



Designation: D4821 – 20

Standard Guide for Carbon Black—Validation of Test Method Precision and Bias¹

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1. Scope

1.1 This guide covers procedures for using the ASTM Standard Reference Blacks² (SRBs) and the HT and INR Iodine Number Standards to continuously monitor the precision of those carbon black test methods for which reference values have been established. It also offers guidelines for troubleshooting various test methods.

1.2 This guide defines the environmental conditions that are required for laboratories that perform carbon black testing activities for those test methods under D24's jurisdiction.

1.3 This guide establishes procedures for the use of x-charts to continuously monitor those tests listed in Section 2 for within-lab precision (repeatability) and between-lab accuracy (reproducibility).

1.4 This guide provides a statistical procedure for improving test reproducibility when a laboratory cannot physically calibrate its apparatus to obtain the reference values of the ASTM reference blacks, within the ranges given in this guide.

1.5 *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:*³

[D1510 Test Method for Carbon Black—Iodine Adsorption Number](#)

¹ This guide 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 Standard Reference Blacks are available from Laboratory Standards & Technologies, Inc., 227 Somerset St., Borger, TX 79007.

³ 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.

- [D1513 Test Method for Carbon Black, Pelleted—Pour Density](#)
- [D1765 Classification System for Carbon Blacks Used in Rubber Products](#)
- [D2414 Test Method for Carbon Black—Oil Absorption Number \(OAN\)](#)
- [D3265 Test Method for Carbon Black—Tint Strength](#)
- [D3324 Practice for Carbon Black—Improving Test Reproducibility Using ASTM Standard Reference Blacks \(Withdrawn 2002\)⁴](#)
- [D3493 Test Method for Carbon Black—Oil Absorption Number of Compressed Sample \(COAN\)](#)
- [D6556 Test Method for Carbon Black—Total and External Surface Area by Nitrogen Adsorption](#)
- [D7854 Test Method for Carbon Black-Void Volume at Mean Pressure](#)
- [E177 Practice for Use of the Terms Precision and Bias in ASTM Test Methods](#)
- [E2282 Guide for Defining the Test Result of a Test Method](#)
- [E2586 Practice for Calculating and Using Basic Statistics](#)

3. Terminology

3.1 *Definitions:*

3.1.1 *accepted reference value, n*—a value that serves as an agreed-upon reference for comparison, and which is derived as: (1) a theoretical or established value, based on scientific principles, (2) an assigned or certified value, based on experimental work of some national or international organization, or (3) a consensus or certified value, based on collaborative experimental work under the auspices of a scientific or engineering group.

3.1.1.1 *Discussion*—A national or international organization, referred to in (2), generally maintains measurement standards to which the reference values obtained are traceable. **E177**

3.1.2 *accuracy, n*—the closeness of agreement between a test result and an accepted reference value.

3.1.2.1 *Discussion*—The term accuracy, when applied to a set of test results, involves a combination of a random

⁴ The last approved version of this historical standard is referenced on www.astm.org.

component and of a common systematic error or bias component. **E177**

3.1.3 *ASTM reference blacks, n*—a set of carbon blacks that span the useful range of the test method for which they are reference materials. **D3324**

3.1.4 *bias, n*—the difference between the expectation of the test results and an accepted reference value.

3.1.4.1 *Discussion*—Bias is the total systematic error as contrasted to random error. There may be one or more systematic error components contributing to the bias. A larger systematic difference from the accepted reference value is reflected by a larger bias value. **E177**

3.1.5 *characteristic, n*—a property of items in a sample or population which, when measured, counted or otherwise observed, helps to distinguish between the items. **E2282**

3.1.6 *coefficient of variation, CV, n*—for a nonnegative characteristic, the ratio of the standard deviation to the mean for a population or sample. **E2586**

3.1.7 *intermediate precision, n*—the closeness of agreement between test results obtained under specified intermediate precision conditions.

3.1.7.1 *Discussion*—The specific measure and the specific conditions must be specified for each intermediate measure of precision; thus, “standard deviation of test results among operators in a laboratory,” or “day-to-day standard deviation within a laboratory for the same operator.”

