Designation: D 1508 – 02$^{\epsilon 1}$

Standard Test Method for Carbon Black, Pelleted Fines and Attrition[1]

This standard is issued under the fixed designation D 1508; 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—Section 7.1.4 was editorially updated in December 2002.

1. Scope

1.1 This test method covers the determination of the fines and attrition of pelleted carbon black.

1.2 *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]

D 5817 Pelleted Carbon Black—Practice for Reducing and Blending Gross Samples of Carbon Black[2]

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

3. Summary of Test Method

3.1 *Method A, Fines*—A sample of carbon black is placed on a 125-µm sieve and shaken in a mechanical or vibratory sieve shaker for 5 min. The pellets, pellet fragments, dust, and unpelletized black that pass through the sieve are defined as carbon black fines. The fines are expressed in percent.

3.2 *Method B, Attrition*—The same test sample is shaken for an additional 15 min to determine the amount of pellet degradation or attrition created during this additional shake interval. The attrition is expressed in percent.

4. Significance and Use

4.1 *Method A, Fines*—The fines content of carbon black is related to the bulk flowability, dustiness, and, in some instances, the level of dispersion. Due to the many other variables that influence dispersion and handling, the significance of fines content must be determined by the user.

4.2 *Method B, Attrition*—By comparing the percent fines and attrition, an indication can be obtained of pellet stability and the amount of fines that may be created by pellet degradation in conveying, handling or transit.

5. Apparatus

5.1 *Mechanical or Vibratory Sieve Shaker.*[4]

5.2 *Sieves*, six 125-µm (U.S. Standard No. 120) having a 200-mm (8-in.) diameter and 25-mm (1-in.) height, or equivalent, conforming to Specification E 11.

5.3 *Sieve Separator Receivers*, five required.

5.4 *Sieve Cover*.

5.5 *Bottom Receiver Pan*.

5.6 *Riffle Sample Splitter*.

5.7 *Small Scoop or Large Spoon*.

5.8 *Balance, 0.1-g sensitivity*.

6. Sampling

6.1 Samples shall be taken in accordance with Practice D 1799 or Test Method D 1900.

6.2 Practice D 5817 shall be used for reduction and blending of samples.

7. Procedure

7.1 *Method A, Fines* and *Method B, Attrition*:

7.1.1 Stack up to six sets of sieves and receivers with a receiver beneath each sieve.

7.1.2 Weigh 25.0 g portions, being careful to dip approximately 25 g of black from the riffle splitter.

[1] This practice 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] *Annual Book of ASTM Standards*, Vol 09.01.

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

[4] A Ro–Tap sieve shaker is satisfactory for this purpose. For a description of this apparatus refer to Test Method D 1511. The Fritsche Analysette 3 Pro vibratory sieve shaker has also been found suitable for this purpose (variable amplitude at 3600 vpm) and is available from Gilson Company, P.O. Box 200, Lewis Center, OH 43035–0200, website: www.globalgilson.com.

NOTE 1—It is not good practice to weigh the sample by pouring it directly out of the black container since the fines and 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.

7.1.3 Transfer each sample to an individual 125-µm sieve.

NOTE 2—Six different materials or samples may be tested when all six sets of sieves are used. In some labs the position of the sieve may affect results with the higher sieves yielding higher fines data. For this reason the center position, sieves 3 and 4, should be used for referee testing.

7.1.4 Assemble up to six sets of sieves and receivers into a stack. Place a cover on top and transfer to the shaker. Tighten the shaker to eliminate any looseness. Refer to the user manual for operation of the vibratory sieve shaker. A vibratory amplitude of 1.3 mm should be selected with the vibratory sieve shakers operating at 3600 vpm. However, products that contain little or no binder may experience excessive attrition at the specified amplitude of 1.3 mm. It is the responsibility of the user to determine the appropriate amplitude for these products in order to match the Ro-Tap[4] values. It should be noted that changing the amplitude for standard products is not recommended and may result in erroneous values.

7.1.5 Start the shaker and allow to shake for 5 min with the hammer operating.

7.1.6 Remove the sieve assembly from the shaker and weigh the carbon black retained in each receiver to the nearest 0.1 g.

NOTE 3—To test only attrition, discard the fines without weighing.

