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## Standard Practice for Carbon Black, Pelleted—Reduction, Blending, and Drying of Gross Samples for Testing<sup>1</sup>

This standard is issued under the fixed designation D5817; 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 ( $\epsilon$ ) 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 **D3053**. The classification system for carbon blacks used in rubber products can be found in Classification System **D1765**.

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, 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:*<sup>2</sup>

**D412** Test Methods for Vulcanized Rubber and Thermoplastic Elastomers—Tension  
**D1506** Test Methods for Carbon Black—Ash Content  
**D1508** Test Method for Carbon Black, Pelleted Fines and Attrition  
**D1509** Test Methods for Carbon Black—Heating Loss  
**D1510** Test Method for Carbon Black—Iodine Adsorption Number  
**D1511** Test Method for Carbon Black—Pellet Size Distribution  
**D1512** Test Methods for Carbon Black—pH Value  
**D1513** Test Method for Carbon Black, Pelleted—Pour Density  
**D1514** Test Method for Carbon Black—Sieve Residue  
**D1618** Test Method for Carbon Black Extractables—Transmittance of Toluene Extract  
**D1619** Test Methods for Carbon Black—Sulfur Content  
**D1765** Classification System for Carbon Blacks Used in Rubber Products  
**D1799** Practice for Carbon Black—Sampling Packaged Shipments  
**D1900** Practice for Carbon Black—Sampling Bulk Shipments  
**D2414** Test Method for Carbon Black—Oil Absorption Number (OAN)  
**D3053** Terminology Relating to Carbon Black  
**D3191** Test Methods for Carbon Black in SBR (Styrene-Butadiene Rubber)—Recipe and Evaluation Procedures  
**D3192** Test Methods for Carbon Black Evaluation in NR (Natural Rubber)  
**D3265** Test Method for Carbon Black—Tint Strength  
**D3493** Test Method for Carbon Black—Oil Absorption Number of Compressed Sample (COAN)

<sup>1</sup> 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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<sup>2</sup> For referenced ASTM standards, visit the ASTM website, [www.astm.org](http://www.astm.org), or contact ASTM Customer Service at [service@astm.org](mailto:service@astm.org). For Annual Book of ASTM Standards volume information, refer to the standard's Document Summary page on the ASTM website.

**D5230 Test Method for Carbon Black—Automated Individual Pellet Hardness**

**D6556 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 The sample must be dried before testing begins for these test methods: Vulcanized Rubber and Thermoplastic Elastomers—Tension (Test Methods **D412**), Ash (Test Methods **D1506**), Iodine Adsorption Number (Test Methods **D1510**), Carbon Black Extractables—Transmittance of Toluene Extract (Test Method **D1618**), Sulfur Content (Test Methods **D1619**), Oil Absorption Number (OAN) (Test Method **D2414**), Carbon Black in SBR (Styrene-Butadiene Rubber)—Recipe and Evaluation Procedures (Test Methods **D3191**), Carbon Black Evaluation in NR (Natural Rubber) (Test Methods **D3192**), Tint Strength (Test Method **D3265**), Oil Absorption Number of Compressed Sample (COAN) (Test Method **D3493**), and Total and External Surface Area by Nitrogen Adsorption (Test Method **D6556**).

3.3.2 For all test methods listed in Section 3, sample drying is not required for these ~~2~~ except: the test methods: Fines and Attrition (Test Method **D1508**), Heating Loss (Test Methods **D1509**), Pellet Size Distribution (Test Method **D1511**), pH Value (Test Methods **D1512**), Pour Density (Test Method **D1513**), Sieve Residue (Test Method **D1514**), Sulfur Content (Test Methods **D1619**), and Pellet Hardness (Test Method **D5230**) 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 32**) 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 A). The blending and reduction must be done before the sample is dried (Test Method C).

### 5. Apparatus

5.1 *Riffle Sample Splitter*<sup>3</sup>—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  $\pm 1^\circ\text{C}$  at  $125^\circ\text{C}$  and temperature uniformity within  $\pm 5^\circ\text{C}$ .

#### 5.3 Desiccator.

<sup>3</sup> 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.