INTERNATIONAL STANDARD

ISO 248-1

Second edition
2021-06

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

Reference number
ISO 248-1:2021(E)

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Published in Switzerland

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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 of 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 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 second edition cancels and replaces the first edition (ISO 248-1:2011), which has been technically revised.

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

— in 6.2.1, update of the tolerance temperature of the oven;

— in 6.3.1 and 6.3.2, update of the procedure: mass of the test sample, drying of the test portion, temperature, requirements on the aluminium tray;

— update of the formulae;

— in Annex B, update of the homogenization procedure for natural rubber;

— in Annex C, addition of the precision data from an ITP conducted in 2020 for natural rubber.

A list of all parts in the ISO 248 series can be found on the ISO website.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

Rubber, raw — Determination of volatile-matter content —

Part 1:
Hot-mill method and oven method

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.

IMPORTANT — Certain procedures specified in this document 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

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

These 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 di-olefins. These 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.

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.

The two methods do not necessarily give identical results. Therefore, in the case 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 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 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.

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

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

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

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 weighted 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 volatile-matter content.

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.

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 method 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 the test portions needed for 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 roll 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 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 to the nearest 0,1 g, followed by passing through the mill for an additional 2 min and reweigh.

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 through the rolls for an additional 2 min 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 Formula (1):

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

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 Formula (2):

$$w_2 = \frac{m_5 - m_6}{m_5} \times 100 \quad (2)$$

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 sample shall simply be dried, without carrying out the homogenizing process.

— Oven method, procedure B: a test sample is sheeted out using a laboratory mill, then a test portion taken from the sheeted test sample is dried in an oven to constant mass. 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 air-circulating type, capable of being maintained at 105 °C ± 3 °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 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_7 and m_8, respectively). Allow to cool to room temperature before the final weighing. Cut the test pieces for 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 at room temperature 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 in the oven, maintained at 105 °C ± 3 °C at least for 3 ½ h, with the ventilators open and with the air-circulating fan, if fitted, switched on. Arrange the test piece 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, which has been weighed to the nearest 1 mg (mass m_0) to facilitate weighing. Weigh the test portion including the watch-glass/tray to the nearest 1 mg (mass m_{11}). Dry the test sample in accordance with 6.3.1.1.4 and weigh the tray including dried test portion to the nearest 1 mg (final mass m_{12}).

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 to the nearest 0,1 g before and after homogenization (masses m_7 and m_8, respectively). Cut the test portion for 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' temperature 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 for 1 h in the oven, maintained at 105 °C ± 3 °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 to the nearest 1 mg. Repeat the heating for further 30 min periods until the mass does not decrease by more than 1 mg between successive weighing (final mass m_{10}).

6.3.1.2.6 If it is difficult to weigh the test portion before and after a homogenization process because it sticks to roll surfaces, take a test portion of about 10 g 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, which has been weighed to the nearest 1 mg (mass m_0), to facilitate weighing. Weigh the test portion including the watch-glass/tray to the nearest 1 mg (mass m_{11}). Dry the test sample in accordance with 6.3.1.2.5 and weigh to the nearest 1 mg (final mass m_{12}).

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, which has been weighed to the nearest 1 mg (mass m_0) to facilitate weighing. Weigh the test portion including the watch-glass/tray to the nearest 1 mg (mass m_{11}). Dry the test sample in accordance with 6.3.1.2.5 and weigh the tray including dried test portion to the nearest 1 mg (final mass m_{12}).

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_{13}). Dry the test portions in accordance with 6.3.1.2.5. Remove the test portions from the oven and cool to room temperature in a desiccator. Reweigh to the nearest 10 mg (mass m_{14}).

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 clean aluminium tray of 15 mm depth and 60 mm diameter, or of similar shape, which has been weighed (mass m_0), and weigh the tray including test portion to the nearest 1 mg (mass m_{15}). Dry the

trays containing the test portions in accordance with 6.3.1.2.5. Remove the trays from the oven and allow to cool in a desiccator to room temperature. Reweigh the tray including dried test portion to the nearest 1 mg (mass m_{16}).

