Designation: D5230 – 21

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

This standard is issued under the fixed designation D5230; 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 a procedure for measuring individual pellet hardness of carbon black by the automated pellet hardness tester.

1.2 The values stated in SI units are to be regarded as the standard. The values 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]

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

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. An appropriate amount of pellets from this portion is selected and placed into the tester. The individual pellets are pressed against a platen with a load cell for measuring force. As pressure is applied the pellet will either break with a rapid force reduction or the pellet will simply compress. The individual pellet hardness is the maximum force prior to a force reduction of at least 3 cN or the maximum force required to compress the pellet to 90 %, whichever comes first.

4. Significance and Use

4.1 Individual 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.

5. Apparatus

5.1 *Automated Pellet Hardness Tester,* capable of achieving an absolute measuring accuracy of ±2 cN (2 gf) for the force measurement and ±0.1 mm for the diameter measurement and a relative accuracy of ±0.5 cN (0.5 gf) for the force measurement and 0.02 mm for the diameter measurement and consisting of the following major components and characteristics.

5.1.1 A means for automatic loading of a pellet on the transport platen for transporting the pellet so as to contact the second platen with a minimum force. Typically one platen contains a force measuring device. The required force to detect the contact shall not exceed 2 cN (2 gf),

5.1.2 A means for applying the force while moving at a constant speed,

5.1.3 A means for transporting the pellet so to minimize its movement during the application of force.

5.1.4 A means for measuring the diameter of the individual pellet under test as measured along the axis of the application of force.

5.1.5 A control device for directing the instrument through the test cycle that includes crushing the pellet under controlled conditions, measuring and storing the results of the initial diameter and crush force determinations, cleaning the fragments from the platen surfaces, and starting the next cycle.

[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.

5.1.6 An algorithm for determining the individual test end point (determination) as the maximum observed force prior to the first occurrence of either a specified reduction in diameter or a specified reduction in force from the maximum force observed,

5.1.7 A program for calculating for a specified number of pellets the data as requested in Section 9, and

5.1.8 A means for identifying, viewing, printing, and storing the data in an ASCII file.

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

5.3 *Sieves,* U.S. Standard No. 12 (1700 µm) and No. 14 (1400 µm) conforming to Specification E11 shall be used to test grades of black that can be segregated in a −12/+14 fraction. For grades of black that are too small to be retained on a No. 14 sieve, i.e., acetylene and thermal blacks, it is acceptable to test with U.S. Standard No. 16 (1180 µm) and No. 18 (1000 µm) sieves.

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

6. Sampling

6.1 Take samples in accordance with Practice D1799 or Practice D1900.

7. Calibration

7.1 Calibrate force and diameter measurement following the manufacturer's instructions.

7.2 *Instrument Parameters:*

7.2.1 Crush diameter, 0.90. A reduction of the pellet diameter to 90 % of the original value is one of two end point criteria.

7.2.2 Force drop. A decrease of 3 cN (3gf) from the maximum force observed is one of two end point criteria.

7.2.3 Rate of piston movement during crush, 0.125 mm/s.

7.2.4 Number of pellets tested; normal applications, 20 pellets, critical applications, 50 pellets. Critical applications are determined by agreement between customer and supplier.

7.2.5 The following ranges of acceptable pellet diameters were established to minimize the number of pellets rejected due to instrument variation and non-spherical pellets.

7.2.5.1 For a −12/+14 fraction, 1.31–1.93 mm.

7.2.5.2 For a −16/+18 fraction, 0.80–1.44 mm.

8. Procedure

8.1 Prepare a sample of carbon black as follows:

8.1.1 Stack the No. 12 above the No. 14 sieve, or to test smaller pellet blacks, stack the No. 16 above the No. 18 sieve with the receiver pan on the bottom.

NOTE 1—It is permissible to use multiples of sieve stacks to screen several samples simultaneously.

8.1.2 Transfer the sample to the coarser top screen, install the cover and transfer the assembly to the mechanical shaker.

