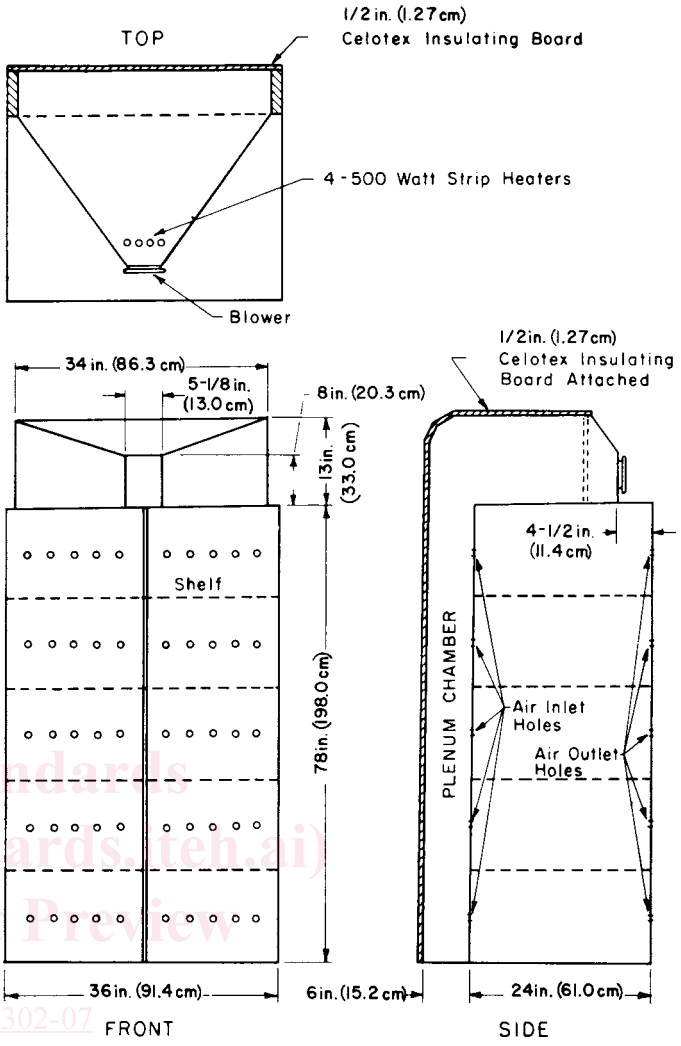

FIG. 2 Air-Drying Oven

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 Practice D 2013, Section 9.2.3 or 9.2.4, observing precautions of Practice 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 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 (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 Practice D 2013, Section 9.2.3 or 9.2.4, observing precautions of Practice 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 Practice 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 is 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.