

Designation: D 1511 – $00^{\epsilon 2}$

Standard Test Method for Carbon Black—Pellet Size Distribution¹

This standard is issued under the fixed designation D 1511; 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.

- ϵ^1 Note—Footnotes 4, 5, and 6 were updated in December 2002.
- ϵ^2 Note—Paragraph 4.5.2 and footnotes 3, 4, and 5 were editorially updated in December 2004.

1. Scope

- 1.1 This test method covers the determination of the pellet size distribution of carbon black.
- 1.2 The values stated in SI units are to be regarded as the standard. The values given in parentheses are for information only.
- 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: ²
- D 1799 Practice for Carbon Black—Sampling Packaged Shipments
- D 1900 Practice for Carbon Black—Sampling Bulk Shipments
- D 4483 Practice for Determining Precision for Test Method Standards in the Rubber and Carbon Black Industries
- D 5817 Practice for Carbon Black, Pelleted-Reduction and Blending of Gross Samples
- E 11 Specification for Wire-Cloth Sieves for Testing Purposes

3. Significance and Use

3.1 The variation in the size of the pellets may relate to the level of dispersion and to the ease of handling. Due to the many

other variables that influence dispersion and handling, the significance of pellet size must be determined by the user.

4. Apparatus

- 4.1 Riffle Sample Splitter as specified in D 5817.
- 4.2 Balance with a sensitivity of 0.1 g.
- 4.3 Sieves—U.S. Standard Sieves or equivalent, conforming to Specification E 11. Sieve Nos. 10, 18, 35, 60, and 120, having openings respectively of 2000, 1000, 500, 250, and 125 μm, shall be used. The sieves shall be 25 mm (1 in.) in height and 200 mm (8 in.) in diameter.
 - 4.4 Bottom receiver pan and top sieve cover.
- 4.5 Sieve Shaker—Any equipment that will vibrate or shake a stack of sieves in a manner that will allow the pellets to separate into size fractions without excessive pellet breakage. The following three types of shakers have been found satisfactory for determining the pellet size distribution of pelleted carbon black.
- 4.5.1 Mechanical Sieve Shaker³—The Ro-Tap Siever imparts a uniform rotary and tapping motion to a stack of sieves as described in 4.3. The shaker machine shall be powered with an electric motor producing 181 to 183 rads/s (1725–1750 r/min). This will produce 140 to 160 raps/min and 280 to 320 rotary motions/min. The cover plate shall be fitted with a cork stopper that shall extend 3–9 mm ($\frac{1}{8}$ to $\frac{3}{8}$ in.) above the metal recess. Materials other than cork, such as rubber or wood, are unacceptable. The height of the RoTap hammer shall be set at 3.30 cm \pm 0.15 cm (1–5/16 in. \pm 1/16 in.).

¹ This test method is under the jurisdiction of ASTM Committee D24 on Carbon Black and is the direct responsibility of Subcommittee D24.51 on Carbon Black Pellet Properties.

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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 The sole source of supply of the Ro-Tap Siever known to the committee at this time is WS Tyler, 8570 Tyler Blvd., Mentor, OH 44060, E-mail: wstyler@wstyler.com. If you are aware of alternative suppliers, please provide this information to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee,³ which you may attend.

- 4.5.2 Vibratory Siever⁴—The Retsch Sieve Shaker AS200 has variable timer and amplitude settings. When set at 3 minutes and 0.5 amplitude, the AS200 provides satisfactory results. It accommodates a stack of sieves as described in 4.3.
- 4.5.3 Automatic Sieve Shaker⁵—The Gradex 2000 automatically performs all of the required steps including the weighing of the sample and the individual fractions retained on each sieve. The equipment consists of a balance, autofeed system, electric motor that imparts a uniform rotary motion, pneumatically operated rods to provide the tapping action, and computer and software to record and perform analyses. Shake time of 1 minute provides satisfactory test results. The test sieves are described in 4.3.
 - Note 1—Top sieve cover is not needed for the Gradex 2000.
- NOTE 2—The Gradex is supplied with one standard tapping rod. It is recommended that two additional tapping rods be installed to provide additional tapping action.

