Designation: D 3175 – 89a (Reapproved 1997)

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

This standard is issued under the fixed designation D 3175; 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 determines the percentage of gaseous products, exclusive of moisture vapor, in the analysis sample which are released under the specific conditions of the test.

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.*

1.3 The values stated in SI units are to be regarded as the standard.

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 2234 Practice for Collection of a Gross Sample of Coal[2]

D 3173 Test Method for Moisture in the Analysis Sample of Coal and Coke[2]

3. Terminology

3.1 *Definition:*

3.1.1 *sparking fuels*—within the context of this test method, fuels that do not yield a coherent cake as residue in the volatile matter determination but do evolve gaseous products at a rate sufficient to mechanically carry solid particles out of the crucible when heated at the standard rate. Such coals normally include all low-rank noncaking coals and lignites but may also include those anthracites, semianthracites, bituminous, chars and cokes that lose solid particles as described above. These are defined as sparking fuels because particles escaping at the higher temperatures may become incandescent and spark as they are emitted.

4. Summary of Test Method

4.1 Volatile matter is determined by establishing the loss in weight resulting from heating a coal or coke under rigidly controlled conditions. The measured weight loss, corrected for moisture as determined in Test Method D 3173 establishes the volatile matter content. Two procedures are described to permit conformity with differences in sample behavior.

5. Significance and Use

5.1 Volatile matter, when determined as herein described, may be used to establish the rank of coals, to indicate coke yield on carbonization process, to provide the basis for purchasing and selling, or to establish burning characteristics.

6. Apparatus

6.1 *Platinum Crucible*, with closely fitting cover, for coal. The crucible shall be of not less than 10 or more than 20-mL capacity, not less than 25 or more than 35 mm in diameter, and not less than 30 or more than 35 mm in height.

6.2 *Platinum Crucible*, with closely fitting cover, for coke. The crucible shall be of 10-mL capacity, with capsule cover having thin flexible sides fitting down into crucible. Or the double-crucible method may be used, in which the sample is placed in 10-mL platinum crucible, which is then covered with another crucible of such a size that it will fit closely to the sides of the outer crucible and its bottom will rest 8.5 to 12.7 mm (⅓ to ½ in.) above the bottom of the outer crucible.

6.3 *Vertical Electric Tube Furnace*, for coal or coke. The furnace may be of the form shown in Fig. 1. It shall be regulated to maintain a temperature of 950 ± 20°C in the crucible, as measured by a thermocouple positioned in the furnace.

6.4 *Balance*, sensitive to 0.1 mg.

7. Procedure

7.1 The sample shall be the material pulverized to No. 60 (250-µm) sieve in accordance with Method D 2013 or Method D 346.

7.2 *Procedure for Nonsparking Coals and Cokes*:

7.2.1 Weigh 1 g of the sample in a weighed platinum crucible, close with a cover which fits closely enough so that the carbon deposit from bituminous, subbituminous, and lignite coals does not burn away from the underside, place on platinum or Nichrome-wire supports and insert directly into the furnace chamber, which shall be maintained at a temperature of 950 ± 20°C, and lower immediately to the 950°C zone. Regulation of the temperature to within the prescribed limits is critical. After the more rapid discharge of volatile matter has subsided, as shown by the disappearance of the luminous

[1] This test method is under the jurisdiction of ASTM Committee D-5 on Coal and Coke and is the direct responsibility of Subcommittee D05.21 on Methods of Analysis.

