



Designation: D 3190 – 9500

Standard Test Method for Rubber—Evaluation of Chloroprene Rubber (CR)¹

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1. Scope

1.1 This test method covers the standard materials, test formulas, mixing procedures and test methods for evaluation and quality control testing of chloroprene rubbers (CR). CR can be generally classified according to the two types of polymerization modifiers used in their manufacture: sulfur modified types and mercaptan modified types.

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:*

¹ This test method is under the jurisdiction of ASTM Committee D-11 on Rubber and is the direct responsibility of Subcommittee D11.23 on Synthetic Rubbers. Current edition approved Feb. 15, 1995; May 10, 2000. Published April 1995; July 2000. Originally published as D 3190 – 73. Last previous edition D 3190 – 94~~5~~.

- D 412 Test Methods for Vulcanized Rubber and Thermoplastic Rubbers and Thermoplastic Elastomers—Tension²
- D 1646 Test Method for Rubber—Viscosity and Vulcanization Characteristics (Mooney Viscometer)²
- D 2084 Test Method for Rubber Property—Vulcanization Characteristics Using Oscillating Disc Cure Meter²
- D 3182 Practice for Rubber—Materials, Equipment and Procedures for Mixing Standard Compounds and Preparing Standard Vulcanized Sheets²
- D 3896 Practice for Rubber from Synthetic Sources— Sampling²
- D 4483 Practice for Determining Precision for Test Method Standards in the Rubber and Carbon Black Industries²
- D 5289 Test Method for Rubber Property – Vulcanization using Rotorless Cure Meters²

3. Summary of Test Method

3.1 Separate standard gum or carbon black filled formulations, or both, using different samples of CR, are prepared on a laboratory mill or in a Miniature Internal Mixer (MIM) in accordance with Practice D 3182.

3.2 See Test Method D 1646 for Mooney scorch times and Test Method D 2084 or D 5289 for cure meter data of the formulations.

3.3 Tensile sheets for stress/strain testing are vulcanized in accordance with Practice D 3182 and 100 and 300 % modulus, tensile strength and elongation at break are measured in accordance with Test Method D 412.

4. Significance and Use

4.1 This test method is intended for referee purposes, but may be used for quality control, research and development testing and comparison of different rubber samples in a standard formula.

4.2 This test method may also be used to obtain values for customer acceptance of rubber.

4.3 This test method is a revision of Test Methods D 3190 (prior to 1991), with 3-methyl thiazolidine thione-2 being substituted for ethylene thiourea (ETU), a suspected carcinogen.

5. Standard Test Formulae

5.1 The Standard Test Formulae are shown in Table 1. Formulae Nos. 1 and 2 shall be used for sulfur modified CR and Nos. 3 and 4 for mercaptan modified CR.

6. Sample Preparation

6.1 Obtain and prepare the test samples in accordance with Practice D 3896.

7. Mixing Procedure

7.1 The following three mixing procedures are offered:

7.1.1 *Mill Method A*— For Formulae Nos. 1 and 2.

7.1.2 *Mill Method B*— For Formulae Nos. 3 and 4.

7.1.3 *Miniature Internal Mixer Method*—For Formulae Nos. 1, 2, 3 and 4.

7.1.4 *Laboratory Banbury*—All Formulae

NOTE 1—It is not implied that comparable results will be obtained by these methods.

NOTE 2—The indicated mill openings are desired and should be maintained in so far as possible to provide a standard for breakdown of the rubber due to milling.

7.2 *Raw Rubber Preparation:*

7.2.1 With the mill roll temperature maintained at $50 \pm 5^\circ\text{C}$ ($122 \pm 9^\circ\text{F}$), set the mill opening at 1.5 mm (0.060 in.) and band 320 g of CR on the slow roll for 6 min. Adjust the mill opening to maintain a rolling bank approximately 12.5 mm (0.5 in.) in diameter.

7.2.2 Remove the rubber from the mill, allow to cool to room temperature and weigh an amount equal to 300 g prior to mixing.

7.3 *Mill Method A*— Procedure for Formulae Nos. 1 and 2 (sulfur modified CR):

7.3.1 For general mixing, weighing and vulcanization procedures, refer to Practice D 3182.

² Annual Book of ASTM Standards, Vol. 09.01.

