Designation: D3186 – 07 (Reapproved 2021)

Standard Test Methods for Rubber—Evaluation of SBR (Styrene-Butadiene Rubber) Mixed With Carbon Black or Carbon Black and Oil[1]

This standard is issued under the fixed designation D3186; 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 cover 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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.*

1.4 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

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

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

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 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 also be used to obtain values for customer acceptance of rubber.

4. Standard Test Formula

4.1 *Standard Formula*—See Table 1.

5. Sample Preparation

5.1 Obtain and prepare the test samples in accordance with Practice D3896.

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 Mix Procedure:*

6.2.1 For general mixing procedures, refer to Practice D3182.

6.2.2 *Mill Mixing Cycle*—See Table 2.

6.2.2.1 After mixing according to Table 2, measure and record the batch mass. If it differs from the theoretical value by more than 0.5 %, discard the batch.

6.2.2.2 If required, cut samples from the batch to allow testing of compound viscosity and processability in accordance

[1] These test methods are under the jurisdiction of ASTM Committee D11 on Rubber and Rubber-like Materials 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.

TABLE 1 Standard Pigmented SBR Test Formula

Material	SRM/IRM No.[A]	Quantity, Parts by Mass
Masterbatch	...	100 + X^B + Y^C
Zinc oxide[D,E]	[A]	3.00
Sulfur[D,E]	[A]	1.75
Stearic acid[D, E]	[A]	1.50
TBBS[E,F]	[A]	1.25
Total		107.50 + X + Y
Batch factor[G]		

[A] Use current IRM/SRM.
[B] X = parts carbon black per 100 parts base polymer.
[C] Y = parts oil per 100 parts base polymer.
[D] For 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.
[E] For 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.
[F] TBBS is *N-tert*-butyl-2-benzothiazolesulfenamide.
[G] For 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.

TABLE 2 Mill Mixing Cycle

	Duration, min	Accumulative, min
Set the mill opening at 1.40 mm (0.056 in.) and band the masterbatch on the slow roll without cutting.	0	0
Add sulfur slowly and evenly across the mill at a uniform rate.	2	2
Add stearic acid. Make one ¾ cut from each side after the stearic acid has been incorporated.	2	4
Add zinc oxide and TBBS accelerator.	3	7
Make three ¾ cuts from each side and cut the batch from mill.	2	9
Set the rolls at 0.8 mm (0.032 in.). Pass the rolled stock endwise through the mill six times.	2	11
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.	1	12

with Test Methods D1646 or D6204, and vulcanization characteristics in accordance with Test Methods D2084 or D5289.

6.2.2.3 If tensile stress strain tests are required, sheet off to a finished thickness of approximately 2.2 mm (0.087 in.) and condition the compound according to Practice D3182.

6.3 *Miniature Internal Mixer (MIM) Procedure:*

6.3.1 For general mixing procedures refer to Practice D3182.

6.3.2 *MIM Mixing Cycle*—See Table 3.

6.3.2.1 After mixing according to Table 3, turn off the motor, raise the ram, remove the mixing chamber and discharge the batch. Record the maximum batch temperature indicated, if desired.

TABLE 3 Miniature Internal Mixer Cycle

	Duration, min	Accumulative, min
Charge the mixing chamber with the masterbatch strips, lower the ram, and start the timer.	0.0	0.0
Masticate the masterbatch.	0.5	0.5
Raise the ram, and add previously blended zinc oxide, sulfur, stearic acid, and TBBS, taking care to avoid any loss. Sweep the orifice and lower the ram.	0.5	1.0
Allow the batch to mix.	8.0	9.0

6.3.2.2 Immediately pass the discharge from the mixer twice through a standard mill maintained at 50 ± 5°C (122 ± 9°F) with a roll separation of 0.5 mm (0.020 in.) once, then twice at a separation of 3 mm (0.12 in.) in order to dissipate heat. Pass the rolled batch endwise through the mill six times with an opening of 0.8 mm (0.032 in.) to enhance the dispersion.

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

6.3.2.4 If required, cut samples from the batch to allow testing of compound viscosity and processability in accordance with Test Methods D1646 or D6204, and vulcanization characteristics in accordance with Test Methods D2084 or D5289.

6.3.2.5 If tensile stress strain tests are required, sheet off to a finished thickness of approximately 2.2 mm (0.087 in.) and condition the compound according to Practice D3182.

6.4 *Internal Mixer Procedure:*

6.4.1 For general mixing procedure refer to Practice D3182.

6.4.2 *Mixing Cycle-Initial Mix*—See Table 4.

6.4.2.1 After mixing according to Table 4, measure and record the batch mass. If it differs from the theoretical value by more than 0.5 %, discard the batch.

6.4.2.2 Pass the batch immediately through the standard laboratory mill three times, with a mill opening of 6.0 mm (0.25 in.) and roll temperature of 40 ± 5°C (104 ± 9°F).

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

6.4.3 *Final Mix*—See Table 5.

6.4.3.1 After mixing according to Table 5, measure and record the batch mass. If it differs from the theoretical value by more than 0.5 %, discard the batch.

6.4.3.2 If required, cut samples from the batch to allow testing of compound viscosity and processability in accordance with Test Methods D1646 or D6204, and vulcanization characteristics in accordance with Test Methods D2084 or D5289.

6.4.3.3 If tensile stress strain tests are required, sheet off to a finished thickness of approximately 2.2 mm (0.087 in.) and condition the compound according to Practice D3182.

7. Preparation and Testing of Vulcanizates

7.1 For stress-strain testing, prepare the test sheets and vulcanize them in accordance with Practice D3182.

7.1.1 The recommended standard cure times for the mill mixes are 25, 35, and 50 min at 145°C (293°F). The recommended cure time for the miniature internal mixer and the internal mixer compounds is 35 min at 145°C (293°F).