



# Standard Test Methods for Carbon Black Evaluation in NR (Natural Rubber)<sup>1</sup>

This standard is issued under the fixed designation D 3192; 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 ( $\epsilon$ ) 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:

- D 412 Test Methods for Vulcanized Rubber and Thermoplastic Rubbers and Thermoplastic Elastomers—Tension<sup>2</sup>
- D 1799 Practice for Carbon Black—Sampling Packaged Shipments<sup>2</sup>
- D 1900 Practice for Carbon Black—Sampling Bulk Shipments<sup>2</sup>
- D 2084 Test Method for Rubber Property—Vulcanization Using Oscillating Disk Cure Meter<sup>2</sup>
- D 3182 Practice for Rubber—Materials, Equipment, and Procedures for Mixing Standard Compounds and Preparing Standard Vulcanized Sheets<sup>2</sup>
- D 4483 Practice for Determining Precision for Test Method Standards in the Rubber and Carbon Black Industries<sup>2</sup>

## 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 <sup>A</sup> No.	Quantity, parts by mass
Natural rubber, SMR L	...	100.00
Stearic acid	21	3.00
Zinc oxide	91	5.00
Benzothiazyl disulfide	2	0.60
Sulfur	31	2.50
Carbon black <sup>B</sup>	...	50.00
Total		161.10
Batch factor: <sup>C</sup>		
Test Method A—Mill		4.00
Test Method B—Internal Mixer		6.00
Test Method C—Miniature Internal Mixer		0.40

<sup>A</sup> 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.

<sup>B</sup> Use 75.00 parts by mass of carbon blacks in the N-800 and N-900 series.

<sup>C</sup> 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 D 1799 or Practice D 1900.

5.2 The carbon black shall be conditioned before weighing and mixing by heating for 1 h in an oven set at  $125 \pm 1^\circ\text{C}$ . 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 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 D 3182. 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.

<sup>1</sup> 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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<sup>2</sup> *Annual Book of ASTM Standards*, Vol 09.01.

	Duration, min	Accumulative, min
<b>6.2 Mill Mix—Test Method A:</b>		
6.2.1 Set the mill opening at 1.4 mm (0.055 in.) and adjust and maintain roll temperature at 70 ± 5°C.	0	0
6.2.2 Add rubber and band on the front roll. Make two ¾ cuts from each side.	2.0	2.0
6.2.3 Set mill opening at 1.65 mm (0.065 in.). Add stearic acid. Make one ¾ cut from each side.	2.5	4.5
6.2.4 Add sulfur, accelerator, and zinc oxide. Make two ¾ cuts from each side.	2.0	6.5
6.2.5 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.		
6.2.6 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
6.2.7 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	
6.2.8 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 D 2084, if desired.		
6.2.9 Open the mill and sheet off to produce a thickness of 2.2 mm (0.085 in.).		
6.2.10 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.		
<b>6.3 Internal Mixer—Test Method B:</b>		
6.3.1 Adjust the internal mixer temperature to create in 6.3.8 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
6.3.2 Add the rubber.	0.5	0.5
6.3.3 Add the benzothiazyl disulfide.	0.5	1.0
6.3.4 Add the stearic acid.	1.0	2.0
6.3.5 Add the zinc oxide and one-half the carbon black.	1.5	3.5
6.3.6 Add the remainder of the carbon black.	1.5	5.0
6.3.7 Add the sulfur. Clean the mixer throat and the top of the ram.	1.0	6.0
6.3.8 Dump at 7 min.	1.0	7.0
Subtotal	7.0	
6.3.9 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
6.3.10 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	
6.3.11 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 D 2084, if desired.		
6.3.12 Open the mill and sheet off to produce a stock thickness of 2.2 mm (0.085 in.).		
6.3.13 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.		
<b>6.4 Miniature Internal Mixer—Test Method C:</b>		
6.4.1 <i>Pigment Masterbatch Preparation—Mill Mix:</i> (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
6.4.2 Add rubber and band on the front roll. Make two ¾ cuts from each side.	2.0	2.0
6.4.3 Set mill opening at 1.65 mm (0.065 in.). Add stearic acid. Make one ¾ cut from each side.	2.5	4.5
6.4.4 Add sulfur, accelerator, and zinc oxide. Make two ¾ cuts from each side.	2.0	6.5
6.4.5 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
6.4.6 Check the batch mass and if outside of the range from 442.2 to 446.6 g, reject the batch.	0.5	9.0
6.4.7 Set the mill opening to 1.5 mm (0.060 in.), band the stock. Sheet off.	1.0	10.0
Total Time	10.0	
6.4.8 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—This pigment masterbatch should be used within 6 weeks or discarded and a new batch prepared.		
6.4.9 <i>Carbon Black—Miniature Internal Mixer:</i> 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).		
6.4.10 Cut the pigment masterbatch prepared in 6.4.1 into strips approximately 20-mm (0.75-in.) wide and weigh out 44.44 g.		
6.4.11 Weigh out 20.00 g of carbon black sample.		
6.4.12 Charge the mixing chamber with the masterbatch strips, and start the timer.	0.0	0.0
6.4.13 Masticate the masterbatch.	0.5	0.5
6.4.14 Add carbon black, use ram to work all of sample into chamber, sweep down orifice and lower ram.	1.0	1.5
6.4.15 Allow the batch to mix.	1.5	3.0
Total Time	3.0	
6.4.16 Turn off the motor, raise the ram, remove the mixing chamber and unload the batch. Record the batch temperature if desired.		
6.4.17 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.		
6.4.18 Check the batch mass and record. If outside of the range from 64.12 to 64.76 g, reject the batch.		
6.4.19 For testing of stress-strain, pass the batch through the mill to produce a stock thickness of 2.2 mm (0.085 in.).		
6.4.20 For testing of curing characteristics in accordance with Test Method D 2084, pass the batch through the mill to produce a minimum stock thickness of 6 mm (0.25 in.).		

