



Designation: **D 5817 – 03a**

Standard Practice for Carbon Black, Pelleted—Reduction, Blending, and Drying of Gross Samples for Testing¹

This standard is issued under the fixed designation D 5817; 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 practice describes the procedure for blending of pelleted carbon black, the procedure for the reduction of gross samples of pelleted carbon black to the appropriate size for testing and the preparation of the sample for testing. These techniques are intended to minimize variations in measured characteristics between test samples. Standard terminology relating to carbon black samples is found in Terminology D 3053.

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.*

¹ This practice is under the jurisdiction of ASTM Committee D24 on Carbon Black and is the direct responsibility of Subcommittee D24.61 on Carbon Black Sampling and Statistical Analysis.

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2. Referenced Documents

2.1 *ASTM Standards:*²

- D 412 Test Methods for Vulcanized Rubber and Thermoplastic Elastomers—Tension
- D 1506 Test Methods for Carbon Black—Ash Content
- D 1508 Test Method for Carbon Black, Pelleted Fines and Attrition
- D 1509 Test Methods for Carbon Black—Heating Loss
- D 1510 Test Method for Carbon Black—Iodine Adsorption Number
- D 1511 Test Method for Carbon Black—Pellet Size Distribution
- D 1512 Test Methods for Carbon Black—pH Value
- D 1513 Test Method for Carbon Black, Pelleted—Pour Density
- D 1514 Test Method for Carbon Black—Sieve Residue
- D 1618 Test Method for Carbon Black Extractables—Transmittance of Toluene Extract
- D 1619 Test Methods for Carbon Black—Sulfur Content
- D 1765 Classification System for Carbon Blacks Used in Rubber Products
- D 1799 Practice for Carbon Black—Sampling Packaged Shipments
- D 1900 Practice for Carbon Black—Sampling Bulk Shipments
- D 2414 Test Method for Carbon Black—Oil Absorption Number
- D 3053 Terminology Relating to Carbon Black
- D 3191 Test Methods for Carbon Black in SBR (Styrene-Butadiene Rubber)—Recipe and Evaluation Procedures
- D 3192 Test Methods for Carbon Black Evaluation in NR (Natural Rubber)
- D 3265 Test Method for Carbon Black—Tint Strength
- D 3493 Test Method for Carbon Black—Oil Absorption Number of Compressed Sample
- D 5230 Test Method for Carbon Black—Automated Individual Pellet Hardness
- D 6556 Test Method for Carbon Black—Total and External Surface Area by Nitrogen Adsorption

3. Summary of Test Methods

3.1 *Test Method A—Reduction of Sample Size:*

3.1.1 The gross bulk sample is poured through the riffle sample splitter. A portion of the sample is reintroduced into the splitter as many times as necessary to reduce the gross sample to the size desired for the intended test portion. The test portion may not be homogeneous.

3.2 *Test Method B—Blending of Carbon Black:*

3.2.1 The gross sample is poured through the riffle sample splitter a minimum of four times in a specific sequence to prepare a homogeneous test portion. The black must be cross-blended to accomplish a homogeneous portion for testing.

3.3 *Test Method C—Drying of Carbon Black:*

3.3.1 For all test methods listed in Section 2 except: the Fines and Attrition (Test Method D 1508), Heating Loss (Test Methods D 1509), Pellet Size Distribution (Test Method D 1511), pH Value (Test Methods D 1512), Pour Density (Test Method D 1513), Sieve Residue (Test Method D 1514), Sulfur Content (Test Methods D 1619), and Pellet Hardness (Test Method D 5230) test methods, the sample must be dried before testing begins.

NOTE 1—Samples that are known to be dry (< 1.0 % moisture) due to prior preparation or production processing may be tested without further drying.

4. Significance and Use

4.1 Several test methods for carbon black require the reduction in sample size or the blending of the test sample to reduce the variation of the measured characteristic. Other factors being equal, larger samples will tend to be more representative of the total supply. This practice provides procedures for reducing the large sample obtained from the bag and bulk sampling practices to a convenient size for conducting a number of tests to describe the material and measure its quality in such a manner that the smaller portion is most likely to be a representation of the bulk sample. Failure to follow the procedures in this practice could result in providing a nonrepresentative sample to be used in subsequent testing. Individual test methods provide for minimum amounts of material to be tested.