3.1.7.2 *Discussion*—Because the training of operators, the agreement of different pieces of equipment in the same laboratory and the variation of environmental conditions with longer time intervals all depend on the degree of within-laboratory control, the intermediate measures of precision are likely to vary appreciably from laboratory to laboratory. Thus, intermediate precisions may be more characteristic of individual laboratories than of the test method. **E177**

3.1.8 *intermediate precision conditions, n*—conditions under which test results are obtained with the same test method using test units or test specimens taken at random from a single quantity of material that is as nearly homogeneous as possible, and with changing conditions such as operator, measuring equipment, location within the laboratory, and time. **E177**

3.1.9 *measured value, n*—an observed test results as opposed to a standard value. **D3324**

3.1.10 *normalization, n*—the practice of applying a statistical correction to test measurements to improve accuracy.

3.1.10.1 *Discussion*—The correction of test data using a straight-line equation (linear regression) where measurements of ASTM reference blacks are analyzed with published accepted reference values to determine a slope and y-intercept. Normalization is a proven technique to improve the accuracy or reproducibility of laboratory data when all other means of calibration do not satisfactorily achieve a desired state of calibration.

3.1.11 *observation, n*—the process of obtaining information regarding the presence or absence of an attribute of a test specimen, or of making a reading on a characteristic or dimension of a test specimen. **E2282**

3.1.12 *observed value, n*—the value obtained by making an observation. **E2282**

3.1.13 *precision, n*—the closeness of agreement between independent test results obtained under stipulated conditions.

3.1.13.1 *Discussion*—Precision depends on random errors and does not relate to the accepted reference value.

3.1.13.2 *Discussion*—The measure of precision usually is expressed in terms of imprecision and computed as a standard deviation of the test results. Less precision is reflected by a larger standard deviation.

3.1.13.3 *Discussion*—“Independent test results” means results obtained in a manner not influenced by any previous result on the same or similar test object. Quantitative measures of precision depend critically on the stipulated conditions. Repeatability and reproducibility conditions are particular sets of extreme stipulated conditions. **E177**

3.1.14 *regression of standard values on measured values, n*—statistical equation derived by the method of least-squares. **D3324**

3.1.15 *repeatability, n*—precision under repeatability conditions.

3.1.15.1 *Discussion*—Repeatability is one of the concepts or categories of the precision of a test method.

3.1.15.2 *Discussion*—Measures of repeatability defined in this compilation are repeatability standard deviation and repeatability limit. **E177**

3.1.16 *repeatability conditions, n*—conditions where independent test results are obtained with the same method on identical test items in the same laboratory by the same operator using the same equipment within short intervals of time.

3.1.16.1 *Discussion*—See precision, the “same operator, same equipment” requirement means that for a particular step in the measurement process, the same combination of operator and equipment is used for every test result. Thus, one operator may prepare the test specimens, a second measure the dimensions and a third measure the mass in a test method for determining density.

3.1.16.2 *Discussion*—By “in the shortest practical period of time” is meant that the test results, at least for one material, are obtained in a time period not less than in normal testing and not so long as to permit significant change in test material, equipment or environment. **E177**

3.1.17 *repeatability limit (r), n*—the value below which the absolute difference between two individual test results obtained under repeatability conditions may be expected to occur with a probability of approximately 0.95 (95 %).

3.1.17.1 *Discussion*—The repeatability limit is times the repeatability standard deviation. This multiplier is independent of the size of the interlaboratory study.

3.1.17.2 *Discussion*—The approximation to 0.95 is reasonably good (say 0.90 to 0.98) when many laboratories (30 or more) are involved, but is likely to be poor when fewer than eight laboratories are studied. **E177**

3.1.18 *repeatability standard deviation (sr), n*—the standard deviation of test results obtained under repeatability conditions.

3.1.18.1 *Discussion*—It is a measure of the dispersion of the distribution of test results under repeatability conditions.

3.1.18.2 *Discussion*—Similarly, “repeatability variance” and “repeatability coefficient of variation” could be defined and used as measures of the dispersion of test results under repeatability conditions.—In an interlaboratory study, this is the pooled standard deviation of test results obtained under repeatability conditions.

