International Standard 2476

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Rubber, butadiene (BR) — Solution-polymerized types — Test recipe and evaluation of vulcanization characteristics

Caoutchouc butadiène (BR) — Types polymérisés en solution — Formule d'essai et évaluation des caractéristiques de vulcanisation

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Foreword

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Draft International Standards adopted by the technical committees are circulated to the member bodies for approval before their acceptance as International Standards by the ISO Council.

International Standard ISO 2476 was developed by Technical Committee ISO/TC 45, *Rubber and rubber products*, and was circulated to the member bodies in October 1978.

It has been approved by the member bodies of the following countries :

Austria	Germany, F.R.	Spain
Belgium	Hungary	Sri Lanka
Brazil	India	Sweden
Bulgaria	Italy	Thailand
China	Korea, Rep. of	Turkey
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Denmark	Poland	USA
Egypt, Arab Rep. of	Romania	USSR
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The member body of the following country expressed disapproval of the document on technical grounds :

Mexico

This second edition cancels and replaces the first edition (i.e. ISO 2476-1975).

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Rubber, butadiene (BR) — Solution-polymerized types — Test recipe and evaluation of vulcanization characteristics

1 Scope and field of application

This International Standard specifies standard materials, equipment and processing methods for evaluating vulcanization characteristics of solution-polymerized butadiene rubbers (BR), including oil extended types (OEBR).

2 References

ISO 37, *Rubber, vulcanized — Determination of tensile stress-strain properties.*

ISO 471, *Rubber — Standard temperatures, humidities and times for the conditioning and testing of test pieces.*

ISO 1795, *Raw rubber in bales — Sampling.*

ISO 1796, *Raw rubber — Sample preparation.*

ISO 2393, *Rubber test mixes — Preparation, mixing and vulcanization — Equipment and procedures.*

ISO 3417, *Rubber — Measurement of vulcanization characteristics with the oscillating disc curemeter.*

3 Test recipe for evaluation of vulcanization characteristics

3.1 Standard test formula

The standard test formula is given in the table.

The material used shall be NBS (National Bureau of Standards of the USA) Standard reference materials as indicated in the table, or shall be in accordance with equivalent national standards.

Material	NBS Standard reference material number	Parts by mass	
		1 Non-oil extended	2 Oil extended
Butadiene rubber (BR)	—	100,00	100,00
Zinc oxide	370	3,00	3,00
Oil furnace black (HAF)[1]	378	60,00	60,00
Stearic acid	372	2,00	2,00
ASTM Type 103 petroleum oil (naphthenic)[2]	—	15,00	—
Sulphur	371	1,50	1,50
TBBS[3]	384	0,90	0,90
		182,40	167,40
Calculated density, Mg/m^3		1,11	1,14 to 1,16[4]

1) The current Industry Reference Black may be used in place of NBS 378, but this may give slightly different results.

2) This oil, relative density 0,92, may be obtained from R.E. Carrol, P.O. Box 139, Trenton, N.J. 08601, USA. Alternative oils such as Circosol 4240 or Shellflex 724 are suitable, but may give slightly different results.

ASTM Type 103 oil has the following characteristics :

Kinematic viscosity at 100 °C, 16,8 ± 1,2 mm^2/s

Viscosity gravity constant, 0,889 ± 0,002

The viscosity gravity constant is calculated from the Saybolt universal viscosity at 37,8 °C and the relative density at 15,5/15,5 °C. Use the following equation to calculate the VGC from the measured properties :

$$VGC = \frac{10\,d - 1{,}075\,2 \log (V - 38)}{10 - \log (V - 38)}$$

where

d is the relative density at 15,5/15,5 °C;

V the Saybolt universal viscosity at 37,8 °C.

3) *N-tert*-butyl-2-benzothiazole sulphenamide. This shall be supplied in powder form having an initial ether- or ethanol-insoluble matter content of less than 0,3 %. The material shall be stored at room temperature in a closed container and the ether- or ethanol-insoluble matter shall be checked every 6 months. If this is found to exceed 0,75 %, the material shall be discarded or recrystallized.

4) Based on 37,5 % oil extended BR.

3.2 Procedure

3.2.1 Equipment and procedure

Equipment and procedure for the preparation, mixing and vulcanization shall be in accordance with ISO 2393, where applicable.

Details of a suitable internal mixer are given in the annex.

