Designation: D3484 – 06 (Reapproved 2016)

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 and health practices and determine the applicability of regulatory limitations prior to use.*

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),

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

[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 June 1, 2016. Published July 2016. Originally approved in 1976. Last previous edition approved in 2010 as D3484 – 06 (2010). DOI: 10.1520/D3484-06R16.

[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.

Copyright © ASTM International, 100 Barr Harbor Drive, PO Box C700, West Conshohocken, PA 19428-2959. United States

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.

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.

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

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.

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.

6.4 *Mill Mixing (Method C):*

6.4.1 For general mixing procedure, refer to Practice D3182.

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

TABLE 4 Mill Final Mixing Cycle

	Duration, min	Accumulative, min
Set the mill opening at 1.50 mm (0.059 in.) and temperature at 40 ± 5°C (104 ± 9°F), band the masterbatch on the slow roll.	0	0
Add the TBBS accelerator, taking care to avoid any loss. Sweep the mill pan and add until all the accelerator is in the batch. Make three ¾ cuts from each side.	3	3
Add the sulfur and that which falls into the mill pan. Make one ¾ cut from each side.	3	6
Cut the batch from the mill and set the mill roll opening at 0.8 mm (0.032 in.). Pass the rolled stock endwise through the mill six times.	2	8
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	9

6.4.2 *Mill Mixing Cycle*—See Table 5.

6.4.2.1 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.

6.4.2.2 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.

6.4.2.3 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.

6.5 *Miniature Internal Mixer (Method D):*

6.5.1 For general mixing procedures, refer to Practice D3182. Mix with the head temperature of the miniature internal mixer maintained at 60 ± 3°C (140 ± 5°F) and the unloaded rotor speed set at 6.3 to 6.6 rad/s (60 to 63 r/min).

6.5.2 Prepare the rubber by passing through a mill one time with the roll temperature set at 40 ± 5°C (104 ± 9°F) and an opening that will give a sheet approximately 0.5 mm (0.02 in.) thick. Cut the sheet into strips that are approximately 25 mm (1 in.) wide, if desired.

6.5.3 *MIM Mixing Cycle*—See Table 6.

6.5.3.1 After mixing according to Table 6, turn off the motor, raise the ram, remove the mixing chamber and discharge the batch. Record the maximum batch temperature indicated, if desired.

6.5.3.2 Immediately pass the discharge from the mixer twice through a standard mill maintained at 40 ± 5°C (104 ± 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.

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

6.5.3.4 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.

6.5.3.5 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.

7. Preparation and Testing of Vulcanizates

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

TABLE 5 Mill Mixing Cycle

NOTE 1—Do not cut any stock while free carbon black is evident in the bank or on the milling surface. Be certain to return any materials that drop through the mill to the milling stock.

	Duration, min	Accumulative, min
With the mill roll temperature set at 40 ± 5°C (104 ± 9°F) and the opening at 1.20 mm (0.047 in.), band the rubber on the slow roll.	0	0
Add the zinc oxide evenly across the rolls. Make two ¾ cuts from each side.	2	2
Add one-half of the carbon black evenly across the mill at a uniform rate. Make two ¾ cuts on each side and then add the stearic acid evenly across the rolls. Continue to make ¾ cuts from each side as necessary during the remaining time.	10	12
Open the rolls to 1.80 mm (0.071 in.) and add the remaining carbon black evenly across the rolls, making sure all materials in the pan are added to the batch. Make two ¾ cuts from each side.	12 to 16	24 to 28
Add the TBBS accelerator and sulfur, taking care to avoid any loss. Sweep the mill pan and add until all ingredients are in the batch.	2	26 to 30
Make three ¾ cuts from each side.	2	28 to 32
Cut the stock from the mill. Set the opening at 0.8 mm (0.032 in.). Pass the rolled stock endwise through the mill six times.	2	30 to 34
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	31 to 35

TABLE 6 Miniature Internal Mixer Cycle

	Duration, min	Accumulative, min
Charge the mixing chamber with the rubber strips, lower the ram, and start the timer.	0.0	0.0
Masticate the masterbatch.	0.5	0.5
Raise the ram and add the previously blended zinc oxide, sulfur, stearic acid, and TBBS, taking care to avoid any loss.	1.0	1.5
Add the carbon black, sweep the orifice, and lower the ram.	1.0	2.5
Allow the batch to mix, raising the ram momentarily to sweep down, if necessary.	6.5	9.0

7.1.1 The recommended standard vulcanization times for the compounds prepared by Methods A, B, and C are 25, 35, and 50 min at 145°C (293°F). The recommended standard vulcanization time for compounds prepared by Method D (MIM) 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 4—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.