3.1.18.3 *Discussion*—The repeatability standard deviation, usually considered a property of the test method, will generally be smaller than the within-laboratory standard deviation. (See **within-laboratory standard deviation**.) **E177**

3.1.19 *reproducibility, n*—precision under reproducibility conditions. **E177**

3.1.20 *reproducibility conditions, n*—conditions where test results are obtained with the same method on identical test items in different laboratories with different operators using different equipment.

3.1.20.1 *Discussion*—Identical material means either the same test units or test specimens are tested by all the laboratories as for a nondestructive test or test units or test specimens are taken at random from a single quantity of material that is as nearly homogeneous as possible. A different laboratory of necessity means a different operator, different equipment, and different location and under different supervisory control. **E177**

3.1.21 *reproducibility limit (R), n*—the value below which the absolute difference between two test results obtained under reproducibility conditions may be expected to occur with a probability of approximately 0.95 (95 %).

3.1.21.1 *Discussion*—The reproducibility limit is times the reproducibility standard deviation. The multiplier is independent of the size of the interlaboratory study (that is, of the number of laboratories participating).

3.1.21.2 *Discussion*—The approximation to 0.95 is reasonably good (say 0.90 to 0.98) when many laboratories (30 or more) are involved but is likely to be poor when fewer than eight laboratories are studied. **E177**

3.1.22 *reproducibility standard deviation (sR), n*—the standard deviation of test results obtained under reproducibility conditions.

3.1.22.1 *Discussion*—Other measures of the dispersion of test results obtained under reproducibility conditions are the “reproducibility variance” and the “reproducibility coefficient of variation.”

3.1.22.2 *Discussion*—The reproducibility standard deviation includes, in addition to between laboratory variability, the repeatability standard deviation and a contribution from the interaction of laboratory factors (that is, differences between operators, equipment and environments) with material factors (that is, the differences between properties of the materials other than that property of interest). **E177**

3.1.23 *standard deviation, n—of a population, σ* , the square root of the average or expected value of the squared deviation of a variable from its mean; *of a sample, s*, the square root of the sum of the squared deviations of the observed values in the sample divided by the sample size minus one. **E2586**

3.1.24 *standard value, n*—the value assigned to a reference black by ASTM Committee D24 on Carbon Black.

3.1.24.1 *Discussion*—Usually this value is calculated as the average test result of an interlaboratory testing program. **D3324**

3.1.25 *test determination, n*—the value of a characteristic or dimension of a single test specimen derived from one or more observed values. **E2282**

3.1.26 *test method, n*—a definitive procedure that produces a test result. **E2282**

3.1.27 *test result, n*—the value of a characteristic obtained by carrying out a specified test method. **E2282**

3.1.28 *test sample, n*—the total quantity of material (containing one or more test specimens) needed to obtain a test result as specified in the test method. See **test result**. **E2282**

3.1.29 *test specimen, n*—the portion of a test sample needed to obtain a single test determination. **E2282**

3.1.30 *trueness, n*—the closeness of agreement between the population mean of the measurements or test results and the accepted reference value.

3.1.30.1 *Discussion*—“Population mean” is, conceptually, the average value of an indefinitely large number of test results. **E177**

3.1.31 *variance, σ^2, s^2, n* —square of the standard deviation of the population or sample. **E2586**

3.1.32 *within-laboratory standard deviation, n*—the standard deviation of test results obtained within a laboratory for a single material under conditions that may include such elements as different operators, equipment, and longer time intervals.

3.1.32.1 *Discussion*—Because the training of operators, the agreement of different pieces of equipment in the same laboratory and the variation of environmental conditions with longer time intervals depend on the degree of within-laboratory control, the within-laboratory standard deviation is likely to vary appreciably from laboratory to laboratory. **E177**

4. Significance and Use

4.1 This guide provides insight into the environmental conditions required for operation of laboratories or to aid in the design of new laboratories that perform carbon black testing activities using those test methods that are under D24’s jurisdiction. This guide does not supersede any specific requirements a laboratory may choose to establish.

