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Standard Test Methods for Carbon Black Evaluation in NR (Natural Rubber)[1]

This standard is issued under the fixed designation D3192; 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 These test methods cover the standard materials, test formulation, mixing procedure, and test methods for the evaluation and production control of carbon blacks in natural rubber (NR).

1.2 The values stated in SI units are to be regarded as the standard. The values 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.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D412 Test Methods for Vulcanized Rubber and Thermoplastic Elastomers—Tension

D1799 Practice for Carbon Black—Sampling Packaged Shipments

D1900 Practice for Carbon Black—Sampling Bulk Shipments

D2084 Test Method for Rubber Property—Vulcanization Using Oscillating Disk Cure Meter

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

D4483 Practice for Evaluating Precision for Test Method Standards in the Rubber and Carbon Black Manufacturing Industries

3. Significance and Use

3.1 The major portion of carbon black consumed by the rubber industry is used to improve the physical properties, life expectancy, and utility of rubber products. These test methods provide a natural rubber formulation and directions for evaluating carbon black intended for use in rubber products.

3.2 These test methods may be used to characterize carbon black in terms of specific properties of the standard compound. These test methods are useful for the quality assurance of carbon black production. They may also be used for the preparation of reference compounds, to confirm the day-to-day reliability of testing operations used in the rubber industry, for the evaluation of experimental compounds, and quality control of production compounds.

4. Standard Test Formula

4.1 *Standard Formula:*

Material	IRM[A] No.	Quantity, parts by mass
Natural rubber[B]	...	100.00
Stearic acid	21	3.00
Zinc oxide	91	5.00
Benzothiazyl disulfide	2	0.60
Sulfur	31	2.50
Carbon black[C]	...	50.00
Total		161.10
Batch factor:[D]		
Test Method A—Mill		4.00
Test Method B—Internal Mixer		6.00
Test Method C—Miniature Internal Mixer		0.40

[A] IRM 91 is available from R. E. Carroll, Inc., 1570 North Olden Ave., Trenton, NJ 08638; (800) 257–9365. IRM 2, IRM 21, and IRM 31 are available from Akron Rubber Development Lab, 2887 Gilchrist Road, Akron, OH 44305; (330) 794–6600.

[B] SMR L and STR L have been found to give satisfactory performance. Other sources of rubber may give satisfactory results but have not been investigated by Subcommittee D24.71. Other sources of rubber should be checked to ensure that results equivalent to SMR L are attained before using in this test method.

[C] Use 75.00 parts by mass of carbon blacks in the N-800 and N-900 series.

[D] Weigh rubber and carbon black to the nearest 1 g, sulfur and accelerator to the nearest 0.02 g, and all other compounding materials to the nearest 0.1 g.

5. Sampling and Sample Preparation

5.1 Samples shall be taken in accordance with Practice D1799 or Practice D1900.

5.2 The carbon black shall be conditioned before weighing and mixing by heating for 1 h in an oven set at 125 ± 1°C. The black shall be placed in an open vessel of suitable dimensions so that the depth of black is no more than 10 mm during

[1] These test methods are under the jurisdiction of ASTM Committee D24 on Carbon Black and are the direct responsibility of Subcommittee D24.71 on Carbon Black Testing in Rubber.

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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.

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conditioning. The black conditioned as above shall be stored in a closed moisture-proof container until ready for mixing.

6. Mixing Procedures

6.1 For general mixing procedure refer to Practice D3182. The following mixing procedures are acceptable in testing carbon black: (*1*) Test Method A—Mill Mix, (*2*) Test Method B—Internal Mixer, and (*3*) Test Method C—Miniature Internal Mixer.

6.1.1 *Test Method A—Mill Mix:*

	Duration, min	Accumulative, min
Set the mill opening at 1.4 mm (0.055 in.) and adjust and maintain roll temperature at 70 ± 5°C.	0	0
Add rubber and band on the front roll. Make two ¾ cuts from each side.	2.0	2.0
Set mill opening at 1.65 mm (0.065 in.). Add stearic acid. Make one ¾ cut from each side.	2.5	4.5
Add sulfur, accelerator, and zinc oxide. Make two ¾ cuts from each side.	2.0	6.5
Add all the black. When that portion of the carbon black that was added has dropped through to the mill pan and the bank is dry, make two ¾ cuts from each side. Open the mill to 1.9 mm (0.075 in.) and add the carbon black from the mill pan until all is incorporated. Make three ¾ cuts from each side.	7.5	14.0
Note—Do not cut any stock while free carbon black is evident in the bank or on the milling surface. Be certain to return any pigments that drop through the mill to the milling stock.		
Set the mill opening at 0.80 mm (0.032 in.) and pass the rolled batch endwise through the mill six times.	2.0	16.0
Open the mill to give a minimum stock thickness of 6 mm (0.25 in.) and pass the stock through the rolls four times, folding it back on itself each time.	1.0	17.0
Total Time	17.0	

6.1.1.1 Check the batch mass and record. If outside of the range from 641.2 to 647.6 g, reject the batch. From this stock, cut enough sample to allow testing of or curing characteristics in accordance with Test Method D2084, if desired.

