



Designation: D 4122 – 02

## Standard Practice for Carbon Black—Evaluation of an Industry Reference Black<sup>1</sup>

This standard is issued under the fixed designation D 4122; 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 offers guidelines for the production and testing for uniformity of a lot of carbon black to be used as an Industry Reference Black (IRB).

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.*

### 2. Referenced Documents

#### 2.1 ASTM Standards:

- D 412 Test Methods for Vulcanized Rubber and Thermoplastic Elastomers—Tension<sup>2</sup>
- D 1506 Test Methods for Carbon Black—Ash Content<sup>2</sup>
- D 1508 Test Method for Carbon Black, Pelleted Fines and Attrition<sup>2</sup>
- D 1509 Test Methods for Carbon Black—Heating Loss<sup>2</sup>
- D 1510 Test Method for Carbon Black—Iodine Adsorption Number<sup>2</sup>
- D 1513 Test Method for Carbon Black, Pelleted—Pour Density<sup>2</sup>
- D 1514 Test Method for Carbon Black—Sieve Residue<sup>2</sup>
- D 1618 Test Method for Carbon Extractables—Transmittance of Toluene Extract<sup>2</sup>
- D 1765 Classification System for Carbon Blacks Used in Rubber Products<sup>2</sup>
- D 2414 Test Method for Carbon Black—Oil Absorption Number<sup>2</sup>
- D 3191 Test Methods for Carbon Black Evaluation in SBR (Styrene-Butadiene Rubber)—Recipe and Evaluation Procedures<sup>2</sup>
- D 3192 Test Methods for Carbon Black Evaluation in NR (Natural Rubber)<sup>2</sup>

- D 3265 Test Method for Carbon Black—Tint Strength<sup>2</sup>
- D 3493 Test Method for Carbon Black—Oil Absorption Number of Compressed Sample<sup>2</sup>
- D 5230 Test Method for Carbon Black—Automated Individual Pellet Hardness<sup>2</sup>
- D 6556 Test Methods for Carbon Black—Total and External Surface Area by Nitrogen Adsorption<sup>2</sup>

### 3. Significance and Use

3.1 These guidelines are intended to ensure that IRBs are evaluated by a standard procedure.

3.2 These guidelines are to be used to establish the average physicochemical and physical rubber properties of a lot of carbon black to be used as an IRB.

### 4. Production, Quality Control, and Quality Assurance

4.1 It is assumed that the manufacturer of the IRB will use state-of-the-art techniques to ensure maximum uniformity throughout the entire production run. The production should be made in one continuous production lot run. The testing called for in this practice is not intended to be a substitute for in-process quality control. This interlaboratory study is only adequate to verify the quality of a homogeneous lot.

4.2 The size of the lot is determined by historical records on the rate of use. The lot should have an expected life of 8 to 10 years at the most recent rate of use.

4.3 The black should be bagged in 50-lb polyethylene bags to reduce moisture incursion. Each pallet of bagged black should be wrapped in plastic to reduce environmental exposure. The bagged black will be segregated into at least twelve equal sized sublots for uniformity testing.

### 5. Sampling

5.1 After a suitable time to allow the black to stabilize, a bag will be selected from the approximate middle of each of the sublots; the bags selected will be numbered from one through  $n$ , where  $n$  is the total number of sublots, in order to represent the corresponding production lot.

5.2  $n$  4-dm<sup>3</sup> (1-gal) samples, numbered from one through  $n$ , and taken from the corresponding bags, will be sent to each participant in the interlaboratory study to evaluate the new IRB.

<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> Annual Book of ASTM Standards, Vol 09.01.

