

SVENSK STANDARD

SS-ISO 6101-2:2019

Gummi – Bestämning av metallhalt genom
flamabsorptionsspektrometri –

Del 2: Bestämning av blyhalt (ISO 6101-2:2019, IDT)

Rubber – Determination of metal content by atomic absorption
spectrometry –

Part 2: Determination of lead content (ISO 6101-2:2019, IDT)



sis Svenska
Institutet för
Standarder

Language: engelska/English

Edition: 2

This preview is downloaded from www.sis.se. Buy the entire
standard via <https://www.sis.se/std-80018826>

Den här standarden kan hjälpa dig att effektivisera och kvalitetssäkra ditt arbete. SIS har fler tjänster att erbjuda dig för att underlätta tillämpningen av standarder i din verksamhet.

SIS Abonnemang

Snabb och enkel åtkomst till gällande standard med SIS Abonnemang, en prenumerationstjänst genom vilken din organisation får tillgång till all världens standarder, senaste uppdateringarna och där hela din organisation kan ta del av innehållet i prenumerationen.

Utbildning, event och publikationer

Vi erbjuder även utbildningar, rådgivning och event kring våra mest sålda standarder och frågor kopplade till utveckling av standarder. Vi ger också ut handböcker som underlättar ditt arbete med att använda en specifik standard.

Vill du delta i ett standardiseringsprojekt?

Genom att delta som expert i någon av SIS 300 tekniska kommittéer inom CEN (europeisk standardisering) och/eller ISO (internationell standardisering) har du möjlighet att påverka standardiseringsarbetet i frågor som är viktiga för din organisation. Välkommen att kontakta SIS för att få veta mer!

Kontakt

Skriv till kundservice@sis.se, besök [sis.se](https://www.sis.se) eller ring 08 - 555 523 10

© Copyright/Upphovsrätten till denna produkt tillhör Svenska institutet för standarder, Stockholm, Sverige. Upphovsrätten och användningen av denna produkt regleras i slutanvändarlicensen som återfinns på [sis.se/slutanvandarlicens](https://www.sis.se/slutanvandarlicens) och som du automatiskt blir bunden av när du använder produkten. För ordlista och förkortningar se [sis.se/ordlista](https://www.sis.se/ordlista).

© Copyright Svenska institutet för standarder, Stockholm, Sweden. All rights reserved. The copyright and use of this product is governed by the end-user licence agreement which you automatically will be bound to when using the product. You will find the licence at [sis.se/enduserlicenseagreement](https://www.sis.se/enduserlicenseagreement).

Upplysningar om sakinnehållet i standarden lämnas av Svenska institutet för standarder, telefon 08 - 555 520 00. Standarder kan beställas hos SIS som även lämnar allmänna upplysningar om svensk och utländsk standard.

Standarden är framtagen av kommittén för Gummi och gummiprodukter, SIS/TK 154.

Har du synpunkter på innehållet i den här standarden, vill du delta i ett kommande revideringsarbete eller vara med och ta fram andra standarder inom området? Gå in på www.sis.se - där hittar du mer information.

Den internationella standarden ISO 6101-2:2019 gäller som svensk standard. Detta dokument innehåller den officiella engelska versionen av ISO 6101-2:2019.

Denna standard ersätter SS-ISO 6101-2, utgåva 1

The International Standard ISO 6101-2:2019 has the status of a Swedish Standard. This document contains the official English version of ISO 6101-2:2019.

This standard supersedes the SS-ISO 6101-2, edition 1

Contents	Page
Foreword	v
1 Scope	6
2 Normative references	6
3 Terms and definitions	6
4 Principle	7
5 Reagents	7
6 Apparatus	8
7 Sampling	8
8 Procedure	9
8.1 Test portion	9
8.2 Preparation of test solution.....	9
8.2.1 Destruction of organic matter	9
8.2.2 Dissolution of inorganic residue.....	9
8.3 Preparation of the calibration graph.....	10
8.3.1 Preparation of calibration solutions	10
8.3.2 Spectrometric measurements	10
8.3.3 Plotting the calibration graph.....	10
8.4 Determination	10
8.4.1 Spectrometric measurements	10
8.4.2 Dilution.....	11
8.5 Blank determination	11
8.6 Number of determinations	11
9 Expression of results	11
10 Test report	12
Annex A (informative) Method of standard additions	14

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 $550\text{ °C} \pm 25\text{ °C}$ 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 $550\text{ °C} \pm 25\text{ °C}$.

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

5.2 Nitric acid, $\rho_{20} = 1,40\text{ Mg/m}^3$.

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

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/dm³ 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 $\geq 99,95\%$) and transfer to a 100 cm³ beaker (6.12). Add 30 cm³ of water and 20 cm³ 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 cm³ or less. No nitrogen oxides should be observed. Otherwise, add water and continue boiling. Transfer to a 1 000 cm³ 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 cm³ 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 cm³ of the standard lead stock solution (5.7) into a 1 000 cm³ 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 cm³ of this standard solution contains 10 µg of Pb.

6 Apparatus

Ordinary laboratory apparatus, plus the following.

6.1 Atomic absorption spectrometer, fitted with a burner fed with acetylene and air, compressed to at least 60 kPa and 300 kPa, respectively, and also fitted with a lead hollow-cathode lamp as the lead emission source. The instrument shall be operated in accordance with the manufacturer's instructions for optimum performance.

Alternatively, an **electrothermal atomization device (graphite furnace)** may be used. It shall be operated by a competent person in accordance with the manufacturer's instructions for optimum performance, during drying, ashing and volatilization of the test portion.

6.2 Balance, accurate to 0,1 mg.

6.3 Muffle furnace, capable of being maintained at $550\text{ °C} \pm 25\text{ °C}$.

6.4 Evaporating dish, of capacity about 100 cm³, made of quartz glass, with a clock-glass cover.

6.5 Conical flask, of capacity 250 cm³, made of silica or borosilicate glass.

6.6 Measuring cylinder, of capacity 25 cm³, graduated in 0,5 cm³ divisions.

6.7 One-mark volumetric flasks, glass-stoppered, of capacities 50 cm³, 100 cm³, 200 cm³, 500 cm³ and 1 000 cm³, in accordance with the requirements of ISO 1042, class A.

6.8 Filter funnel, 65 mm diameter, 60° angle.

6.9 Volumetric pipettes, of capacities 5 cm³, 10 cm³, 20 cm³ and 50 cm³, in accordance with the requirements of ISO 648, class A.

6.10 Electric hotplate or sand bath heated by a gas burner.

6.11 Steam bath.

6.12 Beaker, of capacity 100 cm³.

6.13 Crucible, of platinum, and of capacity 50 cm³ to 150 cm³ depending on the test portion size.

6.14 Crucible, of silica or borosilicate glass, of capacity 50 cm³ to 150 cm³ depending on the test portion size, in accordance with the requirements of ISO 1772.

7 Sampling

Carry out sampling as follows:

- raw rubber: in accordance with ISO 1795;
- latex: in accordance with ISO 123;
- products: to be representative of the whole batch.