INTERNATIONAL STANDARD

ISO 15671

Second edition
2023-02

# Rubber and rubber products — Determination of total sulfur content using an automatic analyser

*Caoutchouc et additifs pour caoutchouc — Dosage du soufre total à l'aide d'un analyseur automatique*

Reference number
ISO 15671:2023(E)

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

Published in Switzerland

# Contents

# 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 15671:2000), which has been technically revised.

The main changes are as follows:

- an alternative detector to determine sulfur dioxide has been added;
- sampling and preparation of the test piece have been added;
- precision statement has been updated in Annex A.

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 and rubber products — Determination of total sulfur content using an automatic analyser

**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 instrumental (automatic analyser) method for the determination of total sulfur in rubber and rubber products.

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

## 3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

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/

**3.1**
**sample**
unit selected to represent the material to be analysed

**3.2**
**test piece**
actual material used in the analysis

**3.3**
**control sample**
material with a recognized content of sulfur, analysed with each set of test pieces

## 4 Principle

**4.1** Specified here is a reliable, rapid, instrumental (automatic analyser) method for determining total sulfur in rubber and rubber products. The sulfur is determined by a single instrumental procedure consisting of weighing a test piece, placing it in the instrument and initiating the (subsequently automatic) analytical process. The analysis may be controlled manually to a limited degree, and a capability to perform computations automatically may be provided by the instrument used to perform the analysis.

**4.2** The actual process can vary substantially from instrument to instrument because a variety of means can be used to meet the primary requirements of the method. The method includes the following:

a) conversion of sulfur-containing materials to sulfur dioxide in an oxygen stream;

b) determination of the sulfur dioxide by one of three detection schemes, and hence the total sulfur content.

**4.3** In the hydrogen peroxide detection configuration, the sulfur dioxide is absorbed in hydrogen peroxide, converting sulfur dioxide to sulfuric acid which is subsequently titrated with standard alkali, enabling the sulfur to be calculated.

**4.4** In the iodine solution detection configuration, the sulfur dioxide is absorbed in standard iodine solution, reducing iodine to iodide which is subsequently measured by a polarized dual platinum electrode system, enabling the sulfur to be calculated.

**4.5** In the sulfur dioxide detection configuration, the sulfur dioxide is passed through an infrared absorption detector or thermal conductivity detector or any equivalent detector and determined using a sulfur dioxide standard, enabling the sulfur to be calculated.

## 5 Requirements for apparatus

**5.1** Because a variety of instrumental (automatic analyser) component configurations can be used satisfactorily for this method, no specifications are presented with regard to overall system design. Functionally, however, the following requirements are specified for all instruments.

**5.2** The conditions for combustion of the sample shall be such that (for the full range of applicable samples) sulfur-containing components shall be converted completely to sulfur dioxide. General instrumental conditions that affect complete combustion include:

a) availability of the oxidant;

b) temperature;

c) time.

**5.3** For the configuration described in 4.3, a correction shall be made for acidity when chlorine is present by titration of a portion of the absorption solution for chloride, which is calculated as hydrochloric acid and subtracted from the total acidity.

**5.4** In the iodine solution and sulfur dioxide detection methods (4.4 and 4.5), the detection system shall determine sulfur without interference and the detector should ideally provide a linear response that correlates directly with sulfur concentration over the full range of possible concentrations from the applicable test samples.

**5.5** The system shall include provisions for evaluating nonlinear response appropriately, so that nonlinear responses can be correlated accurately with concentration. Such provisions can be integral with the instrument or be provided by (auxiliary) computation schemes.

**5.6** Finally, except for those systems in which the sulfur concentration is expressed as a direct output, the instrument shall include an appropriate detector response readout device.

## 6 Reagents

Reagent grade chemicals shall be used in all analyses unless otherwise indicated.

**6.1** **Oxygen**, as specified by the instrument manufacturer.

**6.2** **Additional reagents** as specified by the instrument manufacturer.

This specification refers to the reagents used to meet the requirements cited in 4.3 to 4.5. These reagents can vary substantially for different instruments; in all cases, however, the reagents specified by the instrument manufacturer shall be used.

## 7 Instrument (automatic analyser) preparation and calibration

**7.1** Assemble the instrumental (automatic analyser) system in accordance with the manufacturer's instructions.

**7.2** For the response (drift) adjustment, weigh and analyse (in accordance with the manufacturer's instructions) an appropriate test piece of the sulfur calibrating agent. Repeat this procedure, adjusting instrument response, as recommended by the manufacturer, until the absence of drift is indicated.

