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

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1. Scope

1.1 This guide covers a procedure for using ASTM Standard Reference Blacks² (SRBs) to continuously monitor the precision of those carbon black test methods for which standard values have been established. It also offers guidelines for trouble-shooting various test methods.

1.2 This guide establishes the limits within which the statistical calibration procedure of Practice D 3324 shall be applied and establishes the limits for an out-of-calibration condition beyond which the statistical calibration procedure of Practice D 3324 shall not be applied.

1.3 This guide uses statistical control chart methodology as discussed in STP-15-D³ to determine if a laboratory's test results differ significantly from the accepted values of the SRBs and provides a format for reporting test data for specified tests on the SRB blacks.

1.4 The values stated in SI units are to be regarded as the standard. The values given in parentheses are for information only.

1.5 *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:

- D 1510 Test Method for Carbon Black—Iodine Adsorption Number⁴
- D 2414 Test Method for Carbon Black—Oil Absorption Number⁴
- D 3037 Test Methods for Carbon Black—Surface Area by

Nitrogen Adsorption⁵

- D 3265 Test Method for Carbon Black—Tint Strength⁴
- D 3324 Practice for Carbon Black—Improving Test Reproducibility Using ASTM Reference Blacks⁴
- D 3493 Test Method for Carbon Black—Oil Absorption Number of Compressed Sample⁴
- D 3765 Test Method for Carbon Black—CTAB (Cetyltrimethylammonium Bromide) Surface Area⁴
- D 4820 Test Methods for Carbon Black—Surface Area by Multipoint B. E. T. Nitrogen Adsorption⁶
- D 5816 Test Methods for Carbon Black—External Surface Area by Multipoint Nitrogen Adsorption⁶

3. Significance and Use

3.1 One of the major causes of poor test precision is the lack of calibration or standardization of instruments, apparatus, reagents, and technique among laboratories.

3.2 In addition to the calibration of a test method by physicochemical means, Practice D 3324 describes a statistical method for achieving calibration of a test method.

3.3 This guide outlines the use of control charts to graphically present calibration test data determined for the ASTM SRBs for those test methods given in Section 2. These control charts shall be used to validate the testing precision and bias of an individual laboratory.

4. Procedure

4.1 Select six SRBs from the SRB 4, SRB 5, or SRB 6 series to cover the range of interest, being sure to include one each of the A, B, C, D, E, and F (see Note 1) SRBs. Do not mix materials from different SRB sets. For example, do not use A, B, and C from set 4 with D, E, and F from set 5. This is especially critical for DBP absorptometer calibration. An absorptometer calibrated with F5 (or F5A) must be checked with other members of the 5 set. Likewise, an absorptometer calibrated with F6 must be checked with other members of the 6 set.

NOTE 1—SRB 4 and SRB 5 are depleted and not commercially available. However, they are still in use in some laboratories.

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. Phone/fax: (806) 273-3006. E-mail: jwbal@infinitytx.net.

³ *Symposium on Manual on Presentation of Data and Control Chart Analysis, ASTM STP 15D*, ASTM, 1976.

⁴ *Annual Book of ASTM Standards*, Vol 09.01.

⁵ Discontinued 1999. See 1998 *Annual Book of ASTM Standards*, Vol 09.01.

⁶ Discontinued 2000; replaced by Test Method D 6556; see 1999 *Annual Book of ASTM Standards*, Vol 09.01.



4.2 Prepare a control chart for each of the selected SRBs for each test method as presented by Part 3 of ASTM STP 15D.

4.3 The target values given in Table 1 for SRB 6 are the nominal SRB values reported in Practice D 3324. Values are used as control chart limits plus or minus three single test repeatability standard deviations. These values were determined during the validation of the SRB 6s. Comparable data for the SRB 4 set or SRB 5 sets may be obtained from ASTM International Headquarters by requesting research report RR: D24-1043 for SRB 4 data or RR: D24-1042 for SRB 5 data.

NOTE 2—SRB G5 may be used in conjunction with the SRB 6 series.

4.4 Plot the uncorrected values for the selected six SRBs until a repeatability control limit is exceeded (see Note 3). Perform a retest immediately. If the retest falls outside the repeatability control limits, then begin a search for an assignable cause. (See Section 5 for a list of assignable causes.) If the search for an assignable cause and the subsequent action taken still do not produce results within the control limits, then a statistical regression or correction equation shall be calculated as described in Practice D 3324.

