Designation: D 1412 – 03

Standard Test Method for Equilibrium Moisture of Coal at 96 to 97 Percent Relative Humidity and 30°C[1]

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 method[2] covers determination of the equilibrium moisture of coal in an atmosphere over a saturated solution of potassium sulfate at 30°C.

1.2 *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:*

D 121 Terminology of Coal and Coke[3]
D 388 Classification of Coals by Rank[3]
D 2013 Practice for Preparing Coal Samples for Analysis[3]
D 2234 Practice for Collection of a Gross Sample of Coal[3]
D 3172 Practice for Proximate Analysis of Coal and Coke[3]
D 3173 Test Method for Moisture in the Analysis Sample of Coal and Coke[3]
D 3302 Test Method for Total Moisture in Coal[3]
D 4596 Practice for Collection of Channel Samples of Coal in the Mine[3]

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 ± 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 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 the rate of two times per minute, with the air dried by passing it through H_2SO_4 (sp gr 1.84).

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 (2½in.) in diameter.

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[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, Edgar and Gilbart, 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, Peter, "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] *Annual Book of ASTM Standards*, Vol 05.06.

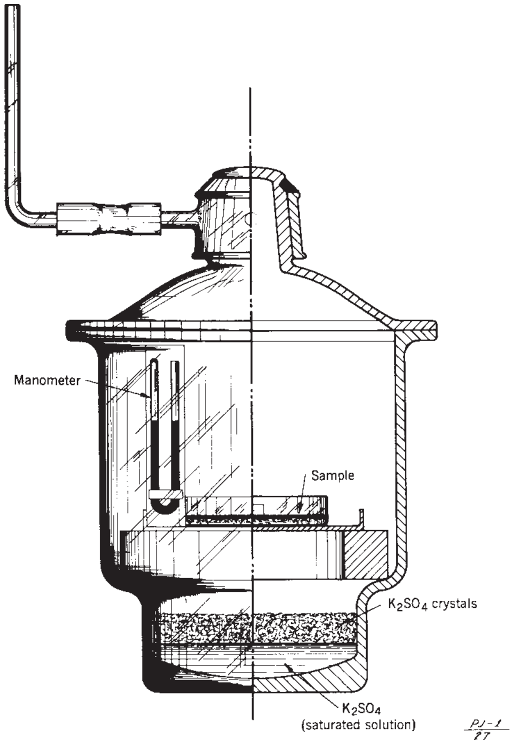

FIG. 1 Vacuum-Type Desiccator

5. Technical Hazards

5.1 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.

5.2 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.

5.3 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.

6. Collection of Gross Samples

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

6.1.1 *Mine Samples*—Take mine samples in accordance with Practice D 4596.

6.1.2 *Tipple or Shipment Samples*—Collect a representative gross sample of coal in accordance with Test Methods 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 Test Methods D 2234.

7. Preparation of Laboratory Samples

7.1 Crush the gross sample to No. 4 (4.75-mm) sieve size in accordance with Method D 2013; however, it is important to also observe the technical hazards stated in Section 5 of this test method.

7.1.1 Divide sample in accordance with Method D 2013.

7.1.2 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.

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

8. Procedure

8.1 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 ± 0.2°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.

8.2 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_2SO_4 (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 0.2 mg. Uncover the weighing bottle, place it in the moisture oven preheated to 105°C, and heat for 1½h. Then remove the weighing bottle from the oven, cover, cool 30 min over H_2SO_4 (sp gr 1.84) in a desiccator, and weigh.

9. Calculation

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

$$\text{Equilibrium Moisture in Analysis Sample, \%} = [(B-C)/(B-A)]\ x\ 100 \quad (1)$$

where:

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

10. Report

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

11. Precision and Bias

11.1 The precision of this test method for the determination of Equilibrium Moisture in Coal are shown in Table 1.

11.1.1 *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 %.

TABLE 1 Concentrations Range and Limits for Repeatability and Reproducibility of Equilibrium Moisture in Coal

Range, %	Repeatability Limit, *r*	Reproducibility Limit, *R*
Under 5	0.3	0.5
5 to 15	0.5	1.0
Over 15	1.0	1.5

11.1.2 *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 %.

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

12. Keywords

12.1 bed moisture; coal; equilibrium moisture; extraneous moisture; surface moisture

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 D 4596). Nevertheless, the procedure does provide a tool for evaluating the relationship between inherent and equilibrium moisture for a given area.[4]

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 moisture is usually feasible. Such samples are considered to contain only inherent moisture (Terminology D 121, Classification D 388).

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

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[4] 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.

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 ($CaSO_4$) 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 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 lbs). 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 weight 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 weight.

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 lbs) each.

X1.12 Analyze Split A for total moisture using Test Method D 3302. 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 (Test Method D 3172), 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 D 1412.

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 to three days as specified in Test Method D 1412.

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.[4] 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 X1.10 from the equilibrium moisture value obtained in X1.11. 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.

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