Designation: D 3568 – 03

Standard Test Methods for Rubber—Evaluation of EPDM (Ethylene Propylene Diene Terpolymers) Including Mixtures With Oil[1]

This standard is issued under the fixed designation D 3568; 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 specify the standard materials, test formulas, mixing procedures, and test methods for the evaluation and production control of ethylene propylene diene rubbers (EPDM).

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]

D 88 Test Method for Saybolt Viscosity

D 412 Test Methods for Vulcanized Rubber and Thermoplastic Elastomers— Tension

D 1646 Test Methods for Rubber—Viscosity, Stress Relaxation, and Pre-Vucanization Characteristics (Mooney Viscometer)

D 2084 Test Method for Rubber Property—Vulcanization Using Oscillating Disk Cure Meter

D 2161 Practice for Conversion of Kinematic Viscosity to Saybolt Universal Viscosity or to Saybolt Furol Viscosity

D 2501 Test Method for Calculation of Viscosity-Gravity Constant (VGC) of Petroleum Oils

D 3182 Practice for Rubber—Materials, Equipment, and Procedures for Mixing Standard Compounds and Preparing Standard Vulcanized Sheets

D 3896 Practice for Rubber from Synthetic Sources—Sampling

D 3900 Test Methods for Rubber Raw—Determination of Ethylene Units in EPM (Ethylene-Propylene Copolymers) and EPDM (Ethylene-Propylene-Diene Terpolymers)

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

D 5289 Test Method for Rubber Property–Vulcanization Using Rotorless Cure Meters

D 6204 Test Method for Rubber–Measurement of Unvulcanized Rheological Properties Using Rotorless Shear Rheometers

3. Significance and Use

3.1 These test methods are intended mainly for referee purposes but may be used for quality control of rubber production. They may also be used in research and development work and for comparison of different rubber samples in a standard formula.

3.2 These test methods may be used to obtain values for customer acceptance of rubber.

4. Standard Test Formulas

4.1 *Standard Formulas:*[3,4]

Material Formula No.	NIST SRM/IRM No.	Quality—Parts by Mass		
		1	2	3
EPDM	...	100.00	100.00	100.00 + Y^A
Zinc oxide[B]	IRM 91	5.00	5.00	5.00
Sulfur[B]	371	1.50	1.50	1.50
Stearic acid[B]	372	1.00	1.00	1.00
Oil furnace black[CD]	SRB-B4	80.00	100.00	80.00
ASTM Type 103 petroleum oil[DE]	...	50.00	75.00	50.00 − Y^A
Tetramethylthiuram disulfide (TMTD)[B]	IRM 1[F]	1.00	1.00	1.00
Mercaptobenzothiazole (MBT)[B]	383	0.50	0.50	0.50
Total		239.00	284.00	239.00
Batch factor[G]				
Mill		2.0	2.0	2.0
Internal mixer		5.5	4.2	5.5
MIM (Cam Head)		0.29	0.25	0.23–0.29[H]
MIM (Banbury Head)		0.25	0.21	0.20–0.26[H]

[A] *Y* = partsoil by mass per 100 parts base polymer in masterbatch. If *Y* is greater than 50 parts oil, do not add oil to Formula 3.

[B] For the MIM procedure, it is recommended that a blend of compounding materials be prepared to improve accuracy in the weighing of these materials. This

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[1] These test methods are under the jurisdiction of ASTM Committee D11 on Rubber and are the direct responsibility of Subcommittee D11.23 on Synthetic Rubbers.

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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.

[3] The Waring blender has been found useful for this blending.

[4] The Whip mixer available from Hobart Corp., Troy, OH 45374, has been found satisfactory for this purpose.

"compounding material blend" is prepared by blending a proportional mass of each material, except carbon black and oil, in a dry powder blender such as a biconical blender or vee blender. A mortar and pestile or blender may be used for blending small quantities.[3]

[C] The current Industry Reference Black may be used in place of NIST 378, although slightly different results may be obtained.