4.2 This guide recommends the use of statistical x-charts to graphically monitor test data determined for the ASTM reference blacks for those test methods given in Section 2. All laboratories are encouraged to utilize statistical x-charts and ASTM reference blacks because this enables a comparison of testing precision within and between laboratories. The guide describes practices for the use of repeatability and reproducibility limits and x-charts.

4.3 In addition to the calibration of a test method by physicochemical means, a statistical method for achieving calibration of a test method is presented (that is, *normalization*).

4.4 Poor test precision can be the result of poor repeatability or poor reproducibility or both. Causes may include inadequate operator training, improperly maintained equipment or laboratory environment, variation in sample preparation or analysis techniques, the lack of calibration or standardization of instrumentation, worn-out apparatus, reagents that do not meet specifications, different sources of instrumentation or equipment, and material heterogeneity. The sum of all sources of testing error is unique for an individual laboratory.

4.5 Precision data for ASTM Reference Blacks are found in **Tables 1-3**. These include standard reference blacks (SRB's) Series 8, HT and INR Iodine Standards. The HT or INR Iodine standards are recommended for monitoring iodine testing.

NOTE 1—Preferred precision values are bolded in **Tables 1-3**.

5. Laboratory Environmental Conditions

5.1 Test methods under the jurisdiction of D24 have no specific requirements for controlling laboratory temperature or relative humidity. The environmental condition in a laboratory is just one of many factors that can impact precision. The following recommendations are offered to help reduce the impact of environmental conditions on a laboratory's testing precision and accuracy.

5.2 It is recommended that laboratories operate in a stable and conditioned environment using standard heating, ventilation, and air conditioning (HVAC) systems since some of the testing requires volumetric measurements that are affected by temperature. A temperature range of 20 to 25°C is recommended since this is the range where most volumetric delivery apparatus and glassware are calibrated.

5.3 Reagents and laboratory equipment should be stored in the laboratory where the testing is being performed prior to use in order to allow proper temperature equilibration.

5.4 Use the standard reference blacks (SRB) and iodine number reference (INR) materials to monitor testing precision and accuracy. It can be assumed that for laboratories that consistently produce results within the requirements of Guide D4821, no further temperature and humidity control are required. For test results outside of these requirements, follow the troubleshooting guide described in Section 10.

6. Guide to Accepted Normalization Practices for Carbon Black Test Methods

6.1 Accepted normalization practices for test methods found in Classification **D1765**, Table 1 are described below.

6.1.1 *Test Method D1510, Iodine Number*—Test Method **D1510** contains instructions on how to perform a normalization of the test results. The HT or INR Iodine reference materials are recommended for monitoring iodine testing. The SRB HT and INR reference materials are specially prepared carbon blacks that have been shown to have stable iodine number values over a period of many years. If normalization is required, it shall be done using only the SRB HT or INR reference material values as given in **Table 2** and **Table 3**. Typically, this test method does not require normalization unless the HT or INR reference material values are not within the published precision or accuracy limits, or both. The

statistical correction described in Section 7 should not be used with the values in **Table 1A** or **Table 4A** due to the known phenomenon that the iodine number can decrease due to aging effects, most likely the result of slow oxygen chemisorption. See **Fig. 1** for an example of material aging.

6.1.2 *Test Method D1513, Pour Density*—Normalization is not possible because accepted reference values have not been established for this test method.

6.1.3 *Test Method D2414, Oil Absorption*—Normalizations are required using the tread and carcass SRBs as discussed in the test method.

6.1.4 *Test Method D3265, Tint Strength*—Normalization is required using the ITRB materials as discussed in the test method. Normalization with the SRBs must not be done (See Section 7).

6.1.5 *Test Method D3493, Oil Absorption of Compressed Sample*—Normalization is required using the tread or carcass SRBs as discussed in the test method.

6.1.6 *Test Method D6556, NSA and STSA*—Normalization to the SRB reference materials is not discussed in the test method. Normalization is allowed if the conditions in Section 7 are satisfied.

