Designation: ~~D6915 – 17ε1~~ D6915 – 23

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

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 reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

~~ε[1] NOTE—Corrected Fig. A1.2 editorially in August 2017.~~

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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.*

1.4 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

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
D8043 Guide for Carbon Black—Shelf Life

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.

[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.

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 m^2/kg (m^2/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} m^3/kg ($cm^3/100$ 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 D3493)*—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 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 D3265)*—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 D6556)*—Report single determination in 10^3 m^2/kg (m^2/g) to the nearest 0.1 unit.

6.4 For the HT SRBs, perform the Iodine Adsorption Number Test (Test Method D1510). 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 D4483, 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 D4483.

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 D4821. 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 D4821.

8.2 Annex A2 provides flowcharts showing the steps and process flow for the identification, production, selection, testing, approval, and documentation of the SRB materials.

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.

9.2 See Guide D8043 for information on the shelf life of commercial grades of carbon black.

10. Keywords

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

~~ANNEX~~ANNEXES

A1. DETERMINATION OF S_r USING LINEAR REGRESSION

A1.1 An appropriate S_r level can be determined using a regression analysis of mean level and S_r values found in the precision table for a given test. The regression may be performed using all six furnace SRBs, or, if more appropriate, using the three tread (hard) blacks or three carcass (soft) blacks as demonstrated in Fig. A1.1. The regression coefficients (slope and intercept) are then used to estimate an S_r for a material's mean level as shown in Fig. A1.2 and Table A1.1. The example is a tread or hard black with an STSA mean level of 160 m^2/g and an estimated S_r of 1.65 m^2/g.

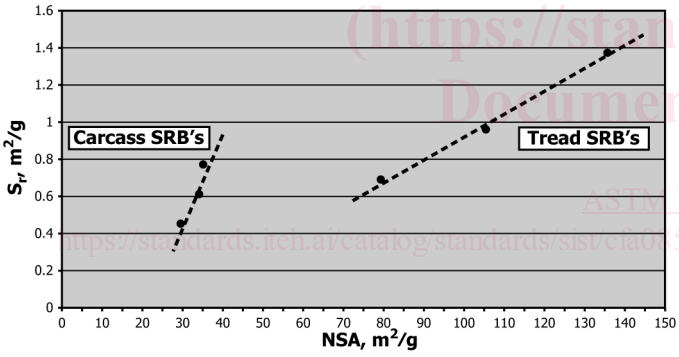

FIG. A1.1 SRB-6 Series STSA Repeatability Standard Deviation

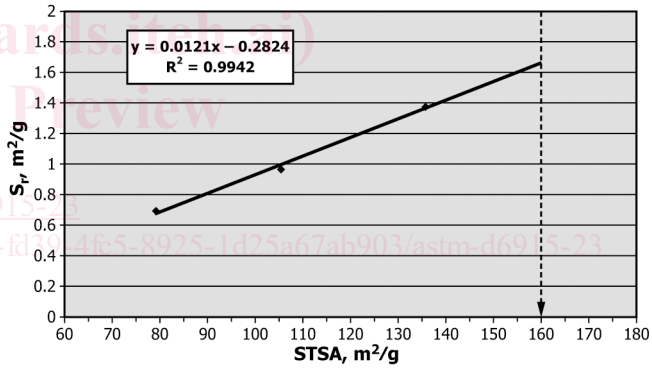

FIG. A1.2 Estimation of STSA S_r for Tread Product Using SRB-6

TABLE A1.1 Precision Data for SRB-6 Tread Standards

SRB	STSA Mean	S_r
A-6	135.7	1.37
B-6	105.4	0.96
C-6	79.2	0.69

Regression Coefficients[A]	
Slope	0.0121
Intercept	–0.2824

[A] Model: $S_r = 0.0121x - 0.2824$
Estimate for $x = 160$
$S_r = 1.65$

A2. SRB APPROVAL FLOWCHARTS

A2.1 See Figs. A2.1-A2.3.

