Designation: D4122 – 17ε1

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

This standard is issued under the fixed designation D4122; 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—Editorial corrections made throughout Annex A1 in June 2017.

1. Scope

1.1 This practice covers guidelines for the production and testing for uniformity of a lot of carbon black to be used as an Industry Reference Black (IRB).

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 and health 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]

D412 Test Methods for Vulcanized Rubber and Thermoplastic Elastomers—Tension

D1506 Test Methods for Carbon Black—Ash Content

D1508 Test Method for Carbon Black, Pelleted Fines and Attrition

D1509 Test Methods for Carbon Black—Heating Loss

D1510 Test Method for Carbon Black—Iodine Adsorption Number

D1513 Test Method for Carbon Black, Pelleted—Pour Density

D1514 Test Method for Carbon Black—Sieve Residue

D1618 Test Method for Carbon Black Extractables—Transmittance of Toluene Extract

D2414 Test Method for Carbon Black—Oil Absorption Number (OAN)

D3182 Practice for Rubber—Materials, Equipment, and Procedures for Mixing Standard Compounds and Preparing Standard Vulcanized Sheets

D3191 Test Methods for Carbon Black in SBR (Styrene-Butadiene Rubber)—Recipe and Evaluation Procedures

D3192 Test Methods for Carbon Black Evaluation in NR (Natural Rubber)

D3265 Test Method for Carbon Black—Tint Strength

D3493 Test Method for Carbon Black—Oil Absorption Number of Compressed Sample (COAN)

D5230 Test Method for Carbon Black—Automated Individual Pellet Hardness

D6556 Test Method for Carbon Black—Total and External Surface Area by Nitrogen Adsorption

3. Significance and Use

3.1 These guidelines are intended to ensure that IRBs are evaluated by a standard procedure.

3.2 These guidelines are to be used to establish the average physicochemical and physical rubber properties of a lot of carbon black to be used as an IRB.

4. Production, Quality Control, and Quality Assurance

4.1 It is assumed that the manufacturer of the IRB 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 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 is determined by historical records on the rate of use. The lot should have an expected life of 8 to 10 years at the most recent rate of use.

4.3 The black should be bagged in 50-lb polyethylene bags to reduce moisture incursion. Each pallet of bagged black should be wrapped in plastic to reduce environmental exposure. Depending on the size of the lot, the bagged black will be

[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.

segregated into equal sized sublots, typically ten to twenty, representing some logical subdivision, such as a truck load.

5. Sampling

5.1 To avoid having to break down stacked and wrapped bags on a pallet, the bags to be used as samples for uniformity and property determination testing are typically removed at appropriate times from the bagging stream. At least one sample per sublot will be collected for uniformity testing. If more than one sample point is identified for each sublot, they should be spaced to represent approximately equal quantities. Typically, two bags are taken at each sampling point. One is retained by the producer for in-house uniformity testing and one is used for samples to be distributed to participants in the rubber testing and uniformity ITP. The bags selected will be numbered from one through *n*, where *n* is the total number of sublots, in order to represent the corresponding production lot or labeled to identify the sublot and its position relative to the whole of the corresponding production lot.

5.2 *n* 4-dm^3 (1-gal) samples, numbered from one through *n*, or marked with the labels from 5.1 as taken from the corresponding bags, will be sent to each participant in the interlaboratory study to evaluate the new IRB.

5.3 Additionally, a 4-dm^3 (1-gal) sample of the *previous* IRB taken from a common blended source will also be sent to each participant.

6. Procedure

6.1 It is preferred that all of the samples be tested on the same day for any of the test methods described in 6.2 and 6.3. If the testing cannot be completed in the same day, it should be completed in consecutive days.

6.2 *Rubber Physical Tests:*

6.2.1 Perform the following physical tests in rubber on both the new and previous IRB. Test samples mixed in accordance with Test Methods D3191, Test Method A, Test Method B, or Test Method C and cure for 50 min at 145°C as well as samples mixed in accordance with Test Methods D3192, Test Method A, Test Method B, or Test Method C and cure for 30 min at 145°C. It is preferred that participating laboratories be found so that data from all test methods can be included for evaluation.

6.2.1.1 In accordance with Test Methods D412, Test Method A, test five dumbbells from each cured sheet and determine the mean values of tensile stress at 300 % elongation, tensile strength, and ultimate elongation.

