



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 (ε) 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

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 ± 0.2 °C.

<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.

\*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*:

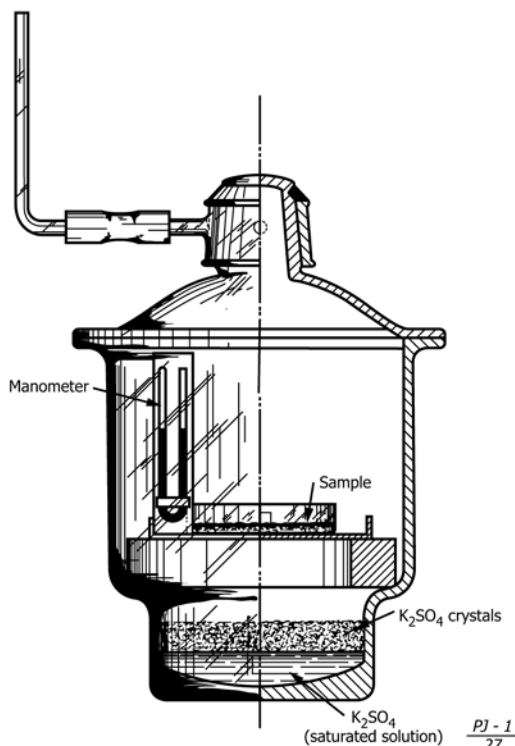
4.7.1 *High Vacuum Desiccator*—Small vacuum-type desiccator, approximately 160 mm in diameter, when used in conjunction with a saturated  $K_2SO_4$  solution provides the necessary humidity for the test conditions (see **Fig. 1**).

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 readsorbed 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

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

<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.

laboratories reporting for a sample, ASTM Practices **E691** and **E177** were followed for the design and analysis of the data; the details are given in ASTM Research Report No. D05-1049.<sup>6</sup>

12.1.1 The precision of this test method for the determination of Equilibrium Moisture in Coal is expressed as best-fit regression trendlines in **Eq 1** and **Eq 2**.

$$\text{Repeatability Limit } (r) = 0.035 \times EQM + 0.216 \quad (1)$$

$$\text{Reproducibility Limit } (R) = 0.057 \times EQM + 0.398 \quad (2)$$

where:

$EQM$  = Equilibrium Moisture, %

12.1.2 *Repeatability Limit (r)*—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 %.

12.1.3 *Reproducibility Limit (R)*—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 %.

12.2 *Bias*—Certified standards or absolute methods are not available for this test; therefore, bias of results cannot be determined.

### 13. Keywords

13.1 bed moisture; coal; equilibrium moisture; extraneous moisture; surface moisture

<sup>6</sup> Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D05-1049. Contact ASTM Customer Service at service@astm.org.

## APPENDIX

### (Nonmandatory Information)

#### X1. PRACTICE FOR COMPARING THE RELATIONSHIP BETWEEN INHERENT AND EQUILIBRIUM MOISTURE

X1.1 The purpose of the equilibrium moisture test is to provide an estimate of the inherent (bed) moisture. However, evidence has shown that equilibrium moisture results on many low rank coals, including most lignite coal, are often lower than inherent moisture. The procedure described in this appendix can be used where there is a question about the applicability of the equilibrium moisture result as an estimator of inherent moisture. The method is straightforward and has proven effective in many situations for examining this moisture relationship.

X1.2 Special coal samples, collected at their inherent moisture level, are analyzed for both total (inherent) and equilibrium moisture. The results are then compared to see if differences exist between the two moisture parameters, and the end user(s) can then determine whether such differences have any practical significance. This procedure does not directly yield inherent moisture values for an entire coal seam or mine, because the samples are not necessarily representative of the full seam as would be the case for face channel samples (Practice **D4596**). Nevertheless, the procedure does provide a tool for evaluating the relationship between inherent and equilibrium moisture for a given area. See **(6)**

X1.3 The most critical step in evaluating this relationship is the collection of samples containing their full complement of inherent moisture. Occasionally, it may be difficult to obtain a channel sample that contains no surface moisture. Also, obtaining a channel sample from thick coal seams, such as those in the Western United States, is generally impractical, especially from a safety standpoint. However, the collection of fresh, unfractured pieces of coal without visible surface mois-

ture is usually feasible. Such samples are considered to contain only inherent moisture (Terminology **D121**, Classification **D388**).

