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METHODS OF TEST FOR
RAW NATURAL RUBBER
PART 10 : DETERMINATION OF VOLATILE MATTER CONTENT BY HOT-MILL METHOD AND OVEN METHOD

SRI LANKA STANDARDS INSTITUTION

Sri Lanka Standard

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SLS 484 Part 10 : 2014
ISO 248 Part 1 : 2011
(superseding SLS 484 : Part 4)

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Sri Lanka Standard
METHODS OF TEST FOR RAW NATURAL RUBBER
PART 10 : DETERMINATION OF VOLATILE MATTER CONTENT BY HOT-MILL METHOD AND OVEN METHOD

NATIONAL FOREWORD

This Sri Lanka Standard was approved by the Sectoral Committee on Chemical and Polymer Technology and authorized for adoption and publication as a Sri Lanka Standard by the council of the Sri Lanka Standards Institution on 2014-09-02.

This Standard superseded by SLS 484 Method of test for raw natural rubber Part 4 : Determination of volatile matter. This Standard is identical with ISO 248 - 1 Rubber raw determination of volatile matter content Part 1 : Hot-mill method and oven method published by the International Organization for standardization in 2011.

TERMINOLOGY AND CONVENTIONS

The text of the International Standard has been accepted as a 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/Publication” appear referring to this 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 line as the decimal marker.

c) Wherever page numbers are quoted, they are ISO/IEC page numbers.

Cross References

International Standard	Corresponding Sri Lanka Standard
ISO 1629 Rubber and lattices Nomenclature	No corresponding Sri Lanka Standard.
ISO 1795 Rubber raw natural and raw synthetic – Sampling and further preparative procedures.	SLS 1297 Method of sampling and further preparative procedures for rubber, raw natural and raw synthetic.
ISO 2393 Rubber test mixes – preparation, mixing and vulcanization- equipment and procedures.	No corresponding Sri Lanka Standard.

SLS 484-10 :2014

INTERNATIONAL STANDARD

ISO 248-1

First edition
2011-10-15

Rubber, raw — Determination of volatile-matter content —

Part 1:
Hot-mill method and oven method

Caoutchouc brut — Détermination des matières volatiles —

Partie 1: Méthode par mélangeage à chaud et méthode par étuvage

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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 248-1 was prepared by Technical Committee ISO/TC 45, *Rubber and rubber products*, Subcommittee SC 2, *Testing and analysis*.

This first edition cancels and replaces ISO 248:2005, which has been technically revised.

ISO 248 consists of the following parts, under the general title *Rubber, raw — Determination of volatile-matter content*:

- *Part 1: Hot-mill method and oven method*
- *Part 2: Thermogravimetric methods using an automatic analyser with an infrared drying unit*

Rubber, raw — Determination of volatile-matter content —

Part 1:
Hot-mill method and oven method

WARNING — Persons using this part of ISO 248 should be familiar with normal laboratory practice. This part of ISO 248 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.

IMPORTANT — Certain procedures specified in this part of ISO 248 might involve the use or generation of substances, or the generation of waste, that could constitute a local environmental hazard. Reference should be made to appropriate documentation on safe handling and disposal after use.

1 Scope

1.1 This part of ISO 248 specifies two methods for the determination of volatile-matter content in raw rubbers by using a hot mill or an oven.

1.2 The methods are applicable to the determination of the volatile-matter content in the “R” group of rubbers listed in ISO 1629. These are rubbers having an unsaturated carbon chain, for example natural rubber and synthetic rubbers derived at least partly from diolefins. The methods can also be applicable to other raw rubbers, but in these cases it is necessary to demonstrate that the change in mass is due solely to loss of actual volatile matter and not to rubber degradation.

1.3 The hot-mill method is not applicable to natural rubber, to synthetic rubbers which are too difficult to handle on a hot mill or to synthetic rubbers in powder or chip form.

1.4 The test methods do not necessarily give identical results. Therefore, in cases of dispute, the oven method, procedure A, is the reference method.

NOTE The applicability of each test method to various types of rubber is summarized in Annex A.

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 1629, *Rubber and latices — Nomenclature*

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

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

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 1795 and the following apply.

3.1
test portion
quantity of rubber taken from the test sample or laboratory sample for the purpose of a single specific test, for example the amount actually weighed out for a single determination of the volatile-matter content

4 Principle

4.1 Hot-mill method

A test portion is sheeted out on a heated mill until all volatile matter is driven off. The loss in mass during milling is calculated and expressed as the volatile-matter content. If the test portion is homogenized in accordance with Annex B before drying, the loss in mass during the homogenization is included in the calculation.