NOTE 3—Selected SRBs from SRB 4 and SRB 5 may be plotted on separate charts. Do not plot SRB 4 and SRB 5, for example D-4 and D-5, on the same chart.

4.5 Continuously monitor the selected SRBs and use the most current six data for each selected SRB to construct the correction equation. Otherwise, the correction equation will not be comparable to the corrections made for other instruments and other laboratories.

4.6 Use the correction equation to obtain corrected values for the selected SRBs and plot the corrected values until the repeatability control limit is again exceeded. When a 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 six test data for each selected SRB.

4.7 If the corrected data using the new correction equation still fall outside the repeatability limits, then no further use should be made of the test method until corrective action brings the SRBs back inside the three standard deviation repeatability control chart limits.

TABLE 1 SRB 6 Control Chart Limits

Test Property	ASTM Standard	SRB	Target Value	3 s Value	Lower Control Limit	Upper Control Limit
Iodine adsorption number, ^A g/kg	D 1510	A 6 (N134)	137.2	3.00	134.20	140.20
		B 6 (N220)	117.9	2.28	115.62	120.18
		C 6 (N326)	82.4	1.08	81.32	83.48
		D 6 (N762)	26.5	1.26	25.24	27.76
		E 6 (N660)	35.3	1.62	33.68	36.92
		F 6 (N683)	33.1	1.44	31.66	34.54
Dibutyl phthalate (DBP) absorption number, 10 ⁻⁵ m ³ /kg (cm ³ /100 g)	D 2414	A 6 (N134)	123.7	1.83	121.87	125.53
		B 6 (N220)	114.3	1.11	113.19	115.41
		C 6 (N326)	70.3	1.05	69.25	71.35
		D 6 (N762)	67.4	1.50	65.90	68.90
		E 6 (N660)	88.2	1.80	86.40	90.00
		F 6 (N683)	133.6	3.33	130.27	136.93
		G 5 (N990)	36.2	0.75	35.45	36.95
DBP absorption number of compressed sample (CDBP), 10 ⁻⁵ m ³ /kg (cm ³ /100 g)	D 3493	A 6 (N134)	101.0	2.46	98.54	103.46
		B 6 (N220)	98.5	1.80	96.70	100.30
		C 6 (N326)	68.1	1.59	66.51	69.69
		D 6 (N762)	60.2	1.59	58.61	61.79
		E 6 (N660)	76.0	2.49	73.51	78.49
		F 6 (N683)	88.6	2.58	86.02	91.18
Surface area by multipoint B.E.T. nitrogen adsorption (NSA), 10 ³ m ² /kg (m ² /g)	D 4820	A 6 (N134)	143.9	2.10	141.80	146.00
		B 6 (N220)	110.0	1.59	108.41	111.59
		C 6 (N326)	78.3	1.20	77.10	79.50
		D 6 (N762)	30.6	0.75	29.85	31.35
		E 6 (N660)	36.0	1.20	34.80	37.20
		F 6 (N683)	35.3	1.41	33.89	36.71
		G 5 (N990)	9.1	0.36	8.74	9.46
Tint Strength	D 3265	A 6 (N134)	129.8	4.11	125.69	133.91
		B 6 (N220)	117.8	3.36	114.44	121.16
		C 6 (N326)	113.1	1.68	111.42	114.78
		D 6 (N762)	56.8	2.01	54.79	58.81
		E 6 (N660)	60.0	1.92	58.08	61.92
		F 6 (N683)	51.7	1.47	50.23	53.17
External surface area by multipoint nitrogen adsorption (STSA), 10 ³ m ² /kg (m ² /g)	D 5816	A 6 (N134)	135.7	4.11	131.59	139.81
		B 6 (N220)	105.4	2.88	102.52	108.28
		C 6 (N326)	79.2	2.07	77.13	81.27
		D 6 (N672)	29.6	1.35	28.25	30.95
		E 6 (N660)	35.1	2.31	32.79	37.41
		F 6 (N683)	34.1	1.83	32.27	35.93
		G 5 (N990)	8.4	0.60	7.80	9.00

^AThe iodine adsorption number of carbon black has been shown to decrease in value as the black ages. Generally, the higher the surface area the faster the rate of change. Therefore, the target values given in this table may not be obtained due to this aging effect. The most current standard value may be obtained by contacting the chairman of Subcommittee D24.61.