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Standard Test Method for Determination as Carbon Dioxide of Carbonate Carbon in Coal¹

This standard is issued under the fixed designation D 1756; 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 reappraisal. A superscript epsilon (ε) indicates an editorial change since the last revision or reappraisal.

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 April 10, 1996; 2002. Published June 1996; December 2002. Originally published as D 1756-60T. Last previous edition ~~D 1756-89 (1993)~~; D 1756-96 (2000).

1. Scope

1.1 This test method covers the determination of carbon dioxide in coal in any form, such as mineral carbonate, from which carbon dioxide is released by action of mineral acids. It applies to high-carbonate and low-carbonate coals.

1.2 The values stated in SI units are to be regarded as 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.*

2. Referenced Documents

2.1 *ASTM Standards:*

D 1193 Specification for Reagent Water²

D 2013 Method of Preparing Coal Samples for Analysis³

D 2234 ~~180~~ Practice for ~~Collection of a Gross Sample of~~ Calculating Coal and Coke Analyses from as Determined to Different Bases³

3. Summary of Test Method

3.1 The determination of carbon dioxide is made by decomposing with acid a weighed quantity of the sample in a closed system and absorbing the carbon dioxide in an absorbent. The increase in weight of the absorbent is a measure of the carbon dioxide in the sample used.

4. Significance and Use

4.1 Small amounts of mineral carbonates occur in many coals and comparatively large amounts in some coals. The determination of these carbonates is the purpose of this test method. The value found for carbon dioxide is used to estimate the mineral matter content, particularly CaCO₃ and MgCO₃, of high-carbonate coals.

5. Apparatus (see Fig. 1)

5.1 The use of apparatus modified from that shown in Fig. 1 shall be considered permissible so long as results agree within the accepted precision.⁴

5.2 *Air-Purifying Train*, consisting of the following units arranged in the order of passage of air: Any convenient form of flow meter (1) connected to a carbon dioxide absorber (2) filled with an acceptable absorbent. The absorber (2) shall be connected to a trap (3).

5.3 *Reaction Unit*, consisting of a 300-mL wide-mouth flask (5) fitted with a three-hole rubber stopper. One hole is to accommodate a 60-mL open-end separatory funnel (4) extending almost to the bottom of the flask (5). The second hole in the stopper is used to connect the purifying train through the trap (3); the tip of the glass tubing shall extend almost to the bottom of flask (5). The third hole of the stopper shall be fitted to a water-cooled condenser (6).

² *Annual Book of ASTM Standards*, Vol 11.01.

³ *Annual Book of ASTM Standards*, Vol 05.06.

⁴ For further information regarding this determination, see Krumin, P. O., "The Meigs Creek No. 9 Coal Bed in Ohio, Part III—Further Study of the Chemical and Physical Properties, and Washability Characteristics, with a Brief Review of New Methods Employed," *Bulletin No. 165*, Ohio State University, Engineering Experiment Station, (1957), pp. 38–46; and Krumin, P. O., and Svanks, K., "Four Methods of Determination of Carbon Dioxide in Solid Fuels," *ASTM Bulletin*, No. 227, January 1958, pp. 51–57.

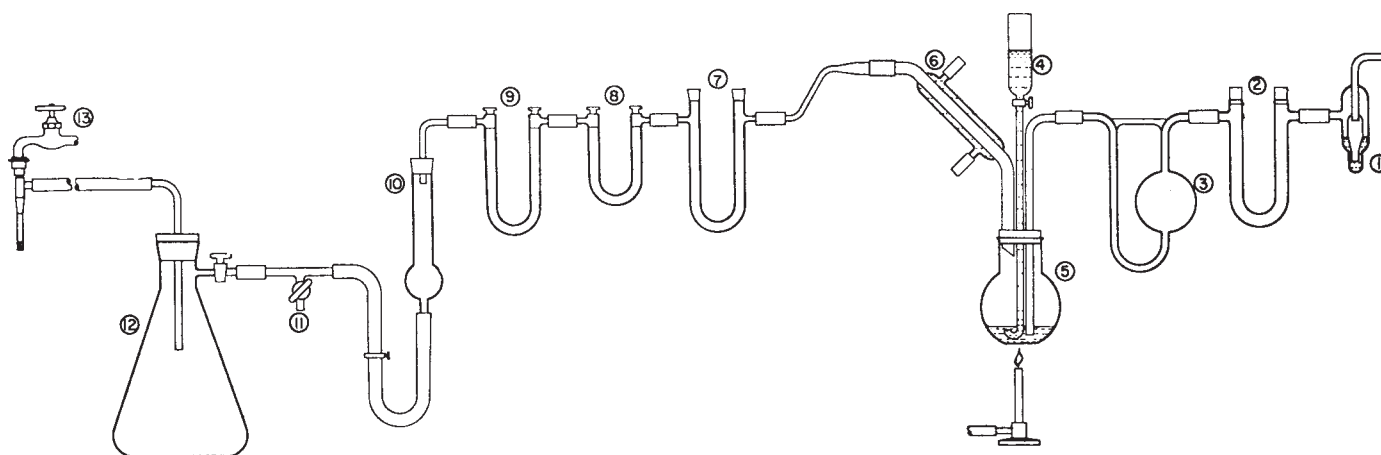


FIG. 1 Apparatus for the Determination of Carbon Dioxide

5.4 *Absorption Unit*, consisting of a water absorber (7) filled with an acceptable desiccant connected to the condenser (6) on the one side and on the other side to absorber (8) containing anhydrous copper sulfate on pumice or granular silver sulfate. The carbon dioxide absorber (9) shall be connected to absorber (8) and to another water absorber (10). The absorber (10) shall be connected to a T-piece stopcock (11) which in turn shall be attached to a filter-flask (12). The system shall be aspirated by a water pump (13).