TABLE 1 Standard Test Formulae

Formula	1	2	3	4
Chloroprene rubber				
Sulfur modified	100.00	100.00
Mercaptan modified	100.00	100.00
Stearic acid, SRM 372 ^A	-0.50	-0.50
Stearic acid, SRM 372 ^{A,B}	0.50	0.50
Magnesium oxide ^{A,B}	-4.00	-4.00	-4.00	-4.00
Magnesium oxide ^{A,B,C}	4.00	4.00	4.00	4.00
IRB No. 6	...	25.00	...	25.00
Zinc oxide, IRM 91 ^A	-5.00	-5.00	-5.00	-5.00
Zinc oxide, IRM 91 ^{A,B}	5.00	5.00	5.00	5.00
3-methyl thiazolidine- thione-2-80 % in polymeric- binder ^C (curative)	-0.45	-0.45
3-methyl thiazolidine- thione-2-80 % in polymeric binder ^D (curative)	0.45	0.45
Total	109.50	134.50	109.45	134.45
Batch factors ^D				
Batch factors ^E				
Laboratory mill	3.00	3.00	3.00	3.00
MIM (Cam Head)	0.76	0.63	0.76	0.63
MIM (Banbury Head)	0.65	0.54	0.65	0.54

^AFor the MIM procedure it is recommended that a blend of the identified ingredients be prepared to improve accuracy in the weighing. The blend is made by mixing a proportional mass of each material in a dry powder blender (see Practice D 3182). A mortar and pestle may be used for small quantities.

^BMaglite D, available from Marine Magnesium Company, 995 Beaver Creek Road, Coraopolis, PA 15064M/SRM.

^CRhenogran MTT 80 Maglite D, available from Rhein Chemie Magnesium Corporation, 1008 Whitehead Rd. Ext., Trenton, NJ 08638 or Rhein Chemie Rheinau GmbH, Mülheimer Str. 24-28, D68 1500 Mannheim 861, Germany.

^DRhenogran MTT 80, available from Rhein Chemie Corporation, 1008 Whitehead Rd. Ext., Trenton, NJ 08638 or Rhein Chemie Rheinau GmbH, Mülheimer Str. 24-28, D6800 Mannheim 81, Germany.

^EFor mill mixes, weigh the rubber and carbon black to the nearest 1.0 g, ingredients identified with footnote^A to the nearest 0.1 g, and the curative^C to the nearest 0.001 g. For MIM mixes weigh the rubber, carbon black and ingredient blend to the nearest 0.1 g, individual ingredients, if used, to the nearest 0.01 g and the curative^C to the nearest 0.001 g. Note that the curative tolerances given are lower than those specified in the referenced Practice D 3182.

7.3.2 Mixing Cycle:

Maintain roll temperatures at 50 ± 5°C (122 ± 9°F).

7.3.2.1 Set the mill opening at about 1.5 mm (0.060 in.), band 300 g of rubber prepared in 7.2 and maintain a rolling bank.

7.3.2.2 Add stearic acid.

7.3.2.3 Add magnesium oxide slowly, spreading it evenly over the entire width of the band. Ensure complete addition before adding the next material.

7.3.2.4 Add carbon black. Open the mill at intervals to maintain a rolling bank.

7.3.2.5 Add zinc oxide.

7.3.2.6 Make three three-quarter cuts from alternate sides and cut stock from the mill.

7.3.2.7 Set the rolls at 0.8 mm (0.032 in.).

Pass the rolled stock endwise through the mill six times.

Total time

	Gum	Duration min	Black
	1		1
	1		1
	2		2
	...		5
	2		2
	2		2
	2		2
	10		15

7.3.1.1 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.

7.3.2.9 Check and record the stock mass. If it differs more than 0.5 % from the total mass, corrected for the batch factor as specified in Table 1, for the carbon black stock and 0.3 % for the gum stock, discard the batch.

7.3.2.10 Cut a specimen to allow testing for scorch time in accordance with Test Method D 1646. The test should be performed between 1 to 2 h after mixing using a test temperature of 125 ± 1°C (257 ± 1.8°F) for a rise of 5 Mooney units above the minimum with the large rotor. If desired, measure the vulcanization characteristics in accordance with Test Method D 2084, conditioning the

specimen for 1 to 24 h at $23 \pm 3^\circ\text{C}$ ($73.4 \pm 5.4^\circ\text{F}$) before testing.

