# INTERNATIONAL STANDARD

# ISO 2476

Sixth edition
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# Butadiene rubber (BR) — Solution-polymerized types — Evaluation procedures

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

Reference number
ISO 2476:2014(E)

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# 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.

The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

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For an explanation on the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the WTO principles in the Technical Barriers to Trade (TBT) see the following URL: Foreword - Supplementary information

The committee responsible for this document is ISO/TC 45, *Rubber and rubber products*, Subcommittee SC 3, *Raw materials (including latex) for use in the rubber industry*.

This sixth edition cancels and replaces the fifth edition (ISO 2476:2009), which has been technically revised with the following changes:

- — ISO 2476 has been aligned with ISO 2393:2014;
- — 5.2.2.1 states that the laboratory internal mixer procedure is the preferred method.

**INTERNATIONAL STANDARD** **ISO 2476:2014(E)**

# Butadiene rubber (BR) — Solution-polymerized types — Evaluation procedures

**WARNING — Persons using this International Standard should be familiar with normal laboratory practice. This International Standard does not purport to address all of the safety problems, if any, associated with its use. It is the responsibility of the user to establish appropriate safety and health practices and to ensure compliance with any national regulatory conditions.**

## 1 Scope

This International Standard specifies:

- physical and chemical tests on raw rubbers;
- standard materials, standard test formulations, equipment, and processing methods for evaluating the vulcanization characteristics of solution-polymerized butadiene rubbers (BR), including oil-extended types (OEBR), and the tensile stress-strain properties of vulcanized mixes.

## 2 Normative references

The following documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

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

ISO 247:2006, *Rubber — Determination of ash*

ISO 248-1, *Rubber, raw — Determination of volatile-matter content — Part 1: Hot-mill method and oven method*

ISO 248-2, *Rubber, raw — Determination of volatile-matter content — Part 2: Thermogravimetric methods using an automatic analyser with an infrared drying unit*

ISO 289-1, *Rubber, unvulcanized — Determinations using a shearing-disc viscometer — Part 1: Determination of Mooney viscosity*

ISO 1795, *Rubber, raw natural and raw synthetic — Sampling and further preparative procedures*

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

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

ISO 6502, *Rubber — Guide to the use of curemeters*

ISO 23529, *Rubber — General procedures for preparing and conditioning test pieces for physical test methods*

## 3 Sampling and further preparative procedures

**3.1** Take a laboratory sample of approximately 1,5 kg by the method described in ISO 1795.

**3.2** Prepare test samples in accordance with ISO 1795.

# 4 Physical and chemical tests on raw rubber

## 4.1 Mooney viscosity

Determine the Mooney viscosity in accordance with ISO 289-1 on a test sample prepared as indicated in ISO 1795 (preferably without milling). If milling is necessary, 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 248-1 and ISO 248-2.

## 4.3 Ash

Determine the ash in accordance with ISO 247.

# 5 Preparation of test mixes for evaluation of butadiene rubbers

## 5.1 Standard test formulations

Two standard test formulations are given in Table 1. The materials used shall be national or International Standard reference materials (or, if no standard reference material is available, as agreed by the interested parties).

**Table 1 — Standard test formulations**

| **Material** | **Parts**<br>mass fraction | |
|---|---|---|
| | **Non-oil-extended** | **Oil-extended** |
| Butadiene rubber | 100,00 | 100,00 + $Y$[a] |
| Zinc oxide | 3,00 | 3,00 |
| IRB[b] | 60,00 | 0,6 × (100 + $Y$) |
| Stearic acid | 2,00 | 2,00 |
| ASTM 103 oil[c] | 15,00 | — |
| Sulfur | 1,50 | 1,50 |
| TBBS[d] | 0,90 | 0,009 × (100 + $Y$) |
| Total | 182,40 | 167,40 + 1,609$Y$ |
| Calculated density, $g/cm^3$ | 1,11 | — |

[a] $Y$ = parts of oil, mass fraction, per 100 parts of base polymer in the oil-extended rubber.

