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

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

Reference number
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ISO copyright office
Ch. de Blandonnet 8 • CP 401
CH-1214 Vernier, Geneva, Switzerland
Tel. +41 22 749 01 11
Fax +41 22 749 09 47
copyright@iso.org
www.iso.org

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Foreword

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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).

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For an explanation on the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT) see the following URL: www.iso.org/iso/foreword.html.

This document 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 2930:2009), which has been technically revised.

The main changes compared to the previous edition are as follows:

— the normative references have been updated;

— a duration of 30 min has been added in Clause 4;

— the tolerance value for oven temperature has been changed to ±0,5 °C in 5.5;

— the description for lightweight aluminium tray and dishes in 5.6 has been modified;

— a new Annex A on the air change in oven for PRI determination has been added;

— Table B.1 has been updated, following the interlaboratory test programme conducted in 2014.

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

WARNING — Persons using this document should be familiar with normal laboratory practice. This document 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 document 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. PRI is not an absolute value and cannot give an absolute classification of plasticity number of different natural rubber after oxidation.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements 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, *Rubber, unvulcanized — Determination of plasticity — Rapid-plastimeter method*

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

ISO 23529:2016, *Rubber — General procedures for preparing and conditioning test pieces for physical test methods*

3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

— IEC Electropedia: available at http://www.electropedia.org/

— ISO Online browsing platform: available at http://www.iso.org/obp

3.1
plasticity number
measure of plasticity, based upon the height of a test piece after being subjected to deformation under specified conditions of compressive force, time and temperature

3.2
plasticity retention index
ratio of the *plasticity number* (3.1) measured after air-oven ageing for 30 min at 140 °C to the plasticity number before oven ageing

4 Principle

The rapid plasticity numbers of unaged test pieces and test pieces aged by heating in an oven at 140 °C for 30 min are determined using a parallel-plate plastimeter with a platen of diameter 10 mm, following the procedure specified in ISO 2007.

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

5 Apparatus

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

5.2 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.

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

5.4 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 ± 0,5) m/min;

— roll speed ratio: 1:1,4;

— temperature: (27 ± 3) °C;

— roll length between guides: (265 ± 15) mm.

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

— The temperature in the vicinity of the test pieces shall be controllable to within ±0,5 °C over a period of 30 min.

— 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 5 min.

— The air shall be changed 10 times per hour. Alternatively, the air flap of the oven can be set as semi-open.

NOTE Additional information on the air change in an oven is given in Annex A.

5.6 Lightweight aluminium dishes and tray, with a low thermal capacity.

A suitable size of tray and/or dishes should be used depending on the size of the oven.

5.7 Tissue paper, as described in ISO 2007, or **cigarette paper** of 22 g/m^2 to 26 g/m^2 cut into two equal pieces (approximately 30 mm × 45 mm).

6 Procedure

6.1 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 (5.4) at (27 ± 3) °C. Then, 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 and running with the nip adjusted so that the final sheet thickness is (3,4 ± 0,2) mm.

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

Cut test pieces, as specified in ISO 2007, from the doubled sheet with the punch (5.2), and measure their thickness with the gauge (5.3) until six pieces are obtained with a thickness of (3,4 ± 0,2) 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 described above, are not obtained, a fresh doubled sheet shall be prepared.

6.2 Ageing

Before ageing is started, check the temperature of the oven (5.5) 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 5.5).

Place the test pieces for ageing test on the dishes in the tray. Quickly insert the tray (5.6), close the oven door and start timing once the oven temperature has reached 140 °C ± 0,5 °C. Care shall be taken to ensure that the dishes and tray are arranged within the calibrated region of the oven. Check if the correct temperature is quickly regained and maintained (5.5).

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

6.3 Determination of plasticity

Carry out in triplicate the rapid plasticity determination, as specified in ISO 2007, using the equipment with a platen of 10 mm diameter, as specified in 5.1, first on the unaged test pieces and then on the aged test pieces.

The laboratory temperature shall be in accordance with ISO 23529:2016, 5.1.

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).

7 Expression of results

Use the median values of the rapid plasticity numbers of the three unaged and three aged test pieces to calculate the plasticity retention index, PRI, from Formula (1):

$$\mathrm{PRI} = \frac{n_{\mathrm{aged}}}{n_{\mathrm{unaged}}} \times 100 \tag{1}$$

where

n_{aged} is the aged rapid plasticity number;

n_{unaged} is the unaged rapid plasticity number.

