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 Oil[1]

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—Tension[2]

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 Meter[2]

D 3182 Practice for Rubber—Materials, Equipment, and Procedures for Mixing Standard Compounds and Preparing Standard Vulcanized Sheets[2]

D 3896 Practice for Rubber from Synthetic Sources—Sampling[2]

D 4483 Practice for Determining Precision for Test Method Standards in the Rubber and Carbon Black Industries[2]

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.

[C] 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.

[D] 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.

[E] TBBS is *N*- *tert*-butyl-2-benzothiazolesulfenamide.

[F] 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.

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.

Current edition approved Nov. 10, 1997. Published April 1998. Originally published as D 3186 – 81. Last previous edition D 3186 – 95.

[2] *Annual Book of ASTM Standards*, Vol 09.01.

6.2.1.1 Mix with the mill roll temperature maintained at 50 ± 5°C (122 ± 9°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

	Duration, min	Accumulative, min
Set the mill opening at 1.40 mm (0.055 in.), and band the masterbatch on the slow roll without cutting.	2	2

6.2.2.2

	Duration, min	Accumulative, min
Add sulfur slowly and evenly across the mill at a uniform rate.	2	4

6.2.2.3

	Duration, min	Accumulative, min
Add stearic acid. Make one ¾ cut from each side after the stearic acid has been incorporated.	2	6

6.2.2.4

	Duration, min	Accumulative, min
Add zinc oxide and accelerator.	3	9

6.2.2.5

	Duration, min	Accumulative, min
Make three ¾ cuts from each side and cut the batch from mill.	2	11

6.2.2.6

	Duration, min	Accumulative, min
Set the rolls at 0.8 mm (0.032 in.). Pass the rolled stock endwise through the mill six times.	2	13

6.2.2.7

	Duration, min	Accumulative, min
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	14

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 ± 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 *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 ± 3°C (140 ± 5°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 ± 5°C (122 ± 9°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

	Duration, min	Accumulative, min
Charge the mixing chamber with the masterbatch strips, lower the ram, and start the timer.	0.0	0.0

6.3.3.2

	Duration, min	Accumulative, min
Masticate the masterbatch.	0.5	0.5

6.3.3.3

	Duration, min	Accumulative, min
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.	0.5	1.0

6.3.3.4

	Duration, min	Accumulative, min
Allow the batch to mix.	8.0	9.0

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 ± 5°C (122 ± 9°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 ± 3°C (73.4 ± 5.4°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 ± 5°C (122 ± 9°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 ± 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.

6.3.3.12 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 Procedure*:

6.4.1 For general mixing procedure refer to Method D 3182.

6.4.2 *Mixing Cycle-Initial Mix*:

6.4.2.1

	Duration, min	Accumulative, min
Adjust the internal mixer temperature to achieve the discharge conditions outlined in 6.2.2.5. Close the discharge gate, start the rotor at 8.1 rad/s (77 rpm) and raise the ram.	0	0

6.4.2.2

	Duration, min	Accumulative, min
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.	.5	.5

6.4.2.3 Allow the batch to mix.

6.4.2.4

	Duration, min	Accumulative, min
Raise the ram and clean the mixer throat and the top of the ram.	3	3.5
Lower the ram.	.5	4

6.4.2.5

	Duration, min	Accumulative, min
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.	2	6

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

6.4.2.7

	Duration, min	Accumulative, min
Pass the batch immediately through the standard laboratory mill three times, set at 6.0 mm (0.25 in.) and 40 ± 5°C (104 ± 9°F).	1	7

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

6.4.3 *Final Mix*:

6.4.3.1

	Duration, min	Accumulative, min
Adjust the internal mixer temperature to 40 ± 5°C (104 ± 9°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.	0	0

6.4.3.2

	Duration, min	Accumulative, min
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	.5	.5

6.4.3.3

	Duration, min	Accumulative, min
Allow the batch to mix until a temperature of 110 ± 5°C (230 ± 9°F) or a total mixing time of 3 min is reached, whichever occurs first. Discharge the batch.	2.5	3

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

6.4.3.5

	Duration, min	Accumulative, min
With the rolls of a standard laboratory mill maintained at 40 ± 5°C (104 ± 9°F) and set at 0.8 mm (0.032 in.) opening, pass the rolled batch endwise through the rolls six times.	2	5

6.4.3.6

	Duration, min	Accumulative, min
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.	1	6

6.4.3.7 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, as described in 7.2, or both, if these are desired. Condition the specimen for 1 to 24 h at 23 ± 3°C (73.4 ± 5.4°F) before testing.

6.4.3.8 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± 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.

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

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 compound is 35 min at 145°C (293°F).

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

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

7.1.3 Prepare test specimens and obtain modulus, tensile and elongation in accordance with Test Method D 412.

7.2 An alternative to measuring vulcanization characteristics by means of tensile strength measurement on vulcanizates is the measurement of vulcanization parameters with a cure meter in accordance with Test Method D 2084.

7.2.1 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, 30 min test time and no preheating.

7.2.2 The recommended standard test parameters are M_H, M_{HF},or M_{HR}, as appropriate, and M_L, t_{S1}, t' 50 and t' 90 as determined in Test Method D 2084.

NOTE 2—It is recommended that M_H, if applicable, be taken as the torque value at 30 min.

TABLE 1 Type 2 Precision for Curemeter 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	6.4 to 8.2	0.17	0.48	6.6	0.92	2.60	35.6
M_H	dN·m	24.2 to 42.7	0.69	1.95	5.8	2.69	7.61	22.7
t_{s1}	min	5.8 to 6.8	0.19	0.54	8.6	0.89	2.52	40.0
t' 50	min	9.3 to 9.9	0.28	0.79	8.2	0.73	2.07	21.6
t' 90	min	15.0 to 15.1	0.40	1.13	7.5	0.86	2.43	16.2

[A] Measured at 160°C, 1.7 Hz, 1° amplitude arc—midpoint of range used for (*r*) and (*R*) calculations.

8. Precision and Bias

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 average value, as specified by these test methods, obtained on one determination(s) or measurement(s).

8.4 Two different materials (SBR rubbers) were used in the interlaboratory program; were tested in six laboratories on two different days, by the internal mixer method. The results of the precision calculations for repeatability and reproducibility are given in Table 1, as conducted according to Test Method D 2084.

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 test parameter, and 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 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 carbon black and oil; mixed with carbon black; SBR