[b] Use the current industrial reference black. Dry the material for 1 h at 125 °C ± 3 °C and store in a tightly closed container.

[c] This oil, density 0,92 $g/cm^3$, is produced by the Sun Refining and Marketing Company and distributed by R.E. Carroll, Inc., 1570 North Olden Avenue Ext, Trenton, NJ 08638-3204, USA. Overseas requests should be directed to Sunoco Overseas, Inc., 1801 Market Street, Philadelphia, PA 19103-1699, USA. This is an example of a suitable product available commercially. This information is given for convenience of users of this document and does not constitute an endorsement by ISO of this product. Alternative oils can be used, but might give slightly different results.

[d] N-tert-butylbenzothiazole-2-sulfenamide. This is supplied in powder form having an initial insoluble-matter content, determined in accordance with ISO 11235, of less than 0,3 %. The material shall be stored at room temperature in a closed container and the insoluble matter checked every 6 months. If this is found to exceed 0,75 %, the material shall be discarded or recrystallized.

## 5.2 Procedure

### 5.2.1 General

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

### 5.2.2 Mixing procedures

#### 5.2.2.1 General

Five mixing procedures are specified, a procedure using a laboratory internal mixer is preferred.

- method A1: single-stage mixing using a laboratory internal mixer;
- method A2: two-stage mixing using a laboratory internal mixer for both initial and final mixing;
- method B: two-stage mixing using a laboratory internal mixer for initial mixing and a mill for final mixing.

  The smaller laboratory internal mixers do not provide enough compound for the final mill mixing since a batch mass of four times the formula mass is required. In these cases, the laboratory internal mixer should be used for the final mixing. The mixer head starting temperature or the batch mass can be adjusted, so that the final temperature of the discharged batch does not exceed 120 °C.
- method C1 and C2: mill mixing.

NOTE 1 These procedures can give different results.

NOTE 2 Mill mixing of solution-polymerized butadiene rubbers is more difficult than with other rubbers, and mixing is best accomplished by using an internal mixer. With some types of butadiene rubber, it is not possible to obtain a satisfactory mix using a mill-mixing procedure.

#### 5.2.2.2 Method A1 — Single-stage mixing using a laboratory internal mixer

For laboratory internal mixers having nominal capacities of 65 cm$^3$ to about 2 000 cm$^3$, the batch mass shall be equal to the nominal mixer capacity, in cubic centimetres, multiplied by the density of the compound. For each batch mixed, the laboratory internal mixer conditions shall be the same during the preparation of a series of identical mixes. At the beginning of each series of test mixes, a machine-conditioning batch shall be mixed using the same formulation as the mixes under test. The laboratory internal mixer shall be allowed to cool down to a specified temperature (60 °C has been found to be suitable) between the end of one test batch and the start of the next. The temperature control conditions shall not be altered during the mixing of a series of test batches.

The mixing technique shall be such as to obtain a good dispersion of all the ingredients.

The temperature of the batch discharged on completion of mixing shall not exceed 120 °C. If necessary, adjust the batch mass or the mixer head starting temperature so that this condition is met.

NOTE 1 The mixing conditions given in Table A.7 for various sizes of laboratory internal mixer might be helpful.

NOTE 2 A general mixing procedure for the laboratory internal mixer is as follows:

| | | Duration (min) | Cumulative time (min) |
|---|---|---|---|
| a) | Charge the chamber with the rubber. Lower the ram. | — | — |
| b) | Masticate the rubber. | 1,0 | 1,0 |
| c) | Raise the ram and add the zinc oxide, the oil, the stearic acid, and half of the carbon black. Then sweep the orifice and lower the ram. | 1,0 | 2,0 |
| d) | Raise the ram and add the TBBS, the sulfur, and the rest of the carbon black. Then sweep the orifice and lower the ram. | 1,0 | 3,0 |
| e) | Allow the batch to mix. | 6,0 | 9,0 |
| f) | Turn off the motor, raise the ram, remove or open the mixing chamber, and discharge the batch. Record the maximum batch temperature.<br>With some types of mixer, the rotors might need to be turning in order to discharge the batch. | | |

Immediately after discharging the mixed batch, pass it through a mill at 50 °C ± 5 °C once at a 0,5 mm mill opening, and then twice at a 3,0 mm mill opening.