4.2 Test Method A, *Reduction of Sample Size*, is typically used to obtain an aliquot from a large sample, splitting a black into two or more equal portions, or reducing a blended black from Test Method B to a specified sample size. The final sample is always smaller than the original sample and would not be considered to be a “well blended” sample.

4.3 Test Method B, *Blending of Carbon Black*, is typically used to prepare a homogeneous sample. The homogeneous sample may be used for several tests (see Note-2) 3) or may be reduced in size by Test Method A. The final sample size is the same as the original.

4.4 When a sample is to be blended and reduced, the sample should be blended (Test Method B) before reduction (Test Method

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*, Vol 09.01, volume information, refer to the standard's Document Summary page on the ASTM website.

A). The blending and reduction must be done before the sample is dried (Test Method C).

5. Apparatus

5.1 *Riffle Sample Splitter*³—Riffle type sample splitters shall have an even number of equal width chutes, but not less than a total of twelve, that discharge alternately to each side of the splitter. The chutes shall be 1.3 cm (½ in.) or less in width. The splitter shall be equipped with two receptacles to hold the two halves of the sample following splitting. It shall also be equipped with a hopper or straight-edged pan that has a width equal to or slightly less than the overall width of the assembly of chutes, by which the sample may be fed at a controlled rate to the chutes. The splitter and accessory equipment shall be so designed that the sample will flow smoothly without restriction or loss of material (Fig. 1).

5.2 *Oven*, gravity-convection type, capable of temperature regulation of within ±1°C at 125°C and temperature uniformity within ±5°C.

5.3 *Desiccator*.

6. Sampling

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

7. Procedure

7.1 *Test Method A—Reduction in Sample Size* (Fig. 2):

7.1.1 Place two empty pans under the riffle sample splitter.

7.1.2 Pour the gross sample through the riffle sample splitter.

NOTE 1²—The gross sample is poured into the hopper and uniformly distributed from edge to edge, so that when it is introduced into the chutes, approximately equal amounts will flow through each chute. The rate at which the sample is introduced shall be such as to allow free flowing through the chutes into the receptacles below (Fig. 1).

7.1.3 Remove one of the pans from under the riffle sample splitter and replace with an empty pan. Pour the carbon black from the pan that was removed into the hopper.

7.1.4 Remove the pan from the same side as in 7.1.3 and replace with an empty pan. Repeat these steps as many times as necessary until the specified test portion size is obtained.

7.1.5 The entire test portion must be used if the original sample was not previously blended by Test Method B or is subsequently blended by Test Method B.

7.2 *Test Method B—Blending of Gross Sample* (Fig. 3):

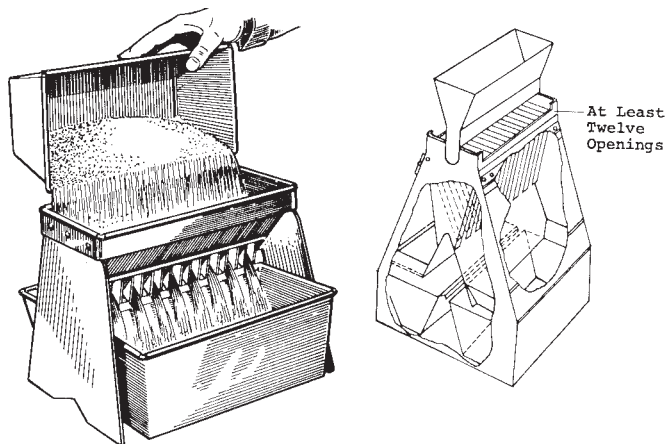
7.2.1 Place two empty pans under the riffle sample splitter.

7.2.2 Pour the gross sample through the riffle sample splitter (see Note 1)–(2).

7.2.3 Remove one of the pans from under the riffle sample splitter and replace with an empty pan. Pour the carbon black from the pan that was removed into the hopper.

7.2.4 Remove the pan from the opposite side of the riffle and replace with an empty pan and reintroduce the riffled black into the hopper. Repeat these steps until the carbon black has passed through the riffle sample splitter a minimum of four times.

