
Rubber, raw natural — Determination of plasticity retention index (PRI)

*Caoutchouc naturel brut — Détermination de l'indice de rétention de
plasticité (PRI)*

Reference number ISO 2930:2009(E)



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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.

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 2930 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 fourth edition cancels and replaces the third edition (ISO 2930:1995), which has been technically revised. In this new edition, ISO 2930:1995/Amd 1:2008 was incorporated and the following modifications were therefore introduced:

- updating of the normative references in Clause 2 and in 4.7;
- addition of a sentence in 5.3 after the first paragraph;
- replacement of Clause 7 “Repeatability of results” with Clause 7 “Precision”, and deletion of Footnote 2);
- addition of a sentence at the end of Clause 6;
- addition of informative Annex A providing an updated precision statement;
- addition of a Bibliography.

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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 a method for determining the plasticity retention index (PRI) of raw natural rubber.

The PRI is a measure of the resistance of raw natural rubber to thermal oxidation. A high resistance to thermal oxidation is shown as a high value of the index.

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

ISO 2007:2007, *Rubber, unvulcanized — Determination of plasticity — Rapid-plastimeter method*

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

ISO 23529:—¹⁾, *Rubber — General procedures for preparing and conditioning test pieces for physical test methods*

3 Principle

The rapid plasticity numbers of unaged test pieces and test pieces aged by heating in an oven at 140 °C are determined using a parallel-plate plastimeter with a platen of diameter 10 mm and following the procedure specified in ISO 2007.

The PRI is the ratio of the rapid plasticity numbers after and before heating multiplied by 100.

4 Apparatus

4.1 Parallel-plate plastimeter, with a platen of diameter 10 mm, as specified in ISO 2007.

1) To be published. (Revision of ISO 23529:2004)

4.2 Punch, capable of compressing a portion of the material being tested to a thickness of approximately 3 mm and cutting out a disc of approximately 13 mm in diameter for the preparation of test pieces, as specified in ISO 2007.

4.3 Thickness gauge, having a scale graduated in unit divisions of 0,01 mm, fitted with a flat contact of diameter 10 mm and operating with a pressure of (20 ± 3) kPa.

4.4 Laboratory mixing mill, in conformity with the requirements of ISO 2393, but with the following characteristics:

- roll diameter: 150 mm to 250 mm;
- linear speed of back (fast) roll: $(14,6 \pm 0,5)$ m/min;
- roll speed ratio: 1:1,4;
- temperature: (27 ± 3) °C;
- roll length between guides: (265 ± 15) mm.

4.5 Oven, meeting the following requirements at 140 °C.

- The temperature in the vicinity of the test pieces shall be controllable to within $\pm 0,2$ °C over a 30 min period. Some ovens may not achieve this close temperature tolerance and may require a temperature tolerance of $\pm 0,5$ °C. This wider tolerance may impair the accuracy of the test. If an oven of tolerance $\pm 0,5$ °C is used, this shall be stated in the test report.
- Following insertion of the tray plus dishes into the oven, the temperature of the oven shall recover, and that of the tray plus dishes increase, to within 1 °C of the set temperature within 2 min.
- The air shall be changed ten times per hour.

4.6 Lightweight aluminium dishes and tray, with a low thermal capacity. The total mass of the tray and dishes shall not exceed 35 g, and their volume shall not exceed 5 % of the volume of the oven chamber. Dishes of diameter 40 mm to 50 mm made of foil of thickness 0,2 mm have been found suitable.

4.7 Tissue paper, as described in ISO 2007:2007, 4.3, or **cigarette paper** of about 22 g/m² cut into two equal pieces (approximately 30 mm × 45 mm).

5 Procedure

5.1 Preparation of test pieces

Homogenize the raw rubber as specified in ISO 1795. Take a test portion of (20 ± 2) g from the homogenized piece and pass twice (doubling the sheet between passes) between the rolls of the mill (4.4) at (27 ± 3) °C, running with the nip adjusted so that the final sheet thickness is about 1,7 mm.

In order to obtain a smooth sheet from old rubber, three passes may be necessary, in which case this shall be stated in the test report.

Immediately double the sheet, which shall be uniform in texture and free from holes, and press the two halves smoothly together by hand, avoiding the formation of air bubbles.

Cut test pieces as specified in ISO 2007 from the doubled sheet with the punch (4.2), and measure their thickness with the gauge (4.3) until six pieces are obtained with a thickness of $(3,4 \pm 0,4)$ mm. Randomly divide these test pieces into sets of three; one set for testing before ageing and the other for testing after ageing.

The preparation of test pieces, as described above, shall be carried out with care, since the PRI is affected by the sheet thickness. The required nip setting shall be ascertained by a preliminary trial. It will vary with the rubber and with the mill. If six test pieces of the required thickness, as above, are not obtained, a fresh doubled sheet shall be prepared.