4.2 Oven method

A test portion is dried in an oven to constant mass. The loss in mass is calculated and expressed as the volatile-matter content. If the test portion is homogenized in accordance with Annex B before drying, the loss in mass during the homogenization is included in the calculation.

5 Hot-mill method

5.1 General

5.1.1 Two procedures are specified, as follows:

- Hot-mill method, procedure A: A test sample is homogenized using a laboratory mill, and a test portion from the homogenized test sample is dried to constant mass using a hot mill.
- Hot-mill method, procedure B: A test portion is dried to constant mass using a hot mill.

NOTE Procedure B is a simplified one which does not include a homogenization process.

5.1.2 When the sample is flaky or becomes sticky on the hot mill, making weighing difficult or impossible, the oven method shall be used.

5.1.3 The number of test portions shall be agreed between the interested parties.

5.2 Apparatus

5.2.1 **Mixing mill**, complying with the requirements of ISO 2393.

5.2.2 **Balance**, capable of weighing to the nearest 0,1 g.

5.3 Procedure

5.3.1 Hot-mill method, procedure A

5.3.1.1 Take a test sample of about 250 g from the laboratory sample in accordance with ISO 1795 and homogenize it in accordance with Annex B. Weigh to the nearest 0,1 g before and after homogenization (masses m_1 and m_2, respectively). Cut test portions needed for other chemical and physical tests from the homogenized test sample, if necessary.

5.3.1.2 Adjust the clearance of the mill rolls to 0,25 mm ± 0,05 mm, using lead strips as specified in ISO 2393. Maintain the surface temperature of the rolls at 105 °C ± 5 °C.

5.3.1.3 Pass a weighed test portion (mass m_3), preferably of 100 g or more, taken from the homogenized test sample repeatedly through the mill for 4 min. Do not allow the test portion to band and take care to prevent any loss of material. Weigh the test portion to the nearest 0,1 g. Pass the test portion through the mill for an additional 2 min and reweigh. If the masses at the end of the 4 min and 6 min periods differ by less than 0,1 g, calculate the volatile-matter content.

If not, continue passing the test portion through the mill for 2 min periods until the mass does not decrease by more than 0,1 g between successive weighings (final mass m_4). Before each weighing, allow the test portion to cool to room temperature in a desiccator.

5.3.2 Hot-mill method, procedure B

5.3.2.1 Take a test portion of about 250 g from the laboratory sample and weigh to the nearest 0,1 g (mass m_5).

5.3.2.2 Adjust the clearance of the mill rolls to 0,25 mm ± 0,05 mm, using lead strips as specified in ISO 2393. Maintain the surface temperature of the rolls at 105 °C ± 5 °C. Pass the test portion through the mill not less than twice, then reweigh to the nearest 0,1 g, followed by passing through the mill not less than twice again and reweighing.

5.3.2.3 If the difference in mass of the test portion before and after it is passed through the rolls is less than 0,1 g, the test portion is considered to be well dried. If it is not well dried, continue passing the test portion twice through the rolls until the mass difference is less than 0,1 g (final mass m_6).

NOTE Cooling in a desiccator before weighing is desirable.

5.4 Expression of results

5.4.1 Hot-mill method, procedure A

The volatile-matter content w_1 is given, as a percent mass fraction, by the formula:

$$w_1 = \left(1 - \frac{m_2 \times m_4}{m_1 \times m_3}\right) \times 100$$

where

m_1 is the mass, in grams, of the test sample before homogenization;

m_2 is the mass, in grams, of the test sample after homogenization;

m_3 is the mass, in grams, of the test portion before milling;

m_4 is the mass, in grams, of the test portion after milling.

5.4.2 Hot-mill method, procedure B

The volatile-matter content w_2 is given, as a percent mass fraction, by the following formula:

$$w_2 = \frac{m_5 - m_6}{m_5} \times 100$$

where

m_5 is the mass, in grams, of the test portion before milling;

m_6 is the mass, in grams, of the test portion after milling.

6 Oven method

6.1 General

6.1.1 Two procedures are specified, as follows:

- Oven method, procedure A: A test sample is homogenized using a laboratory mill, and a test portion taken from the homogenized test sample is dried in an oven to constant mass. If the sample is in powder form or impossible to weigh before and after homogenization, a test portion shall simply be dried, without carrying out the homogenization process.
- Oven method, procedure B: A test sample is sheeted out using a laboratory mill, and a test portion taken from the sheeted test sample is dried in an oven for 1 h. If the sample is in powder form or is difficult to pass through the mill, the test portion shall simply be dried, without carrying out the sheeting process. This procedure is only applicable to synthetic rubbers since natural rubber requires homogenization.

