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Rubber, raw — Determination of volatile-matter content

Caoutchouc brut — Détermination des matières volatiles

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Foreword

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

This fourth edition cancels and replaces the third edition (ISO 248:1991), which has been technically revised.

INTERNATIONAL STANDARD **ISO 248:2005(E)**

Rubber, raw — Determination of volatile-matter content

WARNING 1 — Persons using this International Standard should be familiar with normal laboratory practice. This standard 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 condition.

WARNING 2 — Certain procedures specified in this International Standard may 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 International Standard specifies two methods, a hot-mill method and an oven method, for the determination of moisture and other volatile-matter content in raw rubbers.

These methods are applicable to the determination of the volatile-matter content in the R-group of rubbers listed in ISO 1629 which are rubbers having an unsaturated carbon chain, for example, natural rubber and synthetic rubbers derived at least partly from diolefins.

This International Standard may also be applicable to other rubbers, but in these 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.

The hot-mill method is not applicable to natural and synthetic isoprene rubbers or to rubbers too difficult to handle on a hot mill or to rubbers in powdered or chip form.

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

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*

ISO/TR 9272, *Rubber and rubber products — Determination of precision for test method standards*

3 Principle

3.1 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.

3.2 Oven method

When the sample is not in powder form, a piece is homogenized in accordance with ISO 1795 using a laboratory mill. A test portion, taken either from the comminuted piece or directly from the rubber if in powdered form, is sheeted out and dried in an oven to constant mass. The volatile-matter content is calculated as the mass lost during this procedure, together with the mass lost during any homogenization of the piece.

4 Hot-mill method

4.1 Apparatus

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

4.2 Procedure

4.2.1 Hot-mill method A

4.2.1.1 Sheet out a test piece of about 250 g in accordance with ISO 1795. Weigh to the nearest 0,1 g before and after homogenization (masses m_1, and m_2 respectively).

4.2.1.2 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.

4.2.1.3 Pass a weighed test portion (mass m_3) repeatedly through the mill (4.1.1) for 4 min. Do not allow the test portion to band and take care to prevent any loss of rubber. 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 rubber to cool to room temperature in a desiccator.

4.2.1.4 When the rubber is flaky or becomes sticky in the mill roll, making weighing difficult or impossible, the oven method (procedure 5.2.1.2) shall be used.

4.2.2 Hot-mill method B

Sheet out a test piece of about 250 g and weigh to the nearest 0,1 g (mass m_5). Adjust the surface temperature of the mill roll to 105 °C ± 5 °C and the clearance of the mill roll to 0,25 mm ± 0,05 mm. Pass the test piece 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. When the mass difference before and after roll passing is less than 0,1 g, the test piece is considered to be well dried. If it is not well dried, continue passing the test piece twice through the roll until the mass difference is less than 0,1 g (final mass m_6).

NOTE Although moisture does not affect the result, cooling in a desiccator before weighing is desirable.

4.3 Expression of results

4.3.1 Hot-mill method 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 portion before homogenization;

m_2 is the mass, in grams, of the test portion 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.

4.3.2 Hot-mill method 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 piece before drying;

m_6 is the mass, in grams, of the test piece after drying.

5 Oven method

5.1 Apparatus

5.1.1 Oven, ventilated, preferably air-circulating type, capable of being maintained at 105 °C ± 5 °C.

5.2 Procedure

5.2.1 Oven method A

5.2.1.1 Natural rubber

5.2.1.1.1 When the rubber is not in powder form, select a piece of about 600 g and homogenize in accordance with ISO 1795. Weigh the piece 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.

5.2.1.1.2 Select a test portion of about 10 g from the homogenized test piece and weigh it to the nearest 1 mg (mass m_9).

5.2.1.1.3 With the mill set at 70 °C ± 5 °C and with a mill opening which will produce a sheet of less than 2 mm thickness, pass the test portion twice between the rolls.

5.2.1.1.4 Alternatively, when the rubber is in powdered form, select a test portion of about 10 g taken at random and place it on a watch-glass or an aluminium tray to facilitate weighing. Weigh it to the nearest 1 mg (mass m_9).

