SVENSK STANDARD
SS-ISO 19242:2021

Gummi – Bestämning av total svavelhalt med jonkromatografi (ISO 19242:2021, IDT)

Rubber – Determination of total sulfur content by ion chromatography (ISO 19242:2021, IDT)

Svenska Institutet för Standarder

Language: engelska/English

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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.

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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.

Fastställd: 2021-10-08
ICS: 83.060

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Den internationella standarden ISO 19242:2021 gäller som svensk standard. Detta dokument innehåller den officiella engelska versionen av ISO 19242:2021.

The International Standard ISO 19242:2021 has the status of a Swedish Standard. This document contains the official English version of ISO 19242:2021.

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LÄSANVISNINGAR FÖR STANDARDER

I dessa anvisningar behandlas huvudprinciperna för hur regler och yttre begränsningar anges i standardiseringsprodukter.

Krav

Ett krav är ett uttryck i ett dokuments innehåll som anger objektivt verifierbara kriterier som ska uppfyllas och från vilka ingen avvikelse tillåts om efterlevnad av dokumentet ska kunna åberopas.
Krav uttrycks med hjälpverbet ska (eller ska inte för förbud).

Rekommendation

En rekommendation är ett uttryck i ett dokuments innehåll som anger en valmöjlighet eller ett tillvägagångssätt som bedöms vara särskilt lämpligt utan att nödvändigtvis nämna eller utesluta andra.
Rekommendationer uttrycks med hjälpverbet bör (eller bör inte för avrådanden).

Instruktion

Instruktioner anges i imperativ form och används för att ange hur något görs eller utförs. De kan underordnas en annan regel, såsom ett krav eller en rekommendation. De kan även användas självständigt, och är då att betrakta som krav.

Förklaring

En förklaring är ett uttryck i ett dokuments innehåll som förmedlar information. En förklaring kan uttrycka tillåtelse, möjlighet eller förmåga. Tillåtelse uttrycks med hjälpverbet får (eller motsatsen behöver inte). Möjlighet och förmåga uttrycks med hjälpverbet kan (eller motsatsen kan inte).

READING INSTRUCTIONS FOR STANDARDS

These instructions cover the main principles for the use of provisions and external constraints in standardization deliverables.

Requirement

A requirement is an expression, in the content of a document, that conveys objectively verifiable criteria to be fulfilled, and from which no deviation is permitted if conformance with the document is to be claimed. Requirements are expressed by the auxiliary shall (or shall not for prohibition).

Recommendation

A recommendation is an expression, in the content of a document, that conveys a suggested possible choice or course of action deemed to be particularly suitable, without necessarily mentioning or excluding others. Recommendations are expressed by the auxiliary should (or should not for dissuasion).

Instruction

An instruction is expressed in the imperative mood and is used in order to convey an action to be performed. It can be subordinated to another provision, such as a requirement or a recommendation. It can also be used independently and is then to be regarded as a requirement.

Statement

A statement is an expression, in the content of a document, that conveys information. A statement can express permission, possibility or capability. Permission is expressed by the auxiliary may (its opposite being need not). Possibility and capability are expressed by the auxiliary can (its opposite being cannot).

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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 second edition cancels and replaces the first edition (ISO 19242:2015), of which it constitutes a minor revision. The main changes compared to the previous edition are as follows:

— the CAS numbers have been added for all chemicals listed in this document;

— Clause 3 on “Terms and definitions” has been added.

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.

Introduction

Titration methods using thorin, a toxic arsenic compound as a reagent, have been used to determine the total sulfur content in raw rubber and rubber compounds. In this document, the sulfur in a test piece is burnt and oxidized to sulfur dioxide, which is then absorbed into a hydrogen peroxide solution and converted to sulfuric acid for determination by ion chromatography.

No hazardous reagents are required, thus improving the working environment as well as being eco-friendly. In addition, ion chromatography is used worldwide for its simplicity and high accuracy.

Rubber — Determination of total sulfur content by ion chromatography

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 ensure compliance with any national regulatory conditions.

WARNING 2 — The use of this document pre-supposes sufficient working knowledge of the principles and techniques of ion chromatography for the analyst to perform the operations described and interpret the results correctly.

CAUTION — Certain procedures specified in this document can 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 a method for the determination of the total sulfur content of raw rubber and unvulcanized or vulcanized rubber compounds by ion chromatography following the preparation of a sample solution using either a tubular furnace combustion or an oxygen combustion flask method.

The tubular furnace combustion method is applicable for rubbers with sulfur content less than 0,1 % as it is possible that the oxygen combustion flask method will not give sufficiently accurate results.

