INTERNATIONAL STANDARD

ISO 2476

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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 2476:2009(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.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

The main task of technical committees is to prepare International Standards. Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

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.

ISO 2476 was prepared by Technical Committee ISO/TC 45, *Rubber and rubber products*, Subcommittee SC 3, *Raw materials (including latex) for use in the rubber industry*.

This fifth edition cancels and replaces the fourth edition (ISO 2476:1996), which has been technically revised.

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

WARNING — Persons using this International Standard should be familiar with normal laboratory practice. This 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 referenced documents are indispensable for the application of this document. 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, *Rubber, raw — Determination of volatile-matter content*

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.

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 by mass	
	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, by mass, 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 Information is given for convenience of users of this International Standard and does not constitute an endorsement by ISO of the product named. Alternative oils may 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:

- 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 may be adjusted, so that the final temperature of the discharged batch does not exceed 120 °C.

- Methods C1 and C2: mill mixing.

NOTE 1 These procedures may 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 a laboratory internal mixer having a nominal capacity 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 the mixing chamber and discharge the batch. Record the maximum batch temperature.		

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 (see 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 (see 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 (see 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 procedures

5.2.2.5.1 General

Solution-polymerized butadiene rubbers are difficult to process on a mill. Methods A and B, which give better dispersion of the ingredients, are preferred if a laboratory internal mixer is available. If this is not the case, two mill-mixing procedures may be used:

- method C1, which may 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. NOTE Non-oil-extended rubbers may require longer milling to achieve a good band.	1,0	1,0
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 (see 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
c)	Add successively half of the oil and half of the carbon black. Make seven successive 3/4 cuts from each side.	12,0	16,0
d)	Add successively the remainder of the oil and the remainder of the carbon black. Add any black that has dropped into the mill pan. Make seven 3/4 cuts from each side.	12,0	28,0
e)	Add the TBBS and the sulfur. Make six 3/4 cuts from each side.	4,0	32,0
f)	Cut the batch from the mill. Set the mill opening to 0,7 mm to 0,8 mm and pass the rolled batch endwise through the rolls six times.	3,0	35,0

g) Sheet the batch to approximately 6 mm and check-weigh the batch (see 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.

h) 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.

6 Conditioning of batches

Condition all batches produced by methods A1, A2, B, C1 and C2 for 2 h to 24 h after mixing and prior to vulcanizing, if possible at standard temperature and humidity as defined in ISO 23529.

7 Evaluation of vulcanization characteristics

7.1 Using an oscillating-disc curemeter

Measure the following standard test parameters:

M_L, M_H at defined time, 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° of arc;
— selectivity:	to be chosen to give at least 75 % of full-scale deflection at M_H (note that, with some rubbers, 75 % may not be attainable);
— die temperature:	160 °C ± 0,3 °C;
— pre-heat time:	none.

7.2 Using a rotorless curemeter

Measure the following standard test parameters:

M_L, M_H at defined time, t_{s1}, $t'_c(50)$ and $t'_c(90)$

in accordance with ISO 6502, using the following test conditions:

— oscillation frequency:	1,7 Hz (100 cycles per minute);
— amplitude of oscillation:	0,5° of arc;
— selectivity:	to be chosen to give at least 75 % of full-scale deflection at M_H (note that, 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 or, alternatively, 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 23529.

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

9 Precision

See Annex A.

10 Test report

The test report shall include the following:

a) a reference to this International Standard, i.e. ISO 2476;

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

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

d) the method used for the ash determination (method A or method B of ISO 247:2006);

e) the reference materials used;

f) the standard test formulation used;

g) the mixing procedure used in 5.2.2;

h) the curemeter test used in Clause 7 (ISO 3417 or ISO 6502);

i) the time at which M_H was determined in the curemeter test;

j) the vulcanization temperature and times used in Clause 8;

k) any unusual features noted during the determinations;

l) 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;

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

n) the date of the test.

Annex A
(informative)

Precision

A.1 General

Two interlaboratory test programmes (ITPs) were organized in 1987 and in 2004. The 1987 ITP was conducted with mill mixing. The other was conducted with mill mixing and laboratory internal mixer mixing.

The precision results as determined by these ITPs may not be applied to acceptance or rejection testing of any group of materials or products without documentation that the results of the precision determination actually apply to the products or materials tested.

A.2 Precision obtained for mill mixing in the 1987 ITP

A.2.1 Details of the ITP

A.2.1.1 Formulations containing two types of BR were selected and mixes were prepared in each of the 17 laboratories that participated in the programme on each of two days approximately one week apart. Formula 1 contained a non-oil-extended BR, while formula 2 contained an oil-extended BR.

Only method C1 of this International Standard (mill mixing) was used for preparing the mixes.

The mixes were prepared from special samples of all the necessary materials, sent to each laboratory prior to the actual testing. For each material, the samples were drawn from a homogeneous lot. Stress-strain tests were conducted on cured sheets of each of the mixes or compounds as specified by the test programme.

A.2.1.2 Determinations of modulus (stress at 300 % strain), tensile strength and percent elongation were made, taking as the result the median of five individual determinations, as specified in ISO 37. All 17 laboratories performed the test using dumb-bell test pieces. Five of the laboratories also performed the test using ring test pieces. The precision thus evaluated is a type 2 precision, and the time period for repeatability and reproducibility is on a scale of days.

A.2.2 Precision results

The precision calculations to express repeatability and reproducibility were performed in accordance with ISO/TR 9272:1986.

The precision results are given in Table A.1 for dumb-bell test pieces and in Table A.2 for ring test pieces.

The symbols used in Tables A.1 and A.2 are defined as follows.