8.1.3 Allow the sieve assembly to shake for 60 s with the hammer operating.

8.2 Remove the assembly from the shaking device. Select a sufficiently large sample from the pellets retained on the bottom sieve. The sample size required for testing depends on the apparatus that is used.

8.3 Conduct the test following the instructions in the equipment operation manual.

9. Report

9.1 Report the following information:

9.1.1 Proper identification of the sample,

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

9.1.3 Maximum value in centinewtons (gram force) rounded to a whole number

9.1.4 Number of pellets tested, and

9.1.5 Size of sieves used to prepare the sample.

10. Precision and Bias

10.1 These precision statements have 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 section give an estimate of the precision of this test method with the materials used in the particular interlaboratory program described below. The precision parameters should not be used for acceptance or rejection testing of any group of materials without documentation that they are applicable to those particular materials and the specific testing protocols of the test method.

10.3 A type 1 inter-laboratory precision program was conducted as detailed in Tables 1-4. Both repeatability and reproducibility represent short term (daily) testing conditions. The testing was performed using two operators in each laboratory performing the test once on each of two days (total of four tests). The pellet hardness average test result is the average of all the individual pellet hardness values obtained in a single determination. The pellet hardness maximum test result is the highest individual value of all the individual pellet hardness values obtained in a single determination. Other techniques for obtaining a maximum value, such as the average of x number of the highest individual values or the average of the highest individual values that represent y percent of the total number of pellets tested are not included in these precision calculations. Acceptable difference values were not measured. The between operator component of variation is included in the calculated values for r, (r), R, and (R).

10.4 The results of the precision calculations for this test method are given in Tables 1-4.

10.5 *Repeatability*—The pooled **relative** repeatability, (r), for the pellet hardness average result of this test when testing 20 pellets has been established as 18.6 % (see Table 1). The pooled **relative** repeatability, (r), for the pellet hardness average result of this test when testing 50 pellets has been established as 15.0 % (see Table 2). The pooled **relative** repeatability, (r), for the pellet hardness maximum result of this test when testing 20 pellets has been established as 27.6 % (see Table 3). The pooled **relative** repeatability, (r), for the pellet hardness maximum result of this test when testing 50 pellets has been established as 19.8 % (see Table 4). The best estimate

TABLE 1 Precision Parameters for Test Method D5230, Pellet Hardness Average for 20 Pellets (Type 1 Precision)

		Units	cN (gf)						
Material	Nominal Testing Period	Number of Laboratories (M/H/L)[A]	Mean Level	Sr	r	(r)[B]	SR	R	(R)[B]
SRB-9A	Mar 2013	45(1/2/0)	43.4	2.64	7.48	**17.3**	6.50	18.39	**42.4**
SRB-9A2	Aug 2018	43(0/0/0)	37.9	2.58	7.29	**19.3**	4.89	13.84	**36.6**
SRB-9B	Mar 2016	51(0/1/0)	42.4	2.93	8.31	**19.6**	5.86	16.58	**39.1**
SRB-9B2	Mar 2019	47(0/0/0)	42.0	2.37	6.72	**16.0**	4.67	13.22	**31.5**
SRB-9C	Aug 2019	37(0/0/1)	22.6	1.78	5.04	**22.3**	4.69	13.28	**58.8**
SRB-9D	Mar 2018	47(0/1/0)	34.6	2.37	6.71	**19.4**	4.40	12.45	**36.0**
SRB-9E	Aug 2016	47(0/0/0)	34.6	2.23	6.30	**18.2**	5.34	15.10	**43.7**
SRB-9F	Mar 2015	41(0/0/0)	34.1	2.00	5.67	**16.6**	7.21	20.40	**59.8**
SRB-9G	No Data[C]	0(0/0/0)							
SRB-9H	Mar/Apr 2020	48(0/2/0)	23.0	1.30	3.69	**16.0**	3.06	8.67	**37.6**
Average			34.9						
Pooled Values				2.29	6.49	**18.6**	5.31	15.02	**43.0**

[A] M=Mean outliers; H=High variability outliers; L=Low variability outliers.
[B] Preferred parameter shown in bold.
[C] Not enough 12/14 pellets to be able to perform the test per Test Method D5230 instructions.

