Designation: D1511 – 12 (Reapproved 2023)

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

This standard is issued under the fixed designation D1511; 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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.*

1.4 *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

D5817 Practice for Carbon Black, Pelleted—Reduction, Blending, and Drying of Gross Samples for Testing

E11 Specification for Woven Wire Test Sieve Cloth and Test Sieves

3. Summary of Test Method

3.1 A sample of carbon black is shaken in a sieve shaker to separate the pellets by size with specified series of sieve screens arranged with progressively smaller openings. The percentage, by mass, of carbon black retained on each sieve is weighed to calculate the pellet size distribution.

4. Significance and Use

4.1 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.

5. Apparatus

5.1 Riffle Sample Splitter as specified in Practice D5817.

5.2 Balance with a sensitivity of 0.1 g.

5.3 *Sieves*—U.S. Standard Sieves or equivalent, conforming to Specification E11. 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.

5.4 Bottom receiver pan and top sieve cover.

5.5 *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.

5.5.1 *Mechanical Sieve Shaker*[3]—The Mechanical Sieve Shaker shall impart a uniform rotary and tapping motion to a stack of sieves as described in 5.3. The shaker machine shall be powered with an electric motor producing constant oscillation to the sieve stack providing 280 to 320 rotary motions/min and 140 to 160 taps per minute. The cover plate shall be fitted with

[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 following devices have been found suitable: the Ro-Tap Siever and the AS 200 tap. The sole sources of supply known to the committee at this time are, respectively, WS Tyler, 8570 Tyler Blvd., Mentor, OH 44060, E-mail: wstyler@wstyler.com, and Retsch Inc., 74 Walker Lane, Newton, 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.

a cork stopper that shall extend 3 to 9 mm (⅛ to ⅜ in.) above the metal recess. Materials other than cork, such as rubber or wood, are unacceptable. The amplitude of the hammer shall be set at 3.30 ± 0.15 cm (1–5⁄16 ± 1⁄16 in.).

5.5.2 *Vibratory Siever*[4]—The Retsch Sieve Shaker AS200 has variable timer and amplitude settings. When set at 3 min and 0.5 amplitude, the AS200 provides satisfactory results. It accommodates a stack of sieves as described in 5.3.

5.5.3 *Automatic Sieve Shaker*[5]—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 min provides satisfactory test results. The test sieves are described in 5.3.

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.

6. Sampling

6.1 Lot samples shall be taken in accordance with Practices D1799 or D1900.

6.2 Practice D5817 shall be used for blending or reducing samples.

7. Procedure

7.1 Prepare carbon black for testing as noted in Section 6.

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.

7.2 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.

7.3 *Mechanical and Vibratory Sieve Shakers:*

7.3.1 Weigh 100.0 g of carbon black.

7.3.2 Transfer weighed carbon black to the top sieve.

7.3.3 Install the sieve cover and transfer the sieve assembly to the shaker. The stack in the shaker should be adjusted to eliminate looseness.

7.3.4 Start the shaker and allow it to shake as noted below:

7.3.4.1 *Mechanical Shaker*—1 min with hammer operating.

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).

7.3.4.2 *Vibratory Shaker*—3 min and 0.5 mm amplitude.

7.3.5 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.

7.3.6 Record the data and calculate as noted in Section 8.

7.4 *Automatic Sieve Shaker:*

7.4.1 Follow the manufacturer's instructions to load software and configure the shake time to 1 min.

7.4.2 Transfer the sieve assembly to the automatic sieve shaker.

7.4.3 Weigh or measure approximately 100 g of carbon black.

7.4.4 Transfer measured carbon black to the autofeed container.

7.4.5 Enter the sample identification into the operating program. Repeat steps 7.4.3 – 7.4.5 as required for consecutive samples. Up to six samples may be identified at one time.

7.4.6 Start the testing sequence according to the manufacturer's operating instructions.

7.4.7 Retrieve report from the computer.

8. Calculation

8.1 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	_____	_____

9. Report

9.1 Report the following information:

9.1.1 Proper identification of the sample,

9.1.2 Result obtained from a single determination, reported to the nearest 0.1 %.

9.1.3 Apparatus used to determine test values.

[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.

[5] The 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,[1] which you may attend.

TABLE 2 Precision-Type 1 Carbon Black-Pellet Size Distribution +18[A]

Material	Mean Level, %	Within Laboratories			Between Laboratories		
		Sr	*r*	*(r)*	*SR*	*R*	*(R)*
Sample 2	24.89	1.36	3.84	15.42	6.27	17.75	71.31
Sample 1	45.27	2.14	6.06	13.39	7.50	21.21	46.85
Sample 3	72.75	2.34	6.63	9.11	2.98	8.43	11.59
pooled or average values	47.64	1.99	5.64	11.84	5.90	16.69	35.04

[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).

TABLE 3 Precision-Type 1 Carbon Black-Pellet Size Distribution +35[A]

Material	Mean Level, %	Within Laboratories			Between Laboratories		
		Sr	*r*	*(r)*	*SR*	*R*	*(R)*
Sample 3	20.66	1.43	4.05	19.61	2.03	5.76	27.88
Sample 1	32.48	1.03	2.91	8.96	5.70	16.13	49.65
Sample 2	43.02	0.88	2.48	5.76	1.33	3.77	8.76
pooled or average values	32.05	1.14	3.21	10.03	3.58	10.12	31.59

[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).

10. Precision and Bias[6]

10.1 This precision and bias section has been prepared in accordance with Practice D4483. Refer to Practice D4483 for terminology and other statistical details.

10.2 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.

10.3 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.

TABLE 4 Precision-Type 1 Carbon Black-Pellet Size Distribution +60[A]

Material	Mean Level, %	Within Laboratories			Between Laboratories		
		Sr	*r*	*(r)*	*SR*	*R*	*(R)*
Sample 3	2.97	0.65	1.84	61.94	1.07	3.03	101.92
Sample 1	11.90	1.22	3.45	28.97	3.28	9.29	78.04
Sample 2	21.36	1.12	3.18	14.86	2.21	6.25	29.25
pooled or average values	12.08	1.03	2.91	24.07	2.37	6.70	55.44

[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).

10.4 *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.

10.5 *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 differ by more than *R* must be considered suspect and dictates that some appropriate investigative action be taken.

10.6 *Bias*—Bias is the difference between an average test value and the reference (true) test property value. Reference values do not exist for this test method since the value or level of the test property is exclusively defined by the test method. Therefore, bias cannot be determined.

11. Keywords

11.1 carbon black; pellet size distribution

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