



Designation: D3568 – 03

Standard Test Methods for Rubber—Evaluation of EPDM (Ethylene Propylene Diene Terpolymers) Including Mixtures With Oil¹

This standard is issued under the fixed designation D3568; 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

D88 Test Method for Saybolt Viscosity

D412 Test Methods for Vulcanized Rubber and Thermoplastic Elastomers—Tension

D1646 Test Methods for Rubber—Viscosity, Stress Relaxation, and Pre-Vulcanization Characteristics (Mooney Viscometer)

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

D2161 Practice for Conversion of Kinematic Viscosity to Saybolt Universal Viscosity or to Saybolt Furol Viscosity

D2501 Test Method for Calculation of Viscosity-Gravity Constant (VGC) of Petroleum Oils

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

D3896 Practice for Rubber From Synthetic Sources—Sampling

D3900 Test Methods for Rubber—Determination of Ethylene Units in Ethylene-Propylene Copolymers (EPM) and in Ethylene-Propylene-Diene Terpolymers (EPDM) by Infrared Spectrometry

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

D5289 Test Method for Rubber Property—Vulcanization Using Rotorless Cure Meters

D6204 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

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.

^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 "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 pestle 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 $16.8 \pm 1.2 \text{ mm}^2/\text{s}$ at 100°C in accordance with Test Method **D88** and Practice **D2161**. Viscosity gravity constant is 0.889 ± 0.002 in accordance with Test Method **D2501**. Available in 1 and 5-gal (3.8 and 19-dm³) 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 **D3900**.

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 **D3896**.

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 **D3182**.

6.2.2 *Initial Mix Cycle*:

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.

Charge the rubber, zinc oxide, carbon black, oil, and stearic acid. Lower the ram, start the timer, and allow the batch to mix.

Raise the ram and clean the mixer throat and the top of the ram. Lower the ram.

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.

Immediately pass the batch through a standard laboratory mill, set at $50 \pm 5^\circ\text{C}$ ($122 \pm 9^\circ\text{F}$) 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 $40 \pm 5^\circ\text{C}$ ($104 \pm 9^\circ\text{F}$). Close the discharge gate, start the rotors at 8 rad/s (77 r/min), and raise the ram.

Charge one half of the batch, accelerators and sulfur, and the remaining portion of the batch. Lower the ram.

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.

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 **D1646** or vulcanization characteristics in accordance with Test Method **D2084**, 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 $23 \pm 3^\circ\text{C}$ ($73.4 \pm 5.4^\circ\text{F}$) 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 ± 5 % relative humidity.

Miniature Internal Mixer (MIM) Procedure:

For general mixing procedure, refer to Practice **D3182**. Mix with the head temperature of the miniature internal mixer maintained at $60 \pm 3^\circ\text{C}$ ($140 \pm 5^\circ\text{F}$) 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 $50 \pm 5^\circ\text{C}$ ($122 \pm 9^\circ\text{F}$) 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.

Masticate the rubber.

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.

Duration, min	Accumulative, min
0	0
3.0	3.0
0.5	3.5
1.5	5.0
0	0
0.5	0.5
1.5 max	2.0 max
0	0
1.0	1.0
1.0	2.0