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МЕЖДУНАРОДНАЯ ОРГАНИЗАЦИЯ ПО СТАНДАРТИЗАЦИИ

Rubber, butadiene (BR) — Solution-polymerized types — Evaluation procedure

Caoutchouc butadiène (BR) — Types polymérisés en solution — Méthode d'évaluation

Reference number
ISO 2476 : 1988 (E)

Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

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. They are approved in accordance with ISO procedures requiring at least 75 % approval by the member bodies voting.

International Standard ISO 2476 was prepared by Technical Committee ISO/TC 45, *Rubber and rubber products.*

This third edition cancels and replaces the second edition (ISO 2476 : 1980). The main technical differences introduced in this new edition of ISO 2476 in comparison with the second edition are as follows:

— a new clause covering sampling and sample preparation has been introduced (clause 3);

— a new clause specifying physical and chemical tests on the raw rubber has been introduced (clause 4);

— the use of an internal mixer for the preparation of the test mix is strongly recommended (see 5.2.2);

— when a mill mixer has to be used, the standard mill batch mass has been reduced to three times the formula mass in order to improve mixing efficiency (see 5.2.2.3);

— alternative vulcanization conditions are made possible, and the conditioning period of vulcanized test slabs has been extended to 96 h (see clause 8);

— a new clause giving the required format for a test report has been introduced (clause 9).

Annex A forms an integral part of this International Standard.

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Rubber, butadiene (BR) — Solution-polymerized types — Evaluation procedure

1 Scope

This International Standard specifies

— physical and chemical tests on raw rubbers;

— standard materials, standard test formulae, equipment and processing methods for evaluating the vulcanization characteristics of solution-polymerized butadiene rubbers (BR), including oil-extended types (OEBR).

2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this International Standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the standards listed below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 37 : 1977, *Rubber, vulcanized — Determination of tensile stress-strain properties.*

ISO 247 : 1978, *Rubber — Determination of ash.*

ISO 248 : 1979, *Rubbers, raw — Determination of volatile matter content.*

ISO 289 : 1985, *Rubber, unvulcanized — Determination of Mooney viscosity.*

ISO 471 : 1983, *Rubber — Standard temperatures, humidities and times for the conditioning and testing of test pieces.*

ISO 1795 : 1974, *Raw rubber in bales — Sampling.*

ISO 1796 : 1982, *Rubber, raw — Sample preparation.*

ISO 2393 : 1973, *Rubber test mixes — Preparation, mixing and vulcanization — Equipment and procedures.*

ISO 3417 : 1977, *Rubber — Measurement of vulcanization characteristics with the oscillating disc curemeter.*

3 Sampling and sample preparation

3.1 A sample of mass approximately 1 500 g shall be taken by the method described in ISO 1795.

3.2 Preparation of the test portion shall be in accordance with ISO 1796.

4 Physical and chemical tests on raw rubber

4.1 Mooney viscosity

Determine the Mooney viscosity in accordance with ISO 289 on a test portion prepared as indicated in ISO 1796, but with the following modification: during the massing process, maintain the mill roll surface temperature at 35 °C ± 5 °C. Record the result as ML (1 + 4) at 100 °C.

4.2 Volatile matter

Determine the volatile matter content in accordance with ISO 248.

4.3 Ash content

Determine the ash content in accordance with ISO 247.

5 Preparation of test mixes for evaluation of butadiene rubbers

5.1 Standard test formulae

The standard test formulae are given in table 1.

The materials shall be NBS*) standard reference materials as indicated in table 1, or other, equivalent national or international standard reference materials.

Table 1 — Standard test formulae for evaluation of BR rubbers

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 103 oil[2]	—	15,00	—
Sulfur	371	1,50	1,50
TBBS[3]	384	0,90	0,90
Totals		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, density 0,92 g/cm^3, is produced by the Sun Refining and Marketing Company and distributed by R.E. Carroll Inc., P.O.Box 139, Trenton, NJ 08601, USA. Overseas requests should be directed to Sunoco Overseas Inc., 1801 Market Street, Philadelphia, PA 19103, USA. Alternative oils, such as Circosol 4240 or Shellflex 724, are suitable but may give slightly different results.

ASTM 103 oil has the following characteristics:

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

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

where

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

v is the Saybolt Universal viscosity at 37,8 °C.

3) *N-tert*-butyl-2-benzothiazole sulfenamide. 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.

*) National Bureau of Standards of the USA.

5.2 Procedure

5.2.1 Equipment and procedure

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

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

5.2.2 *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 (method 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.