6.2.1.2 Record data in absolute numbers (not as differences from IRB) on Table 1, reporting tensile stress and tensile strength to the nearest 0.1 MPa and ultimate elongation to the nearest 5 %.

6.3 *Informational Physicochemical Tests:*

6.3.1 Perform the following physicochemical tests on the new IRB:

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 *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.

6.3.2 Record data on Table 2 (or in a form that captures the same information as in Table 2).

TABLE 1 Industry Reference Black Test Data

Laboratory Number __ Test Method _____

Day of Mixing and Date	Sample No.	Tensile Strength, MPa D3191		Tensile Stress at 300 %, MPa D3191	Elongation, % D3191	Tensile Strength, MPa D3192		300 % Modulus, MPa D3192	Elongation, % D3192
Day No.__		50'				30'			
Date ____	Prev. IRB	50'				30'			
Day No.__		50'				30'			
Date ____	Prev. IRB	50'				30'			
Day No.__		50'				30'			
Date ____	Prev. IRB	50'				30'			
Day No.__		50'				30'			
Date ____	Prev. IRB	50'				30'			
Day No.__		50'				30'			
Date ____	Prev. IRB	50'				30'			
Day No.__		50'				30'			
Date ____	Prev. IRB	50'				30'			
Day No.__		50'				30'			
Date ____	Prev. IRB	50'				30'			
Day No.__		50'				30'			
Date ____	Prev. IRB	50'				30'			
Day No.__		50'				30'			
Date ____	Prev. IRB	50'				30'			
Day No.__		50'				30'			
Date ____	Prev. IRB	50'				30'			
Day No.__		50'				30'			
Date ____	Prev. IRB	50'				30'			
Day No.__		50'				30'			
Date ____	Prev. IRB	50'				30'			

TABLE 2 Industry Reference Black Informational Test Data

Laboratory Number__

Day of Mixing and Date	Sample No.	Iodine No., g/kg D1510	Oil Absorption No., 10^{-5} mg^3/kg (cm^3/100 g) D2414
Day No.____ Date ____	___		
Day No.____ Date ____	___		
Day No.____ Date ____	___		
Day No.____ Date ____	___		
Day No.____ Date ____	___		
Day No.____ Date ____	___		
Day No.____ Date ____	___		
Day No.____ Date ____	___		
Day No.____ Date ____	___		
Day No.____ Date ____	___		
Day No.____ Date ____	___		
Day No.____ Date ____	___		

7. Statistical Analysis

7.1 For each test in Table 1, enter the results from each laboratory for each sample into the form shown in Table 3. Then calculate the statistics defined in Table 3.

NOTE 1—Rubber physical test data are to be entered as differences from the previous IRB. For example:

$$Difference = X_1 - X_2 \quad (1)$$

where:

X_1 = measured value for new IRB, and
X_2 = measured value for previous IRB.

7.2 If any row average test result falls outside the interval defined by the upper and lower control limits shown in Table 3, this will indicate that the sublot of IRB represented by that row

TABLE 3 Statistical Analysis Form

NOTE 1—Experience so far has shown that neither a laboratory's test values nor a sample's test values are random values about the grand average, but tend to reflect a persistent bias typified by the average value for the laboratory or the sample. Consequently, it is not appropriate to divide the reproducibility by the square root of L or N as might otherwise be the case when comparing averages of L or N values to the grand average of $L \times N$ values.

Test Method: ASTM D _ _ _

Sample No. ↓	Laboratory No. →	1	2	...	*i*	...	*L*	$\bar{X}_R$
1								
2								
...								
j								
...								
N								
$\bar{X}_C$								$\bar{\bar{X}}$=

Row average $\bar{X}_R = \sum_i X/L$

Grand average test value $\bar{\bar{X}} = \sum_j \bar{X}_R/N$

Upper and lower control limits for row averages $= \bar{\bar{X}} \pm$ reproducibility of the test method.

Column average $\bar{X}_C = \sum_j X/N$

Upper and lower control limits for column averages

$= \bar{\bar{X}} \pm$ reproducibility of the test method.

average may be rejected by Committee D24 as being a nonhomogeneous portion of the production lot.

7.3 If any laboratory average test result (column average) falls outside the upper and lower control limits shown in Table 3, then that laboratory's data for that test should be deleted and Table 3 should be recalculated excluding that laboratory. Such data indicates that the laboratory has a significant reproducibility problem, which needs corrective action.

7.4 After deleting data, the remaining data for each test can be used to provide average differences between the new IRB and the previous one.