7. Procedure for Statistical Calibration or Normalization

7.1 As described in Section 6, Test Methods **D1510**, **D2414**, **D3265**, and **D3493** already contain instructions on how to perform a normalization for those test methods. Therefore, this section only applies to Test Method **D6556**, which does not contain instructions on how to perform normalization on the test results. This section should only be used when the conditions of Sections 8 or 9, or both, are satisfied for the Test Method **D6556** testing. This procedure is recommended only when all other recommended actions have failed to produce acceptable test values for Test Method **D6556**. This action should not be considered to be a substitute for following the applicable ASTM test method, maintaining recommended calibrations, or implementing appropriate corrective actions.

7.2 Test the ASTM Reference Blacks at least four (six preferred) times to establish good estimates of average measured values.

7.3 Calculate the slope and y-intercept of a straight-line equation or linear regression using the ASTM published accepted reference values and the four (or six) measured values.

7.4 Correct the measured (uncorrected) values of all subsequent testing by substituting each measured value into **Eq 1** and calculating the corrected value. A table of numbers may be generated to find the correspondence between a measured value and a corrected value.

7.5 Recalculate the statistical equation whenever replacement apparatus or a new lot of materials or reagents are put into use. Also, recheck it periodically to find changes due to wear or aging.

7.6 The form of the straight-line correction equation (linear regression) is shown in **Eq 1**:

$$\text{Straight - line or Linear Correction Equation: } Y = mx + B \quad (1)$$

TABLE 1 SRB-8 Precision by Test Method

 Table 1A Precision Parameters for Test Method **D1510**, Iodine Number Method A & B, (Type 1 Precision)

Units	g/kg						
Material	Mean Level ^A	Sr	r	(r)	SR	R	(R)
SRB-8B2	146.3	0.57	1.61	1.1	1.70	4.80	3.3
SRB-8C	138.8	0.68	1.92	1.4	2.11	5.96	4.3
SRB-8B	135.6	0.68	1.91	1.4	1.93	5.47	4.0
SRB-8A	80.5	0.36	1.03	1.3	0.88	2.49	3.1
SRB-8A2	78.1	0.88	2.49	3.2	1.33	3.78	4.8
SRB-8F	35.9	0.32	0.89	2.5	0.57	1.61	4.5
SRB-8E	35.8	0.32	0.91	2.5	0.60	1.71	4.8
SRB-8D	21.7	0.28	0.80	3.7	0.55	1.55	7.1
Average	84.1						
Pooled Values		0.48	1.35	1.6	1.32	3.72	4.4

 Table 1B Precision Parameters for Test Method **D6556**, NSA Method, (Type 1 Precision)

Units	10 ³ m ² /kg (m ² /g)						
Material	Mean Level	Sr	r	(r)	SR	R	(R)
SRB-8B	142.0	0.47	1.34	0.9	1.44	4.06	2.9
SRB-8B2	138.0	0.31	0.87	0.6	0.79	2.24	1.6
SRB-8C	126.4	0.44	1.25	1.0	1.07	3.02	2.4
SRB-8A	76.5	0.33	0.94	1.2	0.84	2.38	3.1
SRB-8A2	75.9	0.29	0.81	1.1	0.70	1.98	2.6
SRB-8E	36.7	0.23	0.65	1.8	0.53	1.49	4.1
SRB-8F	36.7	0.21	0.58	1.6	0.38	1.09	3.0
SRB-8D	21.6	0.18	0.52	2.4	0.30	0.85	3.9
Average	81.7						
Pooled Values		0.32	0.91	1.1	0.83	2.35	2.9

 Table 1C Precision Parameters for Test Method **D6556**, STSA Method, (Type 1 Precision)

Units	10 ³ m ² /kg (m ² /g)						
Material	Mean Level	Sr	r	(r)	SR	R	(R)
SRB-8B	133.1	0.71	2.00	1.5	1.39	3.92	2.9
SRB-8B2	126.7	0.56	1.58	1.2	2.02	5.73	4.5
SRB-8C	115.8	0.48	1.35	1.2	1.06	3.01	2.6
SRB-8A	77.2	0.41	1.17	1.5	1.15	3.26	4.2
SRB-8A2	76.0	0.47	1.32	1.7	1.23	3.49	4.6
SRB-8E	35.8	0.34	0.96	2.7	0.71	2.02	5.6
SRB-8F	35.4	0.33	0.93	2.6	0.69	1.95	5.5
SRB-8D	21.2	0.26	0.73	3.5	0.54	1.52	7.2
Average	77.6						
Pooled Values		0.46	1.31	1.7	1.19	3.36	4.3

