INTERNATIONAL STANDARD

ISO 247

Third edition
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Rubber — Determination of ash

Caoutchouc — Détermination du taux de cendres

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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.

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

This third edition cancels and replaces the second edition (ISO 247 : 1978), which contained three methods of determination rather than two methods.

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Rubber — Determination of ash

1 Scope

1.1 This International Standard specifies two methods for the determination of ash from raw rubbers, compounded rubbers and vulcanizates. The methods are applicable to raw, compounded or vulcanized rubbers of the M, N, O, R and U families described in ISO 1629, except as restricted in 1.2 and 1.3 below.

This International Standard does not cover the interpretation of the ash results as to the inorganic chemical content of a compound or vulcanizate. This is the responsibility of the analyst, who has to be aware of the behaviour of rubber additives at elevated temperatures.

1.2 Method A shall not be used for the determination of ash from compounded or vulcanized rubbers containing chlorine, bromine or iodine.

1.3 Method B shall be used for compounded or vulcanized rubbers containing chlorine, bromine or iodine. It shall not be used for uncompounded rubbers.

1.4 Lithium and fluorine compounds may react with silica crucibles to form volatile compounds, giving low ash results. Platinum crucibles shall be used for ashing fluorine-containing and lithium-polymerized rubbers.

1.5 The two methods of ashing do not give identical results in all cases, and it is necessary to state in the test report the method of ashing employed.

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 248 : 1979, *Rubbers, raw — Determination of volatile matter content*.

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

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

3 Principle

3.1 Method A

A weighed test portion is heated in a crucible over a gas burner. After expulsion of the volatile decomposition products, the crucible is transferred to a muffle furnace where it is heated until all the carbonaceous matter has been burnt off and constant mass is attained.

3.2 Method B

A weighed test portion is heated in a crucible in the presence of sulfuric acid, first by means of a gas burner and then in a muffle furnace until all the carbonaceous matter has been burnt off and constant mass is attained.

4 Reagent

Sulfuric acid (for method B only), analytical grade, ϱ 1,84 g/cm^3.

5 Apparatus

Ordinary laboratory apparatus and the following:

5.1 Crucible, of porcelain, silica or platinum, of capacity approximately 50 cm^3. For raw synthetic rubbers, it is permitted to use a crucible of minimum capacity 25 cm^3 per gram of test portion.

5.2 Heat-resistant, thermally insulating board, 100 mm square and of thickness approximately 5 mm, with a central hole to accommodate the crucible (5.1). About two-thirds of the crucible shall project below the board.

5.3 Bunsen burner, or similar type of gas burner.

5.4 Muffle furnace, fitted with a flue and with provision for controlling the air flow through the furnace. (This may be achieved by adjusting the door opening.) A temperature-controlling device is required to maintain a temperature of 550 °C ± 25 °C or 950 °C ± 25 °C.

6 Preparation of the test portion

6.1 Test portions of raw natural rubber shall be cut from the homogenized piece prepared in accordance with ISO 1796. Test portions of raw synthetic rubbers shall be cut from the dried rubber obtained after carrying out the determination of volatile matter content in accordance with ISO 248.

6.2 Test portions of rubber compounds shall be comminuted by hand.

6.3 Test portions of vulcanizates shall be sheeted or crumbed on a mill or comminuted by hand.

6.4 Care shall be taken to ensure that test portions of rubber compounds and vulcanizates are representative of the sample.

7 Procedure

7.1 Method A

Heat the clean empty crucible (5.1) of appropriate size for about 30 min in the muffle furnace (5.4), maintained at 550 °C ± 25 °C, allow to cool to ambient temperature in a desiccator and weigh to the nearest 0,1 mg. Take a test portion of about 5 g of raw rubber or 1 g to 5 g of compounded rubber or vulcanizate, according to the mass of ash to be expected, and weigh to the nearest 0,1 mg. Place the weighed test portion in the crucible mounted in the hole in the heat-resistant, thermally insulating board (5.2). Heat the crucible gently with the burner (5.3) in a hood for proper ventilation, taking care that the rubber does not ignite. If any material is lost due to spurting or frothing, repeat the above procedure with a new test portion.

When the rubber has decomposed to a charred mass, gradually increase the heat from the burner until the volatile decomposition products have been substantially expelled and a dry carbonaceous residue remains. Transfer the crucible and its contents to the muffle furnace, maintained at 550 °C ± 25 °C (see, however, the note), leaving the door of the furnace slightly open to provide sufficient air to oxidize the carbon.

Continue heating until the carbon is completely oxidized and a clean ash is obtained. Remove the crucible and its contents from the furnace, allow to cool to ambient temperature in the desiccator and weigh to the nearest 0,1 mg. Then heat the crucible and its contents again for about 30 min in the muffle furnace, maintained at 550 °C ± 25 °C (or 950 °C ± 25 °C — see the note), allow to cool to ambient temperature in the desiccator and re-weigh to the nearest 0,1 mg. This mass shall not differ from the previous mass by more than 1 mg in the case of raw rubbers or by more than 1 % relative to the amount of ash for compounds and vulcanizates. If this requirement is not fulfilled, repeat the heating, cooling and weighing procedure until the difference between two successive weighings meets this requirement.

NOTE — For compounds and vulcanizates, a temperature of 950 °C ± 25 °C may be used.

7.2 Method B

Heat the clean empty crucible (5.1) of appropriate size for about 30 min in the muffle furnace (5.4), maintained at 950 °C ± 25 °C, allow to cool to ambient temperature in a desiccator and weigh to the nearest 0,1 mg. Take a test portion of about 1 g to 5 g of the compound or vulcanizate and weigh to the nearest 0,1 mg. Place the test portion in the crucible and pour about 3,5 cm^3 of the concentrated sulfuric acid (clause 4) over it so that the rubber is completely wetted. Place the crucible and its contents in the hole in the heat-resistant, thermally insulating board (5.2) and heat gently with the burner in a hood for proper ventilation. If, during the initial reaction, the mixture swells excessively, withdraw the flame to avoid possible loss of material.

When the reaction becomes more gentle, increase the heat from the burner until the excess sulfuric acid is volatilized and a dry, carbonaceous residue remains. Transfer the crucible and its contents to the muffle furnace, maintained at 950 °C ± 25 °C, and heat for about 1 h until all the carbon is completely oxidized and a clean ash is obtained. Remove the crucible and its contents from the furnace, allow to cool to ambient temperature in a desiccator and weigh to the nearest 0,1 mg. Then heat the crucible and its contents again for about 30 min in the muffle furnace, maintained at 950 °C ± 25 °C, allow to cool to ambient temperature in the desiccator and re-weigh to the nearest 0,1 mg.

If this mass differs from the previous mass by more than 1 % relative to the amount of ash, repeat the heating, cooling and weighing procedure until the difference between two successive weighings is less than 1 % relative to the amount of ash.

8 Expression of results

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

$$\frac{m_2 - m_1}{m_0} \times 100$$

where

m_0 is the mass, in grams, of the test portion;

m_1 is the mass, in grams, of the empty crucible;

m_2 is the mass, in grams, of the crucible and ash.

9 Test report

The test report shall include the following particulars:

a) all details required for full identification of the piece or sample;

b) a reference to this International Standard;

c) the method employed — method A or method B;

d) the temperature used and the reason for its choice if 950 °C is used for method A;

e) the ash content of the product tested, as a percentage by mass;

f) the date of the test.

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