7.1.3 Prepare test specimens and obtain the tensile stress, tension, and elongation in accordance with Test Methods D412.

7.2 An alternative to measuring vulcanization characteristics by means of tensile stress measurement on vulcanizates is the measurement of vulcanization characteristics in accordance with Test Method D2084 (Oscillating Disk Cure Meter Method) or Test Method D5289 (Rotorless Cure Meter Method). These methods will not produce equal results.

7.2.1 The recommended Test Method D2084 test conditions are 1.67 Hz (100 cpm) oscillation frequency, 1° oscillation amplitude, 160°C die temperature, 30-min test time, and no preheating. The recommended Test Method D5289 test conditions are 1.67 Hz (100 cpm) oscillation frequency, 0.5° oscillation amplitude, 160°C die temperature, 30-min test time, and no preheating. Test condition tolerances are specified by the test methods.

7.2.2 The recommended standard test parameters are M_L, M_H, t_S1, $t'50$ and $t'90$.

8. Precision and Bias[4]

8.1 The precision and bias section deals with test results obtained in an interlaboratory program organized according to Practice D4483. Refer to this practice for terminology and other statistical calculation details.

8.2 The precision results in this section give an estimate of the precision of this method with the materials used in the particular interlaboratory test program as described below. The precision parameters should not be used for acceptance or rejection testing of any group of materials without documentation that the parameters are applicable to the group of materials and the specific testing protocols of the test method.

8.3 A Class 3, Type 2 interlaboratory test program was conducted in 1996 with ten participating laboratories. One test run included weighing of ingredients, preparation of mill mixes (compounds), testing of compound Mooney viscosities, cure meter testing of compounds, vulcanization of compounds and stress/strain testing of the vulcanizates. Cure meter testing was conducted at 160°C (320°F) and 1.7 Hz (100 cpm) with a 1° arc for the oscillating disk cure meter and a 0.5° arc for the rotorless cure meter. All ingredients were distributed by one laboratory and two complete test runs were conducted with each of two different compound formulations. The two test runs were one week apart. The precision is given in terms of Sr, r, (r), SR, R, and (R) for the measured property values.

8.4 Analysis via Practice D4483 procedures indicated that there were both h and k outliers in the ten laboratory database. These outliers were eliminated and a subsequent analysis conducted with the final number of laboratories indicated in the precision table.

8.5 The precision of the test method may be expressed in the format of the following statements which use an "appropriate value" of r, R, (r), or (R), that is, that value to be used in decisions about test results obtained with this test method. The "appropriate value" is that value of r or R associated with the mean level in Table 7 closest to the mean level under consideration (at any given time, for any given material) in routine testing operations.

8.6 *Repeatability*—The repeatability r of this test method has been established as the appropriate value tabulated in Table 7. 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 non-identical sample populations.

8.7 *Reproducibility*—The reproducibility R of this test method has been established as the appropriate value tabulated in Table 7. 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 value) must be considered to have come from different or non-identical sample populations.

8.8 Repeatability and reproducibility expressed as percent of the mean level, (r) and (R), have equivalent application statements as above for the r and R statements, the difference in the two single test results being expressed as a percent of the arithmetic mean of the two test results.

8.9 In test terminology, bias is the difference between an average test value and the reference (or true) test property value. Reference values do not exist for this test method since the values are exclusively defined by this method. Bias, therefore, cannot be determined.

9. Keywords

9.1 evaluation; oil-extended br; polybutadiene rubber (br); standard formula

[4] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR: D11-1087.