5.2.1.2 Synthetic rubber

5.2.1.2.1 When the sample is not in powder form, select a piece of about 250 g and homogenize in accordance with the procedure for natural rubber specified in ISO 1795. Weigh the piece to the nearest 0,1 g before and after this homogenization (masses m_7 and m_8 respectively).

5.2.1.2.2 With the mill set at 70 °C ± 5 °C and with a mill opening which will produce a sheet of less than 2 mm thickness, pass a test portion of 10 g, taken from the homogenized piece and weighed to the nearest 1 mg (mass m_9), twice between the rolls.

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

5.2.1.2.4 Alternatively, when the rubber is in powdered form, select a test portion of about 10 g taken at random and place it on a watch-glass or an aluminium tray to facilitate weighing. Weigh to the nearest 1 mg (mass m_9).

5.2.1.3 Oven treatment (natural and synthetic rubbers)

Place the test portion, derived in accordance with either 5.2.1.1 or 5.2.1.2, for 1 h in the oven (5.1.1), maintained at 105 °C ± 5 °C, with the ventilators open and with the circulating fan, if fitted, switched on. Arrange the rubber so as to present the largest possible surface area to the hot air. Allow to cool 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 weighing (final mass m_{10}).

5.2.2 Oven method B

5.2.2.1 Weigh a sample of about 250 g and pass it through the mill roll, whose surface temperature is adjusted to about 30 °C and the roll clearance to 0,25 mm ± 0,05 mm, to get thin sheet. Sample randomly two test pieces of about 50 g from this sheet and weigh the mass to the nearest 10 mg (mass m_{11}).

5.2.2.2 When this sheeting is impossible due to the sample sticking to the roll, take two test pieces of about 10 g directly from the sample. Follow by cutting them into small cubes of about 2 mm size. Place them in a tared aluminium tray of 15 mm depth and 60 mm diameter or in a tray with similar shape and weigh the mass to the nearest 1 mg (mass m_{11}). Place the tray containing the samples in an oven maintained at 105 °C ± 5 °C for 1 h. Remove the tray from the oven and cool in a desiccator to room temperature. Reweigh the mass (mass m_{12}).

NOTE Natural rubber requires homogenization, therefore oven method B is not applicable.

5.3 Expression of results

5.3.1 Oven method A

5.3.1.1 When the test portion is taken from a homogenized piece (see 5.2.1.1.2 and 5.2.1.2.2), 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 piece before homogenization;

m_8 is the mass, in grams, of the piece after homogenization;

m_9 is the mass, in grams, of the test portion as taken from the piece;

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

5.3.1.2 When the test portion is taken directly from a sample in powdered form (see 5.2.1.1.4 and 5.2.1.2.4), 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 as taken from the piece;

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

5.3.2 Oven method B

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

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

where

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

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

The test result is the average of duplicate test pieces.

6 Precision

Details of interlaboratory tests, carried out in accordance with ISO/TR 9272, are given in Annex A.

Consult ISO/TR 9272 for precision concepts and nomenclature.

Annex B of this International Standard gives guidance on the use of repeatability and reproducibility.

7 Test report

The test report shall include the following particulars:

a) a reference to this International Standard (ISO 248:2005);

b) all details necessary for the full identification of the sample;

c) the method used (hot-mill or oven);

d) whether the 10 g test portions were taken from a homogenized piece (see 5.2.1.1.2 and 5.2.1.2.2), or directly from the powdered form (see 5.2.1.1.4 and 5.2.1.2.4);

e) the results obtained on each test portion;

f) any unusual features noted during the determination;

g) any operation not included in this International Standard or regarded as optional;

h) the date of test.

Annex A
(informative)

Interlaboratory tests

A.1 Precision details from test programme 1984

A.1.1 An interlaboratory test programme 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”.

A.1.2 For both the blended and the unblended samples, a test result was taken as the mean of three separate determinations.

