



Standard Test Methods for Carbon and Hydrogen in the Analysis Sample of Coal and Coke¹

This standard is issued under the fixed designation D 3178; 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 These test methods cover the determination of total carbon and hydrogen in samples of coal or coke. Both the carbon and hydrogen are determined in one operation. These test methods yield the total percentages of carbon and hydrogen in the coal as analyzed and the results include not only the carbon and hydrogen in the organic matter, but also the carbon present in mineral carbonates and the hydrogen present in the free moisture accompanying the sample as well as hydrogen present as water of hydration of silicates.

NOTE 1—It is recognized that certain technical applications of the data derived from this test procedure may justify additional corrections. These corrections could involve compensation for the carbon present as carbonates, the hydrogen of free moisture accompanying the sample, and the calculated hydrogen present as water of hydration of silicates.

1.2 When data are converted and reported on the “dry” basis, the hydrogen value is corrected for the hydrogen present in the free moisture accompanying the sample.

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.*

1.4 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 Analysis²
- D 1193 Specification for Reagent Water³
- D 2013 Practice for Preparing Coal Samples for Analysis²
- D 3173 Test Method for Moisture in the Analysis Sample of Coal and Coke²
- D 3176 Practice for Ultimate Analysis of Coal and Coke²

¹ These test methods are under the jurisdiction of ASTM Committee D05 on Coal and Coke and are the direct responsibility of Subcommittee D05.21 on Methods of Analysis.

Current edition approved Sept. 29, 1989. Published February 1990. Originally published as D 3178 – 73. Last previous edition D 3178 – 89 (1997).

² *Annual Book of ASTM Standards*, Vol 05.06.

³ *Annual Book of ASTM Standards*, Vol 11.01.

D 3180 Practice for Calculating Coal and Coke Analyses from As-Determined to Different Bases²

TOTAL CARBON AND TOTAL HYDROGEN

3. Summary of Test Methods

3.1 The determination of carbon and hydrogen is made by burning a weighed quantity of sample in a closed system and fixing the products of combustion in an absorption train after complete oxidation and purification from interfering substances. These test methods give the total percentages of carbon and hydrogen in the coal as analyzed, and include the carbon in carbonates and the hydrogen in the moisture and in the water of hydration of silicates.

4. Significance and Use

4.1 Carbon and hydrogen values are used to calculate the amount of oxygen (air) required in combustion processes, and in the calculations of efficiency of combustion processes.

4.2 Carbon and hydrogen determinations are used in material balances on coal conversion processes; also one or the other is frequently used in correlations of chemical and physical properties, such as yields of products in liquefaction, reactivity in gasification, and the density and porosity of coal.

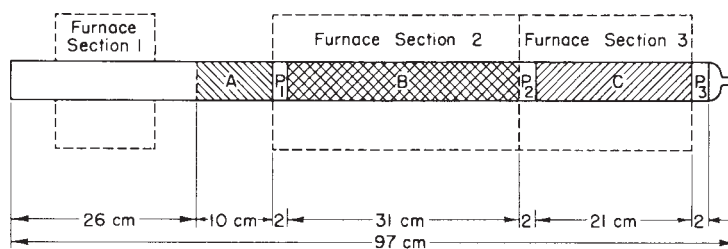
5. Sample

5.1 The sample shall be the material pulverized to pass No. 60 (250- μ m) sieve and well mixed according to Method D 346 or Method D 2013. It may be beneficial to grind the ash, pit ash, calcined coke and high mineral content materials to pass a No. 100 (150- μ m) sieve.

5.2 The test sample shall be weighed just prior to commencing the analysis to minimize chance for moisture change. A change in moisture content would introduce error in the hydrogen analysis. In order to provide the data necessary to correct for the hydrogen present in the moisture and ensuing final calculations of both the hydrogen and carbon, a separate sample shall be weighed out at the same time for a moisture analysis, and analyzed in accordance with Test Method D 3173.

6. Apparatus

6.1 *Oxygen Purifying Train*, consisting of the following units arranged as listed in the order of passage of oxygen:



A—Clear fused quartz section (optional) when a translucent quartz tube is used.
 B—Cupric oxide filling.
 C—Lead chromate or silver filling.
 P₁, P₂, or P₃—oxidized copper gauze plugs.

