



Designation: D1412/D1412M – 17a

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

## 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 and health 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

<sup>1</sup> This test method is under the jurisdiction of ASTM Committee D05 on Coal and Coke and is the direct responsibility of Subcommittee D05.21 on Methods of Analysis.

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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.

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 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  $\pm$  0.2 °C.

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

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  $K_2SO_4$  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.

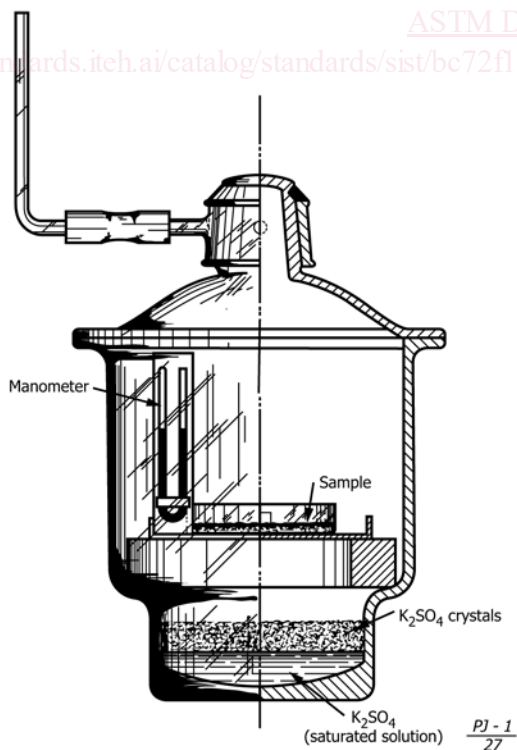


FIG. 1 Vacuum-Type Desiccator

## 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.

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**.

**NOTE 3**—Moisture desorption and adsorption curves for coal are not entirely reversible.<sup>4</sup> The difference between the original and reabsorbed moisture at the maximum relative vapor pressure is due to a failure to close the hysteresis loop, and is often termed the “hysteresis effect.” The effect increases with decreasing coal rank and is believed to be a result of shrinkage due to drying that reduces the coal pore volume,<sup>5</sup> which, in turn, decreases moisture holding capacity. Because of this effect, coal samples subjected to partial drying below their full complement of inherent moisture, or samples of low rank coals (which have large pore sizes) may

<sup>4</sup> Allardice, D. J. and D. G. Evans, “Moisture in coal,” in C. Karr, Jr. (Ed.), *Analytical Methods for Coal and Coal Products*. Vol. 1. New York: Academic Press, 1978: 247–262.

<sup>5</sup> Selvig, W.A. and W. H. Ode. Determination of Moisture-Holding Capacity (Bed Moisture) of Coal or Classification by Rank: US Bureau of Mines, RI-4968, (1953). 10p.

yield anomalously low equilibrium moisture values.

9.2 Place the uncovered weighing bottle in the small vacuum-type desiccator containing a saturated solution of K<sub>2</sub>SO<sub>4</sub> for maintaining the relative humidity of 96 % to 97 %. An excess of crystalline K<sub>2</sub>SO<sub>4</sub> shall extend above the solution level. Evacuate the desiccator to an absolute pressure equivalent to about 30 mm Hg by means of a mechanical vacuum pump and then totally immerse in a constant-temperature water bath or place in an insulated air cabinet maintained at 30 °C ± 0.2 °C for 48 h for all coals higher in rank than subbituminous C. Subbituminous C and lignite coal will require 72 h to reach equilibrium for practical purposes.

9.3 After equilibration of the coal, restore the pressure in the desiccator to atmospheric, with the desiccator still in the bath, by slowly admitting dry air for a period of not less than 15 min. Admit the air to the inlet tube of the desiccator after passing it through a train consisting first of a bubbler containing H<sub>2</sub>SO<sub>4</sub> (sp gr 1.84), then a capillary tube with one end drawn out to a tip having a suitable bore for regulating the rate of air flow, and finally a coiled copper tube placed in the constant-temperature bath. Remove the desiccator from the bath and open immediately. Quickly close the weighing bottle and weigh to the nearest 0.1 mg. Uncover the weighing bottle, place it in the moisture oven preheated to 105 °C, through which passes a current of dry air or nitrogen per **5.1**, and heat for 1½ h. Then remove the weighing bottle from the oven, cool for 30 min, over one of the desiccants specified in **5.2**, in a desiccator, and weigh.

## 10. Calculation

10.1 Calculate the percent equilibrium moisture in the analysis sample as follows:

$$\text{Equilibrium Moisture in Analysis Sample, \%} = \frac{(B - C)}{(B - A)} \times 100$$

where:

- A = mass of weighing bottle, grams,
- B = mass of weighing bottle and wet coal, grams, and
- C = mass of weighing bottle and dried coal, grams.

## 11. Report

11.1 Report the equilibrium or bed moisture to the nearest 0.1 % as the percentage loss in mass of the equilibrated coal.

## 12. Precision and Bias

12.1 The precision of this test method is based on an interlaboratory study of ASTM D1412, Standard Test Method for Equilibrium Moisture of Coal at 96 % to 97 % Percent Relative Humidity and 30 °C, conducted in 2011. A total of 14 laboratories participated in this study, testing nine coal samples (three each of bituminous, subbituminous; and lignite ranks); and using either air or nitrogen as the drying atmosphere. Each of the total 796 “test results” reported represents an individual determination and all participants were asked to report duplicate test results. Except for those analyses with fewer than six