 Table 1D Precision Parameters for Test Method **D2414**, OAN Method, (Type 1 Precision)

Units	10 ⁻⁵ m ³ /kg (cm ³ /100 g)						
Material	Mean Level	Sr	r	(r)	SR	R	(R)
SRB-8C	174.9	0.50	1.41	0.8	1.08	3.04	1.7
SRB-8B2	125.2	0.42	1.19	1.0	0.97	2.74	2.2
SRB-8B	123.5	0.45	1.26	1.0	0.91	2.57	2.1
SRB-8A2	71.5	0.46	1.31	1.8	1.56	4.42	6.2
SRB-8A	70.9	0.46	1.31	1.8	0.93	2.64	3.7
SRB-8F	132.0	0.41	1.16	0.9	0.91	2.59	2.0
SRB-8E	87.8	0.36	1.02	1.2	1.30	3.68	4.2
SRB-8D	36.9	0.26	0.73	1.9	1.09	3.09	8.1
Average	103.0						
Pooled Values		0.42	1.19	1.2	1.11	3.15	3.1

 Table 1E Precision Parameters for Test Method **D3493**, COAN Method, (Type 1 Precision)

Units	10 ⁻⁵ m ³ /kg (cm ³ /100 g)						
Material	Mean Level	Sr	r	(r)	SR	R	(R)
SRB-8C	130.6	0.54	1.52	1.2	1.47	4.17	3.2
SRB-8B2	103.1	0.50	1.42	1.4	1.03	2.92	2.8
SRB-8B	99.4	0.47	1.32	1.3	1.03	2.91	2.9
SRB-8A2	67.5	0.35	0.98	1.5	1.08	3.05	4.5
SRB-8A	66.7	0.42	1.20	1.8	0.87	2.46	3.7
SRB-8F	88.6	0.40	1.12	1.3	0.91	2.58	2.9
SRB-8E	74.7	0.36	1.01	1.3	0.99	2.82	3.8
SRB-8D	36.9	0.26	0.74	1.9	0.96	2.72	7.1
Average	83.7						
Pooled Values		0.42	1.19	1.4	1.06	2.99	3.6

^AThe iodine adsorption number of carbon black has been shown to decrease in value as the carbon black ages. Generally, the higher the surface area the faster the rate of change. Therefore, the target or mean values given in Table 1A may not be obtained due to this aging effect. Iodine Number testing should be monitored using HT or INR iodine standards and their reference values found in Table 2 and Table 3. See Section 10 for additional information.

TABLE 1 SRB-8 Precision by Test Method (continued)

Table 1F Precision Parameters for Test Method D3265 , Tint Strength Method, (Type 1 Precision)							
Units	Tint Strength						
Material	Mean Level	Sr	r	(r)	SR	R	(R)
SRB-8B2	132.1	0.65	1.83	1.4	1.86	5.26	4.0
SRB-8B	131.4	0.43	1.23	0.9	2.12	6.01	4.6
SRB-8C	112.0	0.46	1.29	1.2	1.10	3.11	2.8
SRB-8A2	111.0	0.49	1.39	1.2	1.15	3.25	2.9
SRB-8A	110.6	0.40	1.14	1.0	1.23	3.48	3.1
SRB-8E	61.8	0.30	0.84	1.4	0.95	2.69	4.4
SRB-8F	52.6	0.28	0.80	1.5	0.77	2.18	4.1
SRB-8D	42.5	0.26	0.73	1.7	0.73	2.08	4.9
Average	94.3						
Pooled Values		0.43	1.21	1.3	1.32	3.75	4.0