3.2.2 Mixing procedures

Three mixing procedures are specified.

Method A — Internal mixer for initial and final mixing.

Method B — Internal mixer for initial and mill for final mixing.

Method C — Mill mixing.

NOTE — These procedures may give different results.

The mill handling of solution butadiene rubbers is more difficult than for other rubbers and mixing is best accomplished by using an internal mixer. Because of the difficulty of mill mixing butadiene rubber, it is recommended that one of the internal mixer procedures (methods A or B) be used where such equipment is available. With some types of butadiene rubber it is not possible to get a satisfactory mix using the mill mixing procedure.

3.2.2.1 Method A — Internal mixer for initial and final mixing

3.2.2.1.1 Stage 1 — Initial mixing procedure

	Duration (min)	Cumulative time (min)
a) Adjust the temperature, speed and ram pressure of the internal mixer to achieve the conditions outlined in 3.2.2.1.1 e). Close the discharge gate, start the rotor and raise the ram.....	—	—
b) Charge one-half of the rubber, the zinc oxide, the carbon black, the oil (omit from formula 2 for OEBR), the stearic acid and the balance of the rubber. Lower the ram	0,5	0,5
c) Allow the batch to mix	3,0	3,5
d) Raise the ram and clean the mixer throat and the top of the ram. Lower the ram	0,5	4,0

e) Discharge the batch at a temperature of 170 °C or after a total time of 6 min, whichever occurs first	2,0	6,0
Total time (max.)	6,0	

f) Immediately pass the batch three times through a laboratory mill with a mill opening of 5,0 mm and a temperature of 50 ± 5 °C. Check weigh the batch (see ISO 2393).

g) Rest the batch for at least 30 min and up to 24 h.

3.2.2.1.2 Stage 2 — Final mixing procedure

	Duration (min)	**Cumulative time** (min)
a) Cool the internal mixer to a temperature of 40 ± 5 °C with full cooling water on the rotor. Start the motor and raise the ram	—	—
b) Leave the cooling water on and the steam off. Roll all the sulphur and the TBBS into one-half of the masterbatch and charge into the mixer. Add the remaining portion of the masterbatch. Lower the ram	0,5	0,5
c) Allow the batch to mix until a temperature of 110 °C, or a total time of 3 min is reached, whichever occurs first	2,5	3,0
Total time (max.)	3,0	

d) Immediately pass the batch through a laboratory mill with a mill opening set at 0,8 mm and at a temperature of 50 ± 5 °C.

e) Pass the rolled batch endwise through the rolls six times.

f) Sheet the batch to approximately 6 mm and check weigh (see ISO 2393). Remove sufficient sample for curemeter testing, if required.

g) Sheet the batch to approximately 2,2 mm for preparing test slabs or to the appropriate thickness for preparing ISO ring specimens (see ISO 2393).

3.2.2.2 Method B — Internal mixer for initial and mill for final mixing.

3.2.2.2.1 Stage 1 — Initial mixing procedure

	Duration (min)	**Cumulative time** (min)
a) Adjust the temperature, speed and ram pressure of the internal mixer to achieve the conditions outlined in 3.2.2.2.1 e). Close the discharge gate, start the rotor and raise the ram	—	—
b) Charge one-half of the rubber, the zinc oxide, the carbon black, the oil (omit from formula 2 for OEBR), the stearic acid and the balance of the rubber. Lower the ram	0,5	0,5
c) Allow the batch to mix	3,0	3,5
d) Raise the ram and clean the mixer throat and the top of the ram. Lower the ram	0,5	4,0
e) Discharge the batch at a temperature of 170 °C or after 6 min, whichever occurs first ...	2,0	6,0
Total time (max.)	6,0	

f) Immediately pass the batch three times through a laboratory mill with a mill opening of 5,0 mm and a temperature of 50 ± 5 °C. Check weigh the batch (see ISO 2393).

g) Rest the batch for at least 30 min and up to 24 h.

3.2.2.2.2 Stage 2 — Final mill mixing procedure

Adjust the mass of all material (i.e. masterbatch, sulphur and TBBS) to give a final batch mass of four times the formula mass.

NOTE — All mill openings should be adjusted to maintain a good rolling bank at the nip of the rolls during mixing.