- r = repeatability, in measurement units. This is the value below which the absolute difference between two within-laboratory test results may be expected to lie with a specified probability.
- (r) = repeatability, in percent (relative).

The two within-laboratory test results are obtained with the same method on nominally identical test material under the same conditions (same operator, apparatus and laboratory) and within a specified time period. Unless stated otherwise, the specified probability is 95 %.

— R = reproducibility, in measurement units. This is the value below which the absolute difference between two between-laboratory test results may be expected to lie with a specified probability.

— (R) = reproducibility, in percent (relative).

The two between-laboratory test results are obtained using the same method on nominally identical test materials under different conditions (different operators, apparatus and laboratories) and within a specified time period. Unless stated otherwise, the specified probability is 95 %.

It shall be borne in mind that these precision results apply only to the mill-mixing procedure of ISO 2476:1988, method C1.

Table A.1 — Type 2 precision for dumb-bell test pieces

Formulation	**Average value**	**Within-laboratory**		**Between laboratories**	
		r	(r)	R	(R)
Modulus (at 300 % strain), MPa					
Formula 1	10,9	1,37	12,6	2,61	23,8
Formula 2	13,0	1,66	12,8	2,90	22,3
Tensile strength, MPa					
Formula 1	16,5	1,23	7,47	3,13	18,9
Formula 2	17,7	1,82	10,3	3,93	22,3
Percent elongation					
Formula 1	367	35,1	9,55	76,6	20,8
Formula 2	424	57,8	13,6	127	29,9

Table A.2 — Type 2 precision for ring test pieces

Formulation	**Average value**	**Within-laboratory**		**Between laboratories**	
		r	(r)	R	(R)
Modulus (at 300 % strain), MPa					
Formula 1	10,3	0,82	7,98	4,13	40,2
Formula 2	11,9	0,82	6,93	4,73	39,7
Tensile strength, MPa					
Formula 1	14,4	0,98	6,81	3,03	21,1
Formula 2	15,8	1,40	8,88	4,36	27,6
Percent elongation					
Formula 1	362	62,1	17,2	62,1	17,2
Formula 2	433	51,7	11,9	51,7	11,9

A.3 Precision obtained for both mill and internal mixer in the 2004 ITP

A.3.1 Details of the ITP

The ITP to determine the precision using a laboratory mill and various sizes of internal mixer was conducted on compounds which were based on the formulation for non-oil-extended BR in Table 1, using Neocis BR40, an Nd-catalysed high-cis 1,4-polybutadiene with a cis content of 97 %. The ITP was conducted as part of the revision of ISO 2393:1994.

NOTE The internal mixers used covered those types often described as "miniature", "internal" and "laboratory".

The ITP was carried out using the precision procedures and guidelines described in the newly revised ISO/TR 9272:2005. This revised precision document, which replaces ISO/TR 9272:1986, was under ballot review at the time of the ITP. Reference should be made to ISO/TR 9272:2005 for other details and terminology on precision evaluation.

Two types of measurement were used to evaluate each type of mixing procedure: stress-strain testing and curemeter testing. Stress-strain property precision was determined for stress (modulus) at 100 %, 200 % and 300 % elongation, elongation at break and tensile strength. Curemeter precision was determined for M_H (maximum torque), M_L (minimum torque), t_{s1} (scorch time or time to a 1 dN·m rise), $t'_c(50)$ (time to 50 % of full torque) and $t'_c(90)$ (time to 90 % of full torque). A type 2 precision was determined. The compounds were prepared and tested on a day 1/day 2 basis one week apart.

On each day, homogeneous materials were used to prepare a separate mix for each type of compound, and the appropriate testing (stress-strain and curemeter) was conducted on each of these mixes for each of the two test days. The test result was taken as the average of two replicate test values obtained on each of the two separate test days and the precision was calculated from these test results.

For the internal-mixer testing, 10 laboratories participated in the test programme. However, each laboratory did not have the same type or size of mixer. The sizes of the mixers used in the ITP were 75 cm^3, 80 cm^3, 270 cm^3, 379 cm^3, 422 cm^3, 588 cm^3, 1 500 cm^3, 1 580 cm^3, 1 600 cm^3 and 3 322 cm^3. Some laboratories submitted data from more than one size of mixer, and the data from these separate mixers were incorporated into the ITP as data from individual laboratories. This gave a database equivalent to 17 laboratories. Each of the mixer-laboratory combinations was designated as a pseudo-laboratory.

A.3.2 Precision results

The precision results for both types of mixing, for both stress-strain and curemeter testing, are given in Tables A.3 to A.6. These results were obtained using the option 1 outlier deletion procedures described in ISO/TR 9272:2005. General statements for the use of the precision results are given below. These are given in terms of both the absolute precision, r and R, and also for relative precision, (r) and (R) (see additional discussion below).

Repeatability — The repeatability, or local domain precision, for each of the test methods has been established as the values given in Tables A.3 to A.6 for each measurement parameter listed in the tables. Two individual test results obtained in the same laboratory (by the proper use of this International Standard) that differ by more than the tabulated values for r, in measurement units, or (r), in percent, should be considered as suspect, i.e. to have come from different populations. Such a situation should suggest that some appropriate investigative action be taken.

Reproducibility — The reproducibility, or global domain precision, for each of the test methods has been established as the values given in Tables A.3 to A.6 for each measurement parameter listed in the tables. Two individual test results obtained in different laboratories (by the proper use of this International Standard) that differ by more than the tabulated values for R, in measurement units, or (R), in percent, should be considered as suspect, i.e. to have come from different populations. Such a situation should suggest that some appropriate investigative action be taken.