5.2.2.1 Method A — Internal mixer for initial and final mixing

5.2.2.1.1 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 5.2.2.1.1 e). Close the discharge gate, start the rotor and raise the ram	—	—
b) Load 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 °C ± 5 °C. Check-weigh the batch (see ISO 2393). If the mass of the batch differs from the theoretical value by more than 0,5 %, discard the batch and re-mix.

g) Leave the batch for at least 30 min and up to 24 h, if possible at standard temperature and humidity as defined in ISO 471.

5.2.2.1.2 Stage 2 — Final mixing procedure

	Duration (min)	Cumulative time (min)
a) Cool the internal mixer to a temperature of 40 °C ± 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 sulfur and the TBBS into one-half of the masterbatch and load 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 °C ± 5 °C.

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

f) Sheet the batch to approximately 6 mm. Check-weigh the batch (see ISO 2393). If the mass of the batch differs from the theoretical value by more than 0,5 %, discard the batch and re-mix. Remove sufficient material for oscillating disc curemeter testing.

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

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

5.2.2.2.1 Stage 1 — Initial mixing procedure

Proceed in accordance with 5.2.2.1.1.

5.2.2.2.2 Stage 2 — Final mill mixing procedure

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

A good rolling bank at the nip of the rolls shall be maintained during mixing. If this is not obtained with the nip settings specified hereunder, small adjustments to the mill openings may be necessary.

	Duration (min)	Cumulative time (min)
a) Set and maintain the mill roll temperature at 35 °C ± 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 sulfur 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. Check-weigh the batch (see ISO 2393). If the mass of the batch differs from the theoretical value by more than 0,5 %, discard the batch and re-mix. Remove sufficient material for oscillating disc curemeter testing.

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

5.2.2.3 Method C — Mill mixing procedure

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

Methods A and B, which give better dispersion of the ingredients, are preferred if an internal mixer is available.

A good rolling bank at the nip of the rolls shall be maintained during mixing. If this is not obtained with the nip settings specified hereunder, small adjustments to the mill openings may be necessary.

	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

	Duration (min)	Cumulative time (min)
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 sulfur 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. Check-weigh the batch (see ISO 2393). If the mass of the batch differs from the theoretical value by more than 0,5 %, discard the batch and re-mix. Remove sufficient material for oscillating disc curemeter testing.

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

It is sometimes easier and more practicable to combine steps 5.2.2.3 c) and 5.2.2.3 d), either by premixing the oil and black together and then adding the oiled black directly to the rubber on the mill as described in 5.2.2.3 c) and thus omitting 5.2.2.3 d), or by adding carbon black and oil alternately.

6 Conditioning of compounds

Condition all batches produced by methods A, B or C for 2 h to 24 h after mixing and prior to vulcanizing, if possible at standard temperature and humidity as defined in ISO 471.

7 Evaluation of vulcanization characteristics with the 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 chosen to give at least 75 % of full scale deflection NOTE — With some rubbers, 75 % may not be attainable.
die temperature :	160 °C ± 0,3 °C
pre-heat time :	none

8 Evaluation of tensile stress-strain properties of vulcanized test mixes

Vulcanize sheets at 145 °C for 25 min, 35 min and 50 min. Alternatively, vulcanize sheets at 150 °C for 20 min, 30 min and 50 min.

The three periods of cure shall be chosen to cover the undercure, optimum cure and overcure of the material under test.

Condition the vulcanized sheets for 16 h to 96 h, if possible at standard temperature and humidity as defined in ISO 471.

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

9 Test report

The test report shall include the following:

a) a reference to this International Standard;

b) all details necessary for the identification of the sample;

c) the standard test formula used;

d) the reference materials used;

e) the method used for volatile matter determination (mill or oven);

f) the mixing procedure used in 5.2.2;

g) the vulcanizing temperature and times used in clause 8;

h) any unusual features noted during the determination;

i) any operation not included in this International Standard or in the International Standards to which reference is made, as well as any operation regarded as optional;

j) the results and the units in which they have been expressed;

k) the date of the test.

Annex A
(normative)

Internal mixer

NOTE — This annex will be deleted after publication of the second edition of ISO 2393, in which the use of internal mixers will be specified.

A.1 The internal mixer[1] shall 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 shall be such that the time/temperature programmes set out in 5.2.2.1.1, 5.2.2.1.2 and 5.2.2.2.1 will be accomplished.

A.3 The batch size shall be the nominal capacity of the internal mixer in cubic centimetres, multiplied by the density in grams per cubic centimetre (+ 0, − 10 %).

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

1) A type B Banbury internal mixer has been found to be satisfactory for this purpose. Other internal mixers may be used, if the mass, temperatures and time of mixing are adjusted to give comparable results.

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