**7.3** For the calibration, select calibrating agents and materials specified by the manufacturer that have certified sulfur contents lying within the range of those of the samples to be analysed. At least three such calibrating agents are recommended for each range of sulfur contents to be determined. When possible, two of the calibrating agents shall bracket the range of sulfur contents to be determined, with the third falling within the range.

**7.4** For the calibration procedure, analyse pieces of the calibrating agent chosen (see 7.3) to represent the sulfur content in the samples to be tested. Continue analysing until the results from five consecutive determinations fall within the repeatability interval (see Annex A) of this test method. Calibrate the instrument in accordance with the manufacturer's instructions using these values. The results obtained shall be within the precision limits stated for the calibrating agent, otherwise the calibration procedure shall be repeated.

**7.5** For the periodic calibration verification and recalibration, analyse a control sample on a periodic basis. The results obtained for the control sample shall be within established limits. If not, all results obtained since the last successful control check shall be rejected and the calibration procedure repeated.

## 8 Sampling and preparation of test piece

For solid rubber, take the laboratory sample and prepare the test piece in accordance with the method specified in ISO 1795.

For latex, take the laboratory sample in accordance with ISO 123 and prepare the dry latex test piece as specified in ISO 124.

Prepare a test piece from the sample in accordance with the manufacturer's instructions.

## 9 Procedure

Analyse a test piece of the sample in accordance with the manufacturer's instructions.

Carry out the analysis in duplicate.

## 10 Calculation

Calculate the percent sulfur, $S$, by Formula (1):

$$S = \frac{B \times C}{m} \times 100\ \% \qquad (1)$$

where

$B$ is the detector response for sulfur;

$C$ is the detector response per unit mass established for sulfur during calibration;

$m$ is the mass of the test piece, in grams.

## 11 Precision

See Annex A.

## 12 Test report

The test report shall include the following information:

a) a reference to this document and its year of publication, i.e. ISO 15671:2023;

b) all details necessary for identification of the sample analysed;

c) the procedure used (hydrogen peroxide, iodine solution or sulfur dioxide);

d) the duplicate percent sulfur results to the nearest 0,01 %;

e) the instrument (automatic analyser) make and model;

f) any deviations from the method;

g) the date of the analysis.

# Annex A
(informative)

# Precision

The precision of the test method was determined in accordance with ISO 19983. Refer to this document for terminology and other statistical details.

The precision data are given in Table A.1. The precision parameters should not be used for acceptance or rejection of any group of materials without documentation that the parameters are applicable to those particular materials and specific test protocols of the test method. The precision is expressed on the basis of a 95 % confidence level for the values established for repeatability *r* and reproducibility *R*.

The results contained in Table A.1 are average values and give an estimate of the precision of this test method as determined in an interlaboratory test programme, carried out in 2022, in which three laboratories took part, performing triplicate analyses on two rubber compounds with different sulfur loading, R2 and R3. The prepared compound was sub-sampled into two, labelled A and B (which was tested on two different weeks). Thus, essentially, samples A and B were the same and were treated as such in the statistical computations. Each participating laboratory was required to carry out the test at the specified date.

A type 1 precision was evaluated, based on the method of sampling used for the interlaboratory test programme.

**Repeatability:** The repeatability *r* (in measurement units) of the test method has been established as the appropriate value tabulated in Table A.1. Three single test results, obtained in the same laboratory under normal test method procedures, that differ by more than the tabulated value of *r* (for any given level) should be considered to have come from different, or non-identical, sample populations.

**Reproducibility:** The reproducibility *R* (in measurement units) of the test method has been established as the appropriate value tabulated in Table A.1. Three single test results, obtained in different laboratories under normal test method procedures, that differ by more than the tabulated value of *R* (for any given level) should be considered to have come from different, or non-identical, sample populations.

**Bias:** In test method terminology, bias is the difference between an average test value and the reference (or true) test property value. Reference values do not exist for this test method since the value (of the test property) is exclusively defined by the test method. Bias cannot therefore be determined for this particular method.

**Table A.1 — Precision data for the determination of total sulfur content using an automatic analyser**

| Sample | Average result | Within-laboratory | | Between laboratories | |
|---|---|---|---|---|---|
| | (%) | $s_r$ | $r$ | $S_R$ | $R$ |
| R2 | 1,34 | 0,02 | 0,06 | 0,07 | 0,20 |
| R3 | 1,95 | 0,02 | 0,05 | 0,15 | 0,44 |

$s_r$ is the within-laboratory standard deviation

$r$ is the repeatability (in measurement units)

$S_R$ is the between-laboratory standard deviation

$R$ is the reproducibility (in measurement units)