INTERNATIONAL STANDARD

ISO 9028

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Rubber — Dissolution by acid digestion

Caoutchouc — Dissolution par attaque acide

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Foreword

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The main task of technical committees is to prepare International Standards. 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.

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.

ISO 9028 was prepared by Technical Committee ISO/TC 45, *Rubber and rubber products*, Subcommittee SC 2, *Testing and analysis*.

This second edition cancels and replaces the first edition (ISO 9028:1989), which has been revised to update the normative references.

Rubber — Dissolution by acid digestion

WARNING — Persons using this International Standard should be familiar with normal laboratory practice. This standard 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 ensure compliance with any national regulatory conditions.

1 Scope

This International Standard specifies methods for disintegration of raw rubber and rubber products by nitric acid or by a mixture of nitric and sulfuric acids. This International Standard is generally applicable, but is essential where potentially volatile elements or combinations of elements are present (i.e. As, Sb, Bi, and Zn + Cl, Cu + Cl and Pb + Cl). It is useful in these cases because lower temperatures are involved which result in less loss by volatilization.

These methods will be used in order to produce solutions for the determination of metals, for example as traces, if the application of ISO 247 is not advisable. These methods prevent loss of volatile metal derivatives and the formation of insoluble metal silicates (which may result during dry ashing of halogenated rubbers containing zinc or of rubbers filled with silica).

2 Normative references

The following referenced documents are indispensable for the application 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 124, *Latex, rubber — Determination of total solids content*

ISO 247, *Rubber — Determination of ash*

ISO 1042, *Laboratory glassware — One-mark volumetric flasks*

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

3 Principle

3.1 Method A

Sulfuric acid digestion followed by nitric acid oxidation carried out in open flasks.

Treatment with sulfuric and hydrofluoric acids in order to volatilize any silicon as silicon fluoride and to form metal sulfates is necessary if silicon or silicates are present.

3.2 Method B

Dissolution of the rubber with nitric acid in a polytetrafluoroethylene-coated pressure vessel.

Treatment with sulfuric and hydrofluoric acids in order to volatilize any silicon as silicon fluoride and to form metal sulfates is necessary if silicon or silicates are present.

4 Reagents

WARNING — All recognized health and safety precautions shall be observed during the handling of these reagents.

During the analysis, use only reagents of recognized extra-high-purity analytical grade, and only distilled water or water of equivalent purity.

4.1 Sulfuric acid, $\rho_{20} = 1{,}84$ Mg/m^3.

4.2 Hydrochloric acid, $\rho_{20} = 1{,}18$ Mg/m^3.

4.3 Hydrochloric acid, diluted $1 + 2$.

Dilute 1 volume of concentrated hydrochloric acid (4.2) with 2 volumes of water.

4.4 Hydrofluoric acid, $\rho_{20} = 1{,}12$ Mg/m^3 (38 % to 40 % by mass).

4.5 Nitric acid, $\rho_{20} = 1{,}42$ Mg/m^3.

4.6 Hydrogen peroxide, 38 % (by mass) solution.

5 Apparatus

Ordinary laboratory apparatus, plus the following:

5.1 Balance, accurate to 0,1 mg.

5.2 One-mark volumetric flask, glass-stoppered, of capacity 100 cm^3, class A, complying with the requirements of ISO 1042.

5.3 Platinum crucible, of capacity 50 cm^3.

5.4 Platinum rod, as stirrer.

5.5 Electric heating plate or **gas burner with sand bath**.

5.6 Kjeldahl flask, of capacity 250 cm^3.

5.7 Filter funnel, of diameter 65 mm.

5.8 Pipettes, of capacity 1 cm^3, 5 cm^3, 10 cm^3 and 20 cm^3, respectively. The 5 cm^3 pipette shall be of plastic, not glass, since it is to hold hydrofluoric acid.

5.9 Pressure vessel, non-magnetic, made of high-quality stainless steel, of capacity 20 cm^3 to 50 cm^3, wall-coated with polytetrafluoroethylene, with a magnetic stirrer bar (also coated) and a thermometer.

5.10 Electric hotplate, fitted with a magnetic rotor.

6 Sample preparation

At all stages of sample preparation, take care to avoid contamination.