6.4.21 Cool on a flat, dry metal surface, at a temperature of  $23 \pm 3^\circ\text{C}$  for 1 to 24 h. Unless the relative humidity of the laboratory is controlled at  $50 \pm 5\%$ , 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 D 3182.

7.1.1 The recommended standard cures are 30 min at  $145^\circ\text{C}$  for ASTM N-type carbon black, and 30 and 50 min at  $145^\circ\text{C}$  for ASTM S-type carbon black.

7.1.2 Condition the cured sheets for 1 to 96 h at a temperature of  $23 \pm 3^\circ\text{C}$ .

7.1.3 Prepare test specimens in accordance with Practice D 3182 and obtain tensile stress at 300 % elongation, tensile strength, and ultimate elongation parameters in accordance with Test Methods D 412. 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 D 2084, 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,  $1 \pm 0.03^\circ$  amplitude of oscillation, and  $160 \pm 0.3^\circ\text{C}$  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 <sup>3</sup>

8.1 This precision and bias statement has been prepared in accordance with Practice D 4483. Refer to Practice D 4483 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, etc.) 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 Method D 412 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 D 3192 Method A (using Test Methods D 412 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 D 3192 Method A (using Test Methods D 412 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 D 3192 Method A (using Test Methods D 412 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.

### 8.3.2 Reproducibility:

8.3.2.1 *Tensile Stress at 300 % Elongation*—The pooled reproducibility of Test Methods D 3192 Method A (using Test Methods D 412 Method A) tensile stress at 300 % elongation has been established as 1.09 MPa (158 psi). Two single test results (or determinations) produced in separate laboratories that differ by more than 1.09 MPa (158 psi) must be considered suspect, that is, that they represent different sample populations. Such a decision dictates that appropriate investigative or technical or commercial actions, or both, be taken.

8.3.2.2 *Tensile Strength*—The pooled reproducibility of Test Methods D 3192 Method A (using Test Methods D 412 Method A) tensile strength has been established as 2.07 MPa (300 psi). Two single test results (or determinations) produced in separate laboratories that differ by more than 2.07 MPa (300 psi) must be considered suspect, that is, that they represent different sample populations. Such a decision dictates that appropriate investigative or technical or commercial actions, or both, be taken.

8.3.2.3 *Ultimate Elongation*—The pooled reproducibility of Test Methods D 3192 Method A (using Test Methods D 412 Method A) ultimate elongation has been established as 42.4 %. Two single test results (or determinations) produced in separate laboratories that differ by more than 42.4 % must be considered suspect, that is, that they represent different sample populations. Such a decision dictates that appropriate investigative or technical or commercial actions, or both, be taken.

8.4 *Internal Mixer—Test Method B*—A Type 2 interlaboratory precision program was conducted in 1990. Both repeatability and reproducibility represent short-term testing conditions. Four 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 Method D 412 and are expressed as differences from IRB 6. A test result is the

<sup>3</sup> Supporting data for this precision evaluation are available from ASTM Headquarters. Request RR: D24-1031.

