Designation: D3484 – 06 (Reapproved 2022)

Standard Test Methods for Rubber—Evaluation of Oil-Extended Solution BR (Polybutadiene Rubber)[1]

This standard is issued under the fixed designation D3484; 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 and production control of oil-extended polybutadiene rubber (OE-BR) polymerized in an appropriate solution.

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

2.2 *ISO Standard:*

ISO 2476 Rubber, Butadiene (BR) Solution Polymerized Types—Test Recipe and Evaluation Characteristics[3]

3. Significance and Use

3.1 These tests are mainly intended for referee purposes but may also be used for quality control of rubber production. They may be used in research and development work 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 Formulas

4.1 *Standard Formulas*—See Table 1.

4.2 Formula 1 is written based on 100 parts of rubber while Formula 2 is written on the basis of 100 parts of the masterbatch. Either formula may be used, but these will not give the same results.

NOTE 1—Formula 2 is specified in ISO 2476 for oil extended BR.

5. Sample Preparation

5.1 For test intended for referee purposes obtain and prepare the test samples in accordance with Practice D3896.

6. Mixing Procedures

6.1 The following four mixing test methods are offered:

6.1.1 *Method A*—Internal Mixer Procedure (6.2),

6.1.2 *Method B*—Internal Mixer/Mill Procedure (6.3),

[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.

[3] Available from American National Standards Institute, 25 W. 43rd St., 4th Floor, New York, NY 10036.

TABLE 1 Standard BR Test Formulas

Material	IRM-SRM No.[A]	Quantity, Parts by Mass	
Formula No.		1	2
OE-BR	...	100.00 + Y^B	100.00
Zinc oxide	[A]	3.00	3.00
Sulfur	[A]	1.50	1.50
Stearic acid	[A]	2.00	2.00
Oil furnace black[C]	[A]	60.00 (100 + Y) 0.01	60.00
TBBS[D]	[A]	0.90 (100 + Y) 0.01	0.90
Total		167.40 + Y	167.40
Batch factor for mill mix[E]		4.0 – 0.036 Y	4.0
Batch Factor for Internal Mixer[E]		[1170 (1.020 + 0.00044 Y)/total formula parts]	7.7
Batch Factor for MIM Mix (Formula 1)[F]		[70 (1.020 + 0.00044 Y)/total formula parts]	
Batch Factor for MIM Mix (Cam Head)[F]			0.44
Batch Factor for MIM Mix (Banbury Head)[F]			0.38

[A] Use current IRM/SRM.
[B] Y = parts of oil by mass per 100 parts base polymer in masterbatch.
[C] The current Industry Reference Black (IRB) shall be used.
[D] TBBS is *N-tert*-butyl-2-benzothiazolesulfenamide.
[E] For mill and internal mixer batches, weigh the rubber and carbon black to the nearest 1.0 g, the sulfur and the TBBS accelerator to the nearest 0.02 g, and the other compounding materials to the nearest 0.1 g.
[F] For MIM mixes, 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. For the MIM procedure, it is recommended that a blend of compounding materials, excluding carbon black, be prepared to improve the 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.

6.1.3 *Method C*—Mill Procedure (6.4), and

6.1.4 *Method D*—Miniature Internal Mixer Procedure (6.5).

NOTE 2—It is not implied that comparable results will be obtained by these test methods.

NOTE 3—Since the mill handling characteristics of the solution polybutadiene rubbers are somewhat more difficult than that of other rubbers the use of one of the internal mixer procedures is recommended (Method A, B, or D). The mill procedure (Method C) may be used provided a good carbon black dispersion is obtained.

6.2 *Internal Mixer Initial Mix (Methods A, B):*

6.2.1 For general mixing procedure refer to Practice D3182.

6.2.2 *Internal Mixer Initial Mix*—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 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 70 ± 5°C (158 ± 9°F).

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

6.2.3 *Internal Mixer Final Mix (Method A)*—See Table 3.

6.2.3.1 After mixing according to Table 3, measure and record the batch mass. If it differs from the theoretical value by more than 0.5 %, discard the batch.

6.2.3.2 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.

6.2.3.3 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.

TABLE 2 Internal Mixer - Initial Cycle

	Duration, min	Accumulative, min
Adjust the internal mixer temperature to achieve the discharge conditions outlined below. Close the discharge gate, start the rotors at 8.1 rad/s (77 r/min), and raise the ram.	0.0	0.0
Charge one-half of the rubber, all of the zinc oxide, carbon black, stearic acid, and then the other one-half of the rubber. Lower the ram.	0.5	0.5
Allow the batch to mix.	3.0	3.5
Raise the ram, and clean the mixer throat and the top of the ram. Lower the ram.	0.5	4.0
Allow the batch to mix until a temperature of 170°C (338°F) or a total of 6 min is reached, whichever occurs first. Discharge the batch.	2.0	6.0

TABLE 3 Internal Mixer - Final Cycle

	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 r/min), and raise the ram.	0.0	0.0
Charge one-half of 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.	0.5	0.5
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
With the rolls of a standard laboratory mill maintained at 70 ± 5°C (158 ± 9°F), and set at 0.8 mm (0.032 in.) opening, pass the rolled batch endwise through the mill six times.	2.0	5.0
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.	1.0	6.0

6.3 *Mixing Cycle for Final Mill Mix after Internal Mixer Initial Mix (Method B):*

6.3.1 For general mixing procedures, refer to Practice D3182.

6.3.2 *Mixing Cycle for Mill Final Mix (Method B)*—See Table 4.

6.3.2.1 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.

6.3.2.2 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.

6.3.2.3 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.