[D] For the MIM procedure, it is recommended that a blend of carbon black and oil be prepared to improve accuracy in the weighing and addition of these materials. This "black/oil blend" is prepared by adding a proportional mass of oil to a cavity prepared in a proportional mass of carbon black, covering the oil with the surrounding black and then gently whipping until uniformly blended. Let stand 24 h and blend again before using.[4]

[E] Viscosity is 16.8 ± 1.2 mm^2/s at 100°C in accordance with Test Method D 88 and Practice D 2161. Viscosity gravity constant is 0.889± 0.002 in accordance with Test Method D 2501. Available in 1 and 5-gal (3.8 and 19-dm^3) quantities from Sun Oil Co. and is distributed by R. E. Carroll, P.O. Box 139, Trenton, NJ 08601.

[F] NIST has discontinued supply of SRM 374. A substitute material is available as IRM 1 from Forcoven Products, Inc., P.O. Box 1556, Humble, TX 77338. Reference to research reports RR D04-1034 are available from ASTM Headquarters.

[G] For mill and internal mixer batches, weigh the rubber, carbon black and oil to the nearest 1.0 g, the sulfur and accelerators to the nearest 0.02 g, and all other compounding materials to the nearest 0.1 g. For MIM batches, weigh the rubber and compounding material blend to the nearest 0.01 g and individual compounding material, if used, to the nearest 0.001 g.

[H] Calculate a batch factor to the nearest 0.01, which will provide a 75 % loading of the mixing chamber.

4.1.1 Formula 1 shall apply to the general purpose EPDM types.

4.1.2 Formula 2 shall apply to the EPDM terpolymers with an ethylene content greater than approximately 67 % by mass (which describes high green-strength extrusion grade rubbers). For determining ethylene content, refer to Test Methods D 3900.

4.1.3 Formula 3 shall apply to oil extended EPDM rubbers.

5. Sample Preparation

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

6. Mixing Procedures

6.1 Four mixing procedures are offered:

6.1.1 *Internal Mixer Procedure*,

6.1.2 *Miniature Internal Mixing Procedure*,

6.1.3 *Mill Procedure*, and

6.1.4 *Internal Mixer with Final Curative Addition on Mill Procedure*.

NOTE 1—The mill handling characteristics of the EPDM rubber are somewhat more difficult than for most other rubbers; therefore, mixing is accomplished more easily in an internal mixer. Results from mill mixing in some instances may not correlate with results obtained from internal mixer procedures. Unless it is certain that good carbon black dispersion will be obtained by the mill mixing procedure with the rubber under test, the internal mixer procedures shall be used.

6.2 *Internal Mixer Procedure*:

6.2.1 For general mixing procedure, refer to Practice D 3182.

6.2.2 *Initial Mix Cycle*:

