



Designation: D 3324 – 01a

Standard Practice for Carbon Black—Improving Test Reproducibility Using ASTM Standard Reference Blacks¹

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1. Scope

1.1 This practice provides a statistical procedure for improving test reproducibility when a laboratory cannot physically calibrate its apparatus to obtain the standard values of the ASTM reference blacks, within the ranges given in the precision statement of the test method concerned.

1.2 *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 Number²
- D 2414 Test Method for Carbon Black—Oil Absorption Number²
- D 3037 Test Methods for Carbon Black—Surface Area by Nitrogen Adsorption³
- D 3265 Test Method for Carbon Black—Tint Strength²
- D 3493 Test Method for Carbon Black—Oil Absorption Number of Compressed Sample²
- D 3765 Test Method for Carbon Black—CTAB (Cetyltrimethylammonium Bromide) Surface Area²
- D 4483 Practice for Determining Precision for Test Method Standards in the Rubber and Carbon Black Industries²
- D 4820 Test Methods for Carbon Black—Surface Area by Multipoint B.E.T. Nitrogen Adsorption⁴
- D 4821 Guide for Carbon Black—Validation of Test Method Precision and Bias²
- D 5816 Test Methods for Carbon Black—External Surface Area by Multipoint Nitrogen Adsorption⁴

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 *Annual Book of ASTM Standards*, Vol 09.01.

³ Discontinued 1999; see 1998 *Annual Book of ASTM Standards*, Vol 09.01.

⁴ Discontinued 2000; replaced by Test Method D 6556; see 1999 *Annual Book of ASTM Standards*, Vol 09.01.

3. Terminology

3.1 Definitions:

3.1.1 *ASTM reference blacks, n*—a set of six blacks that span the useful range of the test method for which they are standards.

3.1.2 *measured value, n*—an observed test result as opposed to a standard value.

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

3.1.4 *standard value, n*—the value assigned to a reference black by ASTM Committee D24 on Carbon Black. Usually this value is calculated as the average test result of an interlaboratory testing program.

4. Procedure

4.1 Physically calibrate the test apparatus using the instructions in the test method or the manufacturer's instructions.

4.2 Test the ASTM Standard Reference Blacks (SRB) a sufficient number of times to establish firm measured values. It is recommended that the measured values be control charted in accordance with Guide D 4281.

4.3 Calculate the regression of the standard values on the measured values. This relationship has usually been observed to be linear, although a curvilinear function might conceivably sometimes exist.

4.4 Correct the values measured on all subsequent samples by substituting each measured value into this equation and calculating the corrected value. Because it has been shown that the iodine adsorption number of carbon black decreases in value as the black ages, it is inappropriate to apply a statistical correction factor to the iodine adsorption number, Test Method D 1510.

4.5 Alternatively, a nomograph or a table of numbers may be used to find the correspondence between a measured value and a calibrated value.

4.6 Recheck this regression equation whenever replacement apparatus or a new lot of materials is put into use. Also, recheck it periodically to find changes due to wear or aging.

4.7 Each laboratory is responsible for establishing and maintaining the correlation between the ASTM reference blacks and their measured values.

5. ASTM D24 Standard Reference Blacks⁵

5.1 The target values and control chart limits for the SRB 6 series were determined in accordance with Practice D 4483–99.

5.1.1 Target and control chart limits data on the SRB 6 series, as determined during the validation of the SRB 6 series

in 2001, is given in Table 1.

5.1.2 SRB 4 is now depleted but may still be in use in some laboratories.⁶

5.1.3 SRB 5 is now depleted but may still be in use in some laboratories.⁶

6. Keywords

6.1 ASTM reference blacks; carbon black; carbon black standard values; carbon black test reproducibility

⁵ Standard Reference Blacks SRB 6, are available from Laboratory Standards and Technologies, Inc., 227 Somerset St., Borger, TX 79007. Phone/fax: (806) 273-3006. E-mail: jwbald@infinitytx.net.

⁶ Precision and control chart limits data is available from ASTM International Headquarters. Request RR: D24–1043 for SRB 4 data and D24–1042 for SRB 5 data.

TABLE 1 SRB 6 Control Chart Limits^A

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 (N762)	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.