6. Purity of Reagents

6.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available.⁵ Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

6.2 Unless otherwise indicated, references to water shall be understood to mean Type IV reagent water conforming to Specification D 1193.

7. Reagents and Materials

7.1 *Carbon Dioxide Absorbent*—Sodium or potassium hydroxide (NaOH or KOH) on an inert carrier, 8- to 20-mesh size.⁶

7.2 *Copper Sulfate, Anhydrous, on Pumice*—Crush and sieve the pumice to obtain a fraction passing the No. 6 (3.35-mm) sieve and retained on the No. 20 (850- μ m) sieve. Transfer 60 g of the prepared pumice to a casserole, cover with a saturated solution of copper sulfate (CuSO_4), evaporate to dryness with constant stirring, and then heat 3 to 4 h at 150 to 160°C. Cool in a desiccator and store in a glass-stoppered bottle.

7.3 *Hydrochloric Acid (1 + 1)*—Mix equal volumes of concentrated hydrochloric acid (HCl, sp gr 1.19) and water.

7.4 *Silver Sulfate*, granular Ag_2SO_4 .

7.5 *Sodium Carbonate or Calcium Carbonate*—(Na_2CO_3 or CaCO_3).

7.6 *Water Absorbent*—Anhydrous magnesium perchlorate $\text{Mg}(\text{ClO}_4)_2$ or anhydrous calcium sulfate (CaSO_4), passing No. 8 (2.36-mm) to No. 45 (355- μ m) sieves.⁷

7.7 *Wetting Agent (10 %)*—Any wetting agent suitable for use in acid solution.⁸

8. Preparation of Apparatus

8.1 The various components shall be assembled as shown in Fig. 1. All connections shall be glass to glass with flexible tubing seals or standard ball and socket connections. The carbon dioxide absorbers (2) and (9) shall be filled with layers of the desiccant on each side of the carbon dioxide absorbent. Absorber (8) containing the copper sulfate on pumice or silver sulfate shall have the desiccant on each side of the active material.

8.2 The system shall be calibrated in accordance with the procedure described in Section 9 by substituting 0.5 g of sodium or calcium carbonate for the coal. A variation of not more than 0.1 % from the theoretical value shall be considered satisfactory.

⁵ Reagent Chemicals, American Chemical Society Specifications, Am. Chemical Society, Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see *Analar Standards for Laboratory Chemicals*, BDH Ltd., Poole, Dorset, U.K., and the *United States Pharmacopeia and National Formulary*, U.S. Pharmacopeial Convention, Inc. (USPC), Rockville, MD.

⁶ Ascarite and Mikohbite have been found satisfactory for this purpose.

⁷ Anhydron and Dehydrite have been found satisfactory for this purpose.

⁸ Aerosol and Pluronic L-44 have been found satisfactory for this purpose.

9. Procedure

9.1 The sample shall be the material pulverized to pass No. 60 (250- μ m) sieve and well mixed in accordance with Method D 2013.

9.2 Introduce into the flask (5), 5 g, weighed to the nearest 1.0 mg, of the coal sample. Add 25 mL of cold carbon dioxide-free distilled water and 10 mL of wetting agent to wet the particles thoroughly. Rinse down the sides of the flask with 25 mL of cold carbon dioxide-free distilled water and connect the flask to the assembly.

9.3 Aspirate the apparatus, assembled as in Fig. 1, at the rate of 50 to 75 mL/min for 15 min and then stop the aspiration. Remove absorber (9), wipe with a clean cloth, and allow to come to room temperature (15 to 20 min). Weigh to the nearest 0.2 mg and then reconnect to absorbers (8) and (10). During this time keep absorbers (8) and (9) closed.

9.4 Measure 25 mL of HCl (1 + 1) into the separatory funnel (4) and start aspirating at the rate of 50 to 75 mL/min. Then add the acid slowly to the flask (5) taking care not to allow aspiration through the separatory funnel. After the reaction has subsided, heat the flask (5) with a gas burner or electric heater to slow boiling for 5 min, remove the heat, and continue aspirating for 20 min. Stop the aspiration, close absorber (9), remove and wipe it with a clean, dry cloth, and weigh after 15 to 20 min. The increase in weight is the carbon dioxide in the sample.

10. Calculation

10.1 Calculate the percentage of carbon dioxide (CO₂) in the sample as follows:

$$\text{CO}_2, \% = (A/B) \times 100 \quad (1)$$

where:

A = increase in weight of absorber, g, and

B = grams of sample used.

11. Precision and Bias

11.1 ~~The following permissible differences~~ precision of this test method for the determination as Carbon Dioxide of Carbonate Carbon in Coal are shown in Table 1.

11.1.1 Repeatability Limit (r)—the value below which the absolute difference between two test results calculated to a dry basis (Practice D3180) 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 used for judging expected to occur with a probability of approximately 95%.

11.1.2 Reproducibility Limit (R)—the value below which the absolute difference between two test results calculated to a dry basis (Practice D3180) carried out in different laboratories using samples taken at random from a single quantity of results:

Carbon Dioxide, %	Repeatability (Same Laboratory), %	Reproducibility (Different Laboratories), %
— Under 1.0	0.05	0.10
— Over 1.0	0.10	0.20

11.2 ~~The material that is as homogeneous as possible, may be expected to occur with a probability of approximately 95%.~~

11.2 Bias—the bias of this test method cannot be determined at this time.

12. Keywords

12.1 carbon; carbon dioxide; carbonate carbon

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TABLE 1 Concentrations Range and Limits for Repeatability and Reproducibility as Carbon Dioxide of Carbon in Coal.

Range %	Repeatability Limit r	Reproducibility Limit R
<1	0.05	0.10
>1	0.10	0.20