For the determination of metal in rubber, cut at least 2 g of rubber from the sample, if necessary from more than one place, so that proper representation of the whole sample is achieved. Treat the piece or pieces comprising

the test portion in accordance with ISO 1795. Alternatively, for finished products prepare the test portion by cutting the rubber into small portions each weighing approximately 0,1 g. For the determination of metal in latex, take from the sample a portion of thoroughly mixed latex containing at least 2 g of total solids and dry to constant mass as specified in ISO 124. Digestion of this portion can be facilitated by thinly sheeting it by passing it six times between the tightly closed cold rolls of a laboratory mill, rolling the rubber into a cylinder after each pass and presenting the cylinder end on to the rolls for the next pass.

7 Procedures

7.1 General

The digestion shall be conducted in a well ventilated hood or digestion rack so that acid fumes do not enter the laboratory working space.

7.2 Method A — Digestion in open flasks

7.2.1 Place a test portion (see Clause 6) weighing at least 2 g, weighed to the nearest 2 mg, in the Kjeldahl flask (5.6), add 10 cm^3 of sulfuric acid (4.1) and heat moderately until the test portion has disintegrated. Carefully add 5 cm^3 of nitric acid (4.5). If the reaction becomes too vigorous, cool the flask in a beaker of cold water and store at room temperature for at least 2 h before reheating.

Some rubbers cause considerable splashing; in this case, restart the procedure using a larger flask, decompose with sulfuric acid and cool before adding nitric acid. Store the mixture for a longer time, for example overnight, before heating with the nitric acid.

As soon as the initial reaction has subsided, heat the mixture gently until the vigorous reaction has ceased, and then more strongly until the mixture darkens.

Add a 1 cm^3 portion of nitric acid (4.5) and heat until darkening takes place. Continue this treatment until the solution becomes colourless or pale yellow and fails to darken on further heating.

If the digestion is prolonged, it may be necessary to add about 1 cm^3 of sulfuric acid to prevent the contents of the flask from solidifying.

7.2.2 If the digest is free from insoluble matter, cool, add 0,5 cm^3 of hydrogen peroxide solution (4.6) and 2 drops of nitric acid (4.5) and heat to fuming. Repeat the addition and heating until there is no further reduction in the colour of the solution. Cool, dilute with 10 cm^3 of water and heat to fuming.

Finally, cool the solution and add 20 cm^3 of diluted hydrochloric acid (4.3).

Transfer the acid digest to the volumetric flask (5.2), rinse the Kjeldahl flask with three 5 cm^3 portions of water and add to the volumetric flask. Dilute to the mark with water.

7.2.3 If the digest contains insoluble matter, cool it and transfer it with the residue to the platinum crucible (5.3), rinsing with three 5 cm^3 portions of water. Evaporate to dryness, and then ignite until all the carbon has burnt off.

Certain metals, such as mercury and arsenic, might be volatilized and lost. Therefore, for these metals, do not ignite to complete dryness.

Cool, add a few drops of sulfuric acid (4.1) and 5 cm^3 of hydrofluoric acid (4.4). Evaporate to dryness on the heating device (5.5) while stirring with the platinum rod (5.4). Repeat this procedure twice.

Cool the crucible plus residue and add 20 cm^3 of diluted hydrochloric acid (4.3). If the solution is clear, transfer it to the volumetric flask (5.2), rinsing with three 5 cm^3 portions of water. Dilute to the mark with water.

If the solution is not clear, filter into the volumetric flask (5.2). Rinse the crucible five times with a 5 cm^3 portion of water, and pour each over the filter into the volumetric flask. Dilute to the mark with water.

7.3 Method B — Digestion in a pressure vessel

WARNING — During use, the pressure vessel shall be placed behind a suitable protective screen.

7.3.1 Place a test portion (see Clause 6) of about 100 mg, weighed to the nearest 0,2 mg, in the pressure vessel (5.9). Add the magnetic stirrer bar and rinse with 5 cm^3 of sulfuric acid (4.1) and 3 cm^3 of nitric acid (4.5). Close the pressure vessel in accordance with the manufacturer's instructions. Insert the thermometer. Place the pressure vessel on the hotplate with magnetic rotor (5.10). Heat the plate to 135 °C to 145 °C, starting the magnetic rotor at the same time. Continue stirring the pressure vessel at the indicated temperature for 2 h. Allow the pressure vessel to cool to room temperature. Wipe the pressure vessel dry and open it.

7.3.2 In most cases, the rubber will be found to be completely dissolved. If it is not, proceed in accordance with 7.2.3 in an open crucible.

ICS 83.060

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