7.3.2.11 If stress/strain testing is required, sheet the stock from a mill at a setting to give a finished thickness of approximately 2.2 mm (0.085 in.) by passing the folded stock between the mill rolls set at $50 \pm 5^\circ\text{C}$ ($122 \pm 9^\circ\text{F}$) four times, always in the same direction to obtain the grain effect in the mill direction. Cool the sheeted compound on a flat, dry metal surface.

7.3.2.12 For routine laboratory testing, condition the sheeted compound for 1 to 24 h at $23 \pm 3^\circ\text{C}$ ($73.4 \pm 5.4^\circ\text{F}$) and 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 $35 \pm 5\%$ relative humidity.

7.4 Mill Method B— Procedure for Formulae Nos. 3 and 4 (mercaptan modified CR):

7.4.1 For general mixing, weighing and vulcanization procedures, refer to Practice D 3182.

7.4.2 Mixing Cycle:

Maintain roll temperatures at $50 \pm 5^\circ\text{C}$ ($122 \pm 9^\circ\text{F}$).

7.4.2.1 Set the mill opening at about 1.5 mm (0.060 in.), band 300 g of rubber prepared in 7.2 and maintain a rolling bank.
 7.4.2.2 Add magnesium oxide slowly, spreading it evenly over entire width of the band. Ensure complete addition before adding the next material.
 7.4.2.3 Add carbon black. Open the mill at intervals to maintain a rolling bank.
 7.4.2.4 Add zinc oxide.
 7.4.2.5 Add curative (see Table 1, footnote²).
 7.4.2.6 Make three three-quarter cuts from alternate sides and cut stock from the mill.
 7.4.2.7 Set the rolls at 0.8 mm (0.032 in.). Pass the rolled stock endwise through the mill six times.

	Gum	Duration min	Black
7.4.2.1	1		1
7.4.2.2	2		2
7.4.2.3	...		5
7.4.2.4	2		2
7.4.2.5	1		1
7.4.2.6	2		2
7.4.2.7	2		2
Total time	10		15

7.4.2.8 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.

7.4.2.9 Check and record the stock mass. If it differs more than 0.5 % from the total mass, corrected for the batch factor as specified in Table 1, for the carbon black stock and 0.3 % for the gum stock, discard the batch.

7.4.2.10 Cut a specimen to allow testing for scorch time in accordance with Test Method D 1646. The test should be performed between 1 to 2 h after mixing, using a test temperature of $125 \pm 1^\circ\text{C}$ ($257 \pm 1.8^\circ\text{F}$) for a rise of 5 Mooney units above the minimum with the large rotor. If desired, measure the vulcanization characteristics in accordance with Test Method D 2084, conditioning the specimen for 1 to 24 h at $23 \pm 3^\circ\text{C}$ ($73.4 \pm 5.4^\circ\text{F}$) before testing.

7.4.2.11 If stress/strain testing is required, sheet the stock from a mill at a setting to give a finished gage of approximately 2.2 mm (0.085 in.) by passing the folded stock between the mill rolls set at $50 \pm 5^\circ\text{C}$ ($122 \pm 9^\circ\text{F}$) four times, always in the same direction to obtain the grain effect in the mill direction. Cool the sheeted compound on a flat, dry metal surface.

7.4.2.12 For routine laboratory testing, condition the sheeted compound for 1 to 24 h at $23 \pm 3^\circ\text{C}$ ($73.4 \pm 5.4^\circ\text{F}$) and 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 $35 \pm 5\%$ relative humidity.

7.5 Miniature Internal Mixer (MIM)—Procedure for Formulae Nos. 1, 2, 3 and 4 (sulfur and mercaptan modified CR):

7.5.1 For general mixing, weighing and vulcanization procedures, refer to Practice D 3182.

7.5.1.1 Mix with the head temperature of the MIM maintained at $60 \pm 3^\circ\text{C}$ ($140 \pm 5^\circ\text{F}$) and a rotor speed at 6.3 to 6.6 rad/s (60 to 63 r/min).