Round the result to the nearest whole number.

8 Precision

See Annex B.

9 Test report

The test report shall include the following information:

a) a reference to this document, i.e. ISO 2930;

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 date of the test;

f) any operations not included in this document or other International Standards to which reference is made, and any operations regarded as optional.

Annex A
(informative)

Air change in oven for PRI determination

A.1 Background

Air change in an oven for PRI determination is one of the conditions to be controlled. An optimum air flow can guarantee a good heat distribution and ensure samples are aged evenly. A study to determine an alternative for the existing air flow requirement was conducted in 2016, using the procedures described in ISO 2930. The main objective in this study was to provide a practical option for the users to control the air change in the oven.

The study was conducted using four types of materials with different plasticity retention indices. The test result was taken as an average of 10 replicate determinations carried out for two days. For each replicate determination, the median plasticity number was calculated from three unaged and three aged test pieces.

Three conditions were studied, as an alternative to the requirement of 10 air changes per hour. The openings of an oven's air flap are positioned as follows:

a) fully closed;

b) fully open;

c) semi-open.

Results obtained from each of the above conditions were then compared with the results obtained from 10 air changes per hour. The results obtained were analysed using a *t*-test to determine whether there is a significant difference between the conditions with respect to their average PRI values.

A.2 Results

A.2.1 General

For each of the four materials tested, the *p*-value results for the analysis of 10 air changes per hour with each of the oven's air flap condition at an α level of 0,05 and average PRI for each oven's air change requirement are given in Table A.1.

The condition of a semi-open oven's air flap was found to be comparable with the results of 10 air changes per hour, whereby there are no significant differences between the averages of PRI results between both conditions.

General statements for the use of the *p*-value and α level are given in A.2.2 and A.2.3.

Table A.1 — Average PRI and *p*-value of test for air change control in an oven

Material	Air change requirement in oven						
	10 air changes per hour	Oven's air flap condition					
		Fully closed		Fully open		Semi-open	
	Average PRI	Average PRI	*p*-value	Average PRI	*p*-value	Average PRI	*p*-value
C	61	53	0,013 3	55	$1{,}98 \times 10^{-5}$	61	0,131 0
D	68	62	0,012 5	64	$9{,}67 \times 10^{-8}$	69	0,664 8
E	61	55	0,031 8	57	$1{,}04 \times 10^{-5}$	62	0,327 6
F	70	63	0,026 7	65	$4{,}04 \times 10^{-4}$	68	0,382 8

A.2.2 *p*-value

The *p*-value is the probability when assuming the null hypothesis is true. If the *p*-value is less than (or equal to) α, then the null hypothesis is rejected in favour of the alternative hypothesis. However, if the *p*-value is greater than α, then the null hypothesis is not rejected.

The null hypothesis is the hypothesis that there is no significant difference between specified populations.

An alternative is the hypothesis used in hypothesis testing that is contrary to the null hypothesis.

A.2.3 Alpha level

The alpha level (α) is the probability of rejecting the null hypothesis when it is true. Alpha levels, also denoted as significance levels, are used in hypothesis tests.

Annex B
(informative)

Precision statement for plasticity retention index

B.1 Background

An interlaboratory test programme (ITP) to determine the precision of the method specified in this document was conducted in 2014, using the procedures and guidelines described in ISO/TR 9272.

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

Twelve 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.

B.2 Precision results

B.2.1 General

For each of the two materials tested, the precision results are given in Table B.1. General statements for the use of the precision results are given in B.2.2 and B.2.3. They are given in terms of both the absolute precision, r and R, and the relative precision, (r) and (R).

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

Material	Average PRI	Within laboratory			Between laboratories			Number of laboratories
		s_r	r	(r)	s_R	R	(R)	
Material A	76	0,78	2,22	2,91	1,99	5,64	7,39	12
Material B	81	0,79	2,23	2,76	1,77	5,01	6,21	12

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 average 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 average value).

B.2.2 Repeatability

The repeatability, or local domain precision, for each material is given in Table B.1. Two single average test results obtained in the same laboratory (by the proper use of this document) 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.