6.1.2 The number of test portions shall be agreed between the interested parties.

6.2 Apparatus

6.2.1 **Oven**, ventilated, preferably of the air-circulation type, capable of being maintained at 105 °C ± 5 °C.

6.2.2 **Balance**, capable of weighing to the nearest 0,1 mg.

6.2.3 **Mixing mill**, complying with the requirements of ISO 2393.

6.3 Procedure

6.3.1 Oven method, procedure A

6.3.1.1 Natural rubber

6.3.1.1.1 Take a test sample of about 600 g from the laboratory sample in accordance with ISO 1795 and homogenize it in accordance with Annex B. Weigh the test sample to the nearest 0,1 g before and after this homogenization (masses m_7 and m_8, respectively). Allow to cool to room temperature before the final weighing. Cut test portions needed for other chemical and physical tests from the homogenized test sample, if necessary.

6.3.1.1.2 Take a test portion of about 10 g from the homogenized test sample and weigh it to the nearest 1 mg (mass m_9).

6.3.1.1.3 Pass the test portion twice through the mill with the rolls set at 70 °C ± 5 °C and with a mill opening which will produce a sheet of less than 2 mm thickness.

6.3.1.1.4 Dry the test portion for 1 h in the oven, maintained at 105 °C ± 5 °C, with the ventilators open and with the air-circulating fan, if fitted, switched on. Arrange the test portion so as to present the largest possible surface area to the hot air. Allow to cool to room temperature in a desiccator and weigh. Repeat the heating for further 30 min periods until the mass does not decrease by more than 1 mg between successive weighings (final mass m_{10}).

6.3.1.1.5 If the sample is in powder form, take a test portion of about 10 g at random and place it on a clean watch-glass or a clean aluminium tray to facilitate weighing. Weigh it to the nearest 1 mg (mass m_9). Dry the test portion in accordance with 6.3.1.1.4 and weigh to the nearest 1 mg (final mass m_{10}).

6.3.1.2 Synthetic rubber

6.3.1.2.1 Take a test sample of about 250 g from the laboratory sample in accordance with ISO 1795 and homogenize it in accordance with Annex B. Weigh the test sample to the nearest 0,1 g before and after this homogenization (masses m_7 and m_8, respectively). Cut test portions needed for other chemical and physical tests from the homogenized test sample, if necessary.

6.3.1.2.2 Take a test portion of about 10 g from the homogenized test sample and weigh it to the nearest 1 mg (mass m_9).

6.3.1.2.3 Pass the test portion twice through the mill with the rolls set at 70 °C ± 5 °C and with a mill opening which will produce a sheet of less than 2 mm thickness.

6.3.1.2.4 When sheeting is impossible, take a test portion of about 10 g from the homogenized test sample and cut it by hand into small cubes with edges of 2 mm to 5 mm. Place the cubes on a clean watch-glass or a clean aluminium tray to facilitate weighing. Weigh to the nearest 1 mg (mass m_9).

6.3.1.2.5 Dry the test portion in accordance with 6.3.1.1.4 and weigh to the nearest 1 mg (final mass m_{10}).

6.3.1.2.6 If it is difficult to weigh the test portion before and after the homogenization process because it sticks to the roll surfaces, take a test portion of about 10 g directly from the laboratory sample and cut it by hand into small cubes with edges of 2 mm to 5 mm. Place the cubes on a clean watch-glass or a clean aluminium tray to facilitate weighing. Weigh to the nearest 1 mg (mass m_9). Dry the test portion in accordance with 6.3.1.1.4 and weigh to the nearest 1 mg (final mass m_{10}).

6.3.1.2.7 If the sample is in powder form, take a test portion of about 10 g at random and place it on a clean watch-glass or a clean aluminium tray to facilitate weighing. Weigh to the nearest 1 mg (mass m_9). Dry the test portion in accordance with 6.3.1.1.4 and weigh to the nearest 1 mg (final mass m_{10}).

6.3.2 Oven method, procedure B

6.3.2.1 Take a test sample of about 250 g and pass it through the mill with the surface temperature of the rolls adjusted to about 30 °C and the roll clearance to 0,25 mm ± 0,05 mm to obtain a thin sheet. From the sheeted test sample, take two test portions of about 50 g and weigh each to the nearest 10 mg (mass m_{11}). Dry the test portions for 1 h in the oven, maintained at 105 °C ± 5 °C. Remove the test portions from the oven and cool to room temperature in a desiccator. Reweigh to the nearest 10 mg (mass m_{12}).