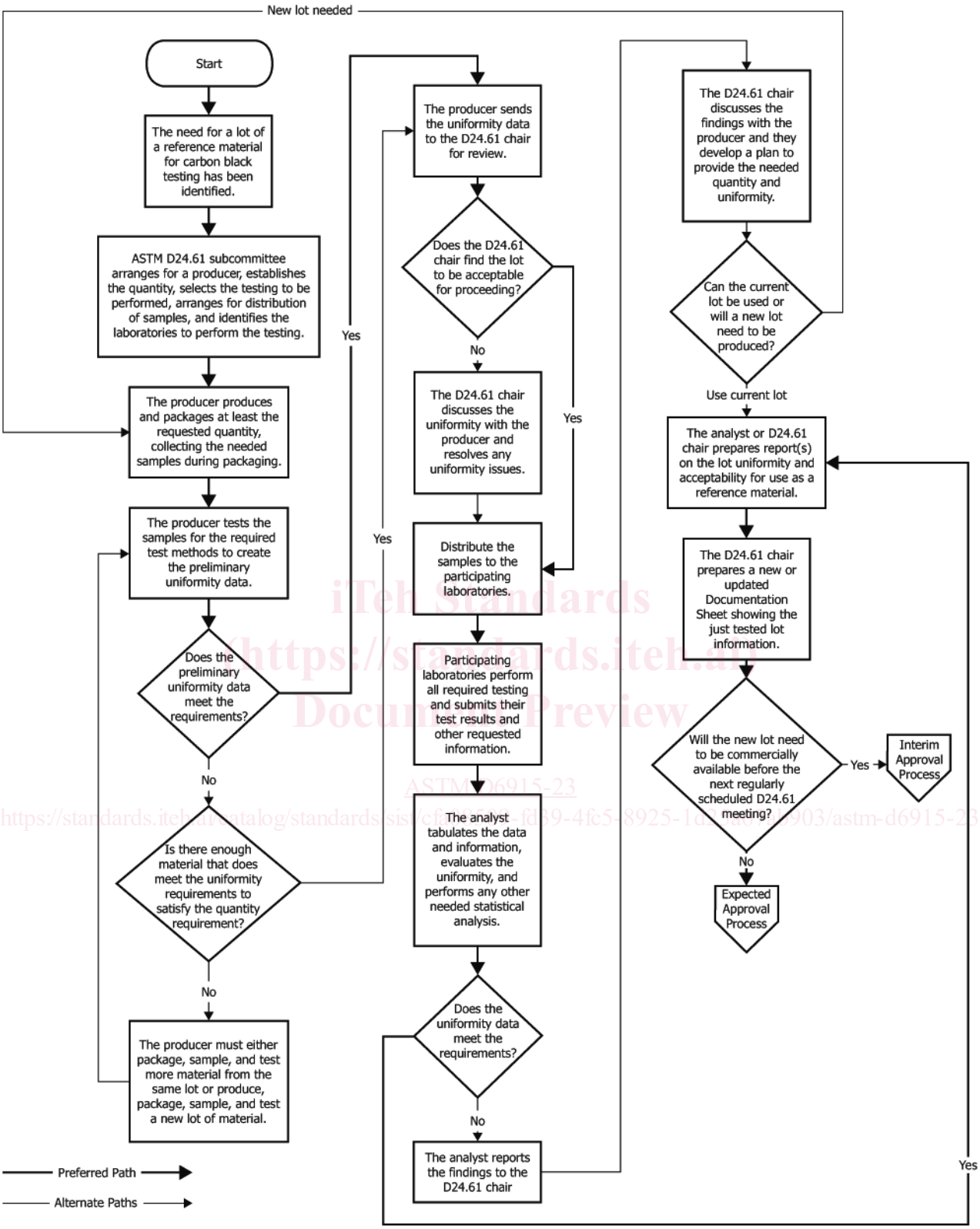

FIG. A2.1 SRB Approval Flowcharts Part 1