7.5.2 *Raw rubber preparation*—Batch factors: Cam head 0.80; Banbury head 0.68:

7.5.2.1 Cut the rubber into small pieces, weigh the appropriate amount and charge it into the mixing chamber. Lower the ram, start the timer and masticate the rubber for 6 min.

7.5.2.2 Turn off the rotors, raise the ram, remove the mixing chamber and discharge the rubber.

7.5.2.3 Cut the rubber into small pieces, allow to cool to room temperature and weigh prior to mixing.

7.5.3 Mixing cycle:

	Gum	Duration min	Black
7.5.3.1 Charge the mixing chamber with the rubber prepared in 7.5.2, lower the ram and start the timer.	0		0
7.5.3.2 Masticate the rubber.	1		1
7.5.3.3 Raise the ram, add the pre-blended powders and curative (see Table 1, footnote ²), taking care to avoid losses. Sweep the orifice and lower the ram.	2		1

7.5.3.4 Raise the ram, add carbon black, lower the ram and allow batch to mix.

...

7

Total time

3

9

7.5.3.5 Turn off the rotors, raise the ram, remove the mixing chamber and discharge the batch. Record the maximum batch temperature, if desired.

7.5.3.6 Pass the batch through a laboratory mill with the roll temperatures set at $50 \pm 5^\circ\text{C}$ ($122 \pm 9^\circ\text{F}$) and 0.5 mm (0.020 in.) mill opening once, then twice at 3 mm (0.125 in.) opening.

7.5.3.7 Check and record the batch mass. If it differs more than 0.5 % from the total mass, corrected for the batch factor as specified in Table 1, for the carbon black stock and 0.3 % for the gum stock, discard the batch.

7.5.3.8 Cut a specimen for testing vulcanization characteristics in accordance with Test Method D 2084. Condition the specimen for 1 to 24 h at $23 \pm 3^\circ\text{C}$ ($73.4 \pm 5.4^\circ\text{F}$) before testing.

7.5.3.9 If either compound scorch or stress/strain testing, or both, are required, pass the rolled stock endwise through the mill six times with the rolls set at 0.8 mm (0.032 in.) and a temperature setting of $50 \pm 5^\circ\text{C}$ ($122 \pm 9^\circ\text{F}$).

7.5.3.10 Cut a specimen to allow testing of compound scorch according to Test Method D 1646. The test should be performed 1 to 2 h after mixing using a test temperature of $125 \pm 1^\circ\text{C}$ ($257 \pm 1.8^\circ\text{F}$) and reporting scorch time to a 5-Mooney unit rise above the minimum with the large rotor.

7.5.3.11 If stress/strain testing is required, sheet the stock from a mill at a setting to give a finished thickness of approximately 2.2 mm (0.085 in.) by passing the folded stock between the mill rolls set at $50 \pm 5^\circ\text{C}$ ($122 \pm 9^\circ\text{C}$) four times, always in the same direction to obtain the grain effect in the mill direction. Cool the sheeted compound on a flat, dry metal surface.

7.5.3.12 For routine laboratory testing, condition the sheeted compound for 1 to 24 h at $23 \pm 3^\circ\text{C}$ ($73.4 \pm 5.4^\circ\text{F}$) and 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 $35 \pm 5\%$ relative humidity.

7.6 Internal Mixer Procedure:

7.6.1 For general mixing procedure refer to Method D 3182.

7.6.2 Mixing Cycle-Initial Mix:

	<u>Duration, Min</u>	<u>Accumula- tive, Min</u>
<u>7.6.2.1</u> Adjust the internal mixer temperature to achieve the discharge conditions outlined in 7.6.2.5. Close the discharge gate, start the rotor at 8.1 rad/s (77 rpm) and raise the ram.	<u>0</u>	<u>0</u>
<u>7.6.2.2</u> Charge one half the rubber, all of the zinc oxide, carbon black, stearic acid, and then the other one half of the rubber. Lower the ram.	<u>.5</u> <u>3.0</u>	<u>.5</u> <u>3.5</u>
<u>7.6.2.3</u> Allow the batch to mix.	<u>.5</u>	<u>4.0</u>
<u>7.6.2.4</u> Raise the ram and clean the mixer throat and the top of the ram. Lower the ram.		
<u>7.6.2.5</u> Allow the batch to mix until a temperature of 170°C (338°F) or a total mixing time of 6 min is reached, whichever occurs first. Discharge the batch.	<u>2.0</u>	<u>6.0</u>

7.6.2.6 Determine and record the batch mass; if the mass differs by more than 0.5 % of the theoretical mass, discard the batch.