NOTE X1.1—The collection of coal at its inherent moisture levels requires some degree of judgement, and the sampler should have the necessary experience. For increased confidence, multiple comparisons are recommended to define the variability of the data.

X1.4 *Characteristics and Conditions of Sampling Locality*—Samples should be obtained from freshly exposed, unweathered mine faces. Avoid coal that exhibits any signs of moisture loss or weathering. There is no single test to determine the degree of weathering of coal under field conditions. However, when an obvious indication of weathering is observed, the sample should be obtained from a different locality or sampling postponed until suitable, fresh coal is available. Collecting a substandard sample simply because it was the best material available will not yield valid results.

NOTE X1.2—Obvious indicators of weathering include, but are not limited to: (1) any discoloration of broken coal surfaces or cleats, (2) presence of sulfate minerals resulting from the oxidation of pyrite, (3) presence of gypsum ( $\text{CaSO}_4$ ) crystals, and (4) presence of dust, dried, crazed, or fragmented condition of the coal blocks resulting from moisture loss from the coal.

A simple field test can be used to identify qualitatively seemingly fresh coal that has lost some inherent moisture but is not yet obviously weathered. Spray or wipe a light coat of water onto the surface of the coal and observe the rate at which it disappears. Rapid disappearance (typically within a few seconds) indicates absorption and demonstrates that the coal contains less than its full complement of inherent moisture. Slower disappearance (taking a minute or more) is characteristic of evaporation and suggests that the pores are filled with moisture. To account for variations in field conditions such as temperature, humidity, different absorption rates by different coals, and so forth, apply the test to a number of coal pieces both before and during the sample collection process.





X1.5 Use heavy equipment such as a backhoe, front end loader, or continuous miner (with spray turned off) to expose a fresh, unweathered coal seam face. Immediately after exposure, collect pieces of coal either by picking from the face or from coal pulled from the face by the machine. The pieces must be solid and unfractured and must exhibit no visible surface moisture. The nominal size of the pieces should be 8 cm to 25 cm [3 to 10 in.]. Larger pieces minimize any effects of surface drying that could reduce the inherent moisture. If there is any doubt that the coal contains its full complement of inherent moisture, select larger pieces 30.48 cm [1 ft] or more in diameter and collect the sample material from the center portion of the larger pieces. Each sample should be comprised of multiple pieces totaling a minimum mass of 8 kg [17.6 lb]. Where practical, collect pieces from various positions in the seam rather than concentrating on a particular horizon. Avoid layers or pieces that are excessively high in mineral matter content, especially clay.

X1.6 An alternative procedure for collecting the pieces is to obtain them from freshly shot coal at the toe of a coal face as the coal is being loaded out. Be especially alert to obtain pieces that were not lying on the surface of the pile of shot coal, and do try to obtain pieces that were well covered by other coal before outloading.

X1.7 Break each piece with a hammer to inspect for any internal moisture-filled fractures. Discard any pieces with visible surface moisture. Remove any fine particles adhering to the coal chunks by wiping or brushing the surfaces.

X1.8 Promptly put the pieces in a polyethylene bag at least 0.2 mm [4 mil] thick. Perform the operation in a manner that minimizes drying of the pieces. Samples should not be left sitting in direct sunlight during collection or transporting. It is recommended that the sample be double bagged for added protection. Promptly ship the samples to the laboratory for analysis. Freezing conditions can affect the pore structure of the coal and, therefore, samples should be protected from freezing during shipment to the laboratory.

X1.9 The samples should be processed immediately upon receipt by the laboratory. Inspect the sample bag for punctures occurring during transit which could cause moisture loss. Discard any samples where this has occurred. Record the mass of the coal and the bag.

