



Designation: D4122 – 06

Standard Practice for Carbon Black—Evaluation of an Industry Reference Black¹

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1. Scope

1.1 This practice covers 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:²

- 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
- 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
- D1765 Classification System for Carbon Blacks Used in Rubber Products
- D2414 Test Method for Carbon Black—Oil Absorption Number (OAN)
- D3182 Practice for Rubber—Materials, Equipment, and

Procedures for Mixing Standard Compounds and Preparing Standard Vulcanized Sheets

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)

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. 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

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

n , where n is the total number of sublots, in order to represent the corresponding production lot.

5.2 n 4-dm³ (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.

5.3 Additionally, a 4-dm³ (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 6.3-6.5.

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 D1510)*—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 D6556)*—Report total and external NSA from a single determination in 10³ m²/kg (m²/g) to the nearest 0.1 unit.

6.3.1.3 *Oil Absorption Number (Test Method D2414)*—Report the result obtained from an individual determination in 10⁻⁵ m³kg (cm³/100 g) to the nearest 0.1 unit.

6.3.1.4 *Oil Absorption Number of Compressed Sample (Test Method D3493)*—Report the result obtained from an individual determination in 10⁻⁵ m³kg (cm³/100 g) to the nearest 0.1 unit.

6.3.1.5 *Tint Strength (Test Method D3265)*—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 D1513)*—Report the result obtained from an individual determination in kg/m³ (lb/ft³) 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 D3191 and cure for 50 min at 145°C as well as samples mixed in accordance with Test Methods D3192, Test Method A, and cure for 30 min at 145°C.

6.4.1.1 In accordance with Test Methods D412, 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 Methods D1506)*—Report results obtained from a single determination to the nearest 0.01 %.

TABLE 1 Industry Reference Black Test Data

Laboratory Number		Tensile Strength, MPa D3191	Tensile Stress at 300 %, MPa D3191	Elongation, % D3191	Tensile Strength, MPa D3192	300 % Modulus, MPa D3192	Elongation, % D3192	Iodine No., g/kg D1510	STSA 10 ³ m ² /kg D6556	NSA Multi-point Adsorption 10 ³ m ² /kg D6556	Oil Absorption, No., 10 ⁻⁵ mg ³ /kg (cm ³ /100 g) D2414	Compressed Oil No., 10 ⁻⁵ mg ³ /kg (cm ³ /100 g) D3493	Tint Strength D3265	Pour Density, kg/m ³ (lb/ft ³) D1513
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'									
Date ___	Prev. IRB	50'			30'									
Day No. ___		50'			30'									
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Day No. ___		50'			30'									
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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'									
Date ___	Prev. IRB	50'			30'									