Designation: D 5230 – 00

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

This standard is issued under the fixed designation D 5230; 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 the crush strength of individual pellets of carbon black by the automated pellet hardness tester.[2]

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 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[3]

D 1799 Practice for Carbon Black—Sampling Packaged Shipments[3]

D 1900 Practice for Carbon Black—Sampling Bulk Shipments[3]

D 4483 Practice for Determining Precision for Test Method Standards in the Rubber and Carbon Black Industries[3]

E 11 Specification for Wire-Cloth Sieves for Testing Purposes[4]

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.

4. Apparatus

4.1 *Automated Pellet Hardness Tester*, [2] 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.

4.1.1 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),

4.1.2 A means for applying the force at a constant rate,

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

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

4.1.5 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.

4.1.6 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,

4.1.7 A program for calculating for a specified number of pellets the data as requested in Section 8, and

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

4.2 *Mechanical Sieve Shaker*, conforming to Test Method D 1511.

4.3 *Sieves*, U.S. Standard No. 12 (1700 µm) and No. 14 (1400 µm) conforming to E-11 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.

4.4 *Sieves*, U.S. standard, or equivalent, conforming to Specification E 11. Sieve Nos. 12 (1700 µm) and 14 (1400 µm) shall be used.

NOTE 1—Pellet size to be tested is 12/14 by program default. Another size may be selected if desired by pressing menu item "6".

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

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[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] The Concarb Titan Automated Pellet Hardness Tester is available from Titan Specialties, Inc., P.O. Box 2316, Pampa, TX 79065. The HITEC IPHT is available from HITEC Luxembourg, 5 rue de L' Eglise, L-1458, Luxembourg.

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

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

4.6 *Special Pellet Selection Tray*, [5] (shallow container), flat and approximately 300 mm (12 in.) long.

5. Sampling

5.1 Take samples in accordance with Practice D 1799 or Practice D 1900.

6. Calibration

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

6.2 *Instrument parameters*:

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

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

6.2.3 Rate of piston movement during crush, 0.125 mm/s

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

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

6.2.5.1 For a -12/+14 fraction, 1.31–1.93 mm.

6.2.5.2 For a -16/+18 fraction, 0.80-1.44 mm.

7. Procedure

7.1 Prepare a sample of carbon black as follows:

7.1.1 Stack the sieves in the following order from bottom to top: bottom receiver pan, No. 14 and No. 12.

NOTE 2—It is permissible to use multiples of sieve stacks to screen several samples simultaneously.

7.1.2 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 reciever pan on the bottom.

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

7.1.4 Transfer the sample to the No. 12 screen, install the cover and transfer the assembly to the mechanical shaker.

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

7.2 Remove the assembly from the shaking device. Select the more spherical pellets retained on the bottom sieve.

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

7.3.1 The more spherical pellets may be selected by use of a special pellet selection tray. Pour approximately 2 g of pellets into the wide end of the tray, tilt the tray slightly to cause the more spherical pellets to roll to the narrow end. Collect the more spherical pellets for testing.

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

8. Report

8.1 Report the following information:

8.1.1 Proper identification of the sample,

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

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

8.1.4 As an option, the average of the highest 10 % of the individual test values in centinewtons (grams force) rounded to a whole number,

8.1.5 Number of pellets tested, and

8.1.6 Size of sieves used to prepare the sample.

9. Precision and Bias

9.1 These precision statements have 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 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. Any appropriate value may be used from Table 1.

TABLE 1 Precision Parameters for D 5230 Automated Individual Pellet Hardness, (Type 1 Precision)

Units	cN (gf)				
Material	Mean Level	Sr	(r)	SR	(R)
N650	23	2	5	4	13
SRB N762	24	2	5	6	16
IRB#6 (N330)	30	2	6	4	13
SRB A5 (N135)	31	2	5	5	13
N550	32	2	6	5	15
Average	28				
Pooled Values		2	50	50	14

9.3 A type 1 inter-laboratory precision program was conducted as detailed in Table 2. 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). A test result is the average value obtained from a single determination. Acceptable difference values were not measured. The between operator component of variation is included in the calculated values for r and R.

9.4 The results of the precision calculations for this test are given in Table 1. The materials are arranged in ascending "mean level" order.

9.5 *Repeatability*—The pooled relative repeatability, (r), of this test has been established as 5 cN (5gf). Any other value in Table 1 may be used as an estimate of repeatability, as

TABLE 2 Interlaboratory Precision Program

Nominal Test Period	Material	Number of Laboratories
March 1996	N650	50
October 1996	IRB#6 (N330)	39
March 1997	SRB N762	46
September 1997	SRB A5 (N135)	41
March 1998	N550	46

[5] A special pellet selection tray is available from Titan Specialties, Inc., P.O. Box 2316, Pampa, TX 79065.

appropriate. 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 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 Table 1 must be suspected of being from different populations and some appropriate action taken.

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.

9.6 *Reproducibility*—The pooled relative reproducibility, (R), of this test has been established as 14 cN (14gf). Any other value in Table 1 may be used as an estimate of reproducibility, as appropriate. 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 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 appropriate value from Table 1 must be suspected of being from different populations and some appropriate investigative or technical/commercial action taken.

9.7 *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; pellet crush strength; pellet hardness