6.1.1.2 Open the mill and sheet off to produce a thickness of 2.2 mm (0.085 in.).

6.1.1.3 Cool on a flat, dry metal surface, at a temperature of 23 ± 3°C for 1 to 24 h. Unless the relative humidity of the laboratory is controlled at 50 ± 5 %, the sheeted stock should be cooled and stored in a closed container to prevent moisture absorption.

6.1.2 *Test Method B—Internal Mixer:*

	Duration, min	Accumulative, min
Adjust the internal mixer temperature to create a dump temperature between 110 and 125°C. Close the discharge gate, start the rotor, raise the ram, and charge the materials as described. Lower the ram after each operation.	0	0
Add the rubber.	0.5	0.5
Add the benzothiazyl disulfide.	0.5	1.0
Add the stearic acid.	1.0	2.0
Add the zinc oxide and one-half the carbon black.	1.5	3.5
Add the remainder of the carbon black.	1.5	5.0
Add the sulfur. Clean the mixer throat and the top of the ram.	1.0	6.0
Dump at 7 min.	1.0	7.0
Subtotal	7.0	
Set the mill opening at 0.80 mm (0.032 in.) and maintain the roll temperature at 70 ± 5°C. Pass the rolled batch endwise through the mill six times.	2.0	9.0
Open the mill to give a minimum stock thickness of 6 mm (0.25 in.) and pass the stock through the rolls four times, folding it back on itself each time.	1.0	10.0
Total Time	10.0	

6.1.2.1 Check the batch mass and record. If outside of the range from 961.8 to 971.4 g, reject the batch. From this stock, cut enough sample to allow testing of curing characteristics in accordance with Test Method D2084, if desired.

6.1.2.2 Open the mill and sheet off to produce a stock thickness of 2.2 mm (0.085 in.).

6.1.2.3 Unless otherwise specified, condition the sheeted compound for 1 to 24 h at 23 ± 3°C (73.4 ± 5.4°F) at a relative humidity not greater than 55 %. For maximum precision, condition for 1 to 24 h in a closed container to prevent absorption of moisture from the air, or in an area controlled at 35 ± 5 % relative humidity in accordance with Practice D3182. Vulcanize and test in accordance with Section 7.

6.1.3 *Test Method C—Miniature Internal Mixer:*

6.1.3.1 *Pigment Masterbatch Preparation—Mill Mix:*

	Duration, min	Accumulative, min
(Batch Factor 4.00) Set the mill opening at 1.4 mm (0.055 in.) and adjust and maintain roll temperature at 70 ± 5°C.	0.0	0.0
Add rubber and band on the front roll. Make two ¾ cuts from each side.	2.0	2.0
Set mill opening at 1.65 mm (0.065 in.). Add stearic acid. Make one ¾ cut from each side.	2.5	4.5
Add sulfur, accelerator, and zinc oxide. Make two ¾ cuts from each side.	2.0	6.5
Set the mill opening at 0.80 mm (0.032 in.), and pass the rolled batch endwise through the mill six times.	2.0	8.5
Check the batch mass and if outside of the range from 442.2 to 446.6 g, reject the batch.	0.5	9.0
Set the mill opening to 1.5 mm (0.060 in.), band the stock. Sheet off.	1.0	10.0
Total Time	10.0	

(1) Cool on a flat, dry metal surface, at a temperature of 23 ± 3°C. Unless the relative humidity of the laboratory is controlled at 50 ± 5 %, this masterbatch should be cooled and stored in a closed container to prevent moisture absorption.

NOTE 1—This pigment masterbatch should be used within 6 weeks or discarded and a new batch prepared.

6.1.3.2 *Carbon Black—Miniature Internal Mixer:*

iTeh Standards (https://standards.iteh.ai) Document Preview
ASTM D3192-09(2014)
https://standards.iteh.ai/catalog/standards/sist/a02ee943-f136-4e43-b6f8-1514cb8b/astm-d3192-092014

(1) Mix with the head temperature of the miniature internal mixer maintained at 60 ± 3°C and the unloaded slow rotor speed at 6.3 to 6.6 rad/s (60 to 63 r/min).

(2) Cut the pigment masterbatch prepared in 6.4.1 into strips approximately 20-mm (0.75-in.) wide and weigh out 44.44 g.

(3) Weigh out 20.00 g of carbon black sample.