	Duration, min	Accumulative, min
Adjust the internal mixer temperature to achieve the discharge conditions outlined in 6.2.2.4. Close the discharge gate, start the rotors at 8.0 rad/s (77 r/min), and raise the ram.	0	0
Charge the rubber, zinc oxide, carbon black, oil, and stearic acid. Lower the ram, start the timer, and allow the batch to mix.	3.0	3.0
Raise the ram and clean the mixer throat and the top of the ram. Lower the ram.	0.5	3.5
Allow the batch to mix until a temperature of 150°C (302°F) or a total mixing time of 5 min is reached, whichever occurs first. Discharge the batch.	1.5	5.0
Immediately pass the batch through a standard laboratory mill, set at 50 ± 5°C (122 ± 9°F) with an opening of 2.5 mm (0.10 in.).		
Check the batch mass and record. If it differs from the theoretical value by more than 0.5 %, discard the batch.		
Cool the masterbatch for 30 min or until it is at room temperature.		
Final Mix Cycle:		
Adjust the chamber and rotors to 40 ± 5°C (104 ± 9°F). Close the discharge gate, start the rotors at 8 rad/s (77 r/min), and raise the ram.	0	0
Charge one half of the batch, accelerators and sulfur, and the remaining portion of the batch. Lower the ram.	0.5	0.5
Allow the batch to mix until a temperature of 110°C (230°F) or a total mixing time of 2 min is reached, whichever occurs first. Discharge the batch.	1.5 max	2.0 max
Check the batch mass and record. If it differs from the theoretical value by more than 0.5 %, discard the batch. Cut enough sample from the batch to allow testing of compound viscosity in accordance with Test Methods D 1646 or vulcanization characteristics in accordance with Test Method D 2084, or both, if these are desired.		
If stress-strain testing is to be conducted, sheet off the stock from the mill at a setting to give a finished thickness of approximately 2.2 mm (0.085 in.). Cool on a flat, dry metal surface.		
For routine laboratory testing, condition the sheeted compound for 1 to 24 h at 23 ± 3°C (73.4± 5.4°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 ± 5 % relative humidity.		
Miniature Internal Mixer (MIM) Procedure:		
For general mixing procedure, refer to Practice D 3182. Mix with the head temperature of the miniature internal mixer maintained at 60 ± 3°C (140 ± 5°F) and rotor speed at 6.3 to 6.6 rad/s (60 to 63 r/min).		
Mixing Cycle:		
Prepare the rubber by passing it through a mill one time with the temperature set at 50 ± 5°C (122 ± 9°F) and an opening that would give an approximately 5 mm (0.2 in.) thick sheet. Cut into strips that are approximately 25 mm (1 in.) wide.		
Charge the mixing chamber with the rubber strips, lower the ram, and start the timer.	0	0
Masticate the rubber.	1.0	1.0
Raise the ram and add the zinc oxide, sulfur, stearic acid, TMTD, and MBT that have previously been blended, taking care to avoid any loss. Sweep the orifice and lower the ram.	1.0	2.0
Raise the ram and add the carbon black/oil blend, lower the ram and allow the batch to mix.	7.0	9.0

6.2.2.1 Turn off the motor, raise the ram, remove the mixing chamber, and discharge the batch. Record the maximum batch temperature indicated, if desired.

6.2.2.2 Pass the batch through the mill set at 50 ± 5°C (122 ± 9°F) and 0.5 mm (0.020 in.), opening once, then twice at 3 mm (0.125 in.) opening.

6.2.2.3 Check the batch mass and record. If it differs from the theoretical value by more than 0.5 %, discard the batch.

6.2.2.4 Cut a specimen for testing of vulcanization characteristics in accordance with Test Method D 2084 or D 5289 as described in 7.1, if required. Condition the specimen for 1 to 24 h at 23 ± 3°C (73.4 ± 5.4°F) before testing.

6.2.2.5 If either compounded viscosity or stress-strain test, or both are required, pass the rolled stock endwise through the mill six times with the mill rolls set at 50 ± 5°C (122 ± 9°F) and 0.8 mm (0.032 in.) opening.

6.2.2.6 Cut a specimen to allow testing of compounded viscosity in accordance with Test Methods D 1646 if required.

6.2.2.7 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 mill rolls set at 50 ± 5°C (122 ± 9°F) four times, always in the same direction to obtain the effects of mill direction. Cool on a flat, dry metal surface.

6.2.2.8 For routine laboratory testing, condition the sheeted compound for 1 to 24 h at 23 ± 3°C (73.4 ± 5.4°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 ± 5 % relative humidity.

6.3 *Mill Mix Procedure* (Note 1):

6.3.1 For general mixing procedure, refer to Practice D 3182.

6.3.2 *Mixing Cycle*:

NOTE 2—The indicated mill openings are desired and should be maintained in so far as possible to provide a good rolling bank at the nip of the rolls during mixing.

NOTE 3—Mix the zinc oxide, carbon black, stearic acid, and oil together before starting to mill the mix.

