Designation: D 6316 – 04

Standard Test Method for Determination of Total, Combustible and Carbonate Carbon in Solid Residues from Coal and Coke[1]

This standard is issued under the fixed designation D 6316; 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 total, combustible, and carbonate carbon remaining in the solid byproducts of combustion from boiler furnaces and similar reactors, including ash, flyash, char, slag, and similar materials.

1.2 This test method is intended for the use of industry to determine the performance of boiler furnaces and similar combustion reactors and aid in determining the quality of the solid residue from combustion.

1.3 This test method comprises the use of any of several methods to determine total carbon content combined with any of several methods to determine carbonate carbon, and the calculation, by difference, of the combustible carbon remaining in a sample.

1.4 Alternatively, this test method applies to the determination of total carbon remaining in a material after acidification with strong acid to evolve carbonate carbon. In this case, the combustible carbon is the total carbon measured in the sample after acidification.

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

1.6 *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 to determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:* [2]

D 121 Terminology of Coal and Coke
D 513 Test Methods for Total and Dissolved Carbon Dioxide in Water
D 1756 Test Method for Determination as Carbon Dioxide of Carbonate Carbon in Coal
D 3178 Test Method for Carbon and Hydrogen in the Analysis Sample of Coal and Coke
D 5373 Test Methods for Instrumental Determination of Carbon, Hydrogen, and Nitrogen in Laboratory Samples of Coal and Coke
E 691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method

2.2 *Other Standards:*

ANSI/ASME Standard, PTC 38-1980 Determining the Concentration of Particulate Matter in A Gas Stream and US EPA Standard CFR 60 Appendix A, Method 17[3]

3. Terminology

3.1 *Definitions:* For additional definitions of terms used in this test method, refer to Terminology D 121.

3.1.1 *carbonate carbon*, *n*—the carbon content present in the solid products derived from the combustion or reaction of coal, coal byproducts, or coke as carbonates and which is non-combustible in standard industry practice.

3.1.2 *combustible carbon*, *n*—carbon content remaining in the solid products derived from the combustion or reaction of coal, coal byproducts, or coke, exclusive of carbonate in any form.

3.1.3 *total carbon*, *n*—carbon content remaining in the solid products derived from the combustion or reaction of coal, coal byproducts, or coke, inclusive of carbonate in any form.

4. Summary of Test Method

4.1 *Total Carbon*—The determination of total carbon is made by the oxidative thermal decomposition of a weighed quantity of sample in a closed system and, after complete oxidation and purification of the resulting gaseous products, measurement of the carbon dioxide produced by one of several methods.

4.1.1 *Absorptive Determination of Total Carbon*—The evolved carbon dioxide is fixed on an absorption train and is measured quantitatively by weighing the absorbent (see Test Methods D 3178).

4.1.2 *Instrumental Determination of Total Carbon*—The carbon dioxide is measured quantitatively by an electronic

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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 D05.29 on Major Elements in Ash and Trace Elements of Coal.

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[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

[3] Available from American National Standards Institute (ANSI), 25 W. 43rd St., 4th Floor, New York, NY 10036.

detection system calibrated against an appropriate reference standard (see Test Methods D 5373).

4.2 *Carbonate Carbon*—The determination of carbonate carbon is made by decomposing a weighed quantity of the sample with a dilute mineral acid, and after the purification of the evolved gases, measuring quantitatively the evolved carbon dioxide.

4.2.1 *Absorptive Determination of Carbonate Carbon*—The carbon dioxide is liberated by acidification and heating. The evolved gases are passed through a purification train and the CO_2 is fixed as Na_2CO_3 on a preweighed absorption unit and is measured gravimetrically (see Test Method D 1756).

4.2.2 *Coulometric Determination of Carbonate Carbon*—The carbon dioxide is liberated by acidification and heating. The evolved gas is swept through a scrubber and into an absorption cell where it is coulometrically titrated (see Part B of Test Methods D 513).

