



Designation: D 3186 – 97 (Reapproved 2002)

Standard Test Methods for Rubber—Evaluation of SBR (Styrene-Butadiene Rubber) Mixed With Carbon Black or Carbon Black and Oil¹

This standard is issued under the fixed designation D 3186; 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 formula, mixing procedures, and test methods for the evaluation and production control of pigmented types of styrene-butadiene rubbers (SBR). This includes the pigmented SBR oil masterbatches.

1.2 The values stated in SI units are to be regarded as the standard. The values given 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 Elastomers—Tension²
- D 1646 Test Methods for Rubber—Viscosity, Stress Relaxation, and Pre-Vulcanization Characteristics (Mooney Viscometer)²
- D 2084 Test Method for Rubber Property—Vulcanization Using Oscillating Disk 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

3. Significance and Use

3.1 These test methods are intended mainly for referee purposes but may be used for quality control of masterbatch

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 Formula

4.1 Standard Formula:

Material	NIST SRM/IRM No.	Quantity, Parts by Mass
Masterbatch	...	$100 + X^A + Y^B$
Zinc oxide ^{C,D}	IRM 91	3.00
Sulfur ^{C,D}	371	1.75
Stearic acid ^{C,D}	372	1.50
TBBS ^{D,E}	384	1.25
Total		$107.50 + X + Y$
Batch factor ^F		

^A X = parts carbon black per 100 parts base polymer.

^B Y = parts oil per 100 parts base polymer.

^CFor the MIM procedure, it is recommended that a blend of compounding materials be prepared to improve accuracy of the weighing of these materials. This material blend is prepared by blending a proportional mass of each material in a dry powder blender such as a biconical blender or vee blender. A mortar and pestle may be used for blending small quantities.

^DFor mill mixes, weigh the rubber to the nearest 1.0 g, the sulfur and the accelerator to the nearest 0.02 g, and all of the other compounding materials to the nearest 0.1 g. For MIM mixes, weigh the rubber and material blend to the nearest 0.01 g and individual pigments, if used, to the nearest 0.001 g.

^ETBBS is *N-tert-butyl-2-benzothiazolesulfenamide*.

^FFor mill mixes, a batch factor should be selected to the nearest 0.5 to give as large a total mass as possible that will not exceed 525.0 g. Calculate all parts to the nearest 0.01 part. For MIM mixes, calculate a batch factor to the nearest 0.01 that will provide a 75 % loading of the mixing chamber.

5. Sample Preparation

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

6. Mixing Procedures

6.1 The compound may be prepared either on a mill, miniature internal mixer, or laboratory internal mixer, although slightly different results may be obtained.

6.2 Mill Procedure:

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

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 *Annual Book of ASTM Standards*, Vol 09.01.

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6.2.1.1 Mix with the mill roll temperature maintained at $50 \pm 5^\circ\text{C}$ ($122 \pm 9^\circ\text{F}$). The indicated mill openings are desired and should be maintained as closely as possible to provide uniformity in the breakdown of the rubber due to milling.

6.2.2 *Mixing Cycle:*

6.2.2.1

Set the mill opening at 1.40 mm (0.055 in.), and band the masterbatch on the slow roll without cutting.

6.2.2.2

Add sulfur slowly and evenly across the mill at a uniform rate.

6.2.2.3

Add stearic acid. Make one $\frac{3}{4}$ cut from each side after the stearic acid has been incorporated.

6.2.2.4

Add zinc oxide and accelerator.

6.2.2.5

Make three $\frac{3}{4}$ cuts from each side and cut the batch from mill.

6.2.2.6

Set the rolls at 0.8 mm (0.032 in.). Pass the rolled stock endwise through the mill six times.

6.2.2.7

Open the mill to give a minimum stock thickness of 6 mm (0.25 in.) and pass the compound through the mill four times, folding it back on itself each time.

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

6.2.2.9 Cut a specimen to allow testing of compound viscosity in accordance with Test Methods D 1646 or vulcanizing characteristics in accordance with Test Method D 2084 as described in 7.2, or both, if these are desired.

6.2.2.10 Sheet off the compound from the mill at a setting to give a finished gage of approximately 2.2 mm (0.085 in.). Cool on a flat, dry, metal surface.

6.2.2.11 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.

6.3 *Miniature Internal Mixer (MIM) Procedure:*

6.3.1 For general mixing procedures refer to Practice D 3182.

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

6.3.2 Prepare the masterbatch 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 will give an approximately 5-mm (0.2-in.) thick sheet. Cut into strips approximately 25 mm (1 in.) wide.

6.3.3 *Mixing Cycle:*

6.3.3.1

Charge the mixing chamber with the masterbatch strips, lower the ram, and start the timer.

6.3.3.2

Masticate the masterbatch.

6.3.3.3

Raise the ram, and add zinc oxide, sulfur, stearic acid, and TBBS that have previously been blended, taking care to avoid any loss. Sweep the orifice and lower the ram.

6.3.3.4

Allow the batch to mix.

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

6.3.3.6 Pass the batch through a mill 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.

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

6.3.3.8 Cut a specimen for testing vulcanization characteristics in accordance with Test Method D 2084, as described in 8.2, if required. 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.

6.3.3.9 If either compound-viscosity or stress-strain testing is required, pass the rolled stock endwise through the mill six times with the rolls set at 0.8 mm (0.032 in.) and $50 \pm 5^\circ\text{C}$ ($122 \pm 9^\circ\text{F}$).

6.3.3.10 Cut a specimen to allow testing of compound viscosity in accordance with Test Methods D 1646, if required.

6.3.3.11 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 $50 \pm 5^\circ\text{C}$ ($122 \pm 9^\circ\text{F}$) four times, always in the same direction to obtain the effects of mill direction. Cool on a flat, dry, metal surface.

6.3.3.12 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.

6.4 *Internal Mixer Procedure:*

