Designation: D7633 – 13 (Reapproved 2022)

Standard Test Method for Carbon Black—Carbon Content[1]

This standard is issued under the fixed designation D7633; 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 instrumental determination of carbon content in a carbon black sample. Values obtained represent the total carbon content.

1.2 The method is applicable to tread, carcass and specialty type carbon blacks obtained from partial combustion or thermal decomposition processes, which typically contain 95 to 100 % carbon.

1.3 The results of these tests can be expressed as mass % carbon.

1.4 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.5 *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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.*

1.6 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D1799 Practice for Carbon Black—Sampling Packaged Shipments

D1900 Practice for Carbon Black—Sampling Bulk Shipments

D4483 Practice for Evaluating Precision for Test Method Standards in the Rubber and Carbon Black Manufacturing Industries

3. Summary of Test Method

3.1 In this test method, a sample is weighed in a combustion boat and the carbon content is determined by placing the boat in a tube furnace operating at 1350°C in a stream of oxygen resulting in complete combustion. Carbon in the sample is oxidized to carbon dioxide. Moisture and particulates are removed from the gas stream by traps filled with anhydrous magnesium perchlorate. The gas stream is then passed through a cell in which carbon dioxide concentration is measured by an infrared (IR) absorption detector at a precise wavelength in the IR spectrum.

3.2 This test method is for use with commercially available carbon analyzers equipped to carry out the combustion and measurement operations automatically.

3.3 The carbon analyzer must be calibrated using an appropriate calibration standard (see 6.4).

4. Significance and Use

4.1 The total carbon content of a carbon black is a requirement for the calculation and reporting of carbon dioxide emissions. It can also be used in calculations to estimate yield of the process.

5. Apparatus

5.1 *Analytical Balance,* or equivalent, capable of a weighing sensitivity of 1 mg or better resolution.

5.2 *Gravity Convection Drying Oven,* capable of maintaining 125 ± 5°C.

5.3 *Measurement Apparatus,* equipped to automatically combust the sample and measure carbon content.

5.4 *Combustion tube and boat* made of a suitable material such as mullite, porcelain or zircon.

6. 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,

[1] This test method is under the jurisdiction of ASTM Committee D24 on Carbon Black and is the direct responsibility of Subcommittee D24.66 on Environment, Health, and Safety.

Current edition approved June 1, 2022. Published July 2022. Originally approved in 2010. Last previous edition approved in 2018 as D7633 – 13 (2018). DOI: 10.1520/D7633-13R22.

[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.

where such specifications are available.[3] 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 *Magnesium Perchlorate.*

6.3 *Oxygen, high purity, 99.9 %.*

6.4 *Calibration standard.*[4]

7. Sampling

7.1 Samples of candidate carbon blacks shall be taken in accordance with Practice D1799 or D1900.

8. Preparation of Apparatus

8.1 Assemble the apparatus according to the manufacturer's instructions.

8.2 Stabilize the furnace temperature at 1350 ± 15°C.

8.3 Make a minimum of two determinations to condition the equipment or follow manufacturer's recommendation to condition equipment prior to calibrating the instrument.

9. Calibration

9.1 Calibrate the instrument per manufacturer's recommendation using a carbon calibration standard.

9.2 *Adjustment of Response of Measurement System*—Weigh approximately 0.1 g of calibration standard or use recommended mass per manufacturer. Analyze the sample (see Section 10). Repeat this procedure. Adjust instrument as recommended by the manufacturer until the absence of drift is indicated.

9.3 *Calibration Procedure*—Follow the calibration procedure recommended by the manufacturer. Confirm the calibration by analyzing a reference material of known carbon concentration.[4] Reference standards should be similar to carbon black with carbon content of approximately 95-100 %. The measured value should be within allowable limits of the known value. If not, repeat the procedure. If acceptable results are not obtained refer to the manufacturer's instructions for calibration.

10. Procedure

10.1 *Sample Preparation*—Dry an adequate sample of the carbon black for at least 1 h in a gravity-convection oven set at 125 ± 5°C, in an open container of suitable dimensions, so that the depth of black is no more than 10 mm. Cool to room temperature in a desiccator before use.

10.2 Stabilize and verify calibration of the analyzer (see 8.1 through 9.3).

10.3 Confirm the furnace temperature is 1350 ± 15°C.

10.4 Weigh approximately 0.1 g of the sample or use recommended mass per manufacturer.

10.5 Spread the sample in the combustion boat and record the sample weight.

10.6 Initiate the analysis and place the sample in the instrument using a boat puller or the auto-sampler mechanism.

10.7 When the analysis is complete, the instrument should indicate the carbon value. Refer to the manufacturer's recommended procedure.

11. Report

11.1 The percent carbon value is obtained directly from the apparatus.

11.2 Report results from individual determinations to the nearest 0.1 %.

NOTE 1—The most representative measurement of carbon content in a carbon black sample is an average value from multiple individual determinations. A good practice is to report a minimum of two individual determinations for a given sample.

12. Precision and Bias[5]

12.1 These precision statements have been prepared in accordance with Practice D4483. Refer to this practice for terminology and other statistical details.

12.2 The precision results in this precision and bias section give an estimate of the precision of this test method with the materials used in the particular interlaboratory program described below. The precision parameters should not be used for acceptance or rejection testing of any group of materials without documentation that they are applicable to those particular materials and the specific testing protocols of the test method. Any appropriate value may be used from Table 1 for the High Temperature Combustion Method. An alternate method for carbon content, CHN, was included in the interlaboratory program used to generate this precision statement since a number labs use this instrumental method. Precision data specific to the CHN method is found in Table 2.

12.3 A type 1 inter-laboratory precision program was conducted. Both repeatability and reproducibility represent short-term (daily) testing conditions. The testing was performed in each laboratory performing the test twice on each of two days (total of four tests). A test result is the value obtained from a single determination. Acceptable difference values were not measured.

12.4 The results of the precision calculations for this test are given in Table 1. The materials are arranged in ascending "mean level" order.

12.5 *Repeatability*—The pooled absolute repeatability, r, of this test has been established as 0.74 %. Any other value in Table 1 may be used as an estimate of repeatability, as appropriate. The difference between two single test results (or determinations) found on identical test material under the repeatability conditions prescribed for this test will exceed the

[3] "Reagent Chemicals," *American Chemical Society Specifications*, American 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.

[4] Purified carbon standards are typically available from the instrument manufacturer.

[5] A research report is pending.