INTERNATIONAL STANDARD

ISO 6101-2

Third edition
2019-11

Rubber — Determination of metal content by atomic absorption spectrometry —

Part 2: Determination of lead content

Caoutchouc — Détermination de la teneur en métal par spectrométrie d'absorption atomique —

Partie 2: Dosage du plomb

Reference number
ISO 6101-2:2019(E)

ISO copyright office
CP 401 • Ch. de Blandonnet 8
CH-1214 Vernier, Geneva
Phone: +41 22 749 01 11
Fax: +41 22 749 09 47
Email: copyright@iso.org
Website: www.iso.org

Published in Switzerland

Contents

Page

Foreword ..... **iv**

1 Scope **1**

2 Normative references **1**

3 Terms and definitions **1**

4 Principle **2**

5 Reagents **2**

6 Apparatus **3**

7 Sampling **3**

8 Procedure **4**

8.1 Test portion 4

8.2 Preparation of test solution 4

8.2.1 Destruction of organic matter 4

8.2.2 Dissolution of inorganic residue 4

8.3 Preparation of the calibration graph 5

8.3.1 Preparation of calibration solutions 5

8.3.2 Spectrometric measurements 5

8.3.3 Plotting the calibration graph 5

8.4 Determination 5

8.4.1 Spectrometric measurements 5

8.4.2 Dilution 6

8.5 Blank determination 6

8.6 Number of determinations 6

9 Expression of results **6**

10 Test report **7**

Annex A (informative) **Method of standard additions** **9**

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 6101-2:1997), of which it constitutes a minor revision to update normative references in Clause 2.

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 2:
Determination of lead 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 lead content of rubbers.

The method is applicable to raw rubber and rubber products. There is no limit to the concentration of lead that can be determined. High or low concentrations can be determined, provided that suitable adjustments are made to the mass of the test portion and/or the concentration 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 247-1:2018, *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*

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

If the rubber does not contain halogenated compounds, a test portion is directly ashed at 550 °C ± 25 °C as specified in ISO 247-1:2018, method A. If the rubber contains halogenated compounds, a test portion is first mineralized with sulfuric and nitric acids, the acids are removed by evaporation, and the portion is ashed at 550 °C ± 25 °C.

NOTE The presence of even small amounts of halogens can lead to the loss of volatile lead salts during dry ashing.

The ash obtained is boiled with ammonium acetate solution to dissolve the lead. Insoluble lead silicates, if present, are converted to chloride by boiling with a mixture of hydrochloric acid, nitric acid and hydrogen peroxide.

The solution is aspirated into an atomic absorption spectrometer and the absorbance is measured at a wavelength of 283,3 nm, using a lead hollow-cathode lamp as the lead emission source.

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 Nitric acid, ρ_{20} = 1,40 Mg/m^3.

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

5.4 Hydrochloric acid, diluted 1 + 2.

Dilute 1 volume of concentrated hydrochloric acid (5.3) with 2 volumes of water.

5.5 Hydrogen peroxide, 30 % (mass fraction) solution.

5.6 Ammonium acetate, 180 g/dm^3 solution.

5.7 Standard lead stock solution, containing 1 g of Pb per cubic decimetre.

Either use a commercially available standard lead solution, or prepare as follows:

Weigh, to the nearest 0,1 mg, 1 g of metallic lead (purity ≥ 99,95 %) and transfer to a 100 cm^3 beaker (6.12). Add 30 cm^3 of water and 20 cm^3 of nitric acid (5.2) and boil on a sand bath (6.10). If the lead is dissolved, continue boiling until the solution is reduced to about 20 cm^3 or less. No nitrogen oxides should be observed. Otherwise, add water and continue boiling. Transfer to a 1 000 cm^3 one-mark volumetric flask (see 6.7) with 1 + 2 hydrochloric acid (5.4) and fill to the mark with 1 + 2 hydrochloric acid (5.4).

1 cm^3 of this standard stock solution contains 1 mg of Pb.

5.8 Standard lead solution, containing 10 mg of Pb per cubic decimetre.

Using a pipette (6.9), carefully introduce 10 cm^3 of the standard lead stock solution (5.7) into a 1 000 cm^3 one-mark volumetric flask (see 6.7). Dilute to the mark with 1 + 2 hydrochloric acid (5.4), and mix thoroughly.

Prepare this solution on the day of use.

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