Designation: D 3185 – 99

Standard Test Methods for Rubber—Evaluation of SBR (Styrene-Butadiene Rubber) Including Mixtures With Oil[1]

This standard is issued under the fixed designation D 3185; 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 formulas, mixing procedures, and test methods for the evaluation of emulsion polybutadiene (EBR) and styrene-butadiene rubbers (SBR) including the emulsion polybutadiene oil masterbatch rubbers, SBR oil masterbatches, and partially crosslinked and high Mooney rubbers.

1.2 These test methods are also applicable to certain types of solution SBR and solution SBR oil masterbatches that are used in applications requiring vulcanization.

1.3 The values stated in SI units are to be regarded as the standard. The values given in parentheses are for information only.

1.4 *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 Rubbers and Thermoplastic Elastomers— Tension[2]

D 1646 Test Method 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]

2.2 *ISO Standard:*

ISO 2322 Rubber, Styrene-Butadiene (SBR)—Emulsion-Polymerized General-Purpose Type—Test Recipe and Evaluation of Vulcanization Characteristics[3]

3. Summary of Test Methods

3.1 These test methods include the standard test formulas, mixing procedures, and test methods for evaluating the following types of synthetic rubbers:

3.1.1 *Non-Oil-Extended Types*, mill and Miniature Internal Mixer (MIM) mixing procedures.

3.1.2 *Oil Masterbatch Types*, mill and Miniature Internal Mixer (MIM) mixing procedures.

3.2 No correlation between results for mill and MIM mixing procedures is implied.

4. Significance and Use

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

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

5. Standard Test Formulas

5.1 *Standard Formulas for Non-Oil-Extended Types*:

Material	IRM-SRM No.	Quantity, Parts by Mass 1A	 2A
SBR or masterbatch	...	100.00	100.00
Zinc oxide	[A]	3.00	3.00
Sulfur	[A]	1.75	1.75
Stearic acid	[A]	1.00	1.00
Oil furnace black[B]	[A]	50.00	35.00
TBBS[C]	[A]	1.00	1.00
Total		156.75	141.75
Batch factor for mill mix[D]		3.0	3.3
Batch factor for MIM mix[E]:			
Cam head		0.47	0.49
Banbury head		0.41	0.46

[A] Use Current IRM/SRM.

[B] The current Industry Reference Black (IRB) which has been dried for 1 h at

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[1] These test methods are under the jurisdiction of ASTM Committee D-11 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.

[3] Available from American National Standards Institute, 11 W. 42nd St., 13th Floor, New York, NY 10036.

125 ± 3°C (257 ± 5°F) may be used in place of NBS 378, although slightly different results may be obtained.

[C] *N-tert*-butyl-2-benzothiazolesulfenamide.

[D] For mill batches, weigh the rubber and carbon black to the nearest 1.0 g, the sulfur and accelerator to the nearest 0.02 g, and the other compounding materials to the nearest 0.1 g.

[E] For MIM batches, weigh the rubber and carbon black to the nearest 0.1 g, the compounding material blend to the nearest 0.01 g, and the individual compounding materials, if used, to the nearest 0.001 g.

5.1.1 For the MIM procedure it is recommended that a blend of compounding materials, excluding carbon black, be prepared to improve accuracy in the weighing of these materials. The compounding 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.

5.1.2 Formula 1A shall apply to the general-purpose nonpigmented, non-oil-extended rubber types. It is also specified in International Standard ISO 2322 for evaluation of general purpose, nonpigmented, non-oil-extended rubbers. Formula 2A shall apply to the partially crosslinked and high-Mooney rubber (>90 ML 1 + 4 at 100°C) for example, SBR 1009, 1018, and 1012 types.

5.2 *Standard Formulas for Oil Masterbatch Types*:

5.2.1 Formulas 1B to 6B shall apply to the general purpose nonpigmented oil-extended rubber types according to the oil content of the rubber. Formula 1A may be used as an alternative to Formulas 1B to 6B. International Standard ISO 2322 specifies Formula 1A for evaluation of general purpose, nonpigmented, oil-extended SBR.

5.2.2 For the MIM procedure it is recommended that a blend of compounding materials, excluding carbon black, be prepared to improve accuracy in the weighing of these materials. The pigment 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.

