INTERNATIONAL STANDARD

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Rubber and rubber additives — 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

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Foreword

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International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 3.

Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

Attention is drawn to the possibility that some of the elements of this International Standard may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

International Standard ISO 15671 was prepared by Technical Committee ISO/TC 45, *Rubber and rubber products*, Subcommittee SC 2, *Testing and analyses*.

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

WARNING — Persons using this International Standard should be familiar with normal laboratory practice. This standard 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.

1 Scope

This International Standard specifies an instrumental (automatic analyser) method for the determination of total sulfur in rubber and rubber additives.

2 Terms and definitions

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

2.1
sample
unit selected to represent the material to be analysed

2.2
test portion
actual material used in the analysis

2.3
control sample
material with a recognized content of sulfur, analysed with each set of test portions

3 Principle

3.1 Specified is a reliable, rapid, instrumental (automatic analyser) method for determining total sulfur in rubber and rubber additives. The sulfur is determined by a single instrumental procedure consisting of weighing a test portion, 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.

3.2 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.

3.3 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.

3.4 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.

3.5 In the **sulfur dioxide detection** configuration, the sulfur dioxide is passed through an infrared absorption detector and determined using a sulfur dioxide standard, enabling the sulfur to be calculated.

4 Requirements for apparatus

4.1 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.

4.2 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.

4.3 For the configuration described in 3.3, 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.

4.4 In the iodine solution and sulfur dioxide detection methods (3.4 and 3.5), 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.

4.5 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.

4.6 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.

5 Reagents

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

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

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

This specification refers to the reagents used to meet the requirements cited in 3.3 to 3.5. These reagents can vary substantially for different instruments; in all cases, however, the reagents specified by the instrument manufacturer shall be used.

6 Instrument (automatic analyser) preparation and calibration

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

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

6.3 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.

6.4 For the calibration procedure, analyse portions of the calibrating agent chosen (see 6.3) 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 9.3) 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.

6.5 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.

7 Procedure

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

Carry out the analysis in duplicate.

8 Calculation

Calculate the percent sulfur S as follows:

$$S = \frac{B \times C}{m} \times 100\ \%$$

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 portion, in grams.

9 Precision

9.1 Although the precision of this test method was calculated from data obtained from the analysis of bituminous coal, it is considered to be directly applicable to the analysis for sulfur in rubber compounds. The precision is expressed in terms as specified in ISO/TR 9272, on the basis of a 95 % confidence level for the values established for repeatability r and reproducibility R .

9.2 The precision data given in this clause give an estimate of the precision of this method with the materials used in the particular interlaboratory test programme. The precision parameters shall not be used for acceptance or rejection testing of any group of materials without documentation that the parameters are applicable to the particular group of materials and the specific test protocols of the method.

9.3 The **repeatability** r, in percent sulfur, has been established as the value in Table 1. Two results, obtained in one laboratory (same instrument, material, operator) under normal procedures, that differ by more than this tabulated value shall be considered to have come from different or non-identical sample populations.

9.4 The **reproducibility** R, in percent sulfur, has been established as the value in Table 1. Two results, obtained in different laboratories (same material, but different instrument and different operator) under normal procedures, that differ by more than this tabulated value shall be considered to have come from different or non-identical sample populations.

9.5 Bias is eliminated when the apparatus is calibrated properly against certified reference standards. Proper calibration includes comparison of test data on calibrating agents that have certified sulfur contents.

Table 1 — Sulfur analysis precision

Within lab r	Between labs R
0,05	0,09
$r = 2{,}83 \times s_r$ where s_r is the repeatability standard deviation. $R = 2{,}83 \times s_R$ where s_R is the reproducibility standard deviation.	

10 Test report

The test report shall include the following information:

a) a reference to this International Standard;

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 0,1 %;

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

f) any deviations from the method;

g) the date of the analysis.