5. Sampling

- 5.1 Lot samples shall be taken in accordance with Practices D 1799 or D 1900.
- 5.2 Practice D 5817 shall be used for blending or reducing samples.

6. Procedure

6.1 Prepare carbon black for testing as noted in Section 5.

Note 3—It is not good practice to weigh out the test portion by pouring it directly from the sample container since the smaller pellets will tend to remain in the container while the larger pellets pour out first. Dipping the black from the container is the preferred technique.

6.2 Prepare the sieve assembly by stacking the sieves in the following order from bottom to top: Bottom receiver pan, No. 120, No. 60, No. 35, No. 18, No. 10, and top sieve cover.

Note 4—Top sieve cover is not needed for the Gradex 2000.

- 6.3 Mechanical and Vibratory Sieve Shakers
- 6.3.1 Weigh 100.0 g of carbon black.
- 6.3.2 Transfer weighed carbon black to the top sieve.
- 6.3.3 Install the sieve cover and transfer the sieve assembly to the shaker. The stack in the shaker should be adjusted to eliminate looseness.
 - 6.3.4 Start the shaker and allow it to shake as noted below:
- 6.3.4.1 Mechanical Shaker—1 minute with hammer operating.
- 6.3.4.2 Vibratory Shaker—3 minutes and 0.5 mm amplitude.

- 6.3.5 Remove the sieve assembly from the apparatus and weigh individually the carbon black retained on each sieve and bottom receiver pan to the nearest 0.1 g.
 - 6.3.6 Record the data and calculate as noted in Section 7.
 - 6.4 Automatic Sieve Shaker
- 6.4.1 Follow the manufacturer's instructions to load software and configure the shake time to 1 minute.
- 6.4.2 Transfer the sieve assembly to the automatic sieve shaker.
- 6.4.3 Weigh or measure approximately 100 g of carbon black.
- 6.4.4 Transfer measured carbon black to the autofeed container.
- 6.4.5 Enter the sample identification into the operating program. Repeat steps 6.4.3-6.4.5 as required for consecutive samples. Up to six samples may be identified at one time.
- 6.4.6 Start the testing sequence according to the manufacturer's operating instructions.
 - 6.4.7 Retrieve report from the computer.

7. Calculation

7.1 Calculate the pellet size distribution of the sample to the nearest 0.1 % as follows:

Sieve No.	Mass Retained, g	Percent Retained
10		
18 0 5		
35		
60		
120	h 311	
Pan	11.41/	
Total		

8. Report

- 8.1 Report the following information:
- 8.1.1 Proper identification of the sample,
- 8.1.2 Result obtained from a single determination, reported to the nearest 0.1 %.
 - 8.1.3 Apparatus used to determine test values.

9. Precision and Bias ⁶

- 9.1 This precision and bias section has been prepared in accordance with Practice D 4483. Refer to Practice D 4483 for terminology and other statistical details.
- 9.2 The precision results in this precision and bias give an estimate of the precision described as follows. The precision parameters should not be used for acceptance/rejection testing of materials without documentation that they are applicable to those particular materials and the specific testing protocols that include this test method.
- 9.3 A Type 1 inter-laboratory precision program was conducted in 1988 to determine the testing precision of three samples (Sample 1, Sample 2, and Sample 3) according to this test method. Both repeatability and reproducibility represent short term testing conditions. The program was conducted by seven laboratories testing three samples twice on each of two different days. A test result is the value obtained from a single determination. Acceptable differences were not measured.

⁴ The sole source of supply of the Retsch Sieve Shaker AS200 known to the committee at this time is Retsch Inc., 74 Walker Lane, Newtown, PA 18940, E-mail: info@retsch.us.com. If you are aware of alternative suppliers, please provide this information to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee, ¹ which you may attend.

⁵The sole source of supply of the Gradex 2000 known to the committee at this time is Rotex, Inc., 1230 Knowlton Street, Cincinnati, OH 45223, E-mail: info@rotex.com. If you are aware of alternative suppliers, please provide this information to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee, which you may attend.

⁶ Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR: D24-1007.