TABLE 2 Precision Parameters for Test Method D5230, Pellet Hardness Average for 50 Pellets (Type 1 Precision)

		Units	cN (gf)						
Material	Nominal Testing Period	Number of Laboratories (M/H/L)[A]	Mean Level	Sr	r	(r)[B]	SR	R	(R)[B]
SRB-9A	Mar 2013	23(0/0/0)	45.1	2.54	7.20	**16.0**	4.85	13.73	**30.5**
SRB-9A2	Aug 2018	24(0/1/0)	40.6	1.57	4.44	**10.9**	4.61	13.03	**32.1**
SRB-9B	Mar 2016	23(0/0/1)	43.7	2.57	7.26	**16.6**	5.14	14.56	**33.3**
SRB-9B2	Mar 2019	24(1/1/1)	44.0	1.58	4.48	**10.2**	3.89	11.02	**25.1**
SRB-9C	Aug 2019	15(0/0/0)	22.3	1.73	4.89	**21.9**	4.18	11.82	**53.0**
SRB-9D	Mar 2018	22(0/1/0)	37.4	1.64	4.63	**12.4**	4.57	12.93	**34.5**
SRB-9E	Aug 2016	21(0/0/0)	35.5	2.39	6.76	**19.0**	5.76	16.30	**45.9**
SRB-9F	Mar 2015	26(0/1/0)	36.6	1.42	4.01	**11.0**	7.22	20.43	**55.9**
SRB-9G	No Data[C]	0(0/0/0)							
SRB-9H	Mar/Apr 2020	15(0/0/0)	21.7	1.47	4.17	**19.2**	2.75	7.78	**35.9**
Average			36.3						
Pooled Values				1.93	5.47	**15.0**	4.92	13.91	**38.3**

[A] M=Mean outliers; H=High variability outliers; L=Low variability outliers.
[B] Preferred parameter shown in bold.
[C] Not enough 12/14 pellets to be able to perform the test per Test Method D5230 instructions.

of the test precision is given by the pooled values and these should be used unless there is a good reason to use a different value. Any other value in Tables 1-4 may be used as an estimate of repeatability, if justified, such as when testing the same material as, or a material similar to, those in the tables. The difference between two single test results (or determinations) found on identical test material under the repeatability conditions prescribed for this test will exceed the repeatability value on an average of not more than once in 20 cases in the normal and correct operation of the method. Two single test results that differ by more than the appropriate value from Tables 1-4 must be suspected of being from different populations and some appropriate action taken.

NOTE 2—See footnote A in Table 3 and Table 4 concerning the validity of drawing any conclusions regarding the precision of the pellet hardness maximum test result.

NOTE 3—Appropriate action may be an investigation of the test method procedure or apparatus for faulty operation or the declaration of a significant difference in the two materials, samples, etc., which generated the two test results.

10.6 *Reproducibility*—The pooled **relative** reproducibility, (R), for the pellet hardness average result of this test when testing 20 pellets has been established as 43.0 % (see Table 1). The pooled **relative** reproducibility, (R), for the pellet hardness average result of this test when testing 50 pellets has been established as 38.3 % (see Table 2). The pooled **relative** reproducibility, (R), for the pellet hardness maximum result of this test when testing 20 pellets has been established as 50.8 % (see Table 3). The pooled **relative** reproducibility, (R), for the pellet hardness maximum result of this test when testing 50 pellets has been established as 43.8 % (see Table 4). The best estimate of the test precision is given by the pooled values and these should be used unless there is a good reason to use a different value. Any other value in Tables 1-4 may be used as an estimate of reproducibility, if justified, such as when testing the same material as, or a material similar to, those in the tables. The difference between two single and independent test results found by two operators working under the prescribed reproducibility conditions in different laboratories on identical test material will exceed the reproducibility value on an average of not more than once in 20 cases in the normal and correct operation of the method. Two single test results produced in different laboratories that differ by more than the