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AMENDMENT 1
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Rubber, butadiene (BR) – Solution-polymerized types – Evaluation procedure

AMENDMENT 1

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

AMENDEMENT 1

Reference number
ISO 2476:1988/Amd.1:1993 (E)

Foreword

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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.

Amendment 1 to International Standard ISO 2476:1988 was prepared by Technical Committee ISO/TC 45, *Rubber and rubber products,* Sub-Committee SC 3, *Raw materials (including latex) for use in the rubber industry.*

Annex B of this amendment to ISO 2476 is for information only.

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

AMENDMENT 1

Page 2

In 5.2.2, first line, replace "three mixing procedures" by "*four mixing procedures*".

In 5.2.2, line 4, replace "Method C" by "Methods C1 and C2".

Pages 3 and 4

Replace 5.2.2.3 by the following text:

5.2.2.3 Methods C1 and C2 – Mill mixing procedures

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

- method C1, which may be used for solution-polymerized butadiene rubbers, whether oil-extended or not;
- method C2, which is limited to non-oil-extended rubbers but allows an easier mixing and leads to a better dispersion of the ingredients.

Methods C1 and C2 will 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 should therefore be used in all cases.

5.2.2.3.1 Method C1

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

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 shall be made.

	Duration (min)	**Cumulative time** (min)
a) Band the rubber with the mill opening set at 1,3 mm	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 the black that has dropped into the mill pan	15,0 to 18,0	18,0 to 21,0
d) Add the oil (omit from formula 2 for OEBR) very slowly drop by drop	8,0 to 10,0	26,0 to 31,0
e) Add the sulfur and the TBBS	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
Total time	32,0 to 37,0	

h) Sheet the batch to approximately 6 mm. Determine the mass of 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.

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.

It is sometimes easier and more practicable to combine steps 5.2.2.3.1 c) and 5.2.2.3.1 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 5.2.2.3.1 c) and thus omitting 5.2.2.3.1 d), or by adding carbon black and oil alternately.

5.2.2.3.2 Method C2

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

	Duration (min)	Cumulative time (min)
a) Band the rubber twice through the rolls with the mill opening set at 0,45 mm ± 0,01 mm and then sheet 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 of the carbon black. Add the 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
Total time	35,0	

g) Sheet the batch to approximately 6 mm and determine the mass of 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 remix. Remove sufficient material for oscillating disc 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.

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Insert the following new clause 9 and renumber the "Test report" clause accordingly.

9 Precision

9.1 General

The precision calculations to express repeatability and reproducibility were performed in accordance with ISO/TR 9272:1986, *Rubber and rubber products – Determination of precision for test method standards.* Consult this for precision concepts and nomenclature. Annex B gives guidance on the use of repeatability and reproducibility.

9.2 Precision details

9.2.1 An interlaboratory test programme (ITP) was organized in 1987. Formulations containing two types of BR were selected and mixes were prepared in each of the 17 laboratories that participated in the ITP, 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 C of ISO 2476:1988 (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 uniform and homogeneous lot. Stress-strain tests were conducted on cured sheets of each of the mixes or compounds as specified by the test programme.

9.2.2 Determinations of modulus (stress at 300 %), tensile strength and percent elongation were made using as a test result the median of five individual test 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.

9.3 Results

9.3.1 The precision results are given in table 2 for dumb-bell test pieces and in table 3 for ring test pieces.

The symbols used in tables 2 and 3 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 test results are obtained with the same method on nominally identical test materials under the same conditions (same operator, apparatus and laboratory) and within a specified time period; unless stated otherwise, the 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 test results are obtained with the same method on nominally identical test materials under different conditions (different laboratories, operators and apparatus) and within a specified time period; unless stated otherwise, the probability is 95 %.

9.3.2 It shall be borne in mind that these precision results apply to the mill mixing procedure of ISO 2476:1988 only (method C).

Table 2 – Type 2 precision for dumb-bell test pieces

Compound or material	Average value	Within lab		Between labs	
		r	(r)	R	(R)
1) Modulus (300 %), 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
2) 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
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 3 – Type 2 precision for ring test pieces

Compound or material	Average value	Within lab		Between labs	
		r	(r)	R	(R)
1) Modulus (300 %), 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
2) 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
3) Percent elongation					
Formula 1	362	62,1	17,2	62,1	17,2
Formula 2	433	51,7	11,9	51,7	11,9

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After annex A, add the following new annex:

Annex B

(informative)

Guidance for using precision results

B.1 The general procedure for using precision results is as follows, with the symbol $|x_1 - x_2|$ designating a positive difference in any two measurement values (i.e. without regard to sign).

B.2 Enter the appropriate precision table (for whatever test parameter is being considered) at an average value (of the measured parameter) nearest to the "test" data average under consideration. This line will give the applicable r, (r), R or (R) for use in the decision process.

B.3 With these r and (r) values, the following general repeatability statements may be used to make decisions.

B.3.1 For an absolute difference: The difference $|x_1 - x_2|$ between two test (value) averages, found on nominally identical material samples under normal and correct operation of the test procedure, will exceed the tabulated repeatability r on average not more than once in twenty cases.

B.3.2 For a percentage difference between two test (value) averages: The percentage difference

$$\left[|x_1 - x_2| / (x_1 + x_2)/2\right] \times 100$$

between two test values, found on nominally identical material samples under normal and correct operation of the test procedure, will exceed the tabulated repeatability (r) on average not more than once in twenty cases.

B.4 With these R and (R) values, the following general reproducibility statements may be used to make decisions.

B.4.1 For an absolute difference: The absolute difference $|x_1 - x_2|$ between two independently measured test (value) averages, found in two laboratories using normal and correct test procedures on nominally identical material samples, will exceed the tabulated reproducibility R not more than once in twenty cases.

B.4.2 For a percentage difference between two test (value) averages: The percentage difference

$$\left[|x_1 - x_2| / (x_1 + x_2)/2\right] \times 100$$

between two independently measured test (value) averages, found in two laboratories using normal and correct test procedures on nominally identical material samples, will exceed the tabulated reproducibility (R) not more than once in twenty cases.