A.1.3 The oven method A was used to determine the volatile matter.

A.1.4 A “type 1” precision was measured in the interlaboratory test programme. The time period for repeatability and reproducibility was on a scale of days. A total of 14 laboratories participated in the “blended” programme for blended samples and a total of 13 laboratories in the programme for unblended samples.

A.2 Precision details from test programme 2003

A.2.1 The interlaboratory round robin tests were done between April and May 2003 with the participation of seven laboratories for the hot-mill method B, and eight laboratories for the oven method B.

A.2.2 Samples of raw rubbers, Sample C (SBR 1500) and Sample D (non-oil extended BR), have been used for both hot-mill method B and oven method B.

A.2.3 The results contained in Table A.3 for oven method B and in Table A.4 for hot-mill method B are averaged values and give an estimate of the precision of this test method, as determined in an interlaboratory round robin consisting of laboratories performing duplicate analyses on two raw rubber samples.

A.3 Precision results

The 1984 precision results for the blended-sample programme are given in Table A.1 and the results for the unblended-sample programme in Table A.2.

The 2003 precision results for the oven method B are given in Table A.3. The precision results for the hot-mill method B are given in Table A.4.

The precision parameters should not be used for acceptance or rejection of any group of materials without documentation that the parameters are applicable to the particular group of materials and the specific test protocols of the test method.

Table A.1 — Oven method A — Blended-sample testing

Rubber sample	Average volatile-matter content	Within-laboratory repeatability		Interlaboratory reproducibility	
	% (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 A.2 — Oven method A — Unblended-sample testing

Rubber sample	Average volatile-matter content	Within-laboratory repeatability		Interlaboratory reproducibility	
	% (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 A.1 for symbol definitions.

Table A.3 — Oven method B — Volatile-matter content

Rubber sample	Average volatile-matter content	Within-laboratory repeatability			Interlaboratory reproducibility		
	% (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 A.1 for other symbol definitions.

Table A.4 — Hot-mill method B — Volatile-matter content

Rubber sample	Average volatile-matter content	Within-laboratory repeatability			Interlaboratory reproducibility		
	% (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 A.1 for other symbol definitions.

Annex B
(informative)

Guidance for using precision results

B.1 General procedure

The general procedure for using precision results is as follows, with the symbol $|\chi_1 - \chi_2|$ designating a positive difference between any two measurement values, i.e. without regard to sign.

a) Enter the appropriate precision table (for whatever test parameter is being considered) at an average value (of the measured parameter) nearest to the “test” data average under consideration. This line gives the applicable r, (r), R or (R) values used in the decision process.

b) With the r and (r) values, the general repeatability statements in B.2 may be used to make decisions.

c) With the R or (R) values, the general reproducibility statements in B.3 may be used to make decisions.

B.2 General repeatability statements

B.2.1 Absolute difference

The difference $|\chi_1 - \chi_2|$ between two test (value) averages, found on nominally identical material samples under normal and correct operation of the test procedure, should not exceed the tabulated repeatability limit r on average more than once in twenty cases.

B.2.2 Percentage difference between two test (value) averages

The percentage difference $[|\chi_1 - \chi_2| / (\chi_1 + \chi_2) / 2] \times 100$ between two test values, found on nominally identical material samples under normal and correct operation of the test procedure, should not exceed the tabulated repeatability limit (r) on average more than once in twenty cases.

B.3 General reproducibility statements

B.3.1 Absolute difference

The absolute difference $|\chi_1 - \chi_2|$ between two independently measured test (value) averages, found in two laboratories using normal and correct test procedures on nominally identical material samples, should not exceed the tabulated reproducibility limit R more than once in twenty cases.

B.3.2 Percentage difference between two test (value) averages

The percentage difference $[|\chi_1 - \chi_2| /(\chi_1 + \chi_2) / 2] \times 100$ between two independently measured test (value) averages, found in two laboratories using normal and correct test procedures on nominally identical material samples, should not exceed the tabulated reproducibility limit (R) more than once in twenty cases.

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