The oxygen combustion flask method is not applicable to rubbers containing a metal salt that forms an insoluble metal sulfate, such as barium sulfate (CAS 7727-43-7).

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

ISO 3696, *Water for analytical laboratory use — Specification and test methods*

ISO 4661-2, *Rubber, vulcanized — Preparation of samples and test pieces — Part 2: Chemical tests*

3 Terms and definitions

No terms and definitions are listed in this document.

ISO and IEC maintain terminology databases for use in standardization at the following addresses:

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

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

4 Principle

4.1 Combustion

4.1.1 Tubular furnace combustion method

A test piece is burnt in an oxygen (CAS 7782-44-7) stream in a tubular furnace with an electrical heater. The sulfur in the test piece is oxidized to sulfur dioxide (CAS 7446-09-5), which is absorbed into hydrogen peroxide (CAS 7722-84-1) solution and converted to sulfuric acid (CAS 7664-93-9).

4.1.2 Oxygen combustion flask method

A test piece is burnt in the presence of oxygen in a flask. The sulfur in the test piece is oxidized to sulfur dioxide, which is absorbed into hydrogen peroxide solution and converted to sulfuric acid.

4.2 Determination

The sulfate from either the tubular furnace combustion or the oxygen combustion flask method is quantitatively analysed by ion chromatography, and the result is converted to the total sulfur content of the original rubber sample.

5 Reagents

Use only reagents of recognized analytical grade and only water as specified in 5.1. The reagents for the tubular furnace combustion method are given in A.2. The reagents for the oxygen combustion flask method are given in B.2.

5.1 Water, of grade 1 or higher as specified in ISO 3696.

5.2 Sulfate stock standard solution, of a commercial standard solution with a certified sulfate concentration, e.g. 1 000 mg/dm^3 of sulfate, traceable to national standards.

Observe the manufacturer's expiry date or recommended shelf-life.

5.3 Sulfate calibration solutions, prepared by dilution of the sulfate stock standard solution (5.2) with water (5.1).

Prepare at least four solutions of different concentration of sulfate covering the expected concentration from the sample. The solutions shall be prepared every analytical day.

5.4 Eluent solution, capable of eluting sulfate to a proper retention time in selected column.

Follow the column manufacturer’s instructions. Some examples of eluent are shown in Annex C.

6 Apparatus

6.1 Balance, of accuracy to the nearest 0,1 mg.

6.2 Apparatus for the tubular furnace combustion method, as specified in A.3.

6.3 Apparatus for the oxygen combustion flask method, as specified in B.3.

6.4 Ion chromatograph, consisting of a pump to supply eluent, injection valve, column and conductivity detector specified as follows:

— pump: capable of delivering a constant flow within the range of 0,1 cm^3/min to 2,0 cm^3/min;

— injection valve: capable of injecting a constant volume of solution;

— column: filled with anion exchange resin suitable for resolving sulfates from other inorganic anions; some examples are shown in Annex C;

— suppressor: used for reducing the conductivity of high ionic eluent;

— detector: for detecting conductivity.

An ion chromatograph fitted with a suppressor is suitable for sensitive analysis (see Figure 1). The suppressor is attached between the column and the detector. However, an ion chromatograph without a suppressor may also be selected (see Figure 2). For the conditions of these methods, see Annex C.

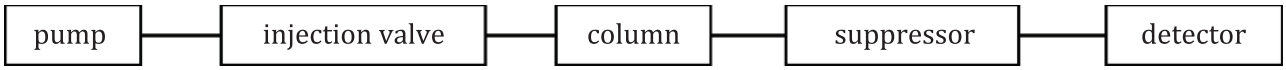

Figure 1 — Example of basic ion chromatography system with suppressor

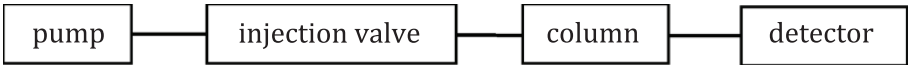

Figure 2 — Example of basic ion chromatography system without suppressor

7 Procedure

7.1 Preparation of sample solutions

7.1.1 Tubular furnace combustion method

Follow the method specified in Annex A. Repeat the procedure to obtain two solutions for each sample.

7.1.2 Oxygen combustion flask method

Follow the method specified in Annex B. Repeat the procedure to obtain two solutions for each sample.

7.2 Determination of sulfate by ion chromatography

7.2.1 Setting up the ion chromatograph

Set up the ion chromatograph in accordance with the manufacturer's instructions.

7.2.2 Measuring calibration solutions

Inject the calibration solutions in order of increasing concentration and measure the conductivity of sulfate peak area in each solution. Determine the correlation formula (calibration curve) by plotting the peak area as a function of concentration by means of linear regression.