Determine the batch mass and record it. If it differs from the theoretical value by more than +0,5 % or −1,5 %, discard the batch.

Prepare a test piece for determining the vulcanization characteristics in accordance with ISO 3417 or ISO 6502, if required. Condition the test piece for 2 h to 24 h, if possible at a standard temperature and humidity as defined in ISO 23529 before testing.

If required, sheet the batch to approximately 2,2 mm for preparing test sheets or the appropriate thickness for preparing ring pieces in accordance with ISO 37. To obtain a grain effect, pass the lengthwise-folded batch in the same direction four times between mill rolls set at an opening between 2,1 mm and 2,5 mm and a temperature of 50 °C ± 5 °C.

#### 5.2.2.3 Method A2 — Two-stage mixing using a laboratory internal mixer for both initial and final mixing

##### 5.2.2.3.1 Stage 1 — Initial mixing procedure

See 5.2.2.2.

The final temperature of the batch shall not exceed 170 °C. If necessary, adjust the batch mass or the mixer head starting temperature so that this condition is met.

NOTE A general mixing procedure for initial mixing using a laboratory mixer is as follows:

| | | Duration (min) | Cumulative time (min) |
|---|---|---|---|
| a) | Adjust the temperature to 50 °C ± 5 °C and the rotor speed and ram pressure of the laboratory internal mixer so that the batch will be discharged under the conditions outlined in e) below. Close the discharge gate, start the motor, and raise the ram. | — | — |
| b) | Load one-half of the rubber, followed by the zinc oxide, the carbon black, the oil (omit from oil-extended BR), 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 |

Immediately after discharging the batch, pass it three times through a laboratory mill with a mill opening of 5,0 mm and at a temperature of 50 °C ± 5 °C. Check-weigh the batch in accordance with ISO 2393. If the mass of the batch differs from the theoretical value by more than +0,5 % or −1,5 %, discard the batch and remix.

#### 5.2.2.3.2 Stage 2 — Final mixing procedure

The mixing technique shall be such as to obtain a good dispersion of all the ingredients.

The final temperature of the batch shall not exceed 120 °C. If necessary, adjust the batch mass or the mixer head starting temperature, so that this condition is met.

NOTE A general mixing procedure for final mixing using a laboratory mixer is as follows.

| | | Duration (min) | Cumulative time (min) |
|---|---|---|---|
| a) | Cool the internal mixer to a temperature of 40 °C ± 5 °C, with full cooling water on the rotors. 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 |

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

Pass the rolled batch endwise through the rolls six times.

Sheet the batch to approximately 6 mm. Check-weigh the batch in accordance with ISO 2393. If the mass of the batch differs from the theoretical value by more than +0,5 % or −1,5 %, discard the batch and remix. Remove sufficient material for curemeter testing.

Sheet the batch to approximately 2,2 mm for preparing test sheets or to the appropriate thickness for preparing ISO ring specimens in accordance with ISO 37.

#### 5.2.2.4 Method B — Two-stage mixing using a laboratory internal mixer for initial mixing and a mill for final mixing

##### 5.2.2.4.1 Stage 1 — Initial mixing procedure

Proceed in accordance with 5.2.2.3.1.

