**Designation: D 6915 – 03a**

# Standard Practice for Carbon Black—Evaluation of Standard Reference Blacks[1]

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## 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:*

[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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D 1510 Test Method for Carbon Black—Iodine Adsorption Number[2]
D 1765 Classification System for Carbon Blacks Used in Rubber Products[2]
D 2414 Test Method for Carbon Black—Oil Absorption Number[2]
D 3265 Test Method for Carbon Black—Tint Strength[2]
D 3493 Test Method for Carbon Black—Oil Absorption Number of Compressed Sample[2]
D 4483 Practice for Determining Precision for Test Method Standards in the Rubber and Carbon Black Industries[2]
D 4821 Guide for Carbon Black—Validation of Test Method Precision and Bias[2]
D 6556 Test Method for Carbon Black—Total and External Surface Area by Nitrogen Adsorption[2]

## 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 5 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.

5.3 The producer of the SRB shall be responsible for performing all testing to demonstrate lot uniformity. 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. The producer must submit the uniformity data to subcommittee D24.61

5.4 The samples for determining lot uniformity shall be taken at the same time as when the SRB is packaged. ~~Knowing the lot size, the total~~ The number of ~~22.7-kg (50-lb) bags~~ samples needed to ~~prepare the lot can~~ demonstrate uniformity shall be ~~calculated. For example, for a 4540 kg (10 000 lb) lot this would be 211 bags. The samples are collected in addition~~ determined by subcommittee D24.61 prior to the ~~number needed for~~ production of the ~~lot. Eleven~~ SRB(s).

5.5 The number of samples ~~will be~~ taken ~~consisting of the fifth bag pulled,~~ and ~~one bag representing one-tenth of~~ the ~~lot from there, through the last bag. For example,~~ quantity collected for ~~200 bags the 5th, 25th, 45th, etc., through the 205th bag would~~ each sample shall be ~~set aside~~ adequate to ~~give 200 bags in the lot~~ perform all uniformity testing and ~~eleven bags as samples.~~

~~5.2 Each 22.7-kg (50-lb) bag's~~ have sufficient material such that when all sample material is combined and blended, there will be ~~blended, apportioned, and sent to each participant in~~ enough material that all the ~~interlaboratory study to evaluate the new SRB. To achieve statistical validity and allow for the removal of outliers, at least 20~~ laboratories ~~should participate~~ participating in the ~~evaluation. Each sample will be identified with~~ determination of the mean and control limit testing can perform the test protocol and have material left if additiong bagl testing is sumbserquently 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:

[2] *Annual Book of ASTM Standards*, Vol 09.01.

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 $10^3$ $m^2/kg$ ($m^2/g$) 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 $10^{-5}$ $m^3/kg$ ($cm^3/100$ g) to the nearest 0.1 unit.

NOTE 1—Each participating laboratory must insure 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 $10^{-5}$ $m^3/kg$ ($cm^3/100$ g) to the nearest 0.1 unit.

NOTE 2—Each participating laboratory must insure 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 Methods D 6556)*—Report single determination in $10^3$ $m^2/kg$ ($m^2/g$) to the nearest 0.1 unit.

## 7. Statistical Analysis

7.1 ~~U~~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.~~2~~6 After eliminating the outliers, the remaining data for each test method will be used to provide mean ~~values~~ 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. Keywords

9.1 blending; lot size; physical properties; physicochemical properties; standard reference blacks (SRBs)