Material	IRM-	Quantity, Parts by Mass					
Formula No.	SRM	1B	2B	3B	4B	5B	6B
Parts Oil	No.	25	37.5	50	62.5	75	Base
Masterbatch		125.00	137.50	150.00	162.50	175.00	100 + Y[A]
Zinc oxide	[B]	3.00	3.00	3.00	3.00	3.00	3.00
Sulfur	[B]	1.75	1.75	1.75	1.75	1.75	1.75
Stearic acid	[B]	1.00	1.00	1.00	1.00	1.00	1.00
Oil furnace black[C]	[B]	62.50	68.75	75.00	81.25	87.50	50 (100 + Y)0.01 1
TBBS[D]	384	1.25	1.38	1.50	1.63	1.75	1 (100 + Y)0.01 1
Total		194.50	213.38	232.25	251.13	270.00	
Batch factor for mill mix[E]		2.4	2.2	2.0	1.9	1.7	
Batch factor for MIM mix:							
Cam head[D]		0.37	0.34	0.31	0.29	0.27	
Banbury head[F]		0.328	0.298	0.273	0.252	0.234	

[A] Y = parts oil by mass per 100 parts base polymer in masterbatch.

[B] Use Current IRM/SRM.

[C] The current Industry Reference Black (IRB) which has been dried for 1 h at 125 ± 3°C (257 ± 5°F) may be used in place of NBS 378, although slightly different results may be obtained.

[D] *N-tert*-butyl-2-benzothiazolesulfenamide.

[E] For mill batches, weigh the rubber and carbon black to the nearest 1.0 g, the sulfur and accelerator to the nearest 0.02 g, and the other compounding materials to the nearest 0.1 g.

[F] For MIM batches, weigh the rubber and carbon black to the nearest 0.1 g, the compounding material blend to the nearest 0.01 g and the individual compounding materials, if used, to the nearest 0.001 g.

6. Sample Preparation

6.1 Obtain the test samples in accordance with Practice D 3896.

7. Mixing Procedures

7.1 Two mixing procedures are provided:

7.1.1 *Method A*—Mill mix (7.2).

7.1.2 *Method B*—Miniature internal mixer (MIM) mix (7.3).

NOTE 1—It is not implied that comparable results will be obtained by these test methods.

7.2 *Method A—Mill Mix Procedure*:

7.2.1 For general mixing procedures refer to Practice D 3182.

7.2.2 Mix with the mill roll temperature maintained at 50 ± 5°C (122 ± 9°F). The indicated mill openings are desired and should be maintained insofar as possible to provide uniformity in the breakdown of the rubber due to milling.

7.2.3 *Mixing Cycle*:

	Duration, min	Accumulative, min
7.2.3.1		
Set the mill opening at 1.15 mm (0.045 in.) and band the rubber on the slow roll. Make ¾ cuts every ½min from alternate sides.	7	7
7.2.3.2		
Add the sulfur slowly and evenly across the mill at a uniform rate.	2	9

7.2.3.3 Add the stearic acid. Make one ¾ cut from each side after the stearic acid has been incorporated.	2	11
7.2.3.4 Add the carbon black evenly across the mill at a uniform rate. When about half the black is incorporated, open the mill to 1.25 mm (0.050 in.) and make one ¾cut from each side. Then add the remainder of the carbon black. When all the black has been incorporated, open the mill to 1.40 mm (0.055 in.) and make one ¾cut from each side.	10	21

NOTE 2—Do not cut any stock while free carbon black is evident in the bank or on the milling surface. Be certain to return to the milling stock any ingredients that drop through the mill.

7.2.3.5 Add the other materials at the 1.40-mm (0.055-in.) setting.	3	24
7.2.3.6 Make three ¾ cuts from each side and cut the batch from the mill.	2	26
7.2.3.7 Set the rolls at 0.8 mm (0.032 in.). Pass the rolled batch endwise through the mill six times.	2	28

	Duration, min	Accumulative, min
7.2.3.8 Open the mill to give a minimum batch thickness of 6 mm (0.25 in.) and pass the stock through the mill four times, folding it back on itself each time.	1	29

7.2.3.9 Determine and record the batch mass. If it differs from the theoretical value by more than 0.5 % discard the batch.