7.2 *Method A, Fines*:

7.2.1 If testing only fines, empty and clean thoroughly all sieves in preparation for the next test.

NOTE 4—If attrition is to be tested, retain the pellets on the sieve and discard the fines on the receiver. Proceed to Method B, Attrition.

7.3 *Method B, Attrition*:

7.3.1 Reassemble the sieves and transfer the stack back to the shaker. Shake for an additional 15 min with the hammer operating.

7.3.2 Remove the sieve assembly and weigh the carbon black retained on each receiver to the nearest 0.1 g.

7.3.3 Empty and clean thoroughly all sieves in preparation for the next test.

8. Calculation

8.1 Calculate the fines content to the nearest 0.1 % as follows:

$$F = (WF/S) \times 100 \quad (1)$$

8.2 Calculate attrition to the nearest 0.1 % as follows:

$$A = (WA/S) \times 100 \quad (2)$$

where:

A = attrition, %,
F = fines content, %,
WF = mass of carbon black in receiver after 5 min shake, g,
WA = mass of carbon black in receiver after additional 15 min shake, total 20 min g, and
S = mass of black tested, g.

TABLE 1 Test Method Precision-Type 1: Five Minute Fines (%)

Materials	Mean Level %	Within Laboratories			Between Laboratories		
		Sr	*r*	*(r)*	*SR*	*R*	*(R)*
Material C	1.5	0.31	0.9	60.2	0.51	1.5	98.9
Material A	2.2	0.53	1.5	69.2	0.68	1.9	88.4
Material D	4.5	0.89	2.5	55.7	1.03	2.9	64.4
Material B	9.8	0.87	2.5	25.2	0.91	2.6	26.4
Pooled Values	4.5	0.70	2.0	43.8	0.81	2.3	51.1

9. Report

9.1 Report the following information:

9.1.1 Proper identification of the sample.

9.1.2 Time duration of shaking.

9.1.3 Results obtained, reported to the nearest 0.1 %.

10. Precision and Bias

10.1 These precision statements have been prepared in accordance with Practice D 4483. 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 for fines and attrition testing. 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 interlaboratory precision program was conducted in May 1998. Both repeatability and reproducibility represent short term testing conditions. Seven laboratories tested four carbon blacks twice on two different days. A test result is the 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*.

10.4 The results of the precision calculations for this test are given in Table 1 and Table 2 respectively. The materials are arranged in ascending “mean level” order.

10.5 Precision is dependent upon the amount of fines or attrition. It can be estimated by selecting a material from Table 1 and Table 2 with properties similar to the test material and reading the corresponding precision figures.

10.6 *Repeatability*—The test repeatability of fines, *r* or attrition *(r)* has been established as any appropriate value in Table 1 or Table 2 Two single test results that differ by more than the tabulated value must be considered suspect. Appropriate action should be taken.

10.7 *Reproducibility*—The test reproducibility of fines, *R* or attrition, *(R)* has been established as any appropriate value in Table 1 or Table 2. Two single test results that differ by more than the tabulated value must be considered suspect. Appropriate action should be taken.

10.8 *Bias*—Reference values do not exist for fines and attrition since the levels of the test properties are defined exclusively by the test method. Therefore bias cannot be determined.

10.9 Test precision using vibratory sieve shakers has not been determined.

TABLE 2 Test Method Precision-Type 1: Attrition (%)

NOTE 1—
Sr = repeatability standard deviation, in measurement units.
r = repeatability, in measurement units.
(r) = repeatability, (relative) percent.
SR = reproducibility standard deviation, in measurement units.
R = reproducibility, in measurement units.
(R) = reproducibility, (relative) percent.

Materials	Mean Level %	Within Laboratories			Between Laboratories		
		Sr	*r*	*(r)*	*SR*	*R*	*(R)*
Material B	0.9	0.14	0.4	46.1	0.24	0.7	78.1
Material A	0.9	0.15	0.4	47.2	0.23	0.6	69.6
Material D	1.8	0.25	0.7	39.7	0.54	1.5	85.2
Material C	2.3	0.19	0.6	24.0	0.61	1.7	75.4
Pooled Values	1.5	0.19	0.5	36.7	0.44	1.2	84.8

11. Keywords

11.1 attrition; carbon black; fines; pellet quality