	Duration, min	Accumulative, min
Band the rubber on the fast roll with the mill set at 23 ± 5°C (73 ± 9°F) and 0.8 mm (0.032 in.) opening.	1.0	1.0
Add the mixture of zinc oxide, carbon black, stearic acid, and oil evenly across the mill with a spatula. When about half of the mixture is incorporated, open the mill to 1.3 mm (0.05 in.). Make one ¾ cut from each side, then add the remainder of the mixture. When all the mixture has been incorporated, make three ¾ cuts from each side. 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.	13.0	14.0
Add the accelerators and sulfur evenly across the rolls still set at 1.3 mm (0.05 in.) opening.	3.0	17.0
Make three ¾ cuts from each side, allowing 15 s between each cut.	2.0	19.0
Cut the stock from the mill. Set the mill opening at 0.8 mm (0.032 in.) and pass the rolled stock through the opening endwise six times.	2.0	21.0

6.3.2.1 Check the batch mass and record. If it differs from the theoretical value by more than 0.5 %, discard the batch.

6.3.2.2 Cut a specimen to allow testing of compound viscosity in accordance with Test Methods D 1646, or D 6204, or vulcanization characteristics in accordance with Test Method D 2084, or D 5289, if these are desired.

6.3.2.3 If stress-strain testing is to be conducted, sheet off the stock from the mill at a setting to give a finished thickness of approximately 2.2 mm (0.085 in.). Cool on a flat, dry metal surface.

6.3.2.4 For routine laboratory testing, condition the sheeted compound for 1 to 24 h at 23 ± 3°C (73.4 ± 5.4°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 ± 5 % relative humidity.

6.4 *Internal Mixer with Final Curative Addition on Mill Procedure*:

6.4.1 For general mixing procedure, refer to Practice D 3182.

6.4.2 *Initial Mix Cycle in Internal Mixer*:

6.4.2.1 Follow the mix cycle stated in 6.2.2.1 to 6.2.2.7.

6.4.3 *Final Mix Cycle on Mill*:

6.4.3.1 The mill batch mass shall be based on twice the formula mass.

	Duration, min	Accumulative, min
Set the mill temperature at 50± 5°C (122 ± 9°F) with a mill opening of 1.5 mm (0.06 in.). Band the masterbatch on the fast roll and add the sulfur and accelerators evenly across the rolls.	1.0	1.0
Make three |n^ cuts from each side, allowing 15 s between each cut.	2.0	3.0
Cut the batch from the mill. Set the mill opening to 0.8 mm (0.032 in.) and pass the rolled stock through the opening endwise six times.	2.0	5.0

6.4.3.2 Check the batch mass and record. If it differs from the theoretical value by more than 0.5 %, discard the batch. Cut enough sample from the batch to allow testing of compound viscosity in accordance with Test Method D 1646, or D 6204, or vulcanization characteristics in accordance with Test Method D 2084, or D 5289, if these are desired.

6.4.3.3 If stress-strain testing is to be conducted, sheet off the stock from the mill at a setting to give a finished thickness of approximately 2.2 mm (0.085 in.). Cool on a flat dry metal surface.

6.4.3.4 For routine laboratory testing, condition the sheeted compound for 1 to 24 h at 23 ± 3°C (73.4 ± 5.4°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 ± 5 % relative humidity.

7. Preparation and Testing of Vulcanizates

7.1 The measurement of vulcanization parameters is carried out in accordance with Test Method D 2084 or Test Method D 5289.

7.1.1 When using D 2084, the recommended standard test conditions are 1.7 Hz (100 cpm) oscillation frequency, 1 ± 0.03° amplitude of oscillation, 160 ± 0.3°C (320 ± 0.5°F) die temperature, and no preheat.

