INTERNATIONAL STANDARD

ISO 248-2

Second edition
2019-03

Rubber, raw — Determination of volatile-matter content —

Part 2: Thermogravimetric methods using an automatic analyser with an infrared drying unit

Caoutchouc brut — Détermination des matières volatiles — Partie 2: Méthodes thermogravimétriques utilisant un analyseur automatique avec une unité de séchage infrarouge

Reference number
ISO 248-2:2019(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 248-2:2012), which has been technically revised.

The main change compared to the previous edition is the addition of precision data for isoprene rubber in Table B.2, following an additional ITP conducted by Japan in 2017.

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 2: Thermogravimetric methods using an automatic analyser with an infrared drying unit

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.

CAUTION — Some procedures specified in this document could 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 document specifies two thermogravimetric methods for the determination of moisture and other volatile-matter content in raw rubbers by using an automatic analyser with an infrared drying unit.

1.2 These methods are applicable to the determination of volatile-matter content in synthetic rubbers (SBR, NBR, BR, IR, CR, IIR, halogenated IIR and EPDM) listed in ISO 1629 and to various forms of raw rubber, such as bale, block, chip, pellet, crumb, powder and sheet. These methods might also be applicable to other raw rubbers only when the change in mass is proven to be due solely to loss of original volatile matter and not to rubber degradation.

1.3 The methods are not applicable to raw rubbers which need homogenizing as specified in ISO 1795.

1.4 The hot-mill method and the oven method specified in ISO 248-1 and the methods specified in this document might not give identical results. In cases of dispute, therefore, the oven method, procedure A, specified in ISO 248-1:2011, is the referee method.

NOTE These methods can be useful for routine determinations, e.g. quality control, when the measurement conditions for the automatic analyser are fixed for a particular raw rubber or grade of raw rubber.

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 248-1, *Rubber, raw — Determination of volatile-matter content — Part 1: Hot-mill method and oven method*

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

3 Terms and definitions

No terms and definitions are listed in this document.

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/

4 Principle

A test portion is continuously weighed to constant mass by a thermogravimetric method using an automatic analyser with infrared drying. The volatile-matter content is calculated as the mass lost during this procedure.

5 Reagents

Use only reagents of recognized analytical grade and only distilled water or water of equivalent purity.

5.1 Sodium L-tartrate dihydrate, purity ≥99 %, for use as a standard reference material.

6 Apparatus

Use ordinary laboratory apparatus and the following.

6.1 Automatic analyser

The automatic analyser shall consist of the following components:

a) an infrared drying unit or a far-infrared drying unit or a near-infrared drying unit;

b) a balance, capable of weighing to the nearest 1 mg;

c) a microprocessor, capable of controlling drying conditions such as the temperature and the drying end point, and of continuously calculating volatile-matter content as the mass lost during drying.

The accuracy of the system shall be demonstrated by performing 10 successive determinations on the standard reference material sodium L-tartrate dihydrate (5.1). The mean of the 10 determinations shall be (15,66 ± 0,5) %. The relative standard deviation, obtained by Formula (1), shall be less than 1,0 %.

$$s_{\text{rel}} = \frac{S}{W} \times 100 \tag{1}$$

where

s_{rel} is the relative standard deviation, in percentage;

S is the standard deviation;

W is the mean volatile-matter content, in mass %.

7 Sampling and preparation of test portion

Take a laboratory sample in accordance with the method specified in ISO 1795, and then prepare a test portion of between 2 g and 15 g from the laboratory sample. The actual mass of the test portion depends on the type of analyser, the expected volatile-matter content, and the form of the sample. For raw rubbers in bale form, the test portion shall be cut into small pieces of volume less than about 350 mm^3 (in the ideal case of a cubic piece, the length of a side should be about 7 mm). This operation shall be carried out as quickly as possible so as not to lose volatile matter.

The test result is taken to be the value from a single determination of the volatile-matter content.

8 Procedure

8.1 General

Either method A (which uses a pre-defined drying time) or method B (in which drying ends when the mass loss rate has decreased to a pre-defined level) can be chosen, provided the automatic analyser used offers the choice. The endpoint (the pre-defined drying time for method A or the pre-defined mass loss rate for method B) shall be determined for each of the two methods for each type or grade of rubber to be analysed.

Examples of test conditions are given in Annex A.

8.2 Determination of endpoints for method A and method B

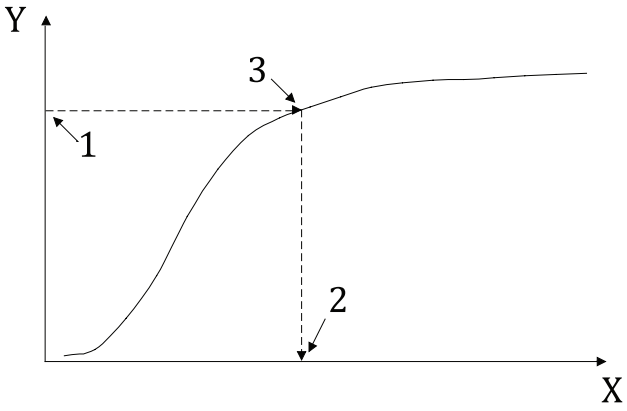
8.2.1 First take a typical sample of the type or grade of rubber being analysed and determine its volatile-matter content in accordance with one of the methods described in ISO 248-1.

8.2.2 Then, for method A, take a test portion of between 2 g and 15 g and weigh it to the nearest 1 mg. Operating the automatic analyser in accordance with the manufacturer's instructions, set a drying temperature (preferably between 100 °C and 120 °C) and obtain a drying profile (X-axis: time in min, Y-axis: volatile matter lost, in %). From the drying profile (see Figure 1), determine the time at which the volatile-matter content determined by the automatic analyser is equal to that determined in 8.2.1. Take this drying time as the pre-defined drying time to be used in method A for this particular type or grade of rubber.

8.2.3 For method B, take a test portion of between 2 g and 15 g and weigh it to the nearest 1 mg. Operating the automatic analyser in accordance with the manufacturer's instructions, set a drying temperature (preferably between 100 °C and 120 °C) and obtain a drying profile (X-axis: time, Y-axis: mass). From the drying profile (see Figure 2), determine the mass loss rate at the point on the profile where the volatile-matter content corresponds to that determined in 8.2.1. Take this value of the mass loss rate as the endpoint for use in method B for this particular type or grade of rubber.

NOTE The automatic analyser can be programmed to calculate this value automatically.

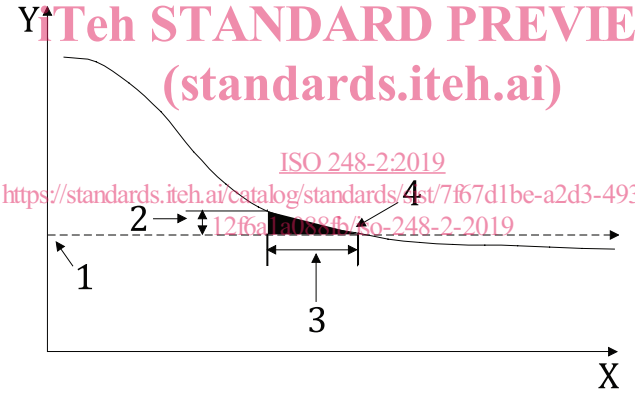
8.2.4 8.2.1 and either 8.2.2 or 8.2.3 shall be performed individually for each type or grade of raw rubber to be analysed.



Key

X time, min
Y volatile matter lost from test portion, %
1 volatile-matter content determined using ISO 248-1
2 drying time
3 drying endpoint

Figure 1 — Drying profile and drying endpoint for method A



Key

X time
Y mass of test portion
1 final (dry) mass of test portion when volatile-matter content is determined using ISO 248-1
2 mass loss increment, mg
3 mass measurement interval, s
4 drying endpoint

Figure 2 — Drying profile and drying endpoint for method B

8.3 Method A (pre-defined drying time method)

8.3.1 Operate the apparatus in accordance with the manufacturer's instructions. A general procedure is described in 8.3.2 to 8.3.7.

8.3.2 Input the drying temperature and the pre-defined drying time determined in 8.2.2 into the microprocessor of the apparatus.

8.3.3 Position an empty sample tray in the designated place and set the balance to zero.

8.3.4 Take a test portion of about the same mass (within ±10 %) as was taken to determine the drying profile in 8.2.2, spread it on the sample tray as uniformly and quickly as possible and press the start button. The initial mass (m_A) of the test portion before drying starts is measured and recorded automatically. Drying starts immediately.

8.3.5 When the drying time reaches the pre-defined value, drying stops automatically.

8.3.6 The final mass (m_B) of the test portion after drying is measured and recorded automatically.

8.3.7 The volatile-matter content is calculated automatically in accordance with 8.5.

8.4 Method B (in which drying ends when the mass loss rate has decreased to a pre-defined level)

8.4.1 Operate the apparatus in accordance with the manufacturer's instructions. A general procedure is described in 8.4.2 to 8.4.8.

8.4.2 Input the drying temperature and the pre-defined mass loss rate determined in 8.2.3 into the microprocessor of the apparatus.

8.4.3 Position an empty sample tray in the designated place and set the balance to zero.

8.4.4 Take a test portion of about the same mass (within ±10 %) as was taken to determine the drying profile in 8.2.3, spread it on the sample tray as uniformly and quickly as possible and press the start button. The initial mass (m_A) of the test portion before drying starts is measured and recorded automatically. Drying starts immediately.

8.4.5 The mass of the test portion is continuously measured and recorded during drying, and the mass loss rate continuously calculated.

8.4.6 When the mass loss rate reaches the pre-defined value, drying stops automatically.

8.4.7 The final mass (m_B) of the test portion after drying is measured and recorded automatically.

8.4.8 The volatile-matter content is calculated automatically in accordance with 8.5.

8.5 Calculation of volatile-matter content

The volatile-matter content is calculated according to Formula (2):

$$W = \frac{(m_A - m_B)}{m_A} \times 100 \qquad (2)$$

where

W is the volatile-matter content, in mass %;

m_A is the mass of the test portion before drying, in g;

m_B is the mass of the test portion after drying, in g.