6.3.2.2 If the sample is in powder form or is impossible to sheet because it sticks to the roll surfaces or because it is flaky, take two test portions of about 10 g directly from the laboratory sample. Cut the test portions by hand into small cubes with edges of 2 mm to 5 mm, if necessary. Place each test portion in a tared clean aluminium tray of 15 mm depth and 60 mm diameter, or of similar shape, and weigh to the nearest 1 mg (mass m_{11}). Place the trays containing the test portions in the oven, maintained at 105 °C ± 5 °C, for 1 h. Remove the trays from the oven and allow to cool in a desiccator to room temperature. Reweigh to the nearest 1 mg (mass m_{12}).

6.4 Expression of results

6.4.1 Oven method, procedure A

6.4.1.1 When the test portion is taken from a homogenized test sample (see 6.3.1.1.2, 6.3.1.2.2 and 6.3.1.2.4), the volatile-matter content w_3 is given, as a percent mass fraction, by the formula:

$$w_3 = \left(1 - \frac{m_8 \times m_{10}}{m_7 \times m_9}\right) \times 100$$

where

m_7 is the mass, in grams, of the test sample before homogenization;

m_8 is the mass, in grams, of the test sample after homogenization;

m_9 is the mass, in grams, of the test portion before drying;

m_{10} is the mass, in grams, of the test portion after drying.

6.4.1.2 When the sample is in powder form or sticks to the roll surface (see 6.3.1.1.5, 6.3.1.2.6 and 6.3.1.2.7), the volatile-matter content w_4 is given, as a percent mass fraction, by the formula:

$$w_4 = \frac{m_9 - m_{10}}{m_9} \times 100$$

where

m_9 is the mass, in grams, of the test portion before drying;

m_{10} is the mass, in grams, of the test portion after drying.

6.4.2 Oven method, procedure B

The volatile-matter content w_5 is given, as a percent mass fraction, by the formula:

$$w_5 = \frac{m_{11} - m_{12}}{m_{11}} \times 100$$

where

m_{11} is the mass, in grams, of the test portion before drying;

m_{12} is the mass, in grams, of the test portion after drying.

The test result is the average of the results for the duplicate test portions.

7 Precision

See Annex C.

8 Test report

The test report shall include the following particulars:

a) all details necessary for full identification of the raw rubber tested;

b) test method:

    1) a reference to this part of ISO 248,

    2) the method used (hot-mill method, procedure A, hot-mill method, procedure B, oven method, procedure A, or oven method, procedure B);

c) details of the test:

    1) the number of test portions tested,

    2) details of any operation not specified in this part of ISO 248 or regarded as optional,

    3) any unusual features noted during the determination;

d) results of the test;

e) the date of the test.

Annex A
(informative)

Selection of appropriate test method

A.1 Rubbers in the "R" group listed in ISO 1629

Table A.1 summarizes the applicability of the test methods specified in this part of ISO 248 for rubbers in the "R" group listed in ISO 1629.

Table A.1 — Rubbers and applicable test methods

Method		**Rubbers in the "R" group listed in ISO 1629**					
		Natural rubber		**Synthetic rubber**			
		With homogenization	In powder form	Possible to weigh before and after homogenization		Impossible to weigh before and after homogenization	In powder form
		Sheeted out		Possible to sheet out	Impossible to sheet out	Sticks to roll surface	
Hot mill	Procedure A	N	N	Y	N	N	N
	Procedure B	N	N	Y	N	N	N
Oven	Procedure A	Y	Y	Y	Y	Y	Y
	Procedure B	N	N	Y	Y	Y	Y

Y: applicable.

N: not applicable.

A.2 Other rubbers which are not in the "R" group listed in ISO 1629

When the test method specified in this part of ISO 248 is used for rubbers other than those in the "R" group, it will be necessary to demonstrate that the change in mass is due solely to loss of original volatile matter and not to rubber degradation. Table A.1 may be used to select the test method for the rubber under test.

Annex B
(normative)

Homogenization

B.1 Apparatus

B.1.1 **Roll mill**, as specified in ISO 2393, for homogenizing test samples.

B.2 Procedure

B.2.1 Weigh the test sample specified for the test method concerned to the nearest 0,1 g and homogenize it by passing it ten times between the surfaces of the mill rolls with the nip set at 1,3 mm ± 0,15 mm and with the surface temperature of the rolls maintained at 70 °C ± 5 °C.