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

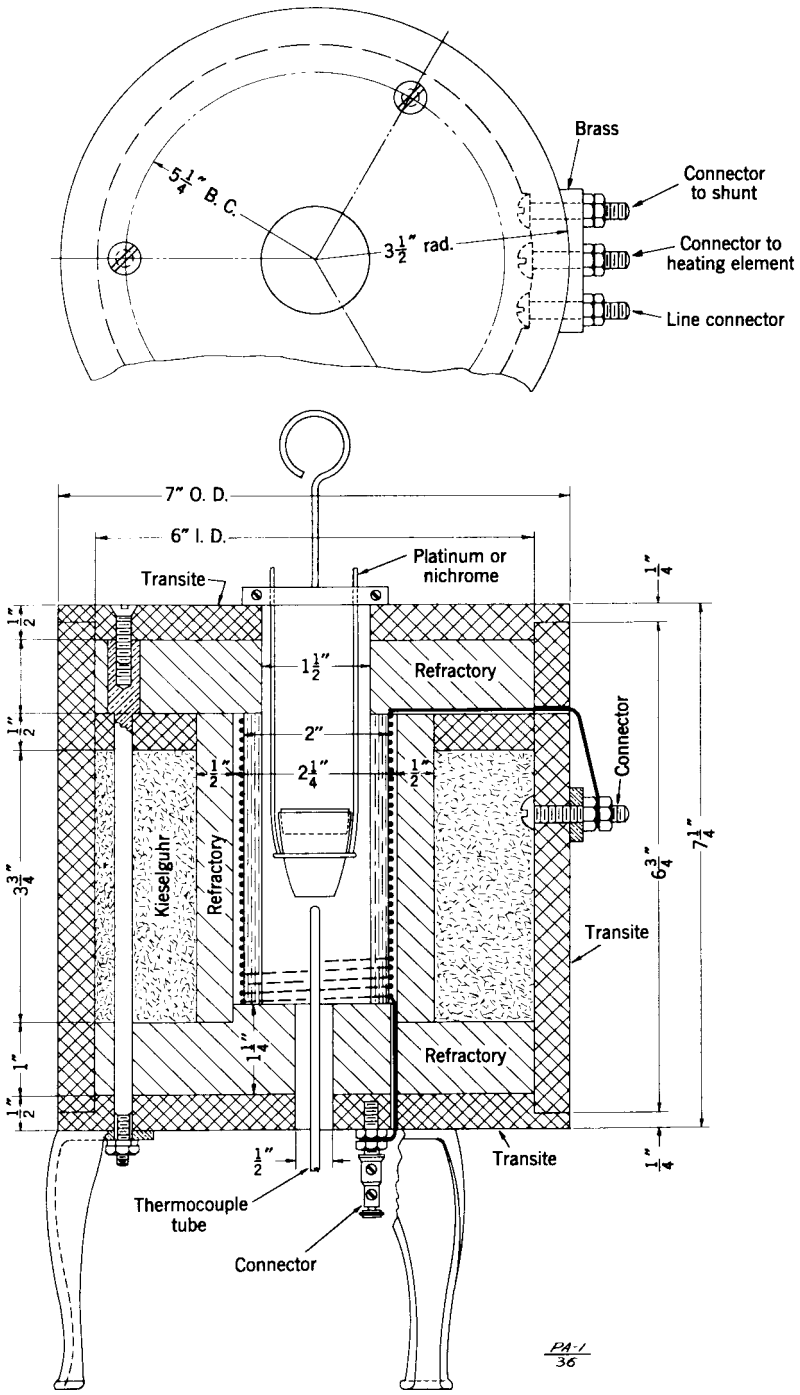

FIG. 1 Electric Furnace for Determining Volatile Matter

flame, or in the case of coke, after 2 or 3 min, inspect the crucible (see Note 1) to verify that the lid is still properly sealed. If necessary, reseat the lid to guard against the admission of air into the crucible. Do this as rapidly as possible by raising the crucible to the top of the furnace chamber, reposition the lid to more perfectly seal the crucible, then lower the crucible immediately back to the 950°C zone. After heating for a total of exactly 7 min, remove the crucible from the furnace and without disturbing the cover, allow it to cool. Coke should be cooled in a desiccator. Weigh as soon as cold. The precentage loss of weight minus the percentage moisture equals the volatile matter. With some strongly caking low-volatile and medium-volatile bituminous coals, the coke button may be broken with explosive violence due to the liberation of volatile matter within the button. This is usually designated as popping. Such popping may blow the lid off the crucible and cause mechanical losses of the coked material. When such popping is observed, the determination shall be rejected and the test repeated until popping does not occur.

NOTE 1—Inspection of the crucible may be aided by the use of a mirror held above the furnace well.

7.3 *Modified Procedure for All Sparking Fuels*:

7.3.1 Fuels that do not cake or cake weakly when volatile matter is determined shall be watched closely for sparking during the heating period (Note 2); also, at the end of the test the crucible cover shall be inspected for ash deposits, and the presence of such deposits shall be considered as evidence of sparking.

7.3.2 All fuels that spark when the volatile matter is determined by the methods described in 7.1 shall be treated as follows: The sample shall be given a preliminary gradual heating such that a temperature of 600 ± 50°C is reached in 6 min (Note 2). After this preliminary heating the sample shall be heated for exactly 6 min at 950 ± 20°C. If sparking is then observed, the determination shall be rejected and the test repeated until no sparking occurs either during the preliminary heating or during the 6-min period at 950°C. Remove the crucible from the furnace, cool on a metal cooling block, and weigh. To ensure uniformity of results, keep the cooling period constant and do not prolong beyond 15 min. The percentage loss in weight minus the percent moisture in accordance with Test Method D 3173, is the volatile matter. All analyses by this test method shall be so marked when reported to indicate that the modified procedure was used.

NOTE 2—If a tubular furnace of the Fieldner type (Fig. 1) is used for the determination of volatile matter, the preliminary gradual heating may be accomplished by moving the crucible to predetermined positions in the cooler top zone of the furnace. Due to variations in the heating characteristics of the furnace, the operator must predetermine by thermocouple the proper positions to meet a preliminary heating rate as specified in 7.3.2. A mechanical device to lower the crucible into the furnace may be used to facilitate control of the lowering operation.

8. Calculation

8.1 Calculate the weight loss percent as follows:

$$\text{Weight loss, \%} = [(A - B)/A] \times 100 \quad (1)$$

where:
A = weight of sample used, g, and
B = weight of sample after heating, g.

8.2 Calculate the volatile matter percent in the analysis samples as follows:

$$\text{Volatile matter in analysis sample , \%} = C - D \quad (2)$$

where:
C = weight loss, %, and
D = moisture, %.

9. Precision and Bias

9.1 *Precision*:

9.1.1 *Repeatability*—Duplicate results by the same laboratory should not be considered suspect unless they differ by more than the following percentages:

High-temperature coke	0.2
Anthracite	0.3
Semianthracite, bituminous coal, low-temperature coke, and chars	0.5
Subbituminous	0.7
Lignite and peat	1.0

9.1.2 *Reproducibility*—The results submitted by two or more laboratories should not be considered suspect unless they differ by more than the following percentages:

High-temperature coke	0.4
Anthracite	0.6
Semianthracite, bituminous coal, low-temperature coke, and chars	1.0
Subbituminous	1.4
Lignite and peat	2.0

9.2 *Bias*—Since this is an empirical test method, the degree of absolute bias cannot be determined.