Designation: D 3313 – 99

Standard Test Method for Carbon Black—Individual Pellet Hardness[1]

This standard is issued under the fixed designation D 3313; 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 standard describes a method for measuring the crush strength of individual pellets 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 1511 Test Method for Carbon Black—Pellet Size Distribution[2]

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]

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

3. Significance and Use

3.1 Pellet crush strength is related to several carbon black characteristics. Among these are mass strength and attrition. The subsequent level of dispersion obtained in some mixed compounds containing the carbon black may be affected by pellet crush strength. Acceptable pellet hardness must be agreed to by the user and the producer.

NOTE 1—Test Method D 5230 is the preferred standard for testing of individual pellet crush strength. It is recognized that Test Method D 3313 relies on operator judgement, thus adding an additional source of variation for this test.

4. Apparatus

4.1 *Pellet-Hardness Tester*,[4] of a type capable of measuring the crush strength of the individual pellet in grams-force (millinewtons). A suitable tester must exhibit the following characteristics:

4.1.1 Initial contact force must be applied so that the force area rests lightly on the pellet before proceeding,

4.1.2 It must be capable of applying force at a constant rate,

4.1.3 It must possess an accurate means of measuring the applied force, and

4.1.4 During the test, the applied force and base plate must be controlled so as not to cause the pellet to move or roll prior to crushing.

NOTE 2—A two-pan torsion balance with 100-g dial and transparent foot mounted so it can be moved directly over the pellet will serve to convert a balance to a pellet strength tester, as needed.

4.2 *Mechanical Sieve Shaker*,[5] (see Test Method D 1511).

4.3 *Sieves*—U.S. Standard sieves or equivalents, conforming to Specification E 11. Sieves Nos. 12 (1700-µm) and 14 (1400-µm) shall be used.

4.4 *Bottom-Receiver Pan and Top-Sieve Cover*.

4.5 *Sample Splitter*, single-stage, riffle-type.

4.6 *Container*, shallow, flat approximately 305 mm (12 in.) long.

4.7 *Forceps*, fitted with sponge tips. A very low-density urethane foam sponge has been found to be acceptable.

5. Sampling

5.1 Samples shall be taken in accordance with Practices D 1799 or D 1900.

6. Calibration

6.1 Equipment used for this test shall be calibrated by dead weights or a force-measuring device that will verify the accuracy of the equipment over the range to be tested.

7. Procedure

7.1 Prepare a sample of carbon black, as follows:

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[1] This practice is under the jurisdiction of ASTM Committee D-24 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 Model-S Pellet Strength Tester, A. L. Sweigart, Technical Service Shop, 1206 Hemlock St., Borger, TX 79007, with lab jack has been found satisfactory for this purpose.

[5] A Ro-Tap sieve shaker has been found satisfactory for this purpose.

7.1.1 Pass the gross sample through the single stage, riffle-type, sample splitter to obtain a representative sample of approximately 100 g. The amount of riffled sample may be varied if the pellet fraction being collected is known or found to be at extremes.

7.1.2 Stack the sieves in the following order from bottom to top: Bottom receiver pan, No. 14 (1400-µm) and No. 12 (1700-µm). It is permissible to use multiples of sieves to screen several samples simultaneously.

7.1.3 Transfer the 100-g riffled black to the No. 12 (1700-µm) screen, install the cover and transfer the assembly to the mechanical shaker.

7.1.4 Allow the sieve assembly to shake for 60, − 0 + 10 s, with the hammer operating.

7.2 Remove the assembly from the shaking device and pour approximately 2 g of pellets retained on the No. 14 (1400-µm) sieve into one end of the shallow container. Tilt the container slightly to cause the most spherical pellets to roll to the opposite end.

7.3 Take 20 of the spherical pellets for testing. Handle individual pellets, when transported or positioned for test, or both, with the sponge tipped forceps.

7.4 Position pellet to be tested on a solid vibration-free surface so as to be near the center area of the applied force.

7.5 Bring the measuring device in contact with the pellet, but do not apply any force. This step is most important when testing soft pellets, which tend to fracture immediately or prematurely due to impact on initial contact.

7.6 Apply force at a slow, constant rate until the pellet is fractured. Record the indicated value to the nearest whole number.

7.7 Repeat 7.4 to 7.6 until 20 pellets have been tested.

8. Report

8.1 The report shall include the following:

8.1.1 Proper identification of the sample,

8.1.2 Average value for the 20 pellets tested, reported to the nearest whole number,

8.1.3 Maximum value for the 20 pellets tested, and

8.1.4 Any other results as determined between the purchaser and the seller.

9. Precision and Bias

9.1 This precision and bias has been prepared in accordance with Practice D 4483. Refer to this practice for terminology and other statistical details.

9.2 The precision results in this precision and bias give an estimate of the precision as described below. The precision parameters should not be used for acceptance/rejection testing of any group 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 1990. Both repeatability and reproducibility represent short term testing conditions. Seven laboratories tested three carbon blacks (D, E, and F) twice on two different days. A test result is the value obtained from an average of 20 pellets. Acceptable difference values were not measured.

9.4 The result of the precision calculations were given in Table 1 with the material arranged in ascending “mean level” order.

9.5 The precision for the pooled values for individual pellet crush strength may be expressed as follows:

9.5.1 *Repeatability*— The repeatability, (*r*), of the individual pellet crush strength result has been established as 26.4 %. Two single test results (or determinations) that differ by more than 26.4 % must be considered suspect and dictates that some appropriate investigative action be taken.

9.5.2 *Reproducibility*— The reproducibility, (*R*), of the individual pellet crush strength result has been established as 74.1 %. Two single test results (or determinations) produced in separate laboratories that differ by more than 74.1 % must be considered suspect and dictates that some appropriate investigative or technical/commercial action be taken.

9.6 *Bias*—In test method terminology, 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. Bias, therefore, cannot be determined.

10. Keywords

10.1 carbon black; individual pellet crush strength; dispersion level; pellet hardness tester

TABLE 1 ASTM Test Method Precision: Type 1 Carbon Black—Individual Pellet Crush Strength (D 3313)[A]

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).

Material	Mean Level mN	Within Laboratories			Between Laboratories		
		sr	*r*	(*r*)	*SR*	*R*	(*R*)
D	214.37	26.37	74.63	34.81	61.99	175.43	81.83
E	390.30	34.06	96.39	24.69	102.25	289.38	74.14
F	395.99	32.48	91.92	23.21	92.55	261.90	66.14
pooled or average values	333.56	31.15	88.14	26.43	87.30	247.05	74.07

[A]This is short term precision (days).