



Designation: **D1412/D1412M – 17a** D1412/D1412M – 18

## Standard Test Method for Equilibrium Moisture of Coal at 96 to 97 Percent Relative Humidity and 30 °C<sup>1</sup>

This standard is issued under the fixed designation D1412/D1412M; 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 determination of the equilibrium moisture of coal in an atmosphere over a saturated solution of potassium sulfate at 30 °C.

NOTE 1—For information concerning the experimental work on which this test method is based, see (1-5).<sup>2</sup>

1.2 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.

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

1.4 *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>3</sup>

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

D3172 Practice for Proximate Analysis of Coal and Coke

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

D3302 Test Method for Total Moisture in Coal

D4596 Practice for Collection of Channel Samples of Coal in a Mine

E177 Practice for Use of the Terms Precision and Bias in ASTM Test Methods

E691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method

### 3. Significance and Use

3.1 This test method affords a means of estimating the inherent moisture of either coal that is wet and shows visible surface moisture or coal that may have lost some moisture. It may be used for estimating the surface, or extraneous moisture of wet coal, such moisture being the difference between the total moisture as determined by Test Method D3302 and the equilibrium moisture.

3.2 When samples are collected in conformity with Classification D388, the equilibrium moisture is considered to be equal to bed moisture with the exception of some low rank coals that yield equilibrium moisture values below bed moisture.

3.3 The results obtained by this test method are sensitive to many influences, and therefore, raw (uncorrected) equilibrium moisture data may be of limited value in and of themselves. When working with low rank coals, the results yielded by this test

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<sup>2</sup> The boldface numbers in parentheses refer to a list of references at the end of this standard.

<sup>3</sup> 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.

\*A Summary of Changes section appears at the end of this standard

method require critical assessments. It is recommended that the procedure outlined in the **Appendix X1** be applied, and the results corrected before use in situations where a more reliable estimation inherent or bed moisture for low rank coals is required. The Appendix also provides useful quality assurance information which is applicable to coals of all ranks.

**4. Apparatus**

4.1 *Water Bath or Insulated Air Cabinet*—The bath or cabinet shall be of sufficient size to accommodate several vacuum-type desiccators, and shall be provided with a temperature regulator to maintain a uniform temperature of 30.0 °C ± 0.2 °C.

4.2 *Moisture Oven*—The oven shall be so constructed as to have a uniform temperature in all parts and a minimum of air space. It may be of the type shown in the Apparatus section of Test Method **D3173**. Provision shall be made for renewing the air (or, if desired, dry oxygen-free nitrogen for subbituminous and lignitic coals) in the oven at a rate of two times per minute, with the air dried as defined in **5.1**.

4.3 *Mechanical Vacuum Pump*.

4.4 *Crusher*, laboratory, coffee-mill type.

4.5 *Sieve*, 203 mm [8 in.] diameter, with 2.36 mm [No. 8] openings.

4.6 *Shaking Machine*.

4.7 *Desiccators*:

4.7.1 *High Vacuum Desiccator*—Small vacuum-type desiccator, approximately 160 mm in diameter, when used in conjunction with a saturated K<sub>2</sub>SO<sub>4</sub> solution provides the necessary humidity for the test conditions (see **Fig. 1**).

4.7.2 *Cooling Desiccator*—Cabinet-type desiccator, when used with any of the desiccants (**5.2.1 – 5.2.3**) allows the dried test sample to cool to room temperature without substantial regain in mass from adsorption of atmospheric moisture.

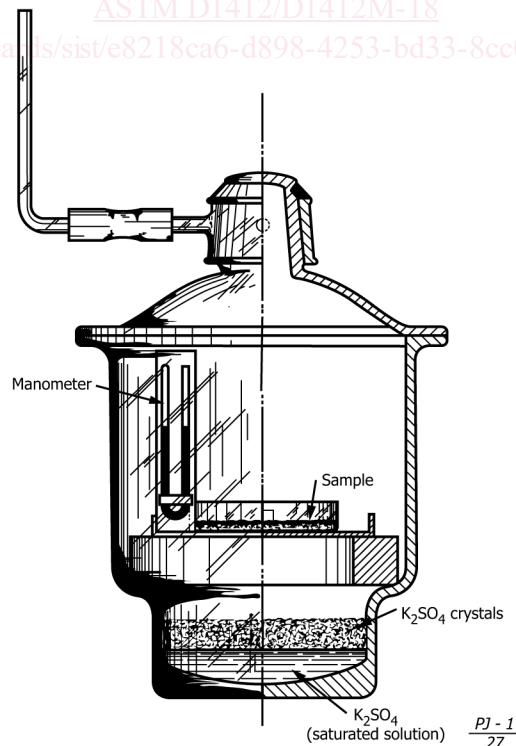
4.8 *Weighing Bottles*, glass, low-form, flat-bottom, cylindrical, 70 mm in diameter, with well-fitting covers.

4.9 *Filter Pump*, aspirator.

