INTERNATIONAL STANDARD

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Third edition
2018-07

Rubber — Determination of metal content by atomic absorption spectrometry —

Part 5: Determination of iron content

Caoutchouc — Dosage du métal par spectrométrie d'absorption atomique —

Partie 5: Dosage du fer

Reference number
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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 third edition cancels and replaces the second edition (ISO 6101-5:2006), which has been technically revised as follows:

— nitric acid has been added as a digestion acid;

— the concentration for sulphuric acid has been specified;

— the procedure for the destruction of organic matter has been further detailed;

— the volume for final solution following dissolution using hydrochloric acid (8.2.2) has been changed to 50 cm^3 to ensure consistency of the formulae used throughout the ISO 6101 series;

— Formulae 1 and 2 in Clause 9 have been corrected for the iron content expressed as percentage by mass and milligram per kilogram, respectively;

— Annex A and Annex B have been added to include method of standard addition and precision statement of the method.

A list of all parts in the ISO 6101 series can be found on the ISO website.

Rubber — Determination of metal content by atomic absorption spectrometry —

Part 5: Determination of iron content

1 Scope

This document specifies an atomic absorption spectrometric method for the determination of the iron content of rubbers.

The method is applicable to raw rubber, rubber products and latex having iron contents of 5 mg/kg to 1 000 mg/kg. Higher concentrations can be determined, provided that suitable adjustments are made to the mass of the test portion and/or the concentrations of the solution used.

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 123, *Rubber latex — Sampling*

ISO 124, *Latex, rubber — Determination of total solids content*

ISO 247-1, *Rubber — Determination of ash — Combustion method*

ISO 648, *Laboratory glassware — Single-volume pipettes*

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

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

A test portion is ashed at 550 °C ± 25 °C in accordance with ISO 247-1. The ash is dissolved in hydrochloric acid or nitric acid and if any silicates are present they are decomposed with a mixture of sulfuric acid and hydrofluoric acid to remove them.

The solution obtained is aspirated into an atomic absorption spectrometer and the absorption is measured at a wavelength of 248,3 nm for concentrations up to 10 mg/kg or 0,001 % (by mass), or 372,0 nm for concentrations of 10 mg/kg to 1 000 mg/kg or 0,001 % (by mass) to 0,1 % (by mass).

5 Reagents

During the analysis, unless otherwise stated, use only reagents of recognized analytical grade, and only distilled water or water of equivalent purity.

5.1 Sulfuric acid, ρ_{20} = 1,84 Mg/m^3, 95 % (m/m) to 97 % (m/m).

5.2 Hydrochloric acid, ρ_{20} = 1,19 Mg/m^3, 37 % (m/m).

5.3 Hydrochloric acid diluted 1 + 3, dilute 1 volume of concentrated hydrochloric acid (5.2) with 3 volumes of water.

5.4 Hydrofluoric acid, ρ_{20} = 1,13 Mg/m^3, 38 % (m/m) to 48 % (m/m).

5.5 Concentrated nitric acid, ρ_{20} = 1,42 Mg/m^3, 69 % (m/m).

5.6 Dilute nitric acid, 1,6 % (by mass), carefully pipette 11,5 cm^3 of concentrated nitric acid (5.5) into a 1 000 cm^3 one-mark volumetric flask, making up to the mark with water, and mix thoroughly.

5.7 Standard iron stock solution, containing 1 g of Fe per cubic decimetre.

Either use a commercially available standard iron solutions, or prepare as follows:

Grind metallic iron, purity greater than 99 % (by mass). Weigh 1 g to the nearest 0,1 mg in a 250 cm^3 conical flask (6.11) and dissolve it in a mixture of 100 cm^3 of 1 + 3 hydrochloric acid (5.3) and 10 cm^3 of concentrated nitric acid (5.5). Transfer it to a 1 000 cm^3 one-mark volumetric flask (6.4), dilute to the mark with 1 + 3 hydrochloric acid (5.3) and mix thoroughly.

1 cm^3 of this standard solution contains 1 000 µg of Fe.

5.8 Standard iron solution, containing 10 mg of Fe per 1 000 cm^3.

Carefully pipette 10 cm^3 of the standard iron stock solution (5.7) using volumetric pipette (6.13) into a 1 000 cm^3 one-mark volumetric flask (6.4) and dilute to the mark with 1 + 3 hydrochloric acid (5.3) or diluted nitric acid (5.6).

1 cm^3 of this primary calibration solution contains 10 µg of Fe.

6 Apparatus

Use ordinary laboratory apparatus and the following, ensuring that all apparatus and laboratory implements are non-ferrous.

6.1 Balance, accurate to 0,1 mg.

6.2 Muffle furnace, capable of being maintained at 550 °C ± 25 °C.

6.3 Beaker, of capacity 250 cm^3.

6.4 One-mark volumetric flasks, glass-stoppered, of capacity 50 cm^3, 100 cm^3, 200 cm^3, 500 cm^3 and 1 000 cm^3, complying with the requirements of ISO 1042, class A.

6.5 Filter funnel.

6.6 Crucible (silica or porcelain), of capacity 150 cm^3 depending on the test portion size.