NOTE 1—All dimensions are given in centimetres. When furnace sections longer than those specified in 6.3 are to be used, changes in the above dimensions shall be in accordance with the provisions of Note 5.

FIG. 1 Arrangement of Tube Fillings for Combustion Tube

6.1.1 *First Water Absorber*—A container for the solid dehydrating reagent. It shall be so constructed that the oxygen must pass through a column of reagent adequate to secure water equilibrium equal to that secured in the prescribed absorption train. A container of large volume and long path of oxygen travel through the reagent will be found to be advantageous where many carbon and hydrogen determinations are made.

6.1.2 *Carbon Dioxide Absorber*—A container for solid carbon dioxide absorbing agent. It shall be constructed as described in 6.1.1 and shall provide for a column of reagent adequate to remove carbon dioxide completely.

6.1.3 *Second Water Absorber*, same as specified in 6.1.1

6.2 *Flowmeter*, used to permit volumetric measurement of the rate of flow of oxygen during the determination. It shall be suitable for measuring flow rates within the range from 50 to 100 mL/min (standard temperature and pressure). The use of a double-stage pressure-reducing regulator with gage and needle valve preceding the first water absorber is recommended to permit easy and accurate adjustment of the rate of flow.

6.3 *Combustion Unit*—The combustion unit shall consist of three electrically heated furnace sections, individually controlled, which may be mounted on rails for easy movement; the upper part of each furnace may be hinged so that it can be opened for inspection of the combustion tube. The three furnace sections shall be as follows:

6.3.1 *Furnace Section 1*, nearest the oxygen inlet end of the combustion tube, approximately 130-mm long and used to heat the inlet end of the combustion tube and the sample. It shall be capable of rapidly attaining an operating temperature of 850 to 900°C. Combustion tube temperature shall be measured by means of a thermocouple placed immediately adjacent to the tube near the center of the tube section.

6.3.2 *Furnace Section 2*, approximately 330 mm in length and used to heat that portion of the tube filled with cupric oxide. The operating temperature shall be 850 ± 20°C. Combustion tube temperature shall be measured by means of a thermocouple placed immediately adjacent to the tube near the center of the tube section.

6.3.3 *Furnace Section 3*, approximately 230 mm-long, and used to heat that portion of the tube filled with lead chromate or silver. The operating temperature shall be 500 ± 50°C. Combustion tube temperature shall be measured by means of a

thermocouple placed immediately adjacent to the tube near the center of the tube section.

6.3.4 *Combustion Tube*—The combustion tube shall be made of fused quartz or high-silica glass⁴ and shall have a nominal inside diameter which may vary within the limits of 19 to 22 mm and a minimum total length of 970 mm. The exit end shall be tapered down to provide a tubulated section for connection to the absorption train. The tubulated section shall have a length of 20 to 25 mm, an internal diameter of not less than 3 mm, and an external diameter of approximately 7 mm. The total length of the reduced end shall not exceed 60 mm. If a translucent fused quartz tube is used, a transparent section 190-mm long, located 250 mm from the oxygen inlet end of the tube, will be found convenient (see Fig. 1).

6.3.5 *Combustion Boat*—This shall be either glazed porcelain, fused silica, or platinum. Boats with internal dimensions of approximately 70 by 8 by 8 mm have been found convenient.

6.4 *Absorption Train*—The absorption train shall consist of the following units arranged as listed in the order of passage of oxygen:

6.4.1 *Water Absorber*, having a capacity for 45 cm³ of solid reagent and a minimum length of gas travel through the reagent of 80 mm.⁵

6.4.2 *Carbon Dioxide Absorber*—If solid reagents are used for carbon dioxide absorption the container shall be as described in 6.4.1. If a solution is used, the container shall be a Vanier bulb.

6.4.3 *Guard Tube*—A container as described in 6.4.1.

7. Reagents

7.1 *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,

⁴ Vycor has been found satisfactory for this purpose.

⁵ Glass-stoppered containers such as the Nesbitt, Schwartz U-tube and the Stetser-Norton bulbs have been found satisfactory.