**TABLE 1 Test Methods D 3192 Test Method Precision—Type 2 (Mill Mix—Method A (Using Test Methods D 412 Method A))<sup>A</sup>**

Tensile Stress at 300 % Elongation, MPa (psi)					
Material	Mean Level	Within Laboratories <sup>B</sup>		Between Laboratories <sup>B</sup>	
		$S_r$	$r$	$S_R$	$R$
D-4	-2.79 (-404)	0.40 (58)	1.13 (164)	0.40 (58)	1.13 (164)
A-4	-0.63 (-92)	0.38 (55)	1.07 (155)	0.41 (59)	1.15 (166)
B-4	-0.28 (-40)	0.29 (43)	0.83 (120)	0.39 (56)	1.10 (159)
F-4	3.23 (468)	0.34 (50)	0.97 (141)	0.35 (50)	0.98 (142)
Average	-0.08 (-12)				
Pooled values		0.36 (52)	1.01 (146)	0.39 (56)	1.09 (158)
Tensile Strength, MPa (psi)					
Material	Mean Level	Within Laboratories <sup>B</sup>		Between Laboratories <sup>B</sup>	
		$S_r$	$r$	$S_R$	$R$
D-4	-2.08 (-301)	0.67 (97)	1.88 (273)	0.67 (97)	1.88 (273)
F-4	-2.01 (-292)	0.79 (114)	2.23 (323)	0.99 (143)	2.79 (405)
B-4	-0.21 (-31)	0.33 (48)	0.93 (135)	0.65 (95)	1.85 (268)
A-4	2.93 (424)	0.52 (75)	1.47 (213)	0.54 (78)	1.53 (221)
Average	-0.34 (-49)				
Pooled values		0.60 (87)	1.70 (246)	0.73 (106)	2.07 (300)
Ultimate Elongation, %					
Material	Mean Level	Within Laboratories <sup>B</sup>		Between Laboratories <sup>B</sup>	
		$S_r$	$r$	$S_R$	$R$
F-4	-94	8.2	23.3	14.5	41.1
B-4	4	11.4	32.2	14.3	40.5
D-4	21	12.1	34.3	12.8	36.3
A-4	34	7.2	20.4	17.8	50.5
Average	-9				
Pooled values		10.0	28.2	15.0	42.4

<sup>A</sup>This is short-term precision (days) with outliers removed from the data set.

<sup>B</sup>Symbols are defined as follows:

$S_r$  = within-laboratory standard deviation,

$r$  = repeatability (in measurement units),

$S_R$  = standard deviation for total between-laboratory variability, and

$R$  = reproducibility (in measurement units).

value obtained from a single determination. Acceptable difference values were not measured (see Table 2).

#### 8.4.1 Repeatability:

8.4.1.1 *Tensile Stress at 300 % Elongation*—The pooled repeatability of Test Methods D 3192 Method B (using Test Methods D 412 Method A) tensile stress at 300 % elongation has been established as 0.68 MPa (99 psi). Two single test results (or determinations) that differ by more than 0.68 MPa (99 psi) must be considered suspect, that is, to have come from different sample populations. Such a decision dictates that some appropriate action be taken.

8.4.1.2 *Tensile Strength*—The pooled repeatability of Test Methods D 3192 Method B (using Test Methods D 412 Method A) tensile strength has been established as 0.88 MPa (128 psi). Two single test results (or determinations) that differ by more than 0.88 MPa (128 psi) must be considered suspect, that is, to have come from different sample populations. Such a decision dictates that some appropriate action be taken.

8.4.1.3 *Ultimate Elongation*—The pooled repeatability of Test Methods D 3192 Method B (using Test Methods D 412 Method A) ultimate elongation has been established as 34.1 %. Two single test results (or determinations) that differ by more than 34.1 % must be considered suspect, that is, to have come from different sample populations. Such a decision dictates that some appropriate action be taken.

#### 8.4.2 Reproducibility:

8.4.2.1 *Tensile Stress at 300 % Elongation*—The pooled reproducibility of Test Methods D 3192 Method B (using Test Methods D 412 Method A) tensile stress at 300 % elongation has been established as 1.37 MPa (198 psi). Two single test results (or determinations) produced in separate laboratories that differ by more than 1.37 MPa (198 psi) must be considered suspect, that is, that they represent different sample populations. Such a decision dictates that appropriate investigative or technical or commercial actions, or both, be taken.

8.4.2.2 *Tensile Strength*—The pooled reproducibility of Test Methods D 3192 Method B (using Test Methods D 412 Method A) tensile strength has been established as 1.67 MPa (242 psi). Two single test results (or determinations) produced in separate laboratories that differ by more than 1.67 MPa (242 psi) must be considered suspect, that is, that they represent different sample populations. Such a decision dictates that appropriate investigative or technical or commercial actions, or both, be taken.