	Duration (min)	**Cumulative time** (min)
a) Set and maintain the mill roll temperature at 35 ± 5 °C and the mill opening at 1,5 mm. Band the masterbatch and band round the front roll	1,0	1,0
b) Add the sulphur and the TBBS slowly to the batch	1,0	2,0
c) Make six 3/4 cuts from each side	1,5	3,5

d) Cut the batch from the mill. Set the mill opening to 0,8 mm and pass the rolled batch endwise through the rolls six times . .	1,5	5,0
Total time	5,0	

e) Sheet the batch to approximately 6 mm and check weigh (see ISO 2393). Remove sufficient sample for curemeter testing, if required.

f) Sheet the batch to approximately 2,2 mm for preparing test slabs or to the appropriate thickness for preparing ISO ring specimens (see ISO 2393).

3.2.2.3 Method C — Mill mixing procedure

The standard laboratory batch mass, in grams, shall be based on four times the formula mass. Adjust the mill roll cooling conditions to maintain a temperature of 35 ± 5 °C throughout the mixing operations.

NOTE — All mill openings should be adjusted to maintain a good rolling bank at the nip of the rolls during mixing.

	Duration (min)	Cumulative time (min)
a) Band the rubber with the mill opening set at 1,3 mm	1	1
b) Add the zinc oxide and the stearic acid evenly across the rolls. Make two 3/4 cuts from each side	2	3
c) Add the carbon black evenly across the rolls at a uniform rate. When about half the black has been incorporated, open the rolls to 1,8 mm and then add the remainder of the black. Make two 3/4 cuts from each side, allowing 30 s between each cut. Be certain to add the black that has dropped into the mill pan	15 to 18	18 to 21
d) Add the oil (omit from formula 2 for OEBR) very slowly drop by drop	8 to 10	26 to 31
e) Add the sulphur and the TBBS	2	28 to 33
f) Make six successive 3/4 cuts from each side	2	30 to 35
g) Cut the batch from the mill. Set the mill opening to 0,8 mm and pass the rolled batch endwise through the rolls six times . .	2	32 to 37
Total time	32 to 37	

h) Sheet the batch to approximately 6 mm and check weigh the batch (see ISO 2393). Remove sufficient sample for curemeter testing if required.

j) Sheet the batch to approximately 2,2 mm for preparing test slabs or to the appropriate thickness for preparing ISO ring specimens (see ISO 2393).

NOTE — It is sometimes easier and more practicable to combine steps 3.2.2.3 c) and 3.2.2.3 d) above, either by premixing the oil and black together and then adding the oiled black directly to the rubber on the mill as described in 3.2.2.3 c) and thus omitting 3.2.2.3 d), or by adding carbon black and oil alternately.

4 Conditioning of compounds

Condition all batches produced by methods A, B, or C at a standard laboratory temperature for 2 to 24 h after mixing and prior to vulcanizing (see ISO 471).

5 Evaluation of vulcanization characteristics

5.1 Evaluation according to stress-strain properties

Vulcanize sheets at 145 °C for 25, 35 and 50 min.

Condition the vulcanized sheets for 16 to 72 h at a standard laboratory temperature and humidity (see ISO 471).

Measure the stress-strain properties in accordance with ISO 37.

5.2 Evaluation according to oscillating disc curemeter test

Measure the following standard test parameters :

M_L, M_H, t_{s1}, t'_c (50) and t'_c (90)

in accordance with ISO 3417, using the following test conditions :

oscillation frequency :	1,7 Hz (100 cycles per minute)
amplitude of oscillation :	1° arc
selectivity :	To be selected to give at least 75 % full scale deflection
die temperature :	160 ± 0,1 °C
pre-heat time :	None (if microdies are used) 1 min (if a large die is used)

Annex

Internal mixer

A.1 The internal mixer[1] should have a nominal capacity of approximately 1 000 cm^3.

A.2 The rotor speed(s), ram pressure and coolant flow of the internal mixer should be such that the conditions specified in 3.2.2.1.1 e), 3.2.2.1.2 c) and 3.2.2.2.1 e) will be accomplished.

A.3 The batch size should be the nominal capacity of the internal mixer measured in cubic centimetres, multiplied by the relative density (+ 0, − 10 %).

NOTE — If an old or worn internal mixer is used, the batch mass should be increased accordingly.

1) A suitable internal mixer is available commercially. Details may be obtained from the Secretariat of ISO/TC 45 (BSI) or from the ISO Central Secretariat.