



Designation: D 1412 – 04

Standard Test Method for Equilibrium Moisture of Coal at 96 to 97 Percent Relative Humidity and 30°C¹

This standard is issued under the fixed designation D 1412; 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 method² covers determination of the equilibrium moisture of coal in an atmosphere over a saturated solution of potassium sulfate at 30°C.

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

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.*

2. Referenced Documents

2.1 ASTM Standards:³

D 121 Terminology of Coal and Coke

D 388 Classification of Coals by Rank

D 2013 Practice for Preparing Coal Samples for Analysis

D 2234 Practice for Collection of a Gross Sample of Coal

D 3172 Practice for Proximate Analysis of Coal and Coke

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

D 3302 Test Method for Total Moisture in Coal

D 4596 Practice for Collection of Channel Samples of Coal in a Mine

3. Significance and Use

3.1 This test method affords a means of estimating the bed 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 D 3302 and the equilibrium moisture.

3.2 When samples are collected in conformity with Classification D 388, 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.

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 $30.0 \pm 0.2^\circ\text{C}$.

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 D 3173. 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 1.18-mm (No. 16) openings.

4.6 *Shaking Machine*.

4.7 *Desiccator*—Small vacuum-type desiccator, 160 mm in diameter (see Fig. 1).

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.

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 information concerning the experimental work on which this test method is based, see the following papers:

Stansfield, E., and Gilbert, K. C., "Moisture Determination for Coal Classification," *Transactions*, American Institute of Mining and Metallurgical Engineers, Coal Division, TAMCA, Vol 101, 1932, pp. 125–43.

Rees, O. W., Reed, F. H., and Land, G. W., "A Study of the Equilibration Method of Determining Moisture in Coal for Classification by Rank," *Report of Investigations No. 58*, Illinois State Geological Survey, ILGIA, 1939, pp. 34.

Krumin, P., "The Determination of Forms of Moisture in Coal," No. 195, Ohio State University, p. 92, 1963.

Kreulen, D. J. W., "The Adsorption Water of Coal," *Chemische en Pharmaceutische Techniek (Dordrecht)*, CHPHA, Vol 7, 1951, pp. 23–24.

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.

³ 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.

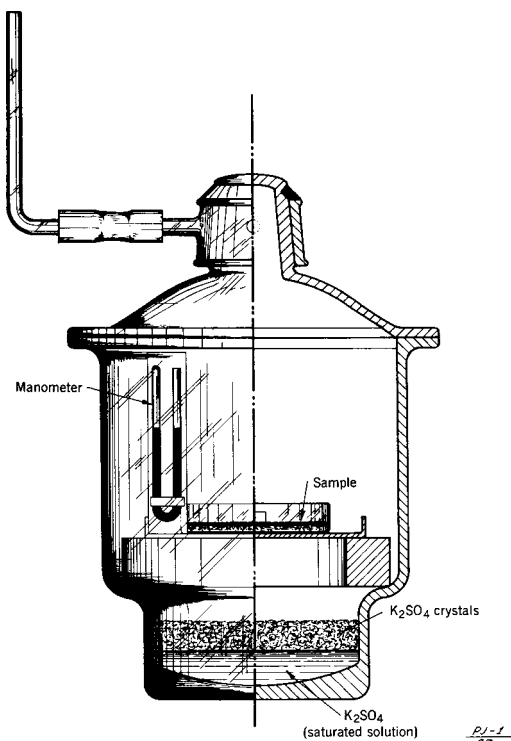


FIG. 1 Vacuum-Type Desiccator

5. Reagents

5.1 *Dry air*—Air used to purge the drying oven should be dried to a moisture content of 1.9 mg/L or less. (Dew point of -10°C or less.) Any desiccant or drying method capable of achieving this degree of dryness is suitable.

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.)

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 D 4596.

7.1.2 *Tipple or Shipment Samples*—Collect a representative gross sample of coal in accordance with Practice D 2234. 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 D 2234.

8. Preparation of Laboratory Samples

8.1 Crush the gross sample to No. 4 (4.75- μm) sieve size in accordance with Practice D 2013; however, it is important to also observe the technical hazards stated in Section 6 of this test method.

8.1.1 Divide sample in accordance with Practice D 2013.

8.1.2 Rapidly stage crush the divided sample to pass a No. 16 (1.18-mm) sieve by means of a coffee-mill-type crusher. This stage crushing produces a minimum amount of fine material; however, it increases segregation so the crushed sample shall be thoroughly mixed.

8.1.3 Divide out the equilibration moisture subsample to be used for testing.

9. Procedure

9.1 Place 20 to 25 g of the crushed coal into a 250-mL Erlenmeyer flask and add 100 mL of recently boiled, cooled, distilled water (Note 1). 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 (2½ 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. 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 weight. Place the uncovered weighing bottle in the small vacuum-type desiccator containing a saturated solution of K_2SO_4 for maintaining the relative humidity of 96 to 97 %. An excess of crystalline K_2SO_4 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 $30 \pm 0.2^{\circ}\text{C}$ for 48 h for all coals higher in rank than lignite. Lignite will require 72 h to reach equilibrium for practical purposes.

NOTE 1—Mine samples and certain coals that deteriorate when treated with water may be equilibrated directly without wetting, provided the samples are collected and prepared with a minimum loss of moisture. Unwetted coals should be equilibrated for varying periods of time, in units of 24 h, in order that equilibrium may be attained.

9.2 After equilibration of the coal, restore the pressure in the desiccator to atmospheric, with the desiccator still in the bath,