INTERNATIONAL STANDARD 247

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Rubber — Determination of ash

Caoutchouc — Détermination des cendres

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

This second edition was submitted directly to the ISO Council, in accordance with clause 6.13.1 of the Directives for the technical work of ISO. It cancels and replaces the first edition (i.e. ISO 247-1976), which had been approved by the member bodies of the following countries :

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

No member body expressed disapproval of the document.

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Rubber — Determination of ash

1 SCOPE AND FIELD OF APPLICATION

1.1 This International Standard 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 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 must be aware of the behaviour of rubber additives at elevated temperatures.

1.2 Methods A and B should not be used for the determination of ash from compounded or vulcanized rubbers containing chlorine, bromine or iodine.

1.3 Method C should not be used for raw rubbers.

1.4 Lithium and fluorine compounds 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 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.

2 REFERENCES

ISO 248, *Rubbers, raw — Determination of volatile matter.*

ISO 1629, *Rubbers and latices — Nomenclature.*

ISO 1795, *Raw rubber in bales — Sampling.*

ISO 1796, *Raw rubber — Sample preparation.*

3 PRINCIPLES OF METHODS

3.1 Method A

Heating of a weighed test portion in a crucible over a gas burner. After expulsion of the volatile decomposition products, transfer of the crucible 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

Heating of a weighed test portion wrapped in ashless filter paper and placed in a crucible, in a muffle furnace until volatile decomposition products have been expelled, all the carbonaceous matter has been burnt off and constant mass is attained.

3.3 Method C

Heating of a weighed test portion in a crucible in the presence of sulphuric 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 APPARATUS

4.1 Crucible, of porcelain, silica or platinum, of capacity approximately 50 ml*. For raw synthetic rubbers, it is permitted to use a crucible of minimum capacity 25 ml per gram of test portion or an aluminium dish or basin of capacity approximately 50 ml.

4.2 Asbestos board (for methods A and C), 100 mm square and of the thickness approximately 5 mm, with a central hole to accommodate the crucible. About two-thirds of the crucible should project below the asbestos board.

4.3 Bunsen burner (for methods A and C), or similar type of gas burner.

* The term millilitre (ml) is commonly used as a special name for the cubic centimetre (cm^3), in accordance with a decision of the Twelfth Conférence générale des poids et mesures. The term millilitre is acceptable, in general, for references in International Standards to capacities of volumetric glassware and to liquid volumes. Apparatus with either type of marking is satisfactory for use with this International Standard.

4.4 Filter paper (for method B only), ashless, of diameter 150 mm.

4.5 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 ± 25 °C or 950 ± 25 °C.

5 REAGENT

Sulphuric acid (for method C only), analytical grade, ρ 1,84 g/ml.

6 PREPARATION OF THE TEST PORTION

6.1 Test portions of raw natural rubber shall be cut from the homogenized piece prepared according to 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.

NOTE — 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 (4.1) of appropriate size for about 30 min in the muffle furnace (4.5), maintained at 550 ± 25 °C, allow to cool to ambient temperature in a desiccator and weigh to the nearest 0,001 g. Take a test portion of about 5 g of raw rubber or 1 to 5 g of compound or vulcanizate, according to the mass of ash to be expected, and weigh to the nearest 0,001 g. Place the weighed test portion in the crucible mounted in the hole in the asbestos board (4.2). Heat the crucible gently with the burner (4.3), 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 ± 25 °C, 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 0,001 g. Then heat the crucible and its contents again for about 30 min in the muffle furnace, maintained at 550 ± 25 °C, allow to cool to ambient temperature in the desiccator and re-weigh to the nearest 0,001 g. This mass should not differ from the previous mass by more than 0,001 g 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.

NOTES

1 For compounds and vulcanizates, a temperature of 950 ± 25 °C may be used. If this temperature is used, aluminium dishes and basins must not be used and the temperature shall be indicated in the test report together with the reason for its use.

2 For raw rubbers, weighings shall be made to an accuracy of 0,000 1 g.

7.2 Method B

Heat the clean empty crucible (4.1) of appropriate size for about 30 min in the muffle furnace (4.5), maintained at 550 ± 25 °C, allow to cool to ambient temperature in a desiccator and weigh to the nearest 0,001 g. Take a test portion of about 5 g of raw rubber or 1 to 5 g of compound or vulcanizate, according to the mass of ash to be expected, and weigh to the nearest 0,001 g. Wrap in ashless filter paper (4.4) and place in the crucible. Transfer the crucible and its contents to the muffle furnace, maintained at 550 ± 25 °C, and close the door rapidly. THE FURNACE DOOR MUST NOT BE OPENED DURING THE FIRST HOUR BECAUSE OF THE RISK OF IGNITING COMBUSTIBLE GASES.

After 1 h, open the door of the furnace slightly to provide sufficient air to oxidize the carbon. Continue heating until the carbon has been 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 0,001 g. Then heat the crucible and its contents again for about 30 min in the muffle furnace, maintained at 550 ± 25 °C, allow to cool to ambient temperature in the desiccator and re-weigh to the nearest 0,001 g. This mass should not differ from the previous mass by more than 0,001 g 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.

NOTES

1 Since the furnace door must be closed rapidly and kept closed after insertion of a crucible, if more than one determination is being made, it is convenient to place the crucibles together on a suitable rack or tray. All the crucibles can be introduced into the furnace in one operation.

2 If the ash line is within 3 mm of the rim of the crucible, the determination shall be abandoned. The test shall then be repeated using either a smaller test portion or a larger crucible. Alternatively, method A may be used in place of method B.

3 For raw rubbers, weighings shall be made to an accuracy of 0,000 1 g.

7.3 Method C

Heat the clean empty crucible (4.1) of appropriate size for about 30 min in the muffle furnace (4.5), maintained at 950 ± 25 °C, allow to cool to ambient temperature in a desiccator and weigh to the nearest 0,001 g. Take a test portion of about 1 to 5 g of the compound or vulcanizate and weigh to the nearest 0,001 g. Pour about 3,5 ml of the concentrated sulphuric acid (clause 5) over the test portion so that the rubber is completely wetted. Place the crucible and its contents in the hole in the asbestos board (4.2) and heat gently with the burner. 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 sulphuric acid is volatilized and a dry, carbonaceous residue remains. Transfer the crucible and its contents to the muffle furnace, maintained at 950 ± 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 0,001 g. Then heat the crucible and its contents again for about 30 min in the muffle furnace, maintained at 950 ± 25 °C, allow to cool to ambient temperature in the desiccator and re-weigh to the nearest 0,001 g.

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 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) reference to this International Standard;

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

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

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

f) date of test.