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Rubber — Determination of metal content by atomic absorption spectrometry —

Part 1: Determination of zinc content

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

Partie 1: Dosage du zinc

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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 of 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 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 6101-1:1991), which has been technically revised.

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

— The procedure for the destruction of organic matter is further detailed.

— Formula (2) in 9.2 has been changed to zinc content being expressed as milligram per kilogram.

— Precision data have been updated in Annex B.

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

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

Rubber — Determination of metal content by atomic absorption spectrometry —

Part 1: Determination of zinc content

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 the applicability of any other restrictions.

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 an atomic absorption spectrometric method for the determination of the zinc content of rubbers.

The method is applicable to raw rubber and rubber products having zinc contents at a minimum of 0,05 % (mass fraction). Zinc contents below this limit can be determined, provided that suitable adjustments are made to the mass of the test portion and/or to the concentrations of the solutions used. The use of the standard additions method might lower the bottom limit of detection.

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 — Part 1: Combustion method*

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

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

ISO 1772, *Laboratory crucibles in porcelain and silica*

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

ISO 4793, *Laboratory sintered (fritted) filters — Porosity grading, classification and designation*

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:

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

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

4 Principle

A test portion is ashed at 550 °C ± 25 °C in accordance with ISO 247-1. The ash is dissolved in hydrochloric or nitric acid. The solution is aspirated into an atomic absorption spectrometer and the absorption is measured at a wavelength of 213,8 nm, using a zinc hollow-cathode lamp as the zinc emission source. Any silicates are volatilized by sulfuric acid and hydrofluoric acid.

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.

5.2 Hydrochloric acid, ϱ_{20} = 1, 18 Mg/m^3.

5.3 Hydrochloric acid solutions.

5.3.1 Hydrochloric acid, diluted 1 + 2.

Dilute 1 volume of the concentrated hydrochloric acid (5.2) with 2 volumes of water.

5.3.2 Hydrochloric acid, diluted 1 + 100.

Dilute 1 volume of the concentrated hydrochloric acid (5.2) with 100 volumes of water.

5.4 Hydrofluoric acid, ϱ_{20} = 1, 12 Mg/m^3.

5.5 Nitric acid, ϱ_{20} = 1,42 Mg/m^3.

5.5.1 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 (6.5), making up to the mark with water, and mix thoroughly.

5.6 Zinc standard stock solution (1mg/ml).

Either use a commercially available standard zinc solution, or prepare as follows.

Weigh, to the nearest 0,1 mg, 1 g of pure zinc dust (minimum purity 99,9 %) and dissolve in a minimum amount of the 1 + 2 hydrochloric acid solution (5.3.1). Allow to cool and transfer quantitatively to 1 000 cm^3 one-mark volumetric flask (6.5). Make up to the mark with 1 + 100 hydrochloric acid solution (5.3.2) and mix thoroughly.

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

5.7 Zinc standard solution (10 µg/ml).

Using a pipette (6.6), carefully introduce 10 cm^3 of the zinc standard stock solution (5.6) into 1 000 cm^3 one-mark volumetric flask (6.5). Dilute to the mark with 1 + 2 hydrochloric acid solution (5.3.1) or dilute nitric acid (5.5.1) and mix thoroughly.

Prepare this solution on the day of use.

1 cm^3 of this standard solution contains 10 µg of Zn.