8.4.2.3 *Ultimate Elongation*—The pooled reproducibility of Test Methods D 3192 Method B (using Test Methods D 412 Method A) ultimate elongation has been established as 45.9 %. Two single test results (or determinations) produced in separate laboratories that differ by more than 45.9 % must be considered suspect, that is, that they represent different sample populations. Such a decision dictates that appropriate investigative or technical or commercial actions, or both, be taken.

**TABLE 2 Test Methods D 3192 Test Method Precision—Type 2 (Internal Mixer—Method B (Using Test Methods D 412 Method A))<sup>A</sup>**

Tensile Stress at 300 % Elongation, MPa (psi)					
Material	Mean Level	Within Laboratories <sup>B</sup>		Between Laboratories <sup>B</sup>	
		$S_r$	$r$	$S_R$	$R$
D-4	-2.72 (-395)	0.16 (23)	0.45 (65)	0.34 (49)	0.95 (138)
A-4	-0.64 (-92)	0.25 (36)	0.71 (103)	0.67 (98)	1.91 (277)
B-4	-0.53 (-76)	0.15 (21)	0.42 (61)	0.49 (71)	1.39 (201)
F-4	2.95 (428)	0.35 (51)	1.00 (145)	0.35 (51)	1.00 (145)
Average	-0.24 (-34)	...	...	...	...
Pooled values	...	0.24 (35)	0.68 (99)	0.48 (70)	1.37 (198)

  

Tensile Strength, MPa (psi)					
Material	Mean Level	Within Laboratories <sup>B</sup>		Between Laboratories <sup>B</sup>	
		$S_r$	$r$	$S_R$	$R$
F-4	-2.69 (-390)	0.22 (32)	0.62 (90)	0.81 (118)	2.30 (334)
D-4	-1.75 (-254)	0.52 (75)	1.46 (212)	0.52 (75)	1.46 (212)
B-4	-0.32 (-47)	0.08 (12)	0.24 (34)	0.45 (65)	1.28 (185)
A-4	2.43 (353)	0.26 (37)	0.72 (105)	0.51 (74)	1.44 (209)
Average	-0.58 (-84)	...	...	...	...
Pooled values	...	0.31 (45)	0.88 (128)	0.59 (86)	1.67 (242)

  

Ultimate Elongation, %					
Material	Mean Level	Within Laboratories <sup>B</sup>		Between Laboratories <sup>B</sup>	
		$S_r$	$r$	$S_R$	$R$
F-4	-100	14.3	40.5	15.2	43.0
B-4	1	4.2	11.8	10.6	30.0
D-4	13	8.5	24.1	8.5	24.1
A-4	16	16.9	47.8	25.2	71.4
Average	-13	...	...	...	...
Pooled values	...	12.0	34.1	16.20	45.9

<sup>A</sup>This is short-term precision (days) with outliers removed from the data set.

<sup>B</sup>Symbols are defined as follows:

$S_r$  = within-laboratory standard deviation,

$r$  = repeatability (in measurement units),

$S_R$  = standard deviation for total between-laboratory variability, and

$R$  = reproducibility (in measurement units).

8.5 *Miniature Internal Mixer—Test Method C*—A Type 2 interlaboratory precision program was conducted in 1990. Both repeatability and reproducibility represent short-term testing conditions. Two 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 Method D 412 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 3).

#### 8.5.1 Repeatability:

8.5.1.1 *Tensile Stress at 300 % Elongation*—The pooled repeatability of Test Methods D 3192 Method C (using Test Methods D 412 Method A) tensile stress at 300 % elongation has been established as 0.64 MPa (93 psi). Two single test results (or determinations) that differ by more than 0.64 MPa (93 psi) must be considered suspect, that is, to have come from different sample populations. Such a decision dictates that some appropriate action be taken.

8.5.1.2 *Tensile Strength*—The pooled repeatability of Test Methods D 3192 Method C (using Test Methods D 412 Method A) tensile strength has been established as 0.62 MPa (90 psi). Two single test results (or determinations) that differ by more than 0.62 MPa (90 psi) must be considered suspect, that is, to have come from different sample populations. Such a decision dictates that some appropriate action be taken.

8.5.1.3 *Ultimate Elongation*—The pooled repeatability of Test Methods D 3192 Method C (using Test Methods D 412

Method A) ultimate elongation has been established as 28.7 %. Two single test results (or determinations) that differ by more than 28.7 % must be considered suspect, that is, to have come from different sample populations. Such a decision dictates that some appropriate action be taken.