##### 5.2.2.4.2 Stage 2 — Final mill-mixing procedure

The standard laboratory mill batch mass, in grams, shall be based on 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, make small adjustments to the mill openings.

| | | 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 around the front roll. | 1,0 | 1,0 |
| b) | Add the sulfur and the TBBS slowly to the batch. Sweep up any material which has fallen into the mill pan and add it to the mix. | 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 |
| e) | Sheet the batch to approximately 6 mm. Check-weigh the batch in accordance with ISO 2393. If the mass of the batch differs from the theoretical value by more than +0,5 % or −1,5 %, discard the batch and remix. Remove sufficient material for curemeter testing. | | |
| f) | Sheet the batch to approximately 2,2 mm for preparing test sheets or to the appropriate thickness for preparing ISO ring specimens in accordance with ISO 37. | | |

#### 5.2.2.5 Methods C1 and C2 — Mill-mixing procedure

##### 5.2.2.5.1 General

Solution-polymerized butadiene rubbers are difficult to process on a mill. Methods A1, A2, and B, which give better dispersion of the ingredients, are preferred. If a laboratory internal mixer is not available, a mill mixing procedure can be used:

— method C1, which can be used for all solution-polymerized butadiene rubbers, whether oil-extended or not;

— method C2, which is limited to non-oil-extended rubbers, but gives easier mixing and leads to better dispersion of the ingredients.

Methods C1 and C2 do not necessarily give identical results for non-oil-extended solution-polymerized butadiene rubbers. In laboratory cross-checks or in a series of evaluations, the same procedure shall therefore be used in all cases.

##### 5.2.2.5.2 Method C1

The standard laboratory batch mass, in grams, shall be based on three times the formulation mass (i.e. 3 × 182,40 g = 547,20 g or 3 × 167,40 g = 502,20 g). Adjust the mill roll cooling conditions to maintain a temperature of 35 °C ± 5 °C throughout the mixing.

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, make small adjustments to the mill openings.

| | | Duration (min) | Cumulative time (min) |
|---|---|---|---|
| a) | Band the rubber with the mill opening set at 1,3 mm. | 1,0 | 1,0 |
| | NOTE Non-oil-extended rubbers might require longer milling to achieve a good band. | | |
| b) | Add the zinc oxide and the stearic acid evenly across the rolls. Make two 3/4 cuts from each side. | 2,0 | 3,0 |
| 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 any black that has dropped into the mill pan. | 15,0 to 18,0 | 18,0 to 21,0 |
| d) | Add the oil (omit from oil-extended BR) very slowly drop by drop. | 8,0 to 10,0 | 26,0 to 31,0 |
| e) | Add the sulfur and the TBBS. Sweep up any material which has fallen into the mill pan and add it to the mix. | 2,0 | 28,0 to 33,0 |
| f) | Make six successive 3/4 cuts from each side. | 2,0 | 30,0 to 35,0 |
| 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. | 2,0 | 32,0 to 37,0 |
| h) | Sheet the batch to approximately 6 mm. Check-weigh the batch in accordance with ISO 2393. If the mass of the batch differs from the theoretical value by more than +0,5 % or −1,5 %, discard the batch and remix. Remove sufficient material for curemeter testing. | | |
| i) | Sheet the batch to approximately 2,2 mm for preparing test sheets or to the appropriate thickness for preparing ISO ring specimens in accordance with ISO 37. | | |

### 5.2.2.5.3 Method C2

The standard laboratory batch mass, in grams, shall be based on two times the formulation mass (i.e. 2 × 182,40 g = 364,80 g). Adjust the mill roll cooling conditions to maintain a temperature of 35 °C ± 5 °C throughout the mixing. Add the ingredients to the batch slowly and evenly across the rolls. Do not cut the batch before all the ingredients have been incorporated.

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, make small adjustments to the mill openings.

| | | Duration (min) | Cumulative time (min) |
|---|---|---|---|
| a) | Pass the rubber twice through the rolls with the mill opening set at 0,45 mm ± 0,01 mm and then band it. Make two successive 3/4 cuts from each side. | 2,0 | 2,0 |
| b) | Add the stearic acid and the zinc oxide. Make three successive 3/4 cuts from each side. | 2,0 | 4,0 |