4.2.3 *Instrumental Determination of Carbonate Carbon*—The carbon dioxide is liberated by acidification and heating. The evolved gases are purified and measured quantitatively by an electronic detection system against an appropriate reference stream. This test method resembles that for the instrumental determination of total carbon, with the provision for sample combustion replaced by a provision for acidification. In practice, this test method uses an adaptation to instruments designed to measure total carbon content.

4.3 *Combustible Carbon by Prior Acidification*—The determination of combustible carbon is made directly by first acidifying and heating to dryness a weighed sample. All carbonate carbon present is evolved as carbon dioxide. The dried sample is then analyzed for total carbon content as above. No determination of carbonate carbon is made and the carbon content measured as total carbon is the combustible carbon.

5. Significance and Use

5.1 The combustible carbon content of solid residues is used to calculate efficiency of fuel combustion in boiler furnaces and similar combustors.

5.2 Combustible carbon values are also used to determine the residual fuel value of incompletely combusted coal and coke in fluidized bed furnaces and other reactors which consume carbonaceous fuels.

5.3 The combustible carbon content of flyash is an important parameter in the use of flyash as a cement additive.

6. Interferences

6.1 The interferences for the determination of total carbon content are those specified in the referenced standard methods, Test Methods D 3178 and Test Methods D 5373, with the following added stipulation.

6.1.1 The temperature and conditions of combustion that ensure complete conversion of a residue sample's total carbon content to carbon dioxide may be different than those required for conversion in coal samples. It is absolutely imperative that the instrumental conditions used will ensure complete conversion of mineral carbonate in any amount or form.

6.2 The interferences for the determination of carbonate carbon are those specified in the referenced methods, Test Method D 1756 and Part B of Test Methods D 513 with the following stipulation

6.2.1 Some coal combustion residues contain significant amounts of reactive elements such as CaO which will absorb carbon dioxide on exposure to open air and form mineral carbonates. This will influence the calculated amount of combustible carbon in the sample only if there was carbon dioxide chemisorption from the air between the time of the carbonate determination and the total carbon determination. Therefore, it is necessary that the sample be protected from air exposure.

6.3 The mineral acid and temperature required for prior acidification of the analytical sample shall not interfere with the amount of combustible carbon present in the sample or the analytical determination of carbon. The use of oxidizing acids such as concentrated nitric acid may oxidize a portion of the combustible carbon. Other acids such as hydrochloric acid may be incompatible with the reagents used to purify the gas stream in the instrumental determination of carbon. Sulfuric acid has a relatively high boiling point and is difficult to remove from the sample by drying. A solution of 10 % nitric acid is suggested for use in this procedure.

7. Apparatus

7.1 The apparatus used is specified in the respective referenced method.

7.2 *Total Carbon Content*:

7.2.1 *Absorptive Determination of Total Carbon*—The apparatus consists of an oxygen-purifying train, combustion unit, and absorption train of the type and configuration specified in Test Methods D 3178.

7.2.2 *Instrumental Determination of Total Carbon*—Because a variety of instrumental components and configurations can be used satisfactorily for these test methods, no specifications are presented here with respect to overall system design. However, the apparatus shall be capable of completely converting the carbon content to carbon dioxide, and the detection system shall determine the carbon dioxide individually and without interference.

7.3 *Carbonate Carbon Content*:

7.3.1 *Absorptive Determination of Carbonate Carbon*—The apparatus shall consist of an air purifying train, reaction unit, and absorption unit of the type and configuration specified in Test Method D 1756.

7.3.2 *Coulometric Determination of Carbonate Carbon*—The apparatus shall consist of an evolution unit, a carbon dioxide coulometer, scrubber, and pH meter of the type and configuration specified in Part B of Test Methods D 513.

7.3.3 *Instrumental Determination of Carbonate Carbon*—The apparatus shall consist of a heated acidification unit that is installed by bypassing the combustion system in a device designed for the determination of total carbon content. The gas-purifying system of such instrument shall be capable of completely removing the water content and acid gases produced as byproducts of the carbon dioxide evolution. The detection system shall measure the evolved carbon dioxide as an individual species and without interference.