³ Humboldt Cat H-3980 and Tyler Cat S.S. 50 have been found satisfactory. Humboldt Cat H-3980 available from Humboldt Manufacturing Co., 7300 W. Agatite Ave., Norridge, IL 60656. Tyler Cat S.S. 50 available from C-E Tyler Combustion Engineering, Inc., 8200 Tyler Blvd., Mentor, OH 44060.



NOTE 1—May be constructed as either closed or open type.

FIG. 1 Riffle Sample Splitter

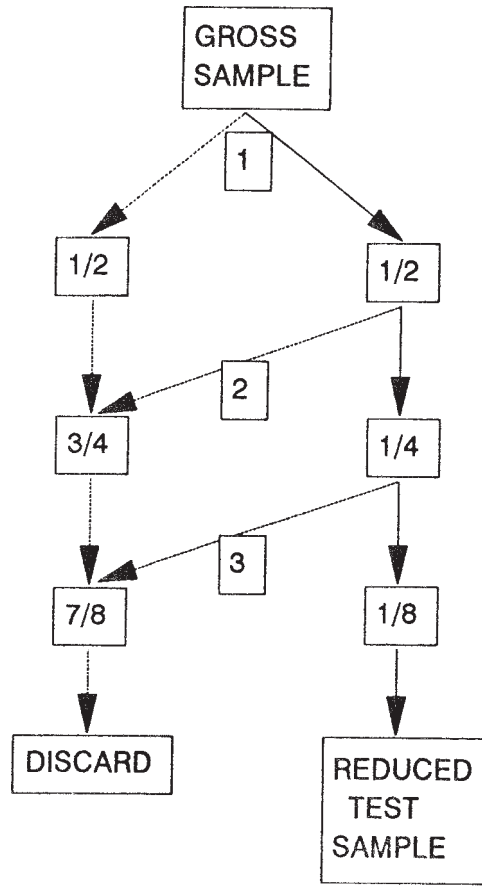


FIG. 2 Reduction of Sample Size

7.2.5 Combine the blended black from the two pans in a common container (see Note 2)-3).

NOTE 23—Pouring pelleted black tends to segregate the black by size of the pellets. Remove test samples in accordance with Test Method A or by dipping.

7.3 Test Method C—Drying of Carbon Black:

7.3.1 Dry an adequate sample of carbon black for 1 h, in a gravity-convection oven set at 125°C, in an open container of suitable dimensions, so that the depth of the black is no more than 10 mm. Cool to room temperature in a desiccator before use.

NOTE 34—Process samples taken under conditions where the material can reasonably be expected to dry (≤0.5 %) or known to be dry from previous testing or sample preparation do not need to be dried again before testing can proceed.

8. Keywords

8.1 carbon black; pelleted carbon black; reducing and blending carbon black

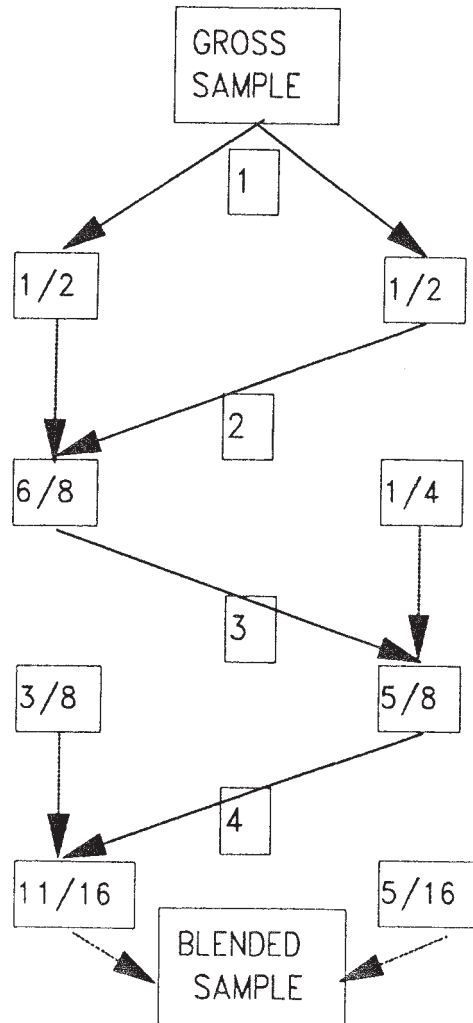


FIG. 3 Blending of Sample

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