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

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

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ISO copyright office
Case postale 56 • CH-1211 Geneva 20
Tel. + 41 22 749 01 11
Fax + 41 22 749 09 47
E-mail copyright@iso.org
Web www.iso.org

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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).

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see www.iso.org/patents).

Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

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.

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 $35\text{ °C} \pm 5\text{ °C}$. 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 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 + 1,609Y
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 $125\text{ °C} \pm 3\text{ °C}$ and store in a tightly closed container.

^c This oil, density 0,92 g/cm³, 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 cm³ to about 2 000 cm³, 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: