INTERNATIONAL STANDARD

ISO 248-2

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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

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

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

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.

CAUTION — Certain procedures specified in this part of ISO 248 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 part of ISO 248 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, but in such cases it is necessary to prove that the change in mass is 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 part of ISO 248 might not give identical results. In cases of dispute, therefore, the oven method, procedure A, specified in ISO 248-1:2011 shall be 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 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 248-1:2011, *Rubber, raw — Determination of volatile-matter content — Part 1: Hot-mill method and oven method*

ISO 1629, *Rubber and latices — Nomenclature*

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

3 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.

4 Reagents

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

5 Apparatus

5.1 Automatic analyser

5.1.1 General

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.

5.1.2 Performance requirements

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

$$\text{Relative standard deviation (\%)} = \frac{s}{w} \times 100$$

where

s is the standard deviation;

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

6 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 will depend 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.

7 Procedure

7.1 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 end point (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.

7.2 Determination of end points for method A and method B

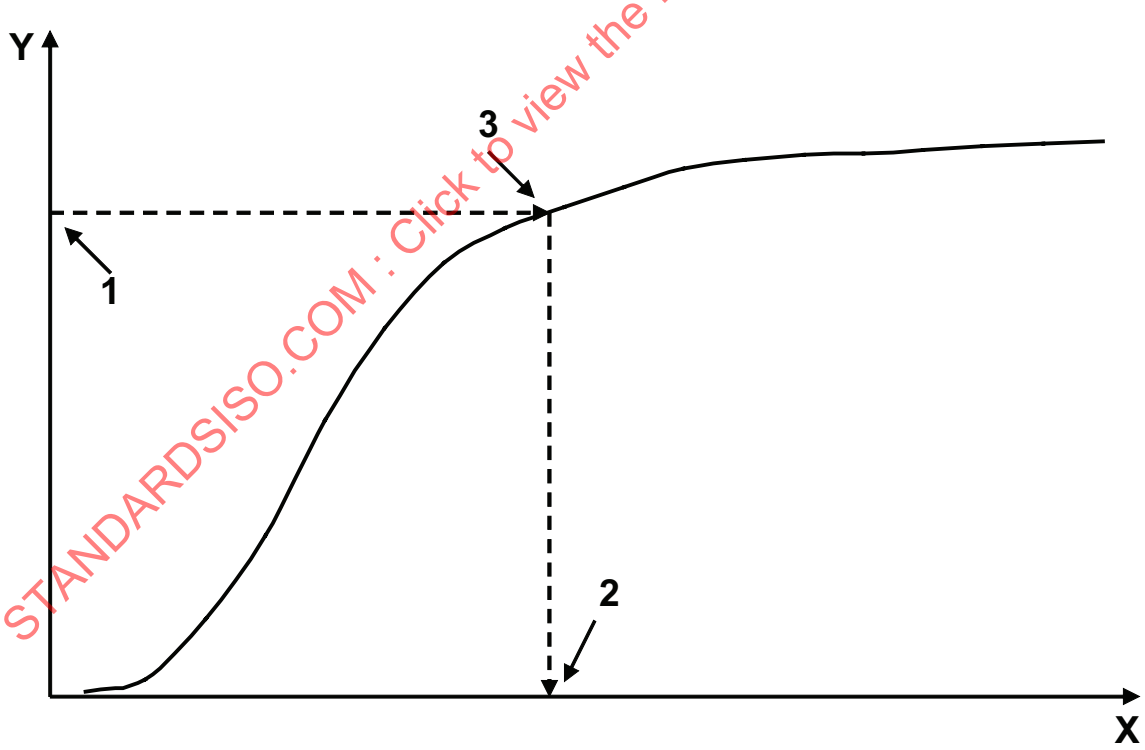
7.2.1 First take a typical sample of the type or grade of rubber being analysed and determine its volatile-matter content in accordance with one of the methods described in ISO 248-1.

7.2.2 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 7.2.1. Take this drying time as the pre-defined drying time to be used in method A for this particular type or grade of rubber.

7.2.3 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 7.2.1. Take this value of the mass loss rate as the end point 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.

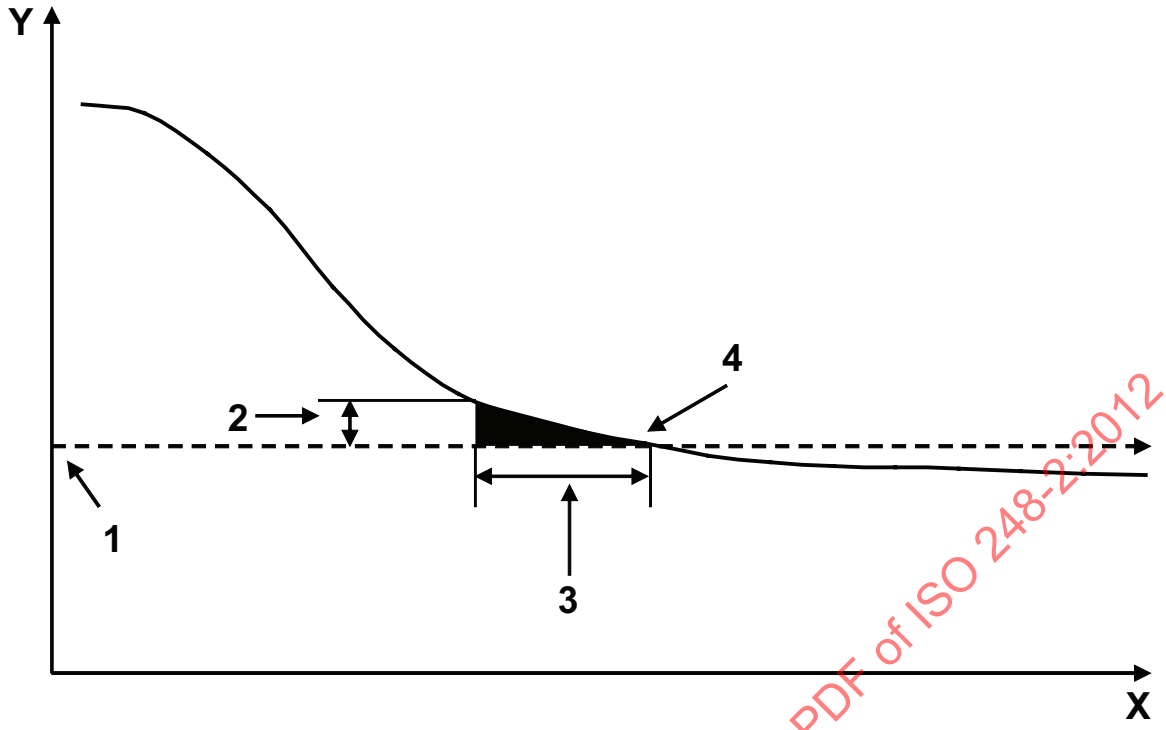
7.2.4 7.2.1 and either 7.2.2 or 7.2.3 shall be performed individually for each type or grade of raw rubber to be analysed.



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 end point

Figure 1 — Drying profile and drying end point 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 end point

Figure 2 — Drying profile and drying end point for method B

7.3 Method A (pre-defined drying time method)

7.3.1 Operate the apparatus in accordance with the manufacturer's instructions. A general procedure is described in 7.3.2 to 7.3.7.

7.3.2 Input the drying temperature and the pre-defined drying time determined in 7.2.2 into the microprocessor of the apparatus.

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

7.3.4 Take a test portion of about the same mass (within ±10 %) as was taken to determine the drying profile in 7.2.2, 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 will be measured and recorded automatically. Drying will start immediately.

7.3.5 When the drying time reaches the pre-defined value, drying will stop automatically.

7.3.6 The final mass (m_B) of the test portion after drying will be measured and recorded automatically.

7.3.7 The volatile-matter content will be calculated automatically in accordance with 7.5.

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

7.4.1 Operate the apparatus in accordance with the manufacturer's instructions. A general procedure is described in 7.4.2 to 7.4.8.

7.4.2 Input the drying temperature and the pre-defined mass loss rate determined in 7.2.3 into the microprocessor of the apparatus.

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

7.4.4 Take a test portion of about the same mass (within ±10 %) as was taken to determine the drying profile in 7.2.3, 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 will be measured and recorded automatically. Drying will start immediately.

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

7.4.6 When the mass loss rate reaches the pre-defined value, drying will stop automatically.

7.4.7 The final mass (m_B) of the test portion after drying will be measured and recorded automatically.

7.4.8 The volatile-matter content will be calculated automatically in accordance with 7.5.

7.5 Calculation of volatile-matter content

The volatile-matter content is calculated as follows:

$$w = \frac{(m_A - m_B)}{m_A} \times 100$$

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.

8 Precision

See Annex B.

9 Test report

The test report shall include the following particulars:

a) full details of the sample of raw rubber tested;

b) test method:

1) a reference to this part of ISO 248,

2) the method used (method A or method B),

3) full details of the apparatus and measurement conditions,

4) the method, and associated procedure, in ISO 248-1 used in determining the pre-defined drying time (method A) or pre-defined mass loss rate (method B);

c) departures from the procedure specified:

1) details of any operation not specified in this part of ISO 248 and of any optional operation;

2) any unusual features noted during the determination;

d) the test result;

e) the date of the test.

Annex A
(informative)

Examples of test conditions

A.1 General

There are differences between automatic analysers, such as the use of the far infrared or near infrared for drying. However, any automatic analyser which meets the performance requirements specified in 5.1 is suitable for the determination of the volatile-matter content of raw rubber by the methods specified in this part of ISO 248.

A.2 Examples of conditions

Examples of test conditions for method A and method B described in Clause 7 are given in Table A.1 and Table A.2, respectively.

Table A.1 — Examples of test conditions for method A

Parameter	Raw-rubber sample				
	SBR-1	SBR-2	BR-1	EPDM-1	NBR-1
Volatile-matter content determined using ISO 248-1[a], %	0,15	0,77	0,07	0,09	0,35
Mass of test portion, g	5	5	5	5	5
Drying temperature, °C	110	120	110	120	120
Pre-defined drying time[b], min	3,0 to 9,0	6,0 to 23,0	1,5 to 11,0	1,0 to 5,0	2,5 to 17,0

[a] Determined using the hot-mill method, procedure B, specified in ISO 248-1.

[b] The value of the pre-defined drying time will depend on the apparatus used.

Table A.2 — Examples of test conditions for method B

Parameter	Raw-rubber sample				
	SBR-1	SBR-2	BR-1	EPDM-1	NBR-1
Volatile-matter content determined using ISO 248-1[a], %	0,15	0,77	0,07	0,09	0,35
Mass of test portion, g	5	5	5	5	5
Drying temperature, °C	110	120	110	120	120
Pre-defined mass loss rate[b], mg/s	1/180 to 3/180	1/180 to 3/180	1/60 to 1/145	1/45 to 2/120	1/60 to 6/120

[a] Determined using the hot-mill method, procedure B, specified in ISO 248-1.

[b] The value of the pre-defined mass loss rate will depend on the apparatus used.

Annex B
(informative)

Precision

B.1 General

An interlaboratory test programme (ITP) to determine the precision of the two test methods specified in this part of ISO 248 was conducted using automatic analysers in August 2007 in Japan. The precision evaluated was a type 1 precision in accordance with ISO/TR 9272.

B.2 Details of the ITP

Five raw rubbers (SBR: two grades, BR: one grade, EPDM: one grade, NBR: one grade) were used in the ITP. A test result was taken to be the value from a single determination of the volatile matter content. Test results were obtained on three different days, with intervals of only one day between each of the three tests in order to avoid volatile-matter loss during the ITP.

Six laboratories participated in the ITP for method A, and two of these used two different types of analyser. For these two laboratories, the data from the separate analysers were included in the ITP as data from individual laboratories. This gave a database of up to eight laboratories or pseudo-laboratories.

Four laboratories participated in the ITP for method B, and one of these used two different types of analyser. For this laboratory, the data from the separate analysers were included in the ITP as data from individual laboratories. This gave a database of up to five laboratories or pseudo-laboratories.

B.3 Precision results

The precision results are given in Table B.1 for method A and Table B.2 for method B. These results were obtained using outlier deletion procedures as described in ISO/TR 9272.

Repeatability: The repeatability r of the test method has been established as the appropriate value tabulated in Table B.1 or Table B.2 for each material. Two single test results that differ by more than this value should be considered suspect and suggest that some appropriate investigative action be taken.

Reproducibility: The reproducibility R of the test method has been established as the appropriate value tabulated in Table B.1 or Table B.2 for each material. Two single test results that differ by more than this value should be considered suspect and suggest that some appropriate investigative action be taken.

The precision results as determined by this ITP 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.