TABLE 2 HT Iodine Standards Precision

Table 2 Precision Parameters for Test Method D1510 , Iodine Number Methods A & B, (Type 1 Precision)							
Units	g/kg						
Material	Mean Level	Sr	r	(r)	SR	R	(R)
HT-1	43.7	0.24	0.68	1.50	0.49	1.38	3.20
HT-2	90.7	0.23	0.65	0.70	0.68	1.94	2.10
HT-3	126.6	0.23	0.64	0.50	0.61	1.73	1.40
Average							
Pooled Values	87.0	0.23	0.66	0.8	0.60	1.70	2.0

TABLE 3 INR Iodine Standards Precision

Table 3 Precision Parameters for Test Method D1510 , Iodine Number Methods A & B, (Type 1 Precision)							
Units	g/kg						
Material	Mean Level	Sr	r	(r)	SR	R	(R)
INR-A	41.5	0.31	0.88	2.1	1.19	3.37	8.1
INR-B	90.8	0.33	0.95	1.0	0.63	1.77	2.0
INR-C	125.8	0.31	0.87	0.7	1.00	2.84	2.3
Average							
Pooled Values	86.0	0.32	0.90	1.0	0.97	2.74	3.2

where:

Y = corrected value

m = slope

x = measured value

B = y-intercept

7.7 A statistical correction or normalization may be applied to any measured test results for test methods found in **Tables 1-3**. However, since Test Methods **D1510**, **D2414**, **D3265**, and **D3493** already contain normalization instructions akin to those in this section, these instructions **MUST** only be used for Test Method **D6556** test results. **NEVER** apply a second statistical correction to data which has already been normalized.

7.8 When it becomes necessary to use statistical calibration, all the SRBs for the range of interest (A-F or A-G) should be used to develop the correction equation, even if some of the SRBs fall outside the range of interest. At least four values (six preferred) should be used for each SRB. An equal number of results must be used for each SRB. If these instructions are not followed, the correction equation will not be comparable to the corrections made for other instruments and other laboratories.

7.9 Continuously monitor the selected SRBs and use the most current data to construct the correction equation.

7.10 Use the correction equation to obtain corrected (normalized) values for the SRBs and graphically monitor the

results with x-charts as described in Sections **8** and **9**. When a normalized test value exceeds the control limit, perform a retest immediately, and if the average of the two remains outside the control limit, then recalculate the statistical correction equation using the most recent measured values for each selected SRB.

7.11 If the statistical equation results in corrected values which still fall outside precision (see Section **8**) or accuracy (see Section **9**) limits, then no further testing should be performed until corrective action(s) are taken to bring the reference blacks inside the limits (see Section **10**).

7.12 This procedure prescribes only the minimum action needed to continuously monitor testing precision or accuracy. A higher testing frequency may be performed with ASTM reference blacks beyond what is described in Guide D4821. Correction equations may be regularly recalculated at individual discretion.

7.13 Discontinue using a statistical correction equation when new or repaired test equipment is put into service, a new lot of ASTM reference blacks are put into use, or other corrective actions yield uncorrected test results that fall within precision or accuracy control limits, or both. Resume using a statistical correction equation only when the conditions of Sections **8** or **9**, or both, are found.

TABLE 4 SRB-8 Between-Lab Accuracy Limits by Test Method

Table 4A Between-Lab Accuracy Limits for Test Method D1510 , Iodine Number Method A & B					
Units	g/kg				
Material	Mean Level ^A	SR	3SR	LCL	UCL
SRB-8B2	146.3	1.70	5.09	141.2	151.4
SRB-8C	138.8	2.11	6.32	132.5	145.2
SRB-8B	135.6	1.93	5.80	129.8	141.4
SRB-8A	80.5	0.88	2.64	77.9	83.2
SRB-8A2	78.1	1.33	4.00	74.1	82.1
SRB-8F	35.9	0.57	1.70	34.2	37.6
SRB-8E	35.8	0.60	1.81	34.0	37.6
SRB-8D	21.7	0.55	1.64	20.0	23.3