**TABLE 1 Industry Reference Black Test Data**

**Laboratory Number**

Day of Mixing and Date	Sample No.	Tensile Strength, MPa D 3191		Tensile Stress at 300 %, MPa D 3191	Elongation, % D 3191	Tensile Strength, MPa D 3192A		300 % Modulus, MPa D 3192A	Elongation, % D 3192A	Iodine No., g/kg D 1510	STSA 10 <sup>3</sup> m <sup>2</sup> /kg D 6556	NSA Multi-point Adsorption 10 <sup>3</sup> m <sup>2</sup> /kg D 6556	Oil Absorption, No., 10 <sup>-5</sup> mg <sup>3</sup> /kg (cm <sup>3</sup> /100 g) D 2414	Compressed Oil No., 10 <sup>-5</sup> mg <sup>3</sup> /kg (cm <sup>3</sup> /100 g) D 3493	Tint Strength, D 3265	Pour Density, kg/m <sup>3</sup> (lb/ft <sup>3</sup> ) D 1513	
Day No. ___		50'				30'											
Date ___	Prev. IRB	50'				30'											
Day No. ___		50'				30'											
Date ___	Prev. IRB	50'				30'											
Day No. ___		50'				30'											
Date ___	Prev. IRB	50'				30'											
Day No. ___		50'				30'											
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Date ___	Prev. IRB	50'				30'											
Day No. ___		50'				30'											
Date ___	Prev. IRB	50'				30'											
Day No. ___		50'				30'											
Date ___	Prev. IRB	50'				30'											

5.3 Additionally, a 4-dm<sup>3</sup> (1-gal) sample of the *previous* IRB taken from a common blended source will also be sent to each participant.

**6. Procedure**

6.1 Test, in order, one of the *n* samples on each of the one to *n* days. These days shall be as near to consecutive as possible.

6.2 Each day a sample is tested, subject it to all of the test methods described in 6.3, 6.4, and 6.5.

**6.3 Physicochemical Tests:**

6.3.1 Perform the following physicochemical tests on both the new and previous IRB:

6.3.1.1 *Iodine Adsorption Number (Test Method D 1510)*—Report the result obtained from an individual determination in grams of iodine per kilogram to the nearest 0.1 unit.

6.3.1.2 *Total and External NSA (Test Method D 6556)*—Report Total and External NSA from a single determination in 10<sup>3</sup> m<sup>2</sup>/kg (m<sup>2</sup>/g) to the nearest 0.1 unit.

6.3.1.3 *Oil Absorption Number (Test Method D 2414)*—Report the result obtained from an individual determination in 10<sup>-5</sup> m<sup>3</sup>/kg (cm<sup>3</sup>/100 g) to the nearest 0.1 unit.

6.3.1.4 *Oil Absorption Number of Compressed Sample (Test Method D 3493)*—Report the result obtained from an individual determination in 10<sup>-5</sup> m<sup>3</sup>/kg (cm<sup>3</sup>/100 g) to the nearest 0.1 unit.

6.3.1.5 *Tint Strength (Test Method D 3265)*—Report the result obtained from an individual determination in percent of ITRB to the nearest 0.1 unit.

6.3.1.6 *Pour Density (Test Method D 1513)*—Report the result obtained from an individual determination in kg/m<sup>3</sup> (lb/ft<sup>3</sup>) to the nearest whole unit.

6.3.2 Record data on Table 1.

**6.4 Rubber Physical Tests:**

6.4.1 Perform the following physical tests in rubber on both the new and previous IRB. Test samples mixed in accordance with Test Methods D 3191 and cure for 50 min at 145°C as well as samples mixed in accordance with Test Methods D 3192, Test Method A, and cure for 30 min at 145°C.

6.4.1.1 In accordance with Test Methods D 412, Test Method A, test five dumbbells from each cured sheet and determine the median values of tensile stress at 300 % elongation, tensile strength, and ultimate elongation.

6.4.1.2 Record data in absolute numbers (not as differences from IRB) on Table 1, reporting tensile stress and tensile strength to the nearest 0.1 MPa and ultimate elongation to the nearest 5 %.

**6.5 Informational Physicochemical Tests:**

6.5.1 Perform the following physicochemical tests on the new IRB:

6.5.1.1 *Ash Content (Test Method D 1506)*—Report results obtained from a single determination to the nearest 0.01 %.

6.5.1.2 *Fines and Attrition (Test Method D 1508)*—Report results obtained from a single determination to the nearest 0.1 %.

6.5.1.3 *Heating Loss (Test Method D 1509)*—Report results obtained from a single determination to the nearest 0.1 %.