Designation: D 3173 – 02

Standard Test Method for Moisture in the Analysis Sample of Coal and Coke[1]

This standard is issued under the fixed designation D 3173; 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 the determination of moisture in the analysis sample of coal or coke. It is used for calculating other analytical results to a dry basis. When used in conjunction with the air drying loss as determined in accordance with Method D 2013 or Practice D 346, each analytical result can be calculated to an as-received basis:

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 346 Practice for Collection and Preparation of Coke Samples for Laboratory Analysis[2]

D 2013 Method of Preparing Coal Samples for Analysis[2]

D 3180 Practice for Calculating Coal and Coke Analyses from As-Determined to Different Bases[2]

D 3302 Test Method for Total Moisture in Coal[2]

3. Summary of Test Method

3.1 Moisture is determined by establishing the loss in weight of the sample when heated under rigidly controlled conditions of temperature, time and atmosphere, sample weight, and equipment specifications.

4. Significance and Use

4.1 Moisture as determined by this test method is used for calculating other analytical results to a moisture free basis using procedures in Practice D 3180. Moisture percent determined by this test method may be used in conjunction with the air-dry moisture loss determined in Method D 2013 and Test Method D 3302 to determine total moisture in coal. Total moisture is used for calculating other analytical results to "as received" basis using Practice D 3180. Moisture, ash, volatile matter, and fixed carbon percents constitute the proximate analysis of coal and coke.

5. Analysis Sample

5.1 The analysis sample is that sample which has been pulverized to pass 250-µm (No. 60) sieve as prepared in accordance with Practice D 346 or Method D 2013.

6. Apparatus

6.1 *Drying Oven*, for coal samples:

6.1.1 For determining the moisture of coal, the oven shall be so constructed as to have a uniform temperature in all parts, have a minimum of air space, and be capable of temperature regulation between the limits of 104 and 110°C. It may be of the form shown in Fig. 1. Provision shall be made for renewing the preheated air in the oven at the rate of two to four times a minute, with the air dried as defined in 7.1.

6.1.2 In the oven shown in Fig. 1, the door should contain a hole of approximately ⅛ in. (3.2 mm) in diameter near the bottom to permit a free flow of air through the oven space.

6.2 *Drying Oven*, for coke samples. For determining the moisture of coke, an ordinary drying oven with openings for natural air circulation and capable of temperature regulation between limits of 104 and 110°C may be used.

6.3 *Capsules*, with covers. A convenient form, which allows the ash determination to be made on the same sample, is a porcelain capsule, ⅞ in. (22 mm) in depth and 1¾in. (44 mm) in diameter, or a fused silica capsule of similar shape. These capsules shall be used with a well-fitting flat aluminum cover, illustrated in Fig. 2. Platinum crucibles or glass capsules with ground-glass caps may also be used. They should be as shallow as possible, consistent with convenient handling.

7. Reagents

7.1 *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 − 10°C or less.) Any desiccant or drying method capable of achieving this degree of dryness is suitable.

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

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

7.2.2 *Silica Gel.*

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

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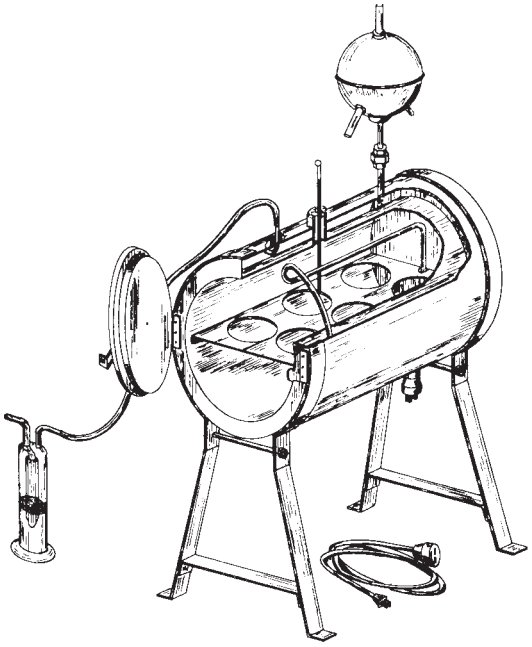
[1] This test method is under the jurisdiction of ASTM Committee D05 on Coal and Coke and is the direct responsibility of Subcommittee D05.21 on Methods of Analysis.

Current edition approved Oct. 10, 2002. Published January 2003. Originally approved in 1973. Last previous edition approved 2002 as D 3173 – 02.

[2] *Annual Book of ASTM Standards*, Vol 05.05.



NOTE 1—Details in U.S. Bureau of Mines Bulletin No. 492, 1951, p 6.