7.6.2.7 Pass the batch immediately through the standard laboratory mill three times, set at 6.0 mm (0.25 in.) and $40 \pm 5^\circ\text{C}$ ($104 \pm 9^\circ\text{F}$).

7.6.2.8 Allow the batch to rest for 1 to 24 h.

7.6.3 Final Mix:

7.6.3.1 Adjust the internal mixer temperature to $40 \pm 5^\circ\text{C}$ ($104 \pm 9^\circ\text{F}$), turn off steam and turn on full cooling water to the rotors, start the rotors at 8.1 rad/s (77 rpm), and raise the ram.

Duration,
Min

Accumula-
tive, Min

<u>7.6.3.2</u>	Charge ½ the batch, with all the sulfur and accelerator rolled into this portion of the batch before feeding to the mixer. Add the remaining portion of the batch. Lower the ram	<u>.5</u>	<u>.5</u>
<u>7.6.3.3</u>	Allow the batch to mix until a temperature of $110 \pm 5^{\circ}\text{C}$ ($230 \pm 9^{\circ}\text{F}$) or a total mixing time of 3 min is reached, whichever occurs first. Discharge the batch.	<u>2.5</u>	<u>3.0</u>
<u>7.6.3.4</u>	Determine and record the batch mass; if the mass differs by more than 0.5 % of the theoretical mass, discard it.		
<u>7.6.3.5</u>	With the rolls of a standard laboratory mill maintained at $40 \pm 5^{\circ}\text{C}$ ($104 \pm 9^{\circ}\text{F}$) and set at 0.8 mm (0.032 in.) opening, pass the rolled batch endwise through the rolls six times.	<u>2.0</u>	<u>5.0</u>
<u>7.6.3.6</u>	Open the rolls to give a minimum thickness of 6 mm (0.25 in.) and pass the compound through four times, folding it back on itself each time.	<u>1.0</u>	<u>6.0</u>

7.6.3.7 Cut enough sample for testing of compound viscosity in accordance with Test Method D 1646 or vulcanizing characteristics in accordance with Test Method D 2084 or D 5289. Condition the specimen for 1 to 24 h at $23 \pm 3^{\circ}\text{C}$ ($73.4 \pm 5.4^{\circ}\text{F}$) before testing.

7.6.3.8 If tensile stress is required, sheet off the compound from the mill at a setting to give a finished gage of approximately 2.2 mm (0.085 in.) by passing the folded stock between the rolls set at $50 \pm 5^{\circ}\text{C}$ ($122 \pm 9^{\circ}\text{F}$) four times always in the same direction to obtain the effects of mill direction. Cool on a flat, dry metal surface.

7.6.3.9 For routine laboratory testing, condition the sheeted compound for 1 to 24 h at $23 \pm 3^{\circ}\text{C}$ ($73.4 \pm 5.4^{\circ}\text{F}$) and 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 $35 \pm 5\%$ relative humidity.

8. Preparation and Testing of Vulcanizates

8.1 For stress/strain testing, prepare the test sheets and vulcanize them in accordance with Practice D 3182.

8.1.1 The recommended standard vulcanization time and temperature is 15 min at 160°C (320°F).

8.1.2 Condition the vulcanized sheets for 16 to 96 h at a temperature of $23 \pm 3^{\circ}\text{C}$ ($73.4 \pm 5.4^{\circ}\text{F}$) prior to testing.

NOTE 3—Quality control of rubber production may require testing within 1 to 6 h to provide close surveillance, however, slightly different results may be obtained.

8.1.3 Prepare test specimens and obtain modulus, tensile strength and elongation at break in accordance with Test Method D 412.

8.2 An alternative to measuring ~~stress/strain properties~~ vulcanization characteristics by means of tensile stress measurement on vulcanizates is the measurement of vulcanization ~~p~~ characteristics with an Oscillating Disk cure meters in accordance with Test Method D 2084 or a Rotorless Cure Meter in accordance with Test Method D 5289. These methods will not produce equal results.

