Designation: D 1511 – 00

Standard Test Method for Carbon Black—Pellet Size Distribution[1]

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.

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:*

D 1799 Practice for Carbon Black—Sampling Packaged Shipments[2]

D 1900 Practice for Carbon Black—Sampling Bulk Shipments[2]

D 4483 Practice for Determining Precision for Test Method Standards in the Rubber and Carbon Black Industries[2]

D 5817 Practice for Carbon Black, Pelleted-Reduction and Blending of Gross Samples[2]

E 11 Specification for Wire-Cloth Sieves for Testing Purposes[3]

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 5817D 5817.

4.2 Balance with a sensitivity of 0.1 g.

4.3 *Sieves*—U.S. Standard Sieves or equivalent, conforming to Specifications E 11E 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 Shaker*[4]—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 (⅛ to ⅜ 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 ± 0.15 cm (1–5/16 in ± 1/16 in).

4.5.2 *Vibratory Siever*[5]—The Retch/Brinkmann Vibratory Siever AS200 has variable timer and amplitude settings. When set at 3 minutes and 0.5 amplitude, the vibratory siever provides satisfactory results. The siever accommodates a stack of sieves as described in 4.3.

4.5.3 *Automatic Sieve Shaker*[6]—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

[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] *Annual Book of ASTM Standards*, Vol 09.01.

[3] *Annual Book of ASTM Standards*, Vol 14.02.

[4] The Ro-Tap Siever is available from WS Tyler, 8570 Tyler Blvd., Mentor, OH 44060, Ph. 1-800-321-6188.

[5] The Retsch/Brinkmann Vibratory Siever AS200 is available from Brinkmann Instruments, Inc., One Cantiague Rd., Westbury, NY 11590, Ph. 1-800-645-3050.

[6] The Gradex 2000 is available from Rotex, Inc., 1230 Knowlton Street, Cincinnati, OH 45223, Ph. 1-513-541-1236.

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 1799D 1799 or D 1900D 1900.

5.2 Practice D 5817D 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	_____	_____
35	_____	_____
60	_____	_____
120	_____	_____
Pan	_____	_____
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 [7]

9.1 This precision and bias section has been prepared in accordance with Practice D 4483D 4483. Refer to Practice D 4483D 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.

9.4 *Repeatability*— The repeatability, *r*, of the specific screen fraction has been established as the value tabulated in Tables 1-6. Two single test results (or determinations) that differ by more than *r* must be considered suspect and dictates that some appropriate investigative action be taken.

9.5 *Reproducibility*— The reproducibility, *R*, of the specific screen fraction has been established as the value tabulated in Tables 1-6. Two single test results (or determinations) that

[7] Supporting data are available from ASTM Headquarters. Request RR: D24-1007.

TABLE 1 Precision-Type 1 Carbon Black-Pellet Size Distribution +10[A]

Material	Mean Level, %	Within Laboratories			Between Laboratories		
		Sr	*r*	*(r)*	*SR*	*R*	*(R)*
Sample 2	0.62	0.08	0.21	34.36	0.27	0.76	121.45
Sample 3	1.77	0.23	0.64	36.08	0.49	1.39	78.37
Sample 1	3.52	0.32	0.90	25.58	1.33	3.75	106.64
pooled or average values	1.97	0.23	0.65	32.94	0.83	2.35	119.29

[A] This is short term precision (days) with:
$p = 7$, $q = 3$, and $n = 4$.
Symbols are defined as follows:

Sr = Within laboratory standard deviation,
r = repeatability (in measured units),
(r) = repeatability (in percent),
SR = Between laboratory standard deviation,
R = reproducibility (in measured units), and
(R) = reproducibility (in percent).