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 6.3.1.1.1, 6.3.1.1.2, 6.3.1.1.4, 6.3.1.2.1, 6.3.1.2.2 and 6.3.1.2.5), the volatile-matter content w_3 is given, as a percent mass fraction, by Formula (3):

$$w_3 = \left(1 - \frac{m_8 \times m_{10}}{m_7 \times m_9}\right) \times 100 \tag{3}$$

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 sticky 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 Formula (4):

$$w_4 = \frac{m_{11} - m_{12}}{m_{11} - m_0} \times 100 \tag{4}$$

where

m_0 is the mass, in grams, of the aluminium tray;

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

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

6.4.2 Oven method, procedure B

6.4.2.1 When the test portion is taken from a sheeted test sample (see 6.3.2.1), the volatile-matter content w_5 is given, as a percent mass fraction, by Formula (5):

$$w_5 = \frac{m_{13} - m_{14}}{m_{13}} \times 100 \tag{5}$$

where

m_{13} is the mass, in grams, of the test portion before drying;

m_{14} is the mass, in grams, of the test portion after drying.

6.4.2.2 When the sample is in powder form or sticky to the roll surface (see 6.3.2.2), the volatile-matter content w_6 is given, as a percent mass fraction, by Formula (6):

$$w_6 = \frac{m_{15} - m_{16}}{m_{15} - m_0} \times 100 \quad (6)$$

where

m_0 is the mass, in grams, of the aluminium tray;

m_{15} is the mass, in grams, of the test portion and the aluminium tray before drying;

m_{16} is the mass, in grams, of the test portion and the aluminium tray after drying.

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 document, i.e. ISO 248-1:2021;

   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 document 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 document for rubbers in the "R" group listed in ISO 1629.

Table A.1 — Rubbers and applicable test method

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 document 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 sample.

B.2 Procedure

B.2.1 Natural rubber

Weigh 250 g ± 5 g of the laboratory sample to the nearest 0,1 g and homogenize it by passing it six times between the surfaces of the mill rolls with the nip set at 1,69 mm ± 0,17 mm and with the surface temperature of the rolls maintained at room temperature. The water passing through the rolls shall not be heated.

In second to fifth passes, roll-up the rubber after passing it through the nip and present the roll endwise to the nip for the next pass. Return to the rubber any solid matter separating from it.

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

NOTE A laboratory sample for homogenizing which is larger than 250 g is acceptable depending on the tests to be carried.

B.2.2 Synthetic rubber

Weigh a test sample as specified for each test method to the nearest 0,1 g and homogenize it by passing 10 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.

In second to ninth passes, 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.

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 express repeatability and reproducibility were performed in accordance with ISO/TR 9272, for the ITP conducted in 2003, and in accordance with ISO 19983, for the ITP conducted in 2020. ISO/TR 9272 and ISO 19983 provide precision concepts and nomenclature. For the general procedure for using precision results, see ISO 19983.

C.2 Details of the ITP conducted in 2020

C.2.1 An interlaboratory test programme (ITP) for the oven method, procedure A, was organized in July 2020. Two separate programmes were conducted, and two types of materials were sent to each laboratory:

a) blended samples of two materials "A - SVR CV" and "B – SVR 10";

b) unblended (normal) samples of the same two materials "A – SVR CV" and "B – SVR 10".

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 A "type 1" precision was measured in the ITP. The precision calculations to express repeatability and reproducibility were performed in accordance with ISO 19983:2017, method B, and the outliers data were treated by using Algorithm A and Algorithm S (see ISO 5725-5). The time period for repeatability and reproducibility was on a scale of days. A total of seven laboratories participated in the programme for blended samples and a total of seven laboratories in the programme for the unblended samples.

C.2.4 The results given in Table C.1 for the blended samples, and Table C.2 for the unblended samples, are averaged values and give an estimate of the precision of the test method, as determined in the ITP, for laboratories performing triplicate analyses on two raw-rubber samples.

C.3 Details of the 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. The precision calculations to express repeatability and reproducibility were performed in accordance with ISO/TR 9272.

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