8.2.1 The recommended standard Oscillating Disk test conditions are: 1.7 Hz (~~100 cpm~~) oscillation frequency, $1 \pm 0.03^{\circ}$ frequency; $\pm 1^{\circ}$ amplitude of oscillation, ~~$160 \pm 0.3^{\circ}\text{C}$ ($320 \pm 0.5^{\circ}\text{F}$)~~ 160°C die temperature, 30-min test time, and no preheating. The recommended test conditions for the Rotorless Cure Meter are: 1.7 Hz oscillation frequency, $\pm 0.5^{\circ}$ of arc for torsional shear cure meters and $\pm 0.05\text{mm}$ for linear shear cure meters, 160°C die temperature, 30 min test time, and no preheating. Tolerances for the listed conditions are included in the specified test methods.

8.2.2 The recommended standard test parameters ~~are MH, MHF or MHR, as appropriate, ML, t_{s1}~~ are: M_L , M_H , t_{s1} , t'_{50} and t'_{90} .

NOTE 4—It is recommended that MH, if applicable, be taken as the torque value at 30 min.

9. Report

9.1 Report the following information:

9.1.1 Proper sample identification.

9.1.2 Mixing procedure used (mill or MIM).

9.1.3 Vulcanization characteristics including Mooney scorch time (ML t_5 at 125°C (257°F)) and ~~oscillating disk~~ cure meter

results (MH, MHF or MHR, ML, ts1 and t'90 at 160°C (320°F)).

9.1.4 Stress/strain data for 15 min cure at 160°C (320°F) (100 % and 300 % modulus, tensile strength and elongation at break).

9.1.5 Identification of any deviation from the specified standard procedures used in mixing, specimen preparation and testing.

10. Precision and Bias ³

10.1 This precision and bias section has been prepared in accordance with Practice D 4483 for terminology and other statistical calculation details.

10.2 The precision results in this precision and bias section give an estimate of the precision of these test methods with the materials (rubbers) used in the particular interlaboratory program as described below. The precision parameters should not be used for acceptance/rejection testing of any group of materials without documentation that they are applicable to those particular materials and the specific testing protocols that include this test method.

10.3 A Type 2 interlaboratory precision program was conducted using the mill mix procedure. Both repeatability and reproducibility are short term; a period of a few days separates replicate test results. A test result is a value, as specified by this test method, obtained for one determination (measurement) of the selected property.

10.4 Two different types of CR were evaluated for precision; sulfur and mercaptan modified CR. Each CR was tested in eight laboratories on two different days. On each of the two days, duplicate tests were obtained. The analysis for precision followed the general procedure as set forth in Annex 5 of Practice D 4483. Each cell of the Practice D 4483 Table 1 basic data format, contained four values (two test days, two test results each day). The estimates for the repeatability parameters therefore contain two undifferentiated sources of variation; replicates within days and between days. The final precision parameters are given in Table 2 of this test method.

10.5 The precision of these test methods may be expressed in the format of the following statements that use an “appropriate value” of r , R , (r) or (R), to be used in decisions about the test results. The appropriate value is that value of r or R associated with a mean level in Table 2 closest to the mean level under consideration at any given time, for any given test and for any given material in routine testing operations.

10.6 *Repeatability*— The repeatability, r , of these test methods has been established as the appropriate value tabulated in Table 2. Two single test results, obtained under normal test method procedures in the same laboratory, that differ by more than this tabulated r (for any given level) must be considered as derived from different populations.

10.7 *Reproducibility*— The reproducibility, R , of these test methods has been established as the appropriate value tabulated in Table 2. Two single test results obtained in two different laboratories, under normal test method procedures, that differ by more than the tabulated R (for any given level) must be considered to have come from different populations.

10.8 Repeatability and reproducibility expressed in percentage of the mean level, (r) and (R), have equivalent application statements as above for r and R . For the (r) and (R) statements, the difference in two single test results is expressed as a percentage of the arithmetic mean of the two test results.

10.9 *Bias*—In test terminology, bias is the difference between an average test value and the reference (or true) test property value. Reference values do not exist for these test methods, since the value (of the test property) is exclusively defined by the test methods. Therefore, bias cannot be determined.

