INTERNATIONAL STANDARD

ISO 248

Third edition
1991-10-15

Rubbers, raw — Determination of volatile-matter content

Caoutchoucs bruts — Détermination des matières volatiles

Reference number
ISO 248:1991(E)

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.

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.

International Standard ISO 248 was prepared by Technical Committee ISO/TC 45, *Rubber and rubber products*.

This third edition cancels and replaces the second edition (ISO 248:1979), of which it constitutes a technical revision.

Annex A of this International Standard is for information only.

International Organization for Standardization
Case Postale 56 • CH-1211 Genève 20 • Switzerland

Printed in Switzerland

Rubbers, raw — Determination of volatile-matter content

1 Scope

1.1 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.

1.2 These methods are suitable for the determination of the volatile-matter content in the R[1] group of rubbers listed in ISO 1629. They may also be used for 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.

1.3 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.

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

2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this International Standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 1629:1987, *Rubber and latices — Nomenclature.*

ISO 1796:1982, *Rubber, raw — Sample preparation.*

ISO 2393:—[2], *Rubber test mixes — Preparation, mixing and vulcanization — Equipment and procedures.*

ISO/TR 9272:1986, *Rubber and rubber products — Determination of precision for test method standards.*

3 Principles

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

If the sample is not in powder form, a piece is homogenized in accordance with ISO 1796 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

Mixing mill, complying with the requirements of ISO 2393.

1) Rubbers having an unsaturated carbon chain, for example natural rubber and synthetic rubbers derived at least partly from diolefins.

2) To be published. (Revision of ISO 2393:1973)

4.2 Procedure

4.2.1 Sheet out a test piece of about 250 g in accordance with ISO 1796. Weigh to the nearest 0,1 g before and after homogenization (masses m_1 and m_2 respectively).

4.2.2 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.

4.2.3 Pass a weighed test portion (mass m_3) repeatedly through the mill (4.1) 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.

4.2.4 If the rubber is flaky or becomes sticky in the mill roll, making weighing difficult or impossible, the oven method (procedure 5.2.2) shall be used.

4.3 Expression of results

The volatile-matter content is given, as a percentage by mass, by the formula

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

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.

5 Oven method

5.1 Apparatus

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

5.2 Procedure

5.2.1 In the case of natural rubber, proceed as follows.

5.2.1.1 If the rubber is not in powder form, select a piece of about 600 g and homogenize in accordance with ISO 1796. Weigh the piece to the nearest 0,1 g before and after this homogenization (masses m_5 and m_6 respectively). Allow to cool to room temperature before the final weighing.

5.2.1.2 Select a test portion of about 10 g from the homogenized test piece and weigh it to the nearest 1 mg (mass m_7).

5.2.1.3 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.

5.2.1.4 Alternatively, if 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_7).

5.2.2 In the case of synthetic rubber, proceed as follows.

5.2.2.1 If 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 1796. Weigh the piece to the nearest 0,1 g before and after this homogenization (masses m_5 and m_6 respectively).

5.2.2.2 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_7), twice between the rolls.

5.2.2.3 If this 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_7).

5.2.2.4 Alternatively, if 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_7).

5.2.3 Place the test portion, derived in accordance with either 5.2.1 or 5.2.2, for 1 h in the oven (5.1), 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 weighings (final mass m_8).

5.3 Expression of results

5.3.1 If the test portion was taken from a homogenized piece (see 5.2.1.2 and 5.2.2.2), the volatile-matter content is given, as a percentage by mass, by the formula

$$\left(\frac{m_5 - m_6}{m_5} + \frac{m_7 - m_8}{m_7} \right) \times 100$$

where

m_5 is the mass, in grams, of the piece before homogenization;

m_6 is the mass, in grams, of the piece after homogenization;

m_7 is the mass, in grams, of the test portion as taken from the piece;

m_8 is the mass, in grams, of the test portion after oven drying.

5.3.2 If the test portion was taken directly from a sample in powdered form (see 5.2.1.4 and 5.2.2.4), the volatile-matter content is given, as a percentage by mass, by the formula

$$\left(\frac{m_7 - m_8}{m_7} \right) \times 100$$

where m_7 and m_8 are as defined in 5.3.1.

6 Precision

6.1 General

The precision calculations to express repeatability and reproducibility were performed in accordance with ISO/TR 9272. Consult this Technical Report for precision concepts and nomenclature. Annex A of this International Standard gives guidance on the use of repeatability and reproducibility.

6.2 Precision details

6.2.1 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".

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

6.2.3 The oven method was used to determine the volatile matter.

6.2.4 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.

6.3 Precision results

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

Table 1 — Type 1 precision — Blended-sample testing

Rubber sample	Average volatile-matter content % (m/m)	Within-laboratory repeatability		Interlaboratory reproducibility	
		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

r = repeatability, in percent by mass

(r) = repeatability, in percent (relative) of the average

R = reproducibility, in percent by mass

(R) = reproducibility, in percent (relative) of the average

Table 2 — Type 1 precision — Unblended-sample testing

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

7 Test report

The test report shall include the following particulars:

a) a reference to this International Standard;

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 5.2.1.2 and 5.2.2.2) or directly from the powdered form (see 5.2.1.4 and 5.2.2.4);

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)

Guidance for using precision results

A.1 The general procedure for using precision results is as follows, with the symbol $|x_1 - x_2|$ designating a positive difference in any two measurement values (i.e. without regard to sign).

A.2 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 will give the applicable r, (r), R or (R) for use in the decision process.

A.3 With these r and (r) values, the following general repeatability statements may be used to make decisions.

A.3.1 For an absolute difference: The difference $|x_1 - x_2|$ between two test (value) averages, found on nominally identical material samples under normal and correct operation of the test procedure, will exceed the tabulated repeatability r on average not more than once in twenty cases.

A.3.2 For a percentage difference between two test (value) averages: The percentage difference

$$[|x_1 - x_2|/(x_1 + x_2)/2] \times 100$$

between two test values, found on nominally identical material samples under normal and correct operation of the test procedure, will exceed the tabulated repeatability (r) on average not more than once in twenty cases.

A.4 With these R and (R) values, the following general reproducibility statements may be used to make decisions.

A.4.1 For an absolute difference: The absolute difference $|x_1 - x_2|$ between two independently measured test (value) averages, found in two laboratories using normal and correct test procedures on nominally identical material samples, will exceed the tabulated reproducibility R not more than once in twenty cases.

A.4.2 For a percentage difference between two test (value) averages: The percentage difference

$$[|x_1 - x_2|/(x_1 + x_2)/2] \times 100$$

between two independently measured test (value) averages, found in two laboratories using normal and correct test procedures on nominallly identical material samples, will exceed the tabulated reproducibility (R) not more than once in twenty cases.

UDC [678.4 + .7].03:543.71

Descriptors: rubber, crude rubber, determination of content, volatile matter.

Price based on 5 pages