TABLE 7 Type 2 Precision Results[A]

					Within Laboratory			Between Laboratory		
No. of Labs	Property	Formula	Mean	S_r	r	(r)	S_R	R	(R)
8	Mooney viscosity (ML1+4, 100°C)	#1	85	3.75	10.5	12.4	3.77	10.6	12.5
8		#2	84	3.57	10.0	12.0	3.70	10.4	12.5
	Cure meter (oscillating disk)								
8	ML (dNm)	#1	12.5	0.63	1.77	14.4	0.92	2.59	20.7
8		#2	12.2	0.50	1.41	11.5	0.91	2.55	20.9
8	MH (dNm)	#1	42.2	0.84	2.36	5.6	3.16	8.84	21.0
8		#2	44.5	1.05	2.94	6.6	3.73	10.40	23.5
9	ts1 (min)	#1	3.7	0.11	0.30	8.1	0.23	0.64	17.3
9		#2	3.5	0.23	0.64	18.1	0.30	0.84	23.8
9	t'50 (min)	#1	7.8	0.38	1.06	13.5	0.38	1.06	13.5
9		#2	7.5	0.27	0.75	10.0	0.28	0.79	10.5
9	t'90 (min)	#1	10.0	0.35	0.99	9.9	0.38	1.05	10.5
9		#2	9.7	0.27	0.75	7.7	0.37	1.05	10.8
	Cure meter (rotorless)								
7	ML (dNm)	#1	3.6	0.30	0.84	23.4	0.33	0.91	25.4
7		#2	3.5	0.18	0.51	14.7	0.28	0.79	22.8
7	MH (dNm)	#1	18.6	0.45	1.27	6.8	0.89	2.50	13.5
7		#2	20.4	0.73	2.03	10.0	1.25	3.51	17.2
7	ts1 (min)	#1	3.2	0.19	0.54	16.7	0.27	0.76	23.6
7		#2	3.1	0.11	0.32	10.3	0.29	0.82	26.5
7	t'50 (min)	#1	6.5	0.38	1.05	16.3	0.38	1.05	16.3
7		#2	6.2	0.17	0.47	7.8	0.20	0.55	8.9
7	t'90 (min)	#1	8.9	0.54	1.52	17.0	0.55	1.53	17.1
7		#2	8.6	0.11	0.32	3.7	0.42	1.16	13.5
	Stress/strain								
8	300 % modulus (MPa)	#1	10.8	0.65	1.81	16.7	0.65	1.81	16.7
9		#2	12.2	0.44	1.24	10.2	0.60	1.69	13.9
8	Tensile strength (MPa)	#1	17.6	0.83	2.32	13.2	1.13	3.16	18.0
9		#2	17.1	1.33	3.72	21.8	1.37	3.84	22.5
8	Ultimate elongation (%)	#1	462	21.2	59.2	12.8	34.6	96.8	21.0
9		#2	408	23.7	66.3	16.3	26.6	74.4	18.2

[A] Interlaboratory Test Program (ITP) [Day 1 - Day 2] replicates 1 week apart
Number of laboratories in ITP after outlier removal indicated in first column
S_r = repeatability standard deviation in measurement units
r = repeatability = 2.83 × S_r (in measurement units)
(r) = repeatability in percent of the mean
S_R = reproducibility standard deviation in measurement units
R = reproducibility = 2.83 × S_R (in measurement units)
(R) = reproducibility in percent of the mean

ASTM International takes no position respecting the validity of any patent rights asserted in connection with any item mentioned in this standard. Users of this standard are expressly advised that determination of the validity of any such patent rights, and the risk of infringement of such rights, are entirely their own responsibility.

This standard is subject to revision at any time by the responsible technical committee and must be reviewed every five years and if not revised, either reapproved or withdrawn. Your comments are invited either for revision of this standard or for additional standards and should be addressed to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee, which you may attend. If you feel that your comments have not received a fair hearing you should make your views known to the ASTM Committee on Standards, at the address shown below.

This standard is copyrighted by ASTM International, 100 Barr Harbor Drive, PO Box C700, West Conshohocken, PA 19428-2959, United States. Individual reprints (single or multiple copies) of this standard may be obtained by contacting ASTM at the above address or at 610-832-9585 (phone), 610-832-9555 (fax), or service@astm.org (e-mail); or through the ASTM website (www.astm.org). Permission rights to photocopy the standard may also be secured from the Copyright Clearance Center, 222 Rosewood Drive, Danvers, MA 01923, Tel: (978) 646-2600; http://www.copyright.com/