

BS ISO 8332:2011



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Rubber compounding ingredients — Sulfur — Methods of test

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National foreword

This British Standard is the UK implementation of ISO 8332:2011. It supersedes BS ISO 8332:2006 which is withdrawn.

The UK participation in its preparation was entrusted to Technical Committee PRI/23, Test methods for rubber and non-black compounding ingredients.

A list of organizations represented on this committee can be obtained on request to its secretary.

This publication does not purport to include all the necessary provisions of a contract. Users are responsible for its correct application.

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ISBN 978 0 580 69692 3

ICS 83.040.20

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This British Standard was published under the authority of the Standards Policy and Strategy Committee on 31 January 2012.

Amendments issued since publication

Date	Text affected
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INTERNATIONAL STANDARD

BS ISO 8332:2011

ISO
8332

Fourth edition
2011-12-01

Rubber compounding ingredients — Sulfur — Methods of test

Ingrédients de mélange du caoutchouc — Soufre — Méthodes d'essai



Reference number
ISO 8332:2011(E)

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ISO copyright office
Case postale 56 • CH-1211 Geneva 20
Tel. + 41 22 749 01 11
Fax + 41 22 749 09 47
E-mail copyright@iso.org
Web www.iso.org

Published in Switzerland

Contents

Page

Foreword	v
Introduction.....	vi
1 Scope	1
2 Normative references	1
3 Physical and chemical properties	1
4 Determination of total sulfur content	2
4.1 Principle	2
4.2 Reagents	2
4.3 Procedure	2
4.4 Expression of results	3
4.5 Precision	3
5 Determination of sieve residue	3
5.1 Principle	3
5.2 Method A	4
5.3 Method B	5
6 Determination of oil content of oil-treated sulfur	6
6.1 Principle	6
6.2 Hexane procedure	7
6.3 Sulfur-saturated hexane procedure	7
7 Determination of insoluble-sulfur content	8
7.1 Principle	8
7.2 Reference procedure using toluene	8
7.3 Alternative procedure using carbon disulfide	9
8 Determination of thermal reversion of insoluble sulfur	10
8.1 General	10
8.2 Method A	10
8.3 Method B	13
8.4 Precision	16
9 Determination of loss in mass at 80 °C	16
9.1 Principle	16
9.2 Apparatus	16
9.3 Procedure	17
9.4 Expression of results	17
9.5 Precision	17
10 Determination of ash at 850 °C to 900 °C	18
10.1 Principle	18
10.2 Apparatus	18
10.3 Procedure	18
10.4 Expression of results	18
10.5 Precision	18
11 Determination of acidity	19
12 Determination of arsenic content	19
13 Test report	19
Annex A (informative) Typical values of the properties of sulfur used as a rubber compounding ingredient	20

Annex B (informative) Precision 22

Bibliography 25

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.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

The main task of technical committees is to prepare International Standards. 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 document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

ISO 8332 was prepared by Technical Committee ISO/TC 45, *Rubber and rubber products*, Subcommittee SC 3, *Raw materials (including latex) for use in the rubber industry*.

This fourth edition cancels and replaces the third edition (ISO 8332:2006), in which the following changes have been made.

- a) the dry procedure for the determination of the sieve residue has been deleted;
- b) the precision data have been moved to informative Annex B;
- c) old Annex B, giving guidance on the use of precision results, has been deleted.

Introduction

Sulfur appears in three allotropic forms:

- a) rhombic;
- b) monoclinic;
- c) amorphous (insoluble in CS₂).

In rubber compounding practice, forms a) and c) are used. Both types may be used in their natural form or may be coated. The coating can be oil, or MgCO₃, or silica, or some other material that aids dispersion in the rubber compound. Sulfur is available in varying degrees of fineness.

Amorphous sulfur (insoluble sulfur) is usually produced in industry through the rapid cooling of molten sulfur and can also contain rhombic sulfur (soluble sulfur), as insoluble sulfur tends to revert back to the rhombic state on storage. Insoluble sulfur is important in the rubber industry as a vulcanizing agent since, when it is used, it can prevent rubber compounds from blooming during storage or during a production process without any adverse impact on the vulcanization process. Grades containing various amounts of insoluble sulfur are available to control blooming today (see Annex A).

Rubber compounding ingredients — Sulfur — Methods of test

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 methods of test for determining the main physical and chemical properties of sulfur used for compounding dry rubber.

NOTE Typical levels for the relevant properties of sulfur for use as a rubber compounding ingredient are contained in Annex A, for information only.

2 Normative references

The following referenced documents are indispensable for the application 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 565, *Test sieves — Metal wire cloth, perforated metal plate and electroformed sheet — Nominal sizes of openings*

ISO 2590, *General method for the determination of arsenic — Silver diethyldithiocarbamate photometric method*

ISO 3704, *Sulphur for industrial use — Determination of acidity — Titrimetric method*

ISO 3705, *Sulphur for industrial use — Determination of arsenic content — Silver diethyldithiocarbamate photometric method*

ISO 4793, *Laboratory sintered (fritted) filters — Porosity grading, classification and designation*

ISO 15528, *Paints, varnishes and raw materials for paints and varnishes — Sampling*

3 Physical and chemical properties

After sampling with a stainless-steel sampling tool in accordance with ISO 15528, the physical and chemical properties of sulfur used as a rubber compounding ingredient shall be determined by the following test methods.

