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METHODS OF TESTING FOR RAW NATURAL RUBBER PART 6: DETERMINATION OF PLASTICITY RETENTION INDEX (PRI)

(Second Revision)

SRI LANKA STANDARDS INSTITUTION

Sri Lanka Standard METHODS OF TESTING FOR RAW NATURAL RUBBER PART 6: DETERMINATION OF PLASTICITY RETENTION INDEX (PRI) (Second Revision)

SLS 484 Part 6: 2018 (ISO 2930: 2017)

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SRI LANKA STANDARDS INSTITUTION
17, Victoria Place
Elvitigala Mawatha
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Sri Lanka Standard METHODS OF TESTING FOR RAW NATURAL RUBBER PART 6: DETERMINATION OF PLASTICITY RETENTION INDEX (PRI) (Second Revision)

FOREWORD

This Sri Lanka Standard was approved by the Sectoral Committee on Chemical and Polymer Technology and was authorized for adoption and publication as a Sri Lanka Standard by the Council of the Sri Lanka Standards Institution on 2018-11-16

This Sri Lanka Standard was first published in 1980 and First Revision which was an adoption of ISO 2930: 1995 was published in 2006. The International Standard ISO 2930: 1995 has been technically revised in 2017. ISO 2930: 2017 has been accepted to adopt as the second revision to **SLS 484: Part 6** to be referred for the determination of plasticity retention index of raw natural rubber.

This Standard is identical with ISO 2930: 2017, Rubber, raw natural — Determination of plasticity retention index (PRI), published by the International Organization for Standardization (ISO).

TERMINOLOGY AND CONVENTIONS:

The text of the International Standard has been accepted as suitable for publication, without deviation, as a Sri Lanka Standard. However, certain terminology and conventions are not identical with those used in Sri Lanka Standards. Attention is therefore drawn to the following:

- a) Wherever the words 'International Standard' appear referring to a particular standard they should be interpreted as "Sri Lanka Standard".
- b) The comma has been used throughout as a decimal marker. In Sri Lanka Standards it is the current practice to use the full point at the base as the decimal marker.
- c) Wherever page numbers are quoted, they are ISO page numbers.

SLS 484 Part 6: 2018 (ISO 2930: 2017)

Cross References

International Standard

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

Corresponding Sri Lanka Standard

SLS 1297 Method of sampling and further preparative procedures for rubber, raw natural and raw synthetic

SLS 484 Part 5 Methods of test for raw natural rubber Part 5: Rapid plasticity test

No corresponding Sri Lanka Standard

SLS 1323 Temperatures, humidities and times for the conditioning and testing of rubber

INTERNATIONAL STANDARD

SLS 484 PART 6: 2018 ISO 2930

Fifth edition 2017-08

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

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





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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.

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).

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. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see www.iso.org/patents).

Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

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 <u>Clause 4</u>;
- 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 <u>5.6</u> has been modified;
- a new Annex A on the air change in oven for PRI determination has been added;
- <u>Table B.1</u> 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 $140\,^{\circ}\text{C}$ for 30 min are determined using a parallel-plate plastimeter with a platen of diameter $10\,\text{mm}$, 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: $(14,6 \pm 0,5)$ 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 <u>Annex A</u>.

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 \pm 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 μ m).

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):

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

where

*n*_{aged} is the aged rapid plasticity number;

 $n_{\rm unaged}$ 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

	Air change requirement in oven							
	10 air	Oven's air flap condition						
Material	changes per hour	Fully closed		Full	y open	Semi-open		
	Average PRI	Average PRI	<i>p</i> -value	Average PRI	<i>p</i> -value	Average PRI	<i>p-</i> value	
С	61	53	0,013 3	55	1,98 × 10 ⁻⁵	61	0,131 0	
D	68	62	0,012 5	64	9,67 × 10 ⁻⁸	69	0,6648	
Е	61	55	0,0318	57	1,04 × 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 <u>Table B.1</u>. General statements for the use of the precision results are given in <u>B.2.2</u> and <u>B.2.3</u>. 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	Within laboratory			Between laboratories			Number of
	PRI	Sr	r	(r)	s_R	R	(R)	laboratories
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 <u>Table B.1</u>. 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.

B.2.3 Reproducibility

The reproducibility, or global domain precision, for each material is given in <u>Table B.1</u>. Two single average test results obtained in different laboratories (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.

B.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, Rubber and rubber products — Determination of precision for test method standards



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