³ Supporting data are available from ASTM Headquarters. Request RR: D11-1073.

TABLE 2 Type 2 Precision, Vulcanization Parameters and Stress/Strain Properties of CR

NOTE 1— S_r = within laboratory standard deviation; r = repeatability (in measured units); (r) = repeatability (as percent of material average); S_R = between laboratory standard deviation; R = reproducibility (in measurement units); (R) = reproducibility (as percent of material average).

Property	Mean	Within Laboratory			Between Laboratory		
		S_r	r	(r)	S_R	R	(R)
CR-Sulfur Grade							
<i>Formula No. 1 (gum):</i>							
ML #5 (min)	29.7	2.31	6.47	21.8	3.83	10.73	36.2
ML (dNm)	3.5	0.19	0.54	15.6	0.77	2.15	61.8
MH (dNm)	32.9	0.55	1.55	4.7	1.72	4.83	14.7
ts1 (min)	3.9	0.16	0.44	11.4	0.43	1.20	31.3
t'90 (min)	9.2	0.24	0.66	7.2	1.88	5.27	57.4
100 % M (MPa)	1.2	0.04	0.10	8.5	0.07	0.19	15.6
300 % M (MPa)	2.2	0.07	0.21	9.6	0.11	0.32	14.8
Tensile (MPa)	26.1	1.13	3.16	12.1	1.75	4.89	18.7
Elongation (%)	875	13.89	38.90	4.4	27.30	76.44	8.7
<i>Formula No. 2 (black):</i>							
ML #5 (min)	19.4	3.72	10.43	53.8	3.72	10.43	53.8
ML (dNm)	5.7	0.28	0.80	14.0	1.16	3.24	56.7
MH (dNm)	53.9	1.03	2.87	5.3	2.97	8.32	15.4
ts1 (min)	2.1	0.22	0.61	28.6	0.51	1.43	66.7
t'90 (min)	8.6	0.52	1.45	16.8	1.36	3.81	44.1
100 % M (MPa)	3.0	0.10	0.27	9.0	0.17	0.48	16.1
300 % M (MPa)	11.8	0.41	1.15	9.8	0.60	1.67	14.2
Tensile (MPa)	26.1	0.77	2.15	8.3	1.66	4.65	17.8
Elongation (%)	597	16.65	46.62	7.8	32.00	89.60	15.0
CR-Mercaptan Grade							
<i>Formula No. 3 (gum):</i>							
ML #5 (min)	26.7	3.16	8.84	33.1	5.37	15.03	56.3
ML (dNm)	4.5	0.18	0.50	11.2	0.90	2.51	55.7
MH (dNm)	26.6	0.55	1.55	5.8	1.00	2.80	10.5
ts1 (min)	4.3	0.37	1.02	23.8	0.58	1.63	37.8
t'90 (min)	10.7	1.37	3.85	36.1	2.15	6.01	56.4
100 % M (MPa)	0.9	0.02	0.07	7.3	0.05	0.14	15.3
300 % M (MPa)	1.7	0.07	0.20	12.2	0.12	0.33	20.2
Tensile (MPa)	17.2	1.35	3.78	21.9	1.98	5.55	32.2
Elongation (%)	780	25.89	72.51	9.3	28.84	80.76	10.4
<i>Formula No. 4 (black):</i>							
ML #5 (min)	11.4	0.80	2.24	19.6	2.29	6.41	56.2
ML (dNm)	7.6	0.27	0.77	10.1	1.02	2.87	37.9
MH (dNm)	47.5	0.69	1.93	4.1	3.31	9.27	19.5
ts1 (min)	2.2	0.10	0.28	12.9	0.32	0.89	41.1
t'90 (min)	10.7	0.87	2.43	24.6	2.47	6.91	69.8
100 % M (MPa)	2.6	0.12	0.34	13.2	0.24	0.67	25.6
300 % M (MPa)	14.5	0.69	1.94	13.4	1.18	3.31	22.8
Tensile (MPa)	24.3	1.24	3.48	14.2	1.51	4.23	17.4
Elongation (%)	441	23.58	66.03	15.0	34.17	95.67	21.7

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