Designation: D 3324 – 01

Standard Practice for Carbon Black—Improving Test Reproducibility Using ASTM Reference Blacks[1]

This standard is issued under the fixed designation D 3324; 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.

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 Number[2]

D 2414 Test Method for Carbon Black—Oil Absorption Number[2]

D 3037 Test Methods for Carbon Black—Surface Area by Nitrogen Adsorption[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 3765 Test Method for Carbon Black—CTAB (Cetyltrimethylammonium Bromide) Surface Area[2]

D 4483 Practice for Determining Precision for Test Method Standards in the Rubber and Carbon Black Industries[2]

D 4820 Test Methods for Carbon Black—Surface Area by Multipoint B.E.T. Nitrogen Adsorption[3]

D 5816 Test Methods for Carbon Black—External Surface Area by Multipoint Nitrogen Adsorption[3]

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 D-24 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 reference blacks a sufficient number of times to establish firm measured values.

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.

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 D-24 Standard Reference Blacks[4]

5.1 The standard values with their limits of uncertainty[5] for the SRB 4 series, and the repeatability precision (as defined in

[1] This practice is under the jurisdiction of ASTM Committee D24 on Carbon Blackand 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 2000; replaced by Test Method D 6556; see *1999 Annual Book of ASTM Standards*, Vol 09.01.

[4] Standard Reference Blacks SRB 4 and SRB 5, are available from Titan Specialties, P.O. Box 2316, Pampa, TX 79066–2316.

[5] The standard values and limits of uncertainty were determined in an interlaboratory study completed in January 1989. "The limits of uncertainty" are shown to indicate ranges within which replicate interlaboratory testing has been able to fix the concensus standard values; they are not to be confused with or interpreted as the repeatability precision shown for the respective test methods.

Practice D 4483), to be expected for each test method are given in Table 1.

5.1.1 Precision data on the SRB 5 series, determined during the validation of the SRB 5 series, is given in Table 2.

6. Keywords

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

TABLE 1 Precision Data[A,B]

Test Property	ASTM Standard	Precision—Single Test Result			Standard Reference Blacks					
		Repeatability			A-4 N326	B-4 N330	C-4 N121	D-4 N762	E-4 N660	F-4 N683
		Sr	*r*	(*r*)						
Iodine adsorption number, /kg	D 1510	0.38	1.08	1.69 %	82.7 (±0.57)[C]	79.3 (±0.65)[C]	121.1 (±1.01)[C]	26.5 (±1.03)[C]	36.0 (±0.38)[C]	37.7 (±0.70)[C]
Surface area by nitrogen adsorption,[D] 10^3 m^2/kg (m^2/g)	D 3037	0.36	1.02	1.62 %	77.3 (±0.53)	74.4 (±1.06)	124.2 (±1.09)	24.1 (±0.25)	34.6 (±0.54)	38.5 (±0.41)
CTAB surface area,[E] 10^3 m^2/kg (m^2/g)	D 3765	0.96[F]	2.72[F]	4.25 %[F]	82.5 (±2.31)[F]	79.1 (±2.03)[F]	120.5 (±2.71)[F]	24.9 (±1.74)[F]	36.6 (±1.13)[F]	40.5 (±1.01)[F]
Dibutyl phthalate absorption number, 10^{-5} m^3/kg (cm^3/100 g)	D 2414	0.53	1.51	1.54 %	70.8 (±0.63)	100.0 (±0.92)	131.0 (±0.97)	64.8 (±0.88)	90.4 (±1.49)	130.6 (±1.27)
Dibutyl phthalate absorption number of compressed sample, 10^{-5} m^3/kg (cm^3/100 g)	D 3493	0.62	1.74	2.15 %	68.3 (±1.30)	86.6 (±1.35)	108.9 (±1.09)	58.9 (±1.11)	75.7 (±1.19)	88.0 (±1.33)
Tint strength	D 3265	0.56[G]	1.57[G]	1.96 %[G]	110.8 (±1.17)[G]	98.5 (±1.35)[G]	119.5 (±1.48)[G]	41.2 (±0.80)[G]	55.2 (±0.68)[G]	55.8 (±0.96)[G]

[A] ( ) Two standard deviations − 95 % confidence. Target values were determined in 1989 analysis of 10 laboratories.
[B] Supporting data are available from ASTM Headquarters. Request RR: D24-1010 and D24-1011.
[C] The iodine adsorption number of carbon black has been shown to decrease in value as the black ages. The most current standard value may be obtained by contacting the chairman of Subcommittee D24.61.
[D] Single point nitrogen surface area (D 3037) targets determined by multipoint nitrogen surface area (D 4820).
[E] Target values based on eight laboratories.
[F] Automatic titration.
[G] Statistics are based on seven laboratories using the Densichron equipment/method and three laboratories using the Erichsen equipment/method.