7.5 For each test in Table 2, enter the results from each laboratory for each sample in the form shown in Table 3. Then calculate the statistics defined in Table 3. The results are for information only and not to determine the uniformity of the lot.

8. Acceptance

8.1 All sublots tested as homogeneous by this practice will be considered acceptable by Committee D24 for use as the new IRB.

9. Shelf Life

9.1 The shelf life of the Industry Reference Black (IRB) 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.

10. Keywords

10.1 blending; industry reference blacks (IRBs); lot size; physical properties; physicochemical properties; shelf life; statistical analysis form; table for IRB test data; uniformity guidelines for production and testing

ANNEX

(Mandatory Information)

A1. LISTING OF THE PROPERTIES OF INDUSTRY REFERENCE BLACKS

A1.1 The listing of properties for Industry Reference Blacks (IRBs) is given in Table A1.1. The absolute values for I_2 number, OAN, COAN, and tint strength are listed. Values for tensile stress at 300 % elongation or "modulus" and tensile strength are given in relation to the previous IRB as a "difference." Since the purpose of an IRB is the elimination of the major part of laboratory-to-laboratory variation, it is used as a reference material within each laboratory to correct actual measured property values in that laboratory.

A1.1.1 The rubber properties of carbon black are typically reported as the difference between the values obtained for the test and those obtained for the current IRB.

NOTE A1.1—IRB No. 8[3] is an N330 type carbon black prepared at one location, then evaluated by ASTM Committee D24 to ensure uniformity. A large volume (115 910 kg (255 000 lb)) of this carbon black was prepared since it is used daily as a standard reference material by carbon black producers. The carbon black has an iodine number of 81.8 g/kg and OAN of 103.0 10^{-5} m^3/kg.

NOTE A1.2—IRB No. 9[3] is an N330 type carbon black prepared at one location, then evaluated by ASTM Committee D24 to ensure uniformity. A large volume (181 818 kg (400 000 lb)) of this carbon black was prepared since it is used daily as a standard reference material by carbon black producers. The carbon black has an iodine number of 82.1 g/kg and OAN value of 98.9 10^{-5} m^3/kg.

NOTE A1.3—Prior to the introduction of IRB8, all rubber testing was done following Method A of Test Methods D3191 and Test Methods D3192. No laboratory in the ITP to validate IRB8 provided data for Method A for either Test Methods D3191 or Test Methods D3192. Therefore, data from Methods B and C were included in the table. In the ITP to validate IRB9, data was provided for all three methods of Test Methods D3191 and Test Methods D3192. The difference from IRB8 to IRB9 data for all three methods has been included in Table A1.1.

NOTE A1.4—All the reported "difference from IRB8 to IRB9" values for all three test methods of Test Methods D3191 and Test Methods D3192 are less than the within laboratory test error reported in the applicable precision statement sections of those two Test Methods. This means that in a given laboratory using these test methods for testing both IRB8 and IRB9, they would not be able to distinguish which sample was which material based on their test results. Therefore, it can be concluded that, within testing error, IRB9 is a virtual drop-in replacement for IRB8, negating the need to revise industry specifications.

A1.2 The user of this table is cautioned against attempting to add the differences listed in the modulus and tensile strength columns to determine the relationship of two carbon blacks not adjacent in time. Such an addition is likely to produce spurious results due to additive errors.

A1.3 The listing of the properties of IRB No. 8 is given in Table A1.2. These values are the average from the producer from their data to evaluate the uniformity of IRB No. 8.

A1.4 The listing of the properties of IRB9 is given in Table A1.3. These values are the average of the test results obtained during the testing of IRB9 in the D24 LPRS program in March 2016 except where noted that the data is from the producer.

[3] Available from Laboratory Standards, 227 Somerset, Borger, TX 79007, www.carbonstandard.com.