	Duration, min	Accumulative, min
Charge the mixing chamber with the masterbatch strips, and start the timer.	0.0	0.0
Masticate the masterbatch.	0.5	0.5
Add carbon black, use ram to work all of sample into chamber, sweep down orifice, and lower ram.	1.0	1.5
Allow the batch to mix.	1.5	3.0
Total Time	3.0	

(4) Turn off the motor, raise the ram, remove the mixing chamber and unload the batch. Record the batch temperature if desired.

(5) With the mill at room temperature, pass the batch through the mill set at 0.80 mm (0.032 in.). Fold it on itself and feed it back through the mill five more times, always keeping the grain in the same direction and folding it on itself each time.

(6) Check the batch mass and record. If outside of the range from 64.12 to 64.76 g, reject the batch.

(7) For testing of stress-strain, pass the batch through the mill to produce a stock thickness of 2.2 mm (0.085 in.).

(8) For testing of curing characteristics in accordance with Test Method D2084, pass the batch through the mill to produce a minimum stock thickness of 6 mm (0.25 in.).

(9) Cool on a flat, dry metal surface, at a temperature of 23 ± 3°C for 1 to 24 h. Unless the relative humidity of the laboratory is controlled at 50 ± 5 %, the sheeted stock should be cooled and stored in a closed container to prevent moisture absorption.

7. Preparation and Testing of Vulcanizates

7.1 For stress-strain testing, prepare test slabs and vulcanize them in accordance with Practice D3182.

7.1.1 The recommended standard cures are 30 min at 145°C for ASTM N-type carbon black, and 30 and 50 min at 145°C for ASTM S-type carbon black.

7.1.2 Condition vulcanizates of compounds at a temperature of 23 ± 2°C (73 ± 3.6°F) for at least 16 h and for not more than 96 h before preparing and testing, unless otherwise specified.

NOTE 2—Quality control of rubber production may require testing within 1 to 6 h to provide close surveillance of the plant operation; however, slightly different results may be obtained.

7.1.3 Prepare test specimens in accordance with Practice D3182 and obtain tensile stress at 300 % elongation, tensile strength, and ultimate elongation parameters in accordance with Test Methods D412. Typically, a test specimen is prepared using the current Industry Reference Black, for example IRB 7, with each set of mixes and the data obtained is reported as a difference from the IRB.

7.2 For measuring vulcanization parameters by the curemeter in accordance with Test Method D2084, use the 6-mm (0.25-in.) thickness samples that were previously prepared.

7.2.1 The recommended standard test conditions are 1.7 Hz (100 cpm) oscillation frequency, 1 ± 0.03° amplitude of oscillation, and 160 ± 0.3°C die temperature using the micro die system.

7.2.2 The recommended standard test parameters are M_L, M_H, t_{s1}, t'_c (50) and t'_c (90).

8. Precision and Bias[3]

8.1 This precision and bias statement has been prepared in accordance with Practice D4483. Refer to Practice D4483 for terminology and other statistical details.

8.2 *Precision*—The precision results in this precision and bias section give an estimate of the precision of this test method with the materials (rubbers, carbon blacks, and so forth) used in the particular interlaboratory program described in 8.3 through 8.5.2.3. The precision parameters should not be used for acceptance or rejection testing of any group of materials without documentation that they are applicable to those particular materials and the specific testing protocols of the test method.

8.3 *Mill Mix—Test Method A*—A Type 2 interlaboratory precision program was conducted in 1990. Both repeatability and reproducibility represent short-term testing conditions. Nine laboratories tested four carbon blacks (SRBs A-4, B-4, D-4, and F-4) once on each of two different days. Test results were obtained in accordance with Test Methods D412 and are expressed as differences from IRB 6. A test result is the value obtained from a single determination. Acceptable difference values were not measured (see Table 1).

8.3.1 *Repeatability:*

8.3.1.1 *Tensile Stress at 300 % Elongation*—The pooled repeatability of Test Methods D3192 Method A (using Test Methods D412 Method A) tensile stress at 300 % elongation has been established as 1.01 MPa (146 psi). Two single test results (or determinations) that differ by more than 1.01 MPa (146 psi) must be considered suspect, that is, to have come from different sample populations. Such a decision dictates that some appropriate action be taken.

8.3.1.2 *Tensile Strength*—The pooled repeatability of Test Methods D3192 Method A (using Test Methods D412 Method A) tensile strength has been established as 1.70 MPa (246 psi). Two single test results (or determinations) that differ by more than 1.70 MPa (246 psi) must be considered suspect, that is, to have come from different sample populations. Such a decision dictates that some appropriate action be taken.

8.3.1.3 *Ultimate Elongation*—The pooled repeatability of Test Methods D3192 Method A (using Test Methods D412 Method A) ultimate elongation has been established as 28.2 %. Two single test results (or determinations) that differ by more than 28.2 % must be considered suspect, that is, to have come from different sample populations. Such a decision dictates that some appropriate action be taken.

[3] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D24-1031.