4.10 *Buchner-Type Funnel*, approximately 64 mm [2½ in.] in diameter.

**5. Reagents**

5.1 *Drying Atmospheres*—The atmosphere used to purge the drying oven is either dry air or dry nitrogen as specified in **5.1.1** or **5.1.2**.



**FIG. 1 Vacuum-Type Desiccator**

5.1.1 *Dry Air*—Air with a moisture content of content of 1.9 mg/L or less passed through a drying column containing any of the appropriate desiccants listed in 5.2.

5.1.2 *Dry Nitrogen*—Compressed nitrogen gas certified having an oxygen content less than 30 µL/L and passed through a drying column containing any of the appropriate desiccants listed in 5.2.

5.2 *Desiccants*—Materials suitable for use in the desiccator may be chosen from the following:

5.2.1 *Anhydrous Calcium Sulfate* (0.004 mg/L).

5.2.2 *Silica Gel*.

5.2.3 *Magnesium Perchlorate* (0.0005 mg/L).

5.2.4 *Sulfuric Acid, Concentrated* (0.003 mg/L).

5.2.5 The desiccant must be kept fresh enough to assure that the air in the desiccator is dry as defined in 5.1. Values in parentheses ( ) are literature values for the residual amount of moisture in air at equilibrium with these desiccants. (**Warning:** Sulfuric acid is corrosive and can cause severe damage to eyes, skin, and clothing. Magnesium perchlorate is a strong oxidant and can react violently with organic materials.)

5.3  $K_2SO_4$ —*Crystalline Potassium Sulfate*

## 6. Technical Hazards

6.1 In collecting, containing, handling, reducing, and dividing the gross moisture sample, all operations must be done expeditiously and in a manner that attempts to preserve the original sample moisture integrity.

6.2 If the gross sample is too wet to allow reduction and division, spread sample in a thin layer and expose to the air of the laboratory. Dry no more than necessary to enable satisfactory reduction and division of sample.

6.3 Take particular care not to overdry low rank coals, especially lignites. Drying will accelerate oxidation and can also result in shrinkage of pore size and volume which will affect the moisture-holding capacity.

## 7. Collection of Gross Samples

7.1 Samples shall not be taken from outcrop, weathered, or oxidized coal.

7.1.1 *Mine Samples*—Take mine samples in accordance with Practice D4596.

7.1.2 *Tipple or Shipment Samples*—Collect a representative gross sample of coal in accordance with Practice D2234/D2234M. If only the equilibrium moisture is desired, use the General Purpose Sampling Procedure. If the surface moisture of wet coal is to be determined, use the procedure for sampling the special total moisture subsample described in Practice D2234/D2234M.

## 8. Preparation of Laboratory Samples

8.1 Crush the sample to a 2.36 mm [No. 8] sieve size in accordance with Practice D2013; however, it is important to also observe the technical hazards stated in Section 6 of this test method.

8.1.1 Divide out a 25 g equilibration moisture subsample in accordance with Practice D2013 to be used for testing.

## 9. Procedure

9.1 Two methods for preparing the sample for equilibration are provided. The wetted procedure has historically been the primary method. However, mine samples and certain coals that deteriorate when treated with water, particularly low rank coals (subbituminous C and lignite), may be equilibrated directly without wetting, provided the samples are collected and prepared with a minimum loss of moisture. Avoid low rank coal samples that have less than their full complement of inherent moisture.

9.1.1 *Wetted Procedure*—Place the 25 g sub-split equilibration sample coal into a 250 mL Erlenmeyer flask and add approximately 100 mL of recently boiled, cooled, distilled water (Note 2). Shake the flask mechanically for 30 min, and then place it in the constant-temperature bath for 3 h at 30 °C. At the end of the wetting period, remove the excess water from the coal by filtering on a Büchner-type funnel approximately 64 mm [2 ½ in.] in diameter, using suction supplied by a water filter pump. Use a minimum amount of water to transfer the coal to the filter. After transfer of the coal, close the funnel with a rubber stopper fitted with a glass tube through which air saturated with water vapor is passed to prevent drying of the coal (Note 2). Cease filtration as soon as the wet sheen on particle surfaces begins to dull or coal particles first begin to disaggregate (particles stop clinging together). Thoroughly mix the wet coal in the funnel with a spoon and place about 5.0 g in a uniform layer in a weighing bottle of known mass. Proceed to section 9.2.

NOTE 2—Over drying the sample during the filtration process to remove excess moisture can result in anomalously low equilibrium moisture values. Conversely, stopping the filtration prematurely may leave the sample too wet. In the latter case, the sample may not reach equilibrium in the desiccator within the prescribed equilibration period.

9.1.2 *Unwetted Procedure*—The use of the unwetted procedure for coal samples that have sustained partial drying is not recommended due to the hysteresis effect (Note 3). Place about 5.0 g of the crushed gross sample coal in a uniform layer in a weighing bottle of known mass. Proceed to section 9.2.