7.2.3.10 Cut enough sample from the batch to allow testing of compound viscosity in accordance with Test Method D 1646 or vulcanizing characteristics in accordance with Test Method D 2084 as described in 8.2, or both, if these are desired.

7.2.3.11 Sheet off the batch 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.

7.2.3.12 For routine laboratory testing, condition the sheeted compound for 1 to 24 h at 23 ± 3°C (73 ± 5°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.

7.3 *Method B—Miniature Internal Mixer Procedure*:

7.3.1 For general mixing procedures refer to Practice D 3182. Mix with the head temperature of the miniature internal mixer maintained at 60 ± 3°C (140 ± 5°F) and the unloaded rotor speed at 6.3 to 6.6 rad/s (60 to 63 rpm).

7.3.2 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 of 0.5 mm (0.02 in.). Cut the sheet into strips that are approximately 25 mm (1 in.) wide, if desired.

7.3.3 *Mixing Cycle*:

	Duration, min	Accumulative, min
7.3.3.1 Charge the mixing chamber with the rubber strips, lower the ram, and start the timer.	0	0
7.3.3.2 Masticate the rubber.	1.0	1.0
7.3.3.3 Raise the ram and add all the zinc oxide, sulfur, stearic acid, and TBBS that have been previously blended, taking care to avoid any loss. Then add the carbon black, sweep the orifice, and lower the ram.	1.0	2.0
7.3.3.4 Allow the batch to mix.	7.0	9.0

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

7.3.3.6 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 (0.12-in.) opening.

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

7.3.3.8 Cut a specimen for testing vulcanization characteristics in accordance with 8.2 of Test Method D 2084, if required. Condition the specimen for 1 to 24 h at 23 ± 3°C (73 ± 5°F) before testing.

7.3.3.9 If either compound viscosity or stress-strain testing is required, pass the rolled batch endwise through the mill six times with the mill roll temperature at 50 ± 5°C (122 ± 9°F) and a roll separation of 0.8 mm (0.032 in.).

7.3.3.10 Cut a specimen to allow testing of compounded viscosity in accordance with Test Method D 1646, if required.

7.3.3.11 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.

7.3.3.12 For routine laboratory testing, condition the sheeted compound for 1 to 24 h at 23 ± 3°C (73 ± 5°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.

7.4 *Method C—Internal Mixer*:

7.4.1 For general mixing procedure refer to Practice D 3182.

7.4.2 *Mixing Cycle-Initial Mix*:

	Duration, min	Accumulative, min
7.4.2.1 Adjust the internal mixer temperature to achieve the discharge conditions outlined in 5.2. Close the discharge gate, start the rotor at 8.1 rad/s (77 rpm) and raise the ram.	0.5	0.5
7.4.2.2 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.	3.0	3.5
7.4.2.3 Allow the batch to mix.	.5	4.0
7.4.2.4 Raise the ram and clean the mixer throat and the top of the ram. Lower the ram.	2.0	6.0
7.4.2.5 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.	2.0	6.0

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

7.4.2.7 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).

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

7.4.3 Final Mix:

7.4.3.1 Adjust the internal mixer temperature to 40 ± 5°C (104 ± 9°F), turn off steam and turn on full cooling water to the rotors, at 8.1 rad/s (77 rpm), and raise the ram.

	Duration, min	Accumulative, min
7.4.3.2 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
7.4.3.3 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	3.0

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

7.4.3.5 With the rolls of a standard laboratory mill maintained at 40 ± 5°C (104 ± 9°F) and set at 0.8 mm (0.0032 in.) opening, pass the rolled batch endwise through the rolls six times.	2.0	5.0
7.4.3.6 Open the rolls to give a minimum thickness of 6 mm (0.25 in.) and pass the compound through four times, folding it back itself each time.	1.0	6.0

7.4.3.7 Cut enough samples 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. Conditions the specimen for 1 to 24 h at 23 ± 3°C (73.4 ± 5.4°F) before testing.

7.4.3.8 If tensile stress is required, sheet off the compound from the mill at a setting to give a finished 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.

