



Designation: ~~D7633—10b~~ **D7633 – 13**

Standard Test Method for Carbon Black—Carbon Content¹

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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 ~~95-100%~~ 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 and health practices and determine the applicability of regulatory limitations prior to use.*

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 $125 \pm 5^\circ\text{C}$.

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

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.

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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.

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, 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 $1350 \pm 15^\circ\text{C}$.

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 $125 \pm 5^\circ\text{C}$, 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 $1350 \pm 15^\circ\text{C}$.

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.

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.