



Standard Practice for Carbon Black—Evaluation of Standard Reference Blacks¹

This standard is issued under the fixed designation D 6915; 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 covers guidelines for the production and testing for uniformity of a set of carbon blacks to be used as Standard Reference Blacks (SRBs).

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

D 1510 Test Method for Carbon Black—Iodine Adsorption Number

D 2414 Test Method for Carbon Black—Oil Absorption Number (OAN)

D 3265 Test Method for Carbon Black—Tint Strength

D 3493 Test Method for Carbon Black—Oil Absorption Number of Compressed Sample (COAN)

D 4483 Practice for Evaluating Precision for Test Method Standards in the Rubber and Carbon Black Manufacturing Industries

D 4821 Guide for Carbon Black—Validation of Test Method Precision and Bias

D 6556 Test Method for Carbon Black—Total and External Surface Area by Nitrogen Adsorption

3. Significance and Use

3.1 This practice is intended to ensure that SRBs are produced and evaluated by a standard procedure.

3.2 This practice is to be used to establish the average physicochemical properties of a set of carbon blacks to be used as SRBs.

3.3 The carbon black grades to be used as SRBs should be selected to give as much coverage of the typical usage range for each test and as nearly evenly spaced across the range as possible. Typically, the carbon black grades selected consist of three tread (hard) type furnace grades (designated A, B, and C), three carcass (soft) type furnace grades (designated D, E, and F), and one thermal type grade (designated G). Subcommittee D24.61 may elect to carry one or more of the existing SRBs into the next set provided there is enough remaining material at the rate of usage to last through the expected life of the next set. Limiting the choice of grades to be used means that not all tests will have an SRB set that is evenly spaced across the range of interest. All the SRB candidates are produced at approximately the same time by the various producers. They are used as a set once they are approved. The sets are consecutively numbered. Values and identification for the current set are given in Guide **D 4821**. Any SRBs carried forward will be renumbered for the new set.

4. Production, Quality Control, and Quality Assurance

4.1 It is assumed that manufacturers of the SRBs 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 and packaging 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 for each SRB is determined by historical records on the rate of use. The lot should have an expected life of about five years at the most recent rate of use. Historically, the lot size has usually been 10 000 lb.

5. Uniformity Sampling and Testing

5.1 It is the producer's responsibility to ensure the SRB is produced under stable conditions using good statistical process control techniques.

5.2 The producer shall maintain all test equipment in proper calibration and be able to supply proof of calibration records, if requested.

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.

Current edition approved May 1, 2007. Published June 2007. Originally approved in 2003. Last previous edition approved in 2005 as D 6915 – 05b.

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.

5.3 The producer of the SRB shall be responsible for performing all testing to demonstrate lot uniformity, and the producer must submit the uniformity data to subcommittee D24.61. The tests to be performed as a minimum are those listed in Section 6. All test results must be ± 2 repeatability standard deviations as determined for each test using any one of the following means of determining S_r :

(1) Any appropriate SRB material's S_r listed in the precision table for a given test.

(2) The average pooled value S_r listed in the precision table for a given test.

(3) Determine a test's S_r using a linear regression from existing precision data for a given test as defined in **Annex A1**.

5.4 The samples for determining lot uniformity shall be taken at the same time as when the SRB is packaged. The number of samples needed to demonstrate uniformity shall be determined by subcommittee D24.61 prior to the production of the SRB(s).

5.5 The number of samples taken and the quantity collected for each sample shall be adequate to perform all uniformity testing and have sufficient material, such that when all sample material is combined and blended, there will be enough material that all the laboratories participating in the determination of the mean and control limit testing can perform the test protocol and have material left if additional testing is subsequently needed.

6. Procedure

6.1 Each laboratory will use the current SRBs to verify that all test methods are in calibration.

6.2 Each sample is tested once on each of two days by two different technicians (total of four test results).

6.3 Physicochemical Tests:

6.3.1 Perform the following physicochemical tests on the new SRB (this does not apply to the testing of the Heat Treated (HT) SRBs):

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 *Multipoint B.E.T. NSA (Test Method D 6556)*—Report a single determination in $10^3 \text{ m}^2/\text{kg}$ (m^2/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 $10^{-5} \text{ m}^3/\text{kg}$ ($\text{cm}^3/100 \text{ g}$) to the nearest 0.1 unit.

NOTE 1—Each participating laboratory must ensure that the absorptometer has been properly calibrated. Reported OAN values must be regressed to the previous SRB set.

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

NOTE 2—Each participating laboratory must ensure that the absorptometer has been properly calibrated. Reported COAN values must be regressed to the previous SRB set.

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 *Statistical Thickness Surface Area (STSA) (Test Method D 6556)*—Report single determination in $10^3 \text{ m}^2/\text{kg}$ (m^2/g) to the nearest 0.1 unit.

6.4 For the HT SRBs, perform the Iodine Adsorption Number Test (Test Method **D 1510**). Report the result obtained from an individual determination in grams of iodine per kilogram to the nearest 0.1 unit.

7. Statistical Analysis

7.1 The uniformity sample material collected by the producer shall be blended, apportioned, and sent to each participant in the interlaboratory study to evaluate the new SRB.

7.2 To achieve statistical validity and allow for the removal of outliers, at least 20 laboratories should participate in the evaluation for each test method.

7.3 Each participating laboratory shall maintain all test equipment in proper calibration and be able to supply proof of calibration and calibration records, if requested.

7.4 The interlaboratory study protocol shall be determined by subcommittee D24.61 prior to the production of the SRB(s).

7.5 The mean and control limit values of the SRBs for each test shall be determined using the methodology of Practice **D 4483**, including the removal of outlier values.

7.6 When calibration data is collected along with the test results for a new SRB and an analysis of the calibration data shows that any given laboratory is not in a state of calibration, the data from that laboratory may be removed from the data set even though the data is not flagged as being an outlier by the techniques of Practice **D 4483**.

7.7 After eliminating the outliers and any laboratories not in a state of calibration, the remaining data for each test method will be used to provide mean and control limit values for tabulation in Guide **D 4821**. This information may also be placed in each test method, if desired. The analysis could be used to develop new precision and bias values for each test method.

8. Acceptance

8.1 Subcommittee D24.61 may reject all or part of a candidate SRB lot as not uniform. All SRB lots tested as homogeneous by this practice will be considered acceptable by Committee D24 for use as a new SRB. The mean values and control limits will be published in Guide **D 4821**.

9. Shelf Life

9.1 The shelf life of the Standard Reference Black (SRB) carbon blacks is indefinite when properly stored in a manner that protects it from exposure to sources of moisture, such as precipitation, other sources of liquid water, or high humidity environments. Iodine number is the only property known to change over an extended period (years). This is due to a slow increase in the oxygen content and is primarily observed with tread blacks and other high surface area carbon blacks. Steps are being taken within ASTM Committee D24 to develop standards that are more stable over a long period.

10. Keywords

10.1 blending; lot size; physical properties; physicochemical properties; shelf life; standard reference blacks (SRBs)