Designation: D3313 – 12$^{\varepsilon 1}$

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

This standard is issued under the fixed designation D3313; 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—Corrected 5.1 editorially in October 2016.

1. Scope

1.1 This test method covers a method for measuring the hardness 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:*[2]

D1511 Test Method for Carbon Black—Pellet Size Distribution

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

D5230 Test Method for Carbon Black—Automated Individual Pellet Hardness

E11 Specification for Woven Wire Test Sieve Cloth and Test Sieves

3. Summary of Test Method

3.1 A sample of carbon black is passed through two sieves to isolate a fraction of uniform size. The most spherical pellets from this portion are selected and brought into contact with a measuring device as force is applied. Pellet hardness is the maximum force required to crush the pellets.

4. Significance and Use

4.1 Pellet hardness 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 hardness. Acceptable pellet hardness must be agreed to by the user and the producer.

NOTE 1—Test Method D5230 is the preferred standard for testing of individual pellet hardness. It is recognized that Test Method D3313 relies on operator judgement, thus adding an additional source of variation for this test.

5. Apparatus

5.1 *Pellet-Hardness Tester,* of a type capable of measuring the pellet hardness of the individual pellet in grams-force (centinewtons). A suitable tester must exhibit the following characteristics:

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

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

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

5.1.4 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 hardness tester, as needed.

5.2 *Mechanical Sieve Shaker,* conforming to Test Method D1511.

5.3 *Sieves*—U.S. Standard sieves or equivalents, conforming to Specification E11. Sieve Nos. 12 (1700-µm) and 14 (1400-µm) shall be used.

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

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

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

[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.

Current edition approved June 1, 2012. Published September 2012. Originally approved in 1974. Last previous edition approved in 2005 as D3313 – 05a. DOI: 10.1520/D3313-12E01.

[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.

6. Sampling

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

7. Calibration

7.1 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.

8. Procedure

8.1 Prepare a sample of carbon black, as follows:

8.1.1 Stack the No. 12 above the No. 14 sieve with the receiver pan on the bottom.

NOTE 3—It is permissible to use multiples of sieves to screen several samples simultaneously.

8.1.2 Place the sample in the top sieve and install the cover. Transfer the assembly to the shaking device.

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

8.2 Remove the assembly from the shaking device. Select the more spherical pellets retained on the No. 14 sieve.

8.3 The more spherical pellets may be selected by pouring approximately 2 g of pellets retained on the No. 14 sieve into one end of the shallow container. Tilt the container slightly to cause the most spherical pellets to roll to the opposite end.

8.4 Take 20 or 50 of the most spherical pellets for testing. Twenty pellets are tested in normal applications. Fifty pellets may be tested for critical applications. Critical applications are determined by agreement between the customer and supplier. Handle individual pellets, when transported or positioned for test, or both, with the sponge-tipped forceps.

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

8.6 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.

8.7 Apply force at a constant rate of approximately 5 cN/s (g force/s) until the pellet is fractured. Record the indicated value to the nearest whole number.

8.8 Repeat 8.5 – 8.7 until the appropriate number of pellets have been tested.

9. Report

9.1 Report the following information:

9.1.1 Proper identification of the sample,

9.1.2 Average value in centinewtons (grams-force) rounded to the nearest millinewton (nearest 0.1 gram-force),

9.1.3 Maximum as the highest individual value in centinewtons (grams-force) rounded to a whole number,

9.1.4 Number of pellets tested, and

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

10. Precision and Bias

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

10.2 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.

10.3 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.

10.4 The result of the precision calculations were given in Table 1 with the material arranged in ascending "mean level" order.

10.5 The precision for the pooled values for individual pellet hardness may be expressed as follows:

10.5.1 *Repeatability*—The repeatability, (*r*), of the individual pellet hardness 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.

10.5.2 *Reproducibility*—The reproducibility, (*R*), of the individual pellet hardness 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.

TABLE 1 ASTM Test Method Precision: Type 1 Carbon Black—Individual Pellet Hardness (D3313)[A]

Material	Mean Level, cN	Within Laboratories		Between Laboratories	
		sr	*(r)*	*SR*	*(R)*
D	21.4	2.64	34.8	6.20	81.8
E	39.0	3.41	24.6	10.2	74.1
F	39.6	3.25	23.2	9.26	66.1
pooled or average values	33.4	3.12	26.4	8.73	74.0

Symbols are defined as follows:

Sr = within laboratory standard deviation,
(r) = repeatability (in percent),
SR = between laboratory standard deviation, and
(R) = reproducibility (in percent).

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

10.6 *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.

11. Keywords

11.1 carbon black; dispersion level; individual pellet hardness; pellet hardness tester