#### 8.5.2 Reproducibility:

8.5.2.1 *Tensile Stress at 300 % Elongation*—The pooled reproducibility of Test Methods D 3192 Method C (using Test Methods D 412 Method A) tensile stress at 300 % elongation has been established as 1.07 MPa (156 psi). Two single test results (or determinations) produced in separate laboratories that differ by more than 1.07 MPa (156 psi) must be considered suspect, that is, that they represent different sample populations. Such a decision dictates that appropriate investigative or technical or commercial actions, or both, be taken.

8.5.2.2 *Tensile Strength*—The pooled reproducibility of Test Methods D 3192 Method C (using Test Methods D 412 Method A) tensile strength has been established as 3.02 MPa (438 psi). Two single test results (or determinations) produced in separate laboratories that differ by more than 3.02 MPa (438 psi) must be considered suspect, that is, that they represent different sample populations. Such a decision dictates that appropriate investigative or technical or commercial actions, or both, be taken.

8.5.2.3 *Ultimate Elongation*—The pooled reproducibility of Test Methods D 3192 Method C (using Test Methods D 412 Method A) ultimate elongation has been established as 86.4 %.



**TABLE 3 Test Methods D 3192 Test Method Precision—Type 2 (Miniature Internal Mixer—Method C (Using Test Methods D 412 Method A))<sup>A</sup>**

Tensile Stress at 300 % Elongation, MPa (psi)					
Material	Mean Level	Within Laboratories <sup>B</sup>		Between Laboratories <sup>B</sup>	
		$S_r$	$r$	$S_R$	$R$
D-4	-2.45 (-355)	0.34 (50)	0.98 (142)	0.34 (50)	0.98 (142)
A-4	-0.81 (-118)	0.07 (10)	0.20 (28)	0.56 (82)	1.59 (231)
B-4	-0.53 (-76)	0.19 (28)	0.55 (79)	0.31 (45)	0.88 (128)
F-4	3.88 (563)	0.21 (30)	0.59 (86)	0.21 (30)	0.59 (86)
Average	0.02 (3)	...	...	...	...
Pooled values		0.23 (33)	0.64 (93)	0.38 (55)	1.07 (156)

  

Tensile Strength, MPa (psi)					
Material	Mean Level	Within Laboratories <sup>B</sup>		Between Laboratories <sup>B</sup>	
		$S_r$	$r$	$S_R$	$R$
F-4	-1.55 (-225)	0.05 (7)	0.14 (20)	1.56 (226)	4.42 (641)
D-4	-0.93 (-135)	0.25 (36)	0.70 (102)	0.75 (109)	2.13 (309)
B-4	0.40 (58)	0.17 (25)	0.49 (71)	1.06 (153)	2.99 (433)
A-4	2.83 (410)	0.31 (45)	0.88 (128)	0.67 (97)	1.90 (275)
Average	0.49 (72)	...	...	...	...
Pooled values	...	0.22 (32)	0.62 (90)	1.07 (155)	3.02 (438)

  

Ultimate Elongation, %					
Material	Mean Level	Within Laboratories <sup>B</sup>		Between Laboratories <sup>B</sup>	
		$S_r$	$r$	$S_R$	$R$
F-4	-104	2.5	7.1	37.2	105.2
D-4	18	18.0	51.0	21.8	61.7
B-4	15	0.0	0.0	21.2	60.0
A-4	11	9.0	25.5	37.7	106.6
Average	-10	...	...	...	...
Pooled values	...	10.2	28.7	30.5	86.4

<sup>A</sup>This is short-term precision (days) with outliers removed from the data set.

<sup>B</sup>Symbols are defined as follows:

$S_r$  = within-laboratory standard deviation,

$r$  = repeatability (in measurement units),

$S_R$  = standard deviation for total between-laboratory variability, and

$R$  = reproducibility (in measurement units).

$S_r$  = within-laboratory standard deviation,

Two single test results (or determinations) produced in separate laboratories that differ by more than 86.4 % must be considered suspect, that is, that they represent different sample populations. Such a decision dictates that appropriate investigative or technical or commercial actions, or both, be taken.

8.6 *Bias*—In test method terminology, bias is the difference between an average test value and the reference (true) test property value. Reference values do not exist for this test method since the value or level of the test property is

exclusively defined by the test method. Bias, therefore, cannot be determined.

## 9. Keywords

9.1 carbon black in NR; evaluation; mixing procedure; preparation and testing of vulcanizates; recipe; standard formula

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