Property	Test method
Total sulfur [% (by mass)]	Clause 4
Fineness (sieve residue) [% (by mass)]	Clause 5
Oil content [% (by mass)]	Clause 6
Insoluble sulfur [% (by mass)]	Clause 7
Thermal reversion (%)	Clause 8
Loss in mass at 80 °C [% (by mass)]	Clause 9
Ash [% (by mass)]	Clause 10
Acidity [as H ₂ SO ₄ , % (by mass)]	Clause 11 (ISO 3704)
Arsenic (mg/kg)	Clause 12 (ISO 3705 and ISO 2590)

NOTE Determination of the arsenic content is not necessary when a sulfur sample is produced through hydro-desulfurization from natural sources such as oil or natural gas.

4 Determination of total sulfur content

4.1 Principle

The sulfur in a test portion is first converted to thiosulfate, to which an excess volume of iodine solution is added, then determined by back titration with a sodium thiosulfate solution.

4.2 Reagents

During the analysis, use only reagents of recognized analytical grade and only distilled water or water of equivalent purity.

4.2.1 Sodium sulfite solution, 50 g/dm³. Dissolve 50 g of anhydrous sodium sulfite in 1 dm³ of water.

4.2.2 Standard volumetric iodine solution, $c(I_2) = 0,05 \text{ mol/dm}^3$, freshly prepared and standardized.

4.2.3 Standard volumetric sodium thiosulfate solution, 0,1 mol/dm³ solution, freshly prepared and standardized.

4.2.4 Formaldehyde solution, 37 % (by mass).

4.2.5 Glacial acetic acid.

4.2.6 Wetting-out solution, consisting of a 20 g/dm³ solution of an alkylaryl sulfonate (e.g. sodium dodecylbenzene sulfonate) in water.

4.2.7 Soluble-starch solution, freshly prepared.

4.3 Procedure

4.3.1 Weigh, to the nearest 0,1 mg, 0,2 g of the sulfur sample (m_0) on a tared watch glass. Using a Pasteur pipette, add approximately 1 cm³ of wetting-out solution (4.2.6). Transfer the mixture to a 200 cm³ flask via a small glass funnel using a stream of the sodium sulfite solution (4.2.1) from a 50 cm³ measuring cylinder. Add a few antibumping granules, fix a reflux condenser in position and place the whole on an electrically heated oil-bath.

Adjust the oil-bath temperature to 130 °C. Stir the contents of the flask for 1 h under reflux, and complete the reaction. If undissolved sulfur remains visible in the flask, continue the heating process as long as necessary. Wash the walls of the condenser with water and collect the washings in the flask. Cool the contents of the flask to 25 °C. Transfer the contents of the flask to a 100 cm³ volumetric flask. Make up to the mark with water.

NOTE Reaction of 20 % oil-treated sulfur takes about 3 h to 4 h.

4.3.2 Place exactly 30 cm³ of iodine solution (4.2.2) into a 200 cm³ conical flask from a burette, and add 10 cm³ of acetic acid (4.2.5) to it using a pipette.

4.3.3 Place 10 cm³ of test solution from the 100 cm³ volumetric flask in 4.3.1, using a pipette, into a 100 cm³ beaker. Add 2 cm³ of formaldehyde solution (4.2.4) and stir using a magnetic stirrer for 2 min. After a 5-min rest, add the solution to the conical flask in 4.3.2. Wash the walls of the beaker and add the washings to the conical flask. While swirling the conical flask, add 1 cm³ of the starch solution (4.2.7) and titrate with the sodium thiosulfate solution (4.2.3) until the dark-blue colour vanishes. V_1 is the volume, in cubic centimetres, of the sodium thiosulfate solution used for this titration.

4.3.4 Carry out a blank determination omitting the test portion. V_0 is the volume, in cubic centimetres, of the sodium thiosulfate solution used for this titration.

4.4 Expression of results

Calculate the total sulfur content (TS_{ts}), expressed as a percentage by mass, from the following formula:

$$\text{TS}_{\text{ts}} = \frac{(V_0 - V_1)}{m_0} \times c \times 10 \times 3,2$$

where

V_0 is the volume, in cubic centimetres, of sodium thiosulfate solution (4.2.3) used to titrate the blank (see 4.3.4);

V_1 is the volume, in cubic centimetres, of sodium thiosulfate solution (4.2.3) used to titrate the test portion (see 4.3.3);

c is the exact concentration, in moles of Na₂S₂O₃ per cubic decimetre, of the sodium thiosulfate solution (4.2.3);

m_0 is the mass, in grams, of the test portion (see 4.3.1).

4.5 Precision

See B.3.2.

5 Determination of sieve residue

5.1 Principle

A test portion is passed through sieves of specified mesh openings, using water to assist in the sieving process, and the residue retained is dried and weighed. Two methods are specified:

- method A, which uses a sieve in a metal funnel and a high-pressure water jet (see Figure 1);
- method B, in which water is poured over the sulfur on the sieve while the sieve is shaken manually.