



Designation: D6915 – 12

Standard Practice for Carbon Black—Evaluation of Standard Reference Blacks¹

This standard is issued under the fixed designation D6915; 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 reappraisal. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reappraisal.

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

D1510 Test Method for Carbon Black—Iodine Adsorption Number

D2414 Test Method for Carbon Black—Oil Absorption Number (OAN)

D3265 Test Method for Carbon Black—Tint Strength

D3493 Test Method for Carbon Black—Oil Absorption Number of Compressed Sample (COAN)

D4483 Practice for Evaluating Precision for Test Method Standards in the Rubber and Carbon Black Manufacturing Industries

D4821 Guide for Carbon Black—Validation of Test Method Precision and Bias

D6556 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.

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 June 1, 2012. Published August 2012. Originally approved in 2003. Last previous edition approved in 2007 as D6915 – 07. DOI: 10.1520/D6915-12.

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.

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 D4821. Any SRBs carried forward will be renumbered for the new set.

4. Production, Packaging, 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.

4.2.1 The actual usage rate of any reference material may result in one or more SRB material being depleted before the remaining materials in a given SRB set. When this happens, Subcommittee D24.61 may elect to have a supplemental lot produced for the needed SRB(s).

4.2.2 It is preferred that any supplemental SRB lots be produced at the same location, on the same production

equipment, under the same production conditions as reasonably possible, and using the same production targets and specifications as was used to produce the initial SRB lot in order to minimize variation between the two lots.

4.2.3 The supplemental lot size must be large enough to meet the expected life of the remaining materials in the SRB set and without a prohibitively large quantity remaining for disposal after the remainder of the set is depleted. Multiples of typical pallet quantities of 1134 kg (2500 lb) are preferred. The total quantity produced must be enough to meet the needed supplemental lot size and have sufficient material to flush the packaging equipment as described in 5.4.

4.2.4 The supplemental SRB lot shall carry the same reference designation as the SRB material it is replacing with the addition of another digit numbering the lot, beginning with the digit “2”. For example, a supplemental lot for the reference material SRB8A would be designated SRB8A2. If the conditions of 4.2.3 are met, there will not be a need for another supplemental lot of any given SRB material.

4.3 The SRB material should be bagged in 22.7 kg (50-lb) polyethylene bags to reduce moisture incursion. Each pallet should have a nominal 50 bags. Each pallet of bagged material should be wrapped in plastic to reduce environmental exposure.

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. Such control techniques include but are not limited to:

5.1.1 Stable statistical process control for at least 24 h prior to isolating the candidate SRB material.

5.1.2 Stable statistical process control during the entire period the candidate SRB material is being produced. If there is an occurrence of instability (process not in statistical control) while the candidate SRB material is being produced, the isolated material must be discarded and a new period of stability demonstrated per 5.1.1.

5.1.3 The storage vessel used to hold the candidate SRB material must be emptied and reasonably cleaned prior to being used to isolate the candidate SRB material. The emptying and cleaning is particularly important if the storage previously held a material of a different grade than the candidate SRB material.

5.2 The producer shall maintain all test equipment in proper calibration and be able to supply proof of calibration records, if requested. It is preferred that the testing laboratory for the production facility be an active participant in D24’s Laboratory Proficiency Rating System (LPRS) program. Active participation shall be demonstrated by submitting test results for at least the previous three LPRS samples.

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 packaging equipment used to package the SRB lot must be flushed to remove any possible contamination from other materials that may remain in the packaging system. This flushing shall be accomplished by packaging at least 10 000 kg (22 000 lb) of the SRB material before isolating the packages to be used for the SRB lot. This flushing is especially critical if the same packaging equipment is used to package other grades of carbon black.

5.5 The samples for determining lot uniformity shall be taken at the same time as when the SRB is packaged. The number of samples and sampling location needed to demonstrate uniformity shall be:

5.5.1 Typically a bag is removed from the packaging stream at the designated sample location without reducing the total bag count on the pallet (nominally 50) so that the total number of bags pulled is the sum of bags on the pallet and bags needed for samples. It may be necessary to remove more than one bag at each sampling event to provide a sufficient quantity for subsequent uniformity testing per 5.6.

5.5.2 If just one pallet of material is being packaged, take a sample at the beginning (bottom layer), middle, and end (top layer) of the pallet.

5.5.3 If more than one pallet of material is being packaged, take a sample at the beginning (bottom layer) and middle of each pallet and the end (top layer) of the last pallet.

5.6 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 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 *Multipoint B.E.T. NSA (Test Method D6556)*— Report a single determination in 10^3 m²/kg (m²/g) to the nearest 0.1 unit.