INTERNATIONAL STANDARD

ISO 247-1

First edition
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Rubber — Determination of ash —

Part 1: Combustion method

Caoutchouc — Détermination du taux de cendres —

Partie 1: Technique de combustion sèche

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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.

The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

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. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see www.iso.org/patents).

Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

For an explanation on the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT) see the following URL: www.iso.org/iso/foreword.html.

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

This first edition of ISO 247-1 cancels and replaces ISO 247:2006, which has been technically revised.

The main changes compared to the previous edition are as follows:

— "Method C" has been included as a new procedure;

— Annex A has been added.

A list of all parts in the ISO 247 series can be found on the ISO website.

Rubber — Determination of ash —

Part 1: Combustion method

WARNING 1 — Persons using this document should be familiar with normal laboratory practice. This document 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 determine applicability of any national regulatory conditions.

WARNING 2 — Certain procedures specified in this document might 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 document specifies three 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 that:

— Method A is not used for the determination of ash from compounded or vulcanized rubbers containing chlorine, bromine or iodine;

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

— Method C is intended to be used for the determination of ash from raw, compounded or vulcanized rubber not containing chlorine, bromine or iodine by wrapping the test portion in ashless filter paper;

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

This document 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.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements 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, *Rubber, raw — Determination of volatile-matter content — Part 1: Hot-mill method and oven method*

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

3 Terms and definitions

No terms and definitions are listed in this document.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

— IEC Electropedia: available at https://www.electropedia.org/

— ISO Online browsing platform: available at https://www.iso.org/obp

4 Principle

4.1 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.

4.2 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.

4.3 Method C

A weighed test portion is wrapped in ashless filter paper, pre-ignited at 300 °C ± 25 °C for 1 h, followed by incineration in a muffle furnace at about 550 °C ± 25 °C for 2 h to 4 h until all the carbonaceous matter has been burnt off and constant mass is attained.

4.4 Test results

The three 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.

5 Reagent

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

6 Apparatus

Ordinary laboratory apparatus, plus the following.

6.1 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.

NOTE Platinum crucibles is used for ashing fluorine-containing and lithium-polymerized rubbers as lithium and fluorine compounds might react with silica crucibles to form volatile compounds, giving low ash results.

6.2 Heat-resistant, thermally insulating board, 100 mm^2 and of thickness approximately 5 mm, with a central hole to accommodate the crucible (6.1). About two-thirds of the crucible shall project below the board.

6.3 Bunsen burner, or similar type of gas burner.

6.4 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 300 °C ± 25°C or 550 °C ± 25°C or 950 °C ± 25 °C.

6.5 Ashless filter paper, of 15 cm diameter.

7 Preparation of the test portion

7.1 Test portions of raw natural rubber shall be cut from the homogenized piece prepared in accordance with ISO 1795. 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 248-1.

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

7.3 Test portions of vulcanizate shall be sheeted or crumbed on a mill or comminuted by hand.

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

8 Procedure

8.1 Method A

Heat a clean empty crucible (6.1) of appropriate size for about 30 min in the muffle furnace (6.4), 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 (6.2). Heat the crucible gently with the burner (6.3) 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 (for compounds and vulcanizates, a temperature of 950 °C ± 25 °C may be used), leaving the door of the furnace slightly open for 1 min 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 (for compounds and vulcanizates, a temperature of 950 °C ± 25 °C may be used), 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 weighing meets this requirement.

8.2 Method B

Heat a clean empty crucible (6.1) of appropriate size for about 30 min in the muffle furnace (6.4), 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 concentrated sulfuric acid (5.1) 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 (6.2) 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, leaving the door of the furnace slightly open for 1 min to provide sufficient air to oxidize the carbon 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 weighing is less than 1 % relative to the amount of ash.

8.3 Method C

Weigh, to the nearest 0,1 mg, a test portion of 5 g to 10 g of the homogenized rubber. Wrap in ashless filter paper (6.5) and place in a crucible (6.1) which has been previously ignited and weighed. Introduce the crucible into a muffle furnace (6.4) at controlled temperature of 300 °C ± 25 °C for 1 h pre-ignition followed by incineration in a muffle furnace at 550 °C ± 25 °C for 2 h to 4 h until free from carbon. When ashing is completed, allow the crucible to cool in a desiccator and weigh to the nearest 0,1 mg.

This mass shall not differ from the previous mass by more than 1 mg. If this requirement is not fulfilled, repeat the heating, cooling and weighing procedure until the difference between two successive weighing meets this requirement.

9 Expression of results

The ash content is given, as a percentage by mass, by Formula (1):

$$\frac{m_2 - m_1}{m_0} \times 100 \tag{1}$$

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.

10 Precision

See Annex A.

11 Test report

The test report shall include the following particulars:

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

b) a reference to this document, i.e. ISO 247-1:2018;

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

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

e) the ash determined for the product tested, as a percentage by mass;

f) the date of the test.