INTERNATIONAL STANDARD 248

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Rubbers, raw — Determination of volatile matter content

Caoutchoucs bruts — Détermination des matières volatiles

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FOREWORD

ISO (the International Organization for Standardization) is a worldwide federation of national standards institutes (ISO member bodies). The work of developing International Standards is carried out through ISO technical committees. Every member body interested in a subject for which a technical committee has been set up 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.

Draft International Standards adopted by the technical committees are circulated to the member bodies for approval before their acceptance as International Standards by the ISO Council.

International Standard ISO 248 was developed by Technical Committee ISO/TC 45, *Rubber and rubber products*, and was circulated to the member bodies in September 1975.

It has been approved by the member bodies of the following countries :

Australia	India	Sri Lanka
Belgium	Italy	Sweden
Brazil	Malaysia	Switzerland
Bulgaria	Mexico	Turkey
Canada	Netherlands	United Kingdom
Czechoslovakia	Poland	U.S.A.
Germany	Romania	U.S.S.R.
Hungary	Spain	Yugoslavia

The member body of the following country expressed disapproval of the document on technical grounds :

France

This International Standard cancels and replaces ISO Recommendation R 248-1962, of which it constitutes a technical revision.

A revised wording for sub-clause 5.2.4 of this International Standard is being submitted for ISO member body ballot. This action follows objections to the present wording raised at a meeting of ISO/TC 45 by experts from several countries.

Printed in Switzerland

Rubbers, raw — Determination of volatile matter content

1 SCOPE AND FIELD OF APPLICATION

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.

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

2 REFERENCES

ISO 1629, *Rubbers and latices — Nomenclature.*

ISO 1796, *Rubber, raw — Sample preparation.*[2]

ISO 2393, *Rubber test mixes — Preparation, mixing and vulcanization — Equipment and procedures.*

3 PRINCIPLE OF METHODS

3.1 Hot-mill method

Sheeting out of a test portion on a heated mill until all the volatile matter is driven off. Calculation of the loss in mass during milling and expression as volatile matter content.

3.2 Oven method

Weighing of a test portion of a piece prepared in accordance with ISO 1796. Sheeting out of the test portion on a laboratory mill or comminution by hand. Drying in an oven to constant mass. Calculation of the volatile matter content as the mass lost during this procedure, together with the mass lost during 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 Weigh, to the nearest 0,1 g, a test portion of at least 250 g from a piece prepared in accordance with ISO 1796.

4.2.2 Adjust the clearance of the mill rolls to 0,25 ± 0,05 mm, using lead strips as specified in ISO 2393. Maintain the surface temperature of the rolls at 100 ± 5 °C.

4.2.3 Pass the test portion 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 re-weigh. If the masses at the end of the 4 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 in successive weighings. Before each weighing, allow the rubber to cool to room temperature in a desiccator.

4.3 Expression of results

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

$$\frac{m_1 - m_2}{m_1} \times 100$$

where

m_1 is the mass, in grams, of the test portion before milling;

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

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

2) At present at the stage of draft. (Revision of ISO 1796-1972.)

5 OVEN METHOD

5.1 Apparatus

5.1.1 Oven, ventilated, preferably air-circulating type, capable of being controlled at 100 ± 5 °C and 160 ± 5 °C.

5.2 Procedure

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

5.2.1.1 Sheet out a test piece of about 600 g, following ISO 1796. Weigh to the nearest 0,1 g before and after homogenization.

5.2.1.2 Select a test portion of about 10 g from the homogenized test piece and weight it to the nearest 0,000 1 g.

5.2.1.3 With the mill set at 70 ± 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.

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

5.2.2.1 Sheet out a test piece of about 250 g, following ISO 1796. Weigh the test piece to the nearest 0,01 g.

Alternatively, use a 10 g test portion, weighed to the nearest 0,000 1 g before and after homogenization.

5.2.2.2 With the mill set at 70 ± 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 (see 5.2.2.3).

5.2.2.3 If sheeting to 2 mm is impossible or if the rubber becomes sticky on the mill roll, take a 10 g test portion directly from the test piece and cut it, by hand, into small cubes with edges of length approximately 2 mm.

Place the test portion on a watch-glass or aluminium tray, to facilitate weighing, and determine the mass to the nearest 0,000 1 g.

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 (5.1.1), controlled at 100 ± 5 °C with the ventilators open and, if fitted, with the circulating fan switched on. Arrange the rubber 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 0,01 % of the initial mass in successive weighings.

5.2.4 If it can be shown that extraneous volatile hydrocarbon oils are present, maintain the oven temperature at 160 ± 5 °C. This test variation is not applicable to oil-extended rubbers.

5.3 Expression of results

5.3.1 If the test portion was taken from a homogenized piece, the volatile matter content is given, as a percentage by mass, by the formula

$$\left(1 - \frac{m_4\, m_6}{m_3\, m_5}\right) \times 100$$

where

m_3 is the mass, in grams, of the piece before homogenization (see ISO 1796);

m_4 is the mass, in grams, of the piece after homogenization (see ISO 1796);

m_5 is the mass, in grams, of the test portion as taken from the piece;

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

5.3.2 If the test portion was taken from an unhomogenized piece, the volatile matter content is given, as a percentage by mass, by the formula

$$\frac{m_5 - m_6}{m_5} \times 100$$

where m_5 and m_6 are as defined in 5.3.1.

6 TEST REPORT

The test report shall include the following particulars :

a) reference to this International Standard;

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

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

d) whether a 10 g or 250 g test portion of synthetic rubber was used in the oven method (see 5.2.2.1);

e) whether the alternative temperature (160°C) was used in the oven method (see 5.2.4);

f) the results obtained on each test portion;

g) any unusual features noted during the determination;

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

i) the date of test.