Table 4B Between-Lab Accuracy Limits for Test Method D6556 , Method NSA					
Units	10 ³ m ² /kg (m ² /g)				
Material	Mean Level	SR	3SR	LCL	UCL
SRB-8B	142.0	1.44	4.31	137.7	146.3
SRB-8B2	138.0	0.79	2.37	135.6	140.4
SRB-8C	126.4	1.07	3.20	123.2	129.6
SRB-8A	76.5	0.84	2.53	74.0	79.0
SRB-8A2	75.9	0.70	2.10	73.8	78.0
SRB-8E	36.7	0.53	1.58	35.1	38.3
SRB-8F	36.7	0.38	1.15	35.5	37.8
SRB-8D	21.6	0.30	0.90	20.7	22.5

Table 4C Between-Lab Accuracy Limits for Test Method D6556 , Method STSA					
Units	10 ³ m ² /kg (m ² /g)				
Material	Mean Level	SR	3SR	LCL	UCL
SRB-8B	133.1	1.39	4.16	128.9	137.2
SRB-8B2	126.7	2.02	6.07	120.7	132.8
SRB-8C	115.8	1.06	3.19	112.6	119.0
SRB-8A	77.2	1.15	3.45	73.8	80.7
SRB-8A2	76.0	1.23	3.70	72.3	79.7
SRB-8E	35.8	0.71	2.14	33.7	38.0
SRB-8F	35.4	0.69	2.06	33.3	37.5
SRB-8D	21.2	0.54	1.61	19.6	22.8

Table 4D Between-Lab Accuracy Limits for Test Method D2414 , Method OAN					
Units	10 ⁻⁵ m ³ /kg (cm ³ /100 g)				
Material	Mean Level ^B	SR	3SR	LCL	UCL
SRB-8C	174.9	1.08	3.23	171.7	178.1
SRB-8B2	125.2	0.97	2.90	122.2	128.1
SRB-8B	123.5	0.91	2.72	120.8	126.2
SRB-8A2	71.5	1.56	4.68	66.8	76.2
SRB-8A	70.9	0.93	2.79	68.1	73.7
SRB-8F	132.0	0.91	2.74	129.2	134.7
SRB-8E	87.8	1.30	3.90	83.9	91.7
SRB-8D	36.9	1.09	3.28	33.6	40.2

Table 4E Between-Lab Accuracy Limits for Test Method D3493 , Method COAN					
Units	10 ⁻⁵ m ³ /kg (cm ³ /100 g)				
Material	Mean Level ^B	SR	3SR	LCL	UCL
SRB-8C	130.6	1.47	4.42	126.2	135.1
SRB-8B2	103.1	1.03	3.09	100.1	106.2
SRB-8B	99.4	1.03	3.09	96.3	102.5
SRB-8A2	67.5	1.08	3.24	64.3	70.8
SRB-8A	66.7	0.87	2.61	64.1	69.3
SRB-8F	88.6	0.91	2.73	85.8	91.3
SRB-8E	74.7	0.99	2.98	71.8	77.7
SRB-8D	36.9	0.96	2.89	34.0	39.8

Table 4F Between-Lab Accuracy Limits for Test Method D3265 , Method Tint Strength					
Units	Tint Strength				
Material	Mean Level	SR	3SR	LCL	UCL
SRB-8B2	132.1	1.86	5.57	126.6	137.7
SRB-8B	131.4	2.12	6.37	125.0	137.7
SRB-8C	112.0	1.10	3.30	108.7	115.3
SRB-8A2	111.0	1.15	3.45	107.6	114.5
SRB-8A	110.6	1.23	3.69	107.0	114.3
SRB-8E	61.8	0.95	2.85	58.9	64.6
SRB-8F	52.6	0.77	2.31	50.3	54.9
SRB-8D	42.5	0.73	2.20	40.3	44.7

^AThe iodine adsorption number of carbon black has been shown to decrease in value as the carbon black ages. Generally, the higher the surface area the faster the rate of change. Therefore, the target or mean values given in Table 7A may not be obtained due to this aging effect. Iodine Number testing should be monitored using HT or INR iodine standards and their reference values found in Table 2 and Table 3. See Section 10 for additional information.

^BValues determined from absorptometers using DBP and various paraffinic oils. Results from using DBP are the majority, representing about 60 % of the data.