X1.10 Most freshly collected samples will exhibit visible moisture which has desorbed from the coal and condensed on the coal surfaces and the inside of the bag. To account for this desorbed inherent moisture, weigh the bag containing the sample. Then, open the bag and allow the coal and bag to air dry at room temperature for 15 min or just until all visible moisture has evaporated. Use caution to prevent overdrying, as that can result in shrinkage of the pore structure, thereby reducing the moisture-holding capacity. Reweigh the coal and bag and record this initial air-drying loss. Also, clean and weigh the bag(s) separately so that the air-drying loss can be

calculated as a percentage of the coal mass.

X1.11 Following the reweighing, immediately reduce the sample to minus 4.75 mm [No. 4] using an enclosed crusher. Work rapidly to minimize moisture loss during this and subsequent handling steps. Use an enclosed riffle to divide the sample into at least two splits (A and B) with a minimum mass of 4000 g [8.8 lb] each.

X1.12 Analyze Split A for total moisture using Test Method **D3302**. Be certain to include the initial air-drying loss (Section **X1.8**) in the calculation of the total moisture.

NOTE X1.3—Although only total moisture is required for comparison with equilibrium moisture, a proximate analysis (Practice **D3172**), a sulfur content analysis, and a calorific value analysis should also be performed. This will allow comparison of the quality of the sample to the quality of the coal from the entire seam or typically shipped from a mine.

X1.13 Analyze Split B for equilibrium moisture using Test Method **D1412/D1412M**.

NOTE X1.4—To ensure that a true equilibrium condition has been reached, it is recommended that, at least for the first few comparisons, portions of Split B be equilibrated for longer than the two days to three days as specified in Test Method **D1412/D1412M**.

NOTE X1.5—As a quality assurance tool, it has been found useful to measure the moisture in the sample just before the start of equilibration(6). To determine this “zero-day” moisture, prepare an extra weighing bottle (equilibration dish) containing 5 g of the sample and immediately measure the moisture using the same procedure as for the equilibrated sample. The zero-day result for wetted samples provides insight as to the amount of excess moisture remaining in the samples after the washing and filtration procedures. This can give an indication of the time required to reach equilibration or detect samples that have been overly dried during filtration that can lead to anomalously low equilibrium moisture values. For nonwetted samples, the zero-day result is useful in monitoring moisture loss during sample preparation as well as identifying suspect inherent (total) and equilibrium moisture data.

X1.14 The difference between inherent (total) moisture and equilibrium moisture (if any) is obtained by subtracting the total moisture value obtained in **X1.12** from the equilibrium moisture value obtained in **X1.13**. If a difference exists, the equilibrium moisture value will typically be lower than the respective inherent moisture value for a sample which has been collected as its inherent moisture level and properly tested. When dealing with low rank coals, if the equilibrium moisture is higher than the inherent moisture, it is recommended to resample and retest to validate or amend the results before using the data for critical purposes. The acceptable range of the difference at which equilibrium moisture may still be considered equivalent to inherent moisture depends on the specific purpose and circumstances. This final decision is left to the user(s) to agree upon.

NOTE X1.6—Based on experience with low rank coals, equilibrium moisture values that exceed inherent moisture values are due to one or both of two situations, either the sample was not collected with its full complement of inherent moisture (partially dried) or an analytical problem occurred, or both. If sampling issues are suspected, refer to **X1.4** for additional guidance. Analytical issues resulting in an unusually high equilibrium moisture value are typically due to the failure of the test sample to reach an equilibrium condition. Extending the equilibration time often remedies this situation.



## REFERENCES

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- (5) Selvig, W. A., and Ode, W. H., “Determination of Moisture-Holding Capacity (Bed Moisture) of Coal for Classification by Rank,” *Report of Investigations No. 4968*, U.S. Bureau of Mines, XMBUA 1953.
- (6) Luppens, J. A., and Hoeft, A. P., “Relationship Between Inherent and Equilibrium Moisture Contents in Coal by Rank,” *Journal Coal Quality*, Vol 10, No. 4, 1991, pp. 133–144.

## SUMMARY OF CHANGES

Committee D05 has identified the location of selected changes to this standard since the last issue (D1412/D1412M-17) that may impact the use of this standard. (Approved August 1, 2017.)

- (1) Language in **Note 3** was revised.

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