7.1.2 When using D 5289, the recommended standard test conditions are 1.7Hz (100 cpm) oscillation frequency, 0.5 ±

TABLE 1 Type 2 Precision for Various Test Parameters

NOTE 1—

Sr = Repeatability standard deviation, in measurement units,
R = Repeatability, in measurement units,
(r) = Repeatability, (relative) percent,
SR = Reproducibility standard deviation, in measurement units,
R = Reproducibility, in measurement units, and
(R) = Reproducibility, (relative) percent.

Property	Units	Range of Values[A]	Within Laboratory			Between Laboratories		
			Sr	*r*	(*r*)	*SR*	*R*	(*R*)
M_L	dN·m	6.7 to 12.4	0.50	1.42	14.8	1.24	3.51	36.6
M_H	dN·m	32.7 to 46.9	1.29	3.65	9.2	3.66	10.4	26.1
T_{s1}	min	2.2 to 2.7	0.11	0.31	12.4	0.38	1.08	43.2
t'_{90}	min	12.6 to 15.6	0.64	1.81	12.8	1.20	3.40	24.1

[A] For Test Method D 2084: 160°C, 1.7 Hz, 1° amplitude arc—midpoint of range used for (*r*) (*R*) calculations.

0.003° amplitude of oscillation, 160 ± 0.3° C (320 ± 0.5°F) die temperature, and no preheat.

7.1.3 The recommended standard test parameters are M_H, M_{HF}, or M_{HR}, as appropriate, M_L, t_{s1}, and t' 90.

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

7.2 An alternative to measuring vulcanization characteristics by the oscillating disk cure meter is the use of stress-strain testing.

7.2.1 For stress-strain testing, prepare test sheets and vulcanize them in accordance with Practice D 3182.

7.2.2 The recommended standard vulcanization times for all EPDM rubbers except the dicyclopentadiene (DCPD) types are 5, 10, and 15 min at 160°C (320°F). For the mill-mixed and miniature internal mixer compounds, it is 10 min at 160°C. For DCPD type, the recommended standard times are 15, 25, and 35 min at 160°C. For the mill-mixed miniature internal mixer compounds, 25 min at 160°C.

7.2.3 Condition the cured sheets for 16 to 96 h at a temperature of 23 ± 2°C (73.4 ± 3.6°F).

NOTE 5—Quality control of rubber production may require testing within 1 to 6 h to provide close surveillance. However, slightly different results may be obtained.

7.2.4 Obtain modulus, tensile, and elongation parameters in accordance with Test Methods D 412.

8. Precision and Bias [5]

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

8.2 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 these test methods.

8.3 A Type 2 (interlaboratory) precision was evaluated. Both repeatability and reproducibility are short term; a period of a few days separates replicate test results. A test result is the value, as specified by these test methods, obtained on determination(s) or measurement(s) of the property or parameter in question.

8.4 Three different materials (EPDM rubbers) were used in the interlaboratory program; these were tested in eight laboratories on two different days in an internal mixture procedure. The results of the precision calculations for repeatability and reproducibility are given in Table 1.

8.5 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 test results. The appropriate value is that value of *r* or *R* associated with a mean level in Table 1 closest to the mean level under consideration at any given time, for any given material in routine testing operations.

8.6 *Repeatability*—The repeatability, *r*, of these test methods has been established as the appropriate value tabulated in Table 1. Two single test results, obtained under normal test method procedures, that differ by more than this tabulated *r* (for any given level) must be considered as derived from different or nonidentical sample populations.

8.7 *Reproducibility*—The reproducibility, *R*, of these test methods has been established as the appropriate value tabulated in Table 1. 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 or nonidentical sample populations.

8.8 Repeatability and reproducibility expressed as a 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 the two single test results is expressed as a percentage of the arithmetic mean of the two test results.

8.9 *Bias*—In test method terminology, bias is the difference between an average test value and the reference (or true) test

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[5] Supporting data are available from ASTM Headquarters. Request RR: D11–1026.

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. Bias therefore cannot be determined.

9. Keywords

9.1 EPDM; mixture with oil

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