TABLE A1.1 Industry Reference Blacks

IRB No.	Producer	Lot Size, lb†	Date of ASTM Acceptance	Iodine Adsorption No., D1510, g/kg	OAN, D2414, 10^{-5} m^3/kg	COAN, D3493, 10^{-5} m^3/kg	Tint Strength, D3265	Difference from previous IRB, MPa (psi)					
									D3192			D3191	
									Modulus	Tensile		Modulus	Tensile
1	Phillips	150 000	12/59	81	97[A]	...	...	15-min	...	...	35-min	...	...
								30-min	...	...	50-min	...	...
2	Continental	200 000	6/65	82	92	83	...	15-min	-0.3 (-50)	+1.9	35-min	...	...
								30-min	-0.2 (-25)	(+275)	50-min	...	...
										+1.2			
										(+175)			
3	Ashland	300 000	6/69	84.8	99.9	87.8	100.0	15-min	+0.7	-0.5 (-75)	35-min	...	...
								30-min	(+100)	-0.5 (-75)	50-min	...	...
									+0.5 (+75)				
4	Cabot	600 000	11/73	82.1	97.1	86.5	107.1	15-min	+0.2 (+30)	+0.9	35-min	...	...
								30-min	+0.5 (+70)	(+130)	50-min	...	...
										+0.8 (+110)			
5	Columbian	500 000	12/79	81.9	102.1	89.6	101.5	15-min	+1.2	0.0 (0.0)	35-min	+1.4	+0.1 (+20)
								30-min	(+170)	-0.6 (-90)	50-min	(+210)	+0.2 (+30)
									+0.9			+1.2	
									(+130)			(+180)	
6	Huber	900 000	6/85	80.0	100.0	87.2	99.2	15-min	-1.7 (-245)	-0.9 (-130)	35-min	-2.6 (-375)	-1.5 (-220)
								30-min	-2.2 (-320)	-0.9 (-130)	50-min	-2.7 (-390)	-1.2 (-175)
7	Sid Richardson	495 000	6/97	83.0	101.6	89.0	106.8	30-min	+3.2	+1.5	50-min	+3.8 (+550	+1.6
									(+460)	(+220)			(+230)
8	Continental	255 000	6/09	81.8	103.0	84.6	102.3	30-min			50-min		
								Method B	-0.09 (-13)	-0.03 (-4)	Method B	-0.63 (-91)	-0.33 (-48)
								Method C	-0.36 (-52)	-0.21 (-30)	Method C	-0.54 (-78)	-0.16 (-23)
9	Orion	400 000	6/16	82.1	98.9	90.1	105.9	30-min			50-min		
								Method A	-0.28 (-41)	-0.54 (-78)	Method A	-0.18 (-26)	-0.35 (-51)
								Method B	-0.08 (-12)	-0.03 (-4)	Method B	0.07 (10)	-0.33 (-48)
								Method C	-0.35 (-51)	-0.42 (-61)	Method C	0.18 (26)	0.35 (51)

[A] Hand oil absorption.
† Editorially corrected.

TABLE A1.2 IRB No. 8 Properties

Property	IRB No. 8
Tint Strength, D3265, % ITRB	102.3
Iodine Adsorption No., D1510, g/kg (mg/g)	81.8
NSA, D6556, 10^3 m^2/kg (m^2/g)	76.3
STSA, D6556, 10^3 m^2/kg (m^2/g)	74.9
OAN, D2414, 10^{-5} m^3/kg	103.0
COAN, D3493, 10^{-5} m^3/kg	84.6
Pour Density, D1513, kg/m^3 (lb/ft^3)	379 (23.7)
Fines Content, D1508, %	1.7
Heating Loss, D1509, %	0.3
Sieve Residue, D1514, mg/kg (ppm)	25
Transmittance of Toluene Extract, D1618, %	97.7
Mean Pellet Hardness, D5230, cN (gf)	27.5 (28.1)
Maximum Pellet Hardness, D5230, cN (gf)	47.1 (48.1)

TABLE A1.3 IRB9 Properties

Property	IRB9
Tint Strength, D3265, % ITRB	105.9
Iodine Adsorption No., D1510, g/kg (mg/g)	82.1
NSA, D6556, 10^3 m^2/kg (m^2/g)	78.1
STSA, D6556, 10^3 m^2/kg (m^2/g)	77.1
OAN, D2414, 10^{-5} m^3/kg	98.9
COAN, D3493, 10^{-5} m^3/kg	90.1
Pour Density, D1513, kg/m^3 (lb/ft^3)	400 (25.0)[A]
Fines Content, D1508, %	4.4[A]
Heating Loss, D1509, %	0.5[A]
Sieve Residue, D1514, mg/kg (ppm)	73
Transmittance of Toluene Extract, D1618, %	95.0
Mean Pellet Hardness, D5230, cN (gf)	41.7 (42.6)
Maximum Pellet Hardness, D5230, cN (gf)	77.5 (79.1)

[A] Test results from producer.

RELATED MATERIAL

D7849 Classification for Nomenclature of Reference Materials of Committee D24