

INTERNATIONAL STANDARD

ISO
2322

Third edition
1985-10-15

AMENDMENT 1
1993-01-15

Rubber, styrene-butadiene (SBR, YSBR) – Emulsion and solution-polymerized types – Test recipes and evaluation of vulcanization characteristics

AMENDMENT 1

*Caoutchoucs butadiène-styrène (SBR, YSBR) – Types polymérisés en émulsion
et en solution – Formules d'essai et évaluation des caractéristiques*

AMENDEMENT 1



Reference number
ISO 2322:1985/Amd.1:1993 (E)

Foreword

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Amendment 1 to International Standard ISO 2322:1985 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*.

Annexes A and B of this amendment to ISO 2322 are for information only.

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Case postale 56 • CH-1211 Genève 20 • Switzerland

Printed in Switzerland

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AMENDMENT 1

Page 3 and subsequent pages

Clause 5

Replace clause 5 by the following text.

5 Precision

5.1 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 A gives guidance on the use of repeatability and reproducibility.

5.2 Precision details

5.2.1 An interlaboratory test programme (ITP) was organized in 1986. Two formulations for series A SBRs were selected:

- A-1 with an oil-extended SBR, type 1712, and
- A-2 with a non-oil-extended SBR, type 1500.

One formulation for series B SBRs was selected:

- A-3 with a high-styrene SBR rubber.

Mixes of these formulations were made in each of the 13 laboratories participating in the ITP, on each of two days approximately one week apart.

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.

5.2.2 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 on dumb-bell 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.

See annex B for some comments on the precision results.

5.3 Precision results

The precision results are given in table 3.

The symbols used in the table 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 %.

Table 3 – Type 2 precision for stress-strain tests

Compound or material	Average value	Within lab		Between labs	
		<i>r</i>	(<i>r</i>)	<i>R</i>	(<i>R</i>)
1) Modulus (300 %), MPa					
SBR A-2	12,3	1,62	13,1	3,83	31,1
SBR A-1	14,6	1,80	12,3	3,86	26,5
SBR A-3	16,0	2,36	14,8	6,12	38,2
2) Tensile strength, MPa					
SBR A-2	20,3	2,05	10,1	3,09	15,2
SBR A-3	23,4	4,70	20,1	4,70	20,1
SBR A-1	25,5	2,50	9,79	3,60	14,1
3) Percent elongation					
SBR A-3	434	52,0	11,9	200	46,2
SBR A-2	481	56,6	11,8	103	21,5
SBR A-1	481	51,6	10,7	66,2	13,8

Clause 6

After clause 5, add the following test report clause:

6 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 vulcanizing temperature and times used in 4.1;
- f) any unusual features noted during the determination;
- g) 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;
- h) the results and the units in which they have been expressed;
- i) the date of the test.

Annexes

After clause 6, add the following two informative annexes:

Annex A

(informative)

Guidance for using precision results

A.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).

A.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.

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

A.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.

A.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.

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

A.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.

A.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.

Annex B

(informative)

Comments on precision results

B.1 Table 3 indicates that the repeatability and the reproducibility are equal for tensile strength for SBR B-1. Such an occurrence can develop with a small data base (only a few laboratories) when the day-1 vs day-2 variations are fairly substantial, combined with the situation in which the day-1 and day-2 average gives rise to a reduced lab-to-lab variation.

B.2 Another way in which repeatability can be equal to reproducibility is a situation in which two main "within-lab" error-generating mechanisms are oper-

ating, i.e. the distribution of the cell standard deviations s_{ij} is bi-modal. The existence of the second upper (higher-value) peak inflates the within-laboratory r and (r) and causes the two values r and R to be equal. For this particular ITP with 13 laboratories, the existence of a bi-modal distribution is the most probable cause.

B.3 The precision results to be expected with SBR types other than those tested in this programme may be different.