TABLE 2 SRB 5 Control Chart Limits[A]

Test Property	ASTM Standard	SRB	Target Value	3 s Value	Lower Control Limit	Upper Control Limit
Iodine adsorption number, g/kg	D 1510	A 5	150.6	1.68	148.92	152.28
		B 5	77.7	0.96	76.74	78.66
		C 5	120.4	1.23	119.17	121.63
		D 5	28.7	1.62	27.08	30.32
		E 5	34.9	1.20	33.70	36.10
		F 5	38.5	1.80	36.70	40.30
		F 5A	34.6	1.80	32.80	36.40
Dibutyl phthalate (DBP) absorption number, 10^{-5} m^3/kg ($cm^3/100$ g)	D 2414	A 5	136.7	1.32	135.38	138.02
		B 5	99.6	1.08	98.52	100.68
		C 5	113.5	1.17	112.33	114.67
		D 5	64.1	1.59	62.51	65.69
		E 5	89.5	1.35	88.15	90.85
		F 5	129.5	1.53	127.97	131.03
		F 5A	129.5	1.53	127.97	131.03
DBP		G 5	36.2	0.75	35.45	36.95
DBP absorption number of compressed sample, 10^{-5} m^3/kg ($cm^3/100$ g) ($cm^3/100$ g)	D 3493	A 5	118.1	1.23	116.87	119.33
		B 5	86.8	1.20	85.60	88.00
		C 5	98.4	1.29	97.11	99.69
		D 5	57.8	1.74	56.06	59.54
		E 5	72.3	1.23	71.07	73.53
		F 5	88.9	2.04	86.86	90.94
		F 5A	88.6	2.04	86.56	90.64
CTAB surface area, 10^3 m^2/kg (m^2/g)	D 3765	A 5	128.2	1.53	126.67	129.73
		B 5	77.4	1.50	75.90	78.90
		C 5	117.3	1.77	115.53	119.07
		D 5	29.4	1.59	27.81	30.99
		E 5	37.5	1.05	36.45	38.55
		F 5	40.9	1.77	39.13	42.67
Surface area by multipoint B.E.T. nitrogen adsorption 10^3 m^2/kg (m^2/g) (m^2/g) NSA	D 4820	A 5 (N135)	141.5	1.74	139.76	143.24
		B 5 (N330)	73.4	1.35	72.05	74.75
		C 5 (N220)	121.6	1.26	120.34	122.86
		D 5 (N762)	27.5	0.48	27.02	27.98
		E 5 (N660)	35.3	0.57	34.73	35.87
		F 5 (N683)	39.1	0.87	38.23	39.97
		F 5 A	38.4	0.87	37.53	39.27
		G 5(N990)	9.1	0.36	8.74	9.46
Tint Strength	D 3265	A 5	120.9	2.41	118.49	123.31
		B 5	98.4	2.05	96.35	100.45
		C 5	115.9	1.99	113.91	117.89
		D 5	49.9	1.26	48.64	51.16
		E 5	55.4	0.98	54.43	56.38
		F 5	57.4	1.60	55.80	59.00
		F 5A	56.8	1.60	55.20	58.40
External surface area by multipoint nitrogen adsorption 10^3 m^2/kg (m^2/g)	D 5816	A 5	124.6	1.62	122.98	126.22
		B 5	73.1	1.68	71.42	74.78
		C 5	111.7	1.65	110.05	113.35
		D 5	27.1	1.67	25.43	28.77
		E 5	34.3	0.93	33.37	35.23
		F 5	37.1	1.99	35.11	39.09
		F 5A	36.5	1.99	34.51	38.49
		G 5	8.4	0.60	7.80	9.00

[A]This table is provided because both the SRB 4 and SRB 5 series are available and in use.