5.2 Ageing

Before ageing is started, check the temperature of the oven (4.5) to ensure that it has been stable for at least 5 min.

To ensure that all test pieces are aged at the correct temperature, the oven shall not be overloaded as this would cause a marked and prolonged decrease in temperature and upset temperature uniformity (see 4.5).

Quickly insert the tray (4.6), close the oven door and start timing. Care shall be taken to ensure that the dishes and tray are arranged within the calibrated region of the oven. Check that the correct temperature is quickly regained and maintained (see 4.5).

After $(30 \pm 0,25)$ min, remove the tray from the oven and the dishes from the tray. Allow them to cool to standard laboratory temperature.

5.3 Determination of plasticity

Carry out in triplicate the rapid plasticity determination as specified in ISO 2007, using the equipment with a platen of diameter 10 mm as specified in 4.1, first on the unaged test pieces and then on the aged test pieces.

The laboratory temperature shall be in accordance with 3.1 of ISO 23529:—¹⁾.

These determinations shall normally be made at least 0,5 h and no more than 2 h after ageing, with the condition that the test pieces have been allowed to cool to room temperature. Plasticity determinations on unaged and aged test pieces should preferably be made concurrently using the same type of paper. The rapid plasticity number shall be read to the nearest 0,5 unit (1 unit corresponds to 10 μm).

6 Expression of results

Use the median values of the rapid plasticity numbers of the three unaged and three aged test pieces to calculate the PRI from the following equation:

$$\text{PRI} = \frac{\text{aged rapid plasticity number}}{\text{unaged rapid plasticity number}} \times 100$$

Round the result to the nearest whole number.

7 Precision

See Annex A.

8 Test report

The test report shall include the following information:

- a) a reference to this International Standard;
- b) all details necessary for identification of each sample tested;
- c) the median rapid plasticity number for the unaged test pieces and for the aged test pieces from each sample tested;
- d) the PRI for each sample tested;
- e) the type of oven used;
- f) the temperature tolerance of the oven used;
- g) the date of the test;
- h) the operator's identification;
- i) any operations not included in this International Standard or in the International Standards to which reference is made, and any operations regarded as optional.

Annex A (informative)

Precision statement for plasticity retention index

A.1 Background

An interlaboratory test programme (ITP) to determine the precision of the method specified in this International Standard was conducted in 2007, using the procedures and guidelines described in ISO/TR 9272.

The ITP was conducted on two types of material with different plasticity retention indices.

Nine laboratories participated in the ITP and a type 1 precision was evaluated. The test result was taken as the average of five replicate determinations carried out on each of two separate test days and the precision calculated using these average values (one for each test day) as the test results. For each replicate determination, the median plasticity number was calculated from three unaged and three aged test pieces.

The precision results obtained by this ITP should not be applied to acceptance or rejection testing of any group of materials or products without documentation that the results obtained from the ITP actually apply to the products or materials tested.

A.2 Precision results

A.2.1 General

For each of the two materials tested, the precision results are given in Table A.1. These results were obtained using the outlier replacement procedures and outlier deletion procedures described in ISO/TR 9272:2005. General statements for the use of the precision results are given in A.2.2 and A.2.3. They are given in terms of both the absolute precision, r and R , and the relative precision, (r) and (R) .

Table A.1 — Precision for plasticity retention index (PRI)

Material	Mean PRI	Within laboratory			Between laboratories			Number of laboratories
		s_r	r	(r)	s_R	R	(R)	
Material A (low PRI)	56	1,29	3,64	6,50	3,33	9,44	16,84	9
Material B (high PRI)	74	1,01	2,86	3,86	5,26	14,90	20,04	9

s_r is the within-laboratory standard deviation (in measurement units);

r is the repeatability (in measurement units);

(r) is the repeatability (in percent of mean value);

s_R is the between-laboratory standard deviation (in measurement units);

R is the reproducibility (in measurement units);

(R) is the reproducibility (in percent of mean value).

A.2.2 Repeatability

The repeatability, or local domain precision, for each material is given in Table A.1. Two single average 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, and (r) , in percent, should be considered as suspect, i.e. to have come from different populations, and should suggest that some appropriate investigative action be taken.

A.2.3 Reproducibility

The reproducibility, or global domain precision, for each material is given in Table A.1. Two single average 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, and (R) , in percent, should be considered as suspect, i.e. to have come from different populations, and should suggest that some appropriate investigative action be taken.

A.2.4 Bias

Bias is the difference between a measured average test result and a reference, or true, value for the measurement in question. Reference values do not exist for this test method and therefore bias cannot be determined.

Bibliography

- [1] ISO/TR 9272:2005, *Rubber and rubber products — Determination of precision for test method standards*

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