B.2.2 In passes two to nine inclusive, roll up the test sample after passing it through the nip and present the roll endwise to the nip for the next pass. Return to the test sample any solid matter separating from it.

B.2.3 On the tenth pass, sheet the test sample, allow it to cool in a desiccator and weigh it again to the nearest 0,1 g.

Annex C
(informative)

Precision

C.1 General

The precision calculations to provide repeatability and reproducibility values were performed in accordance with ISO/TR 9272:1986. Consult this for precision concepts and nomenclature.

C.2 Details of an ITP conducted in 1984

C.2.1 An interlaboratory test programme (ITP) was organized in late 1984 by the Rubber Research Institute of Malaysia. Two separate programmes were conducted, one in March and one in July. Two types of material were sent to each laboratory:

a) blended samples of two rubbers “A” and “B”;

b) unblended (normal) samples of the same two materials “A” and “B”.

C.2.2 For both the blended and the unblended samples, the test result was taken as the mean of three separate determinations.

C.2.3 The oven method, procedure A, was used.

C.2.4 A “type 1” precision was measured in the ITP. The time period for repeatability and reproducibility was on a scale of days. A total of 14 laboratories participated in the programme for blended samples and a total of 13 laboratories in the programme for unblended samples.

C.3 Details of an ITP conducted in 2003

C.3.1 An ITP was conducted between April and May 2003 with the participation of seven laboratories for the hot-mill method, procedure B, and eight laboratories for the oven method, procedure B.

C.3.2 Two samples of raw rubber, sample C (SBR 1500) and sample D (non-oil-extended BR), were used for both methods.

C.3.3 The results given in Table C.3 for the oven method, procedure B, and in Table C.4 for the hot-mill method, procedure B, are averaged values and give an estimate of the precision of these test methods, as determined in the ITP, for laboratories performing duplicate analyses on two raw-rubber samples.

C.4 Precision results

The results of the 1984 ITP for the blended samples are given in Table C.1 and the results for the unblended samples in Table C.2.

The results of the 2003 ITP for the oven method, procedure B, are given in Table C.3 and those for the hot-mill method, procedure B, in Table C.4.

The precision results as determined by these ITPs should not be applied to acceptance or rejection testing of any group of materials or products without documentation that the results of this precision evaluation actually apply to the products or materials tested.

Table C.1 — Precision data for the oven method, procedure A — Blended-sample testing

Sample	**Average volatile-matter content**	**Within-lab**		**Between labs**	
	% (mass fraction)	r	(r)	R	(R)
A	0,37	0,031	8,54	0,154	41,9
B	0,37	0,032	8,71	0,151	40,7
Pooled values	0,37	0,032	8,62	0,152	41,3

Symbols are as follows:

r is the repeatability limit, in percent mass fraction;

(r) is the repeatability limit, in percent (relative) of the average;

R is the reproducibility limit, in percent mass fraction;

(R) is the reproducibility limit, in percent (relative) of the average.

Table C.2 — Precision data for the oven method, procedure A — Unblended-sample testing

Sample	**Average volatile-matter content**	**Within-lab**		**Between labs**	
	% (mass fraction)	r	(r)	R	(R)
A	0,35	0,081	22,9	0,257	73,1
B	0,40	0,091	23,1	0,299	74,5
Pooled values	0,37	0,086	23,0	0,279	74,6

See Table C.1 for symbol definitions.

Table C.3 — Precision data for the oven method, procedure B

Sample	**Average volatile-matter content**	**Within lab**			**Between labs**		
	% (mass fraction)	s_r	r	(r)	s_R	R	(R)
C (SBR)	0,10	0,02	0,04	45,7	0,02	0,06	67,6
D (BR)	0,22	0,03	0,08	35,1	0,08	0,22	99,2

s_r is the repeatability standard deviation;

s_R is the reproducibility standard deviation.

See Table C.1 for other symbol definitions.

Table C.4 — Precision data for the hot-mill method, procedure B

Sample	Average volatile-matter content	Within-lab			Between labs		
	% (mass fraction)	s_r	r	(r)	s_R	R	(R)
C (SBR)	0,07	0,02	0,07	97,8	0,03	0,10	137,3
D (BR)	0,23	0,04	0,10	44,7	0,06	0,18	80,5

s_r is the repeatability standard deviation;

s_R is the reproducibility standard deviation.

See Table C.1 for other symbol definitions.

Bibliography

[1] ISO/TR 9272:1986, *Rubber and rubber products — Determination of precision for test method standards* [now withdrawn]

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