FIG. 1 Moisture Oven

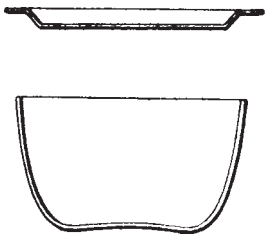


FIG. 2 Capsule for Use in Determining Moisture

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

7.2.5 The desiccant must be kept fresh enough to assure that the air in the desiccator is dry as defined in 7.1. 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.)

8. Procedure for Sample Passing a 250-µm (No. 60) Sieve

8.1 Heat the empty capsules under the conditions at which the sample is to be dried, place the stopper or cover on the capsule, cool over a desiccant for 15 to 30 min, and weigh. Dip out with a spoon or spatula from the sample bottle approximately 1 g of the sample. Put this quickly into the capsule, close, and weigh at once to the nearest ±0.1 mg.

8.2 An alternative procedure for weighing the sample (more subject to error) is as follows: After transferring an amount of the sample slightly in excess of 1 g, bring to exactly 1 g in weight (±0.5 mg) by quickly removing the excess weight of the sample with a spatula. The utmost dispatch must be used to minimize the exposure of the sample until the weight is determined.

8.3 After removing the covers, quickly place the capsules in a preheated oven (at 104 to 110°C) through which passes a current of dry air. (The current of dry air is not necessary for coke.) Close the oven at once and heat for 1 h. Open the oven, cover the capsules quickly, cool in a desiccator over desiccant, and weigh as soon as the capsules have reached room temperature.

8.4 Use the percentage of moisture in the sample passing a 250-µm (No. 60) sieve to calculate the results of the other analyses to a dry basis.

9. Calculations

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

$$\text{Moisture in analysis sample, \%} = [(A - B)/A] \times 100 \quad (1)$$

where:

A = grams of sample used and
B = grams of sample after heating.

10. Precision and Bias [3]

10.1 *Precision*—The precision of this method for the determination of moisture in the analysis sample of coal and coke is shown in Table 1. The precision characterized by repeatability (S_r, r) and reproducibility (S_R, R) is described in Table A1.1 in Annex A1.

10.1.1 *Repeatability Limit (r)*—The value below which the absolute difference between two test results calculated to separate 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 homogenous material, may be expected to occur with a probability of approximately 95%.

10.1.2 *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 nearly homogenous as possible, may be expected to occur with a probability of approximately 95%.

10.2 *Bias*—Certified Reference Materials are not available for the determination of bias by this test method.

NOTE 1—One coke sample was analyzed in the interlaboratory study. The average moisture of the coke sample was 0.36 %. The repeatability limit for this sample is 0.10. The reproducibility limit for this sample is 0.15. The repeatability standard deviation for this sample is 0.036. The reproducibility standard deviation for this sample is 0.053.

[3] An interlaboratory study, designed consistent with Practice E 691, was conducted in 1995. Twelve labs participated. Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR: **D05-1020**.

TABLE 1 Precision

Range	Repeatability Limit (r)	Reproducibility Limit (R)
1.0 – 21.9 %	$0.09 + 0.01\bar{x}^A$	$0.23 + 0.02\bar{x}^A$

[A] Where $\bar{x}$ is the average of two single test results.

ANNEX

(Mandatory Information)

A1. PRECISION STATISTICS

A1.1 The precision of this test method, characterized by repeatability (S_r, r) and reproducibility (S_R, R) has been determined for the following materials as listed in Table A1.1.

A1.2 *Repeatability Standard Deviation (S_r)*—The standard deviation of test results obtained under repeatability conditions.

A1.3 *Reproducibility Standard Deviation (S_R)*—The standard deviation of test results obtained under reproducibility conditions.

TABLE A1.1 Repeatability (S_r, r) and Reproducibilty (S_R, R) Parameters Used for Calculation of Precision Statement

NOTE—The R-Squared statistic for both the repeatability (r) and reproducibility (R) indicates that the model as fitted explains 62% of the variability in the two variables.

Material	Average	S_r	S_R	r	R
91-2Ivb	0.9683	0.0479	0.0853	0.1341	0.2388
91-1 hvAb	1.5689	0.0569	0.1387	0.1593	0.3884
91-5 hvAb	3.1178	0.0559	0.0977	0.1564	0.2736
89-4 hvCb	5.4428	0.0601	0.1408	0.1682	0.3943
91-4 hvCb	9.6392	0.0636	0.1004	0.1781	0.2811
90-1 subB	11.9	0.1252	0.2193	0.3505	0.614
89-7 subA	12.4556	0.0798	0.1188	0.2234	0.3326
91-6 subA	15.1753	0.0577	0.1737	0.1615	0.4865
89-6 subC	21.9461	0.1949	0.2843	0.5456	0.796