7.4.3.9 Four 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.

8. Testing Procedures

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

8.1.1 The recommended standard vulcanization times for the mill mixed compound are 25, 35, and 50 min at 145°C (293°F). The recommended standard vulcanization time for the miniature internal mixer compound is 35 min at 145°C.

8.1.2 Condition the cured sheets for 16 to 96 h at a temperature of 23 ± 3°C (73 ± 5°F).

NOTE 3—Quality control of rubber production may require testing within 1 to 6 h to provide close surveillance of the plant operations; however, slightly different results may be obtained.

8.1.3 Prepare test specimens and obtain tensile stress, tensile strength, and elongation in accordance with Test Methods D 412.

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

8.2.1 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.

8.2.2 The recommended standard test parameters are M_L, M_H, t_{sl}, t′50 and t′90.

9. Precision and Bias

9.1 These precision statements have been prepared in accordance with Practice D 4483. Please refer to this practice for terminology and other testing and statistical concept explanations. The precision results in this precision and bias section give an estimate of the precision of the test method with the materials 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

TABLE 1 Type 2 Test Precision

NOTE 1—Symbols are defined as follows:
Sr = within laboratory standard deviation.
r = repeatability (in measured units).
(*r*) = repeatability (in percent).
SR = between laboratory standard deviation.
R = reproducibility (in measurements units).
(*R*) = reproducibility (in percent).

Property	Units	Range of Values	Within Laboratories			Among (Between) Laboratories		
			Sr	*r*	(*r*)^A	*SR*	*R*	(*R*)^A
Precision estimates for Test Method D 2084 at 160°C, 1.7 Hz, and 1° amplitude are:								
M_L	dN·m	9.7–9.8	0.21	0.594	6.1	0.52	1.47	15.2
M_H	dN·m	32.5–43.8	0.77	2.18	5.7	2.21	6.25	16.4
t_{s1}	min	3.5–4.9	0.13	0.368	8.8	0.67	1.90	45.2
t' 50	min	8.2–8.3	0.20	0.566	6.9	0.74	2.09	25.3
t' 90	min	13.6–16.4	0.48	1.36	9.1	1.12	3.17	21.1
Precision estimates for Test Methods D 412 at 145°C for 35 min are:								
300 % modulus, MPa		8.7–13.8	0.62	1.75	15.6	1.55	4.39	39.0
Tensile strength, MPa		21.9–25.9	0.83	2.35	9.8	1.40	3.96	16.6
Elongation, %		504–599	16.2	45.8	8.3	67.7	191.0	34.7
Precision estimates for Test Method D 1646 are:								
M_L 1 + 4 at 100°C		63.8–70.3	1.51	4.27	6.4	6.61	18.7	27.9

[A] Mid-point of range used to calculate (*r*) and (*R*).

without documentation that they are applicable to those particular materials and the specific testing protocols that include these test methods.

9.2 The Type 2 precision of these test methods was determined from an interlaboratory study of 2 rubbers, Type 1712 and Type 1502. Seven laboratories mixed and tested each rubber by the miniature internal mixer method on 2 days.

9.3 Table 1 gives the Basic Precision (BT) data as obtained in the interlaboratory program. The cure meter testing was conducted in accordance with Test Method D 2084. In Practice D 4483 terminology, Type 2 Precision includes as components of variation the mixing and curing operations within each laboratory.

9.4 A test result for cure meter testing (Test Method D 2084) represents one measurement with a cure meter.

9.5 A test result for tension testing (Test Methods D 412) is the median of 3 dumbbell specimens.

9.6 A test result for viscosity testing (Test Method D 1646) represents one measurement with a viscometer.

9.7 The precision of these test methods may be expressed in the format of the following statements which use what is called an "appropriate value" of *r, R, (r)*, or (*R*), that is, that value to be used in decisions about test results (obtained with the test method). 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.

9.8 *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.

9.9 *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.

9.10 Repeatability and reproducibility expressed as a percentage of the mean level, *(r)* and *(R)*, have equivalent application statements 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.

9.11 *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.

10. Keywords

10.1 SRB; Evaluation of Solution BR; Butadiene Rubber

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