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Rubber compounding ingredients — Sulfur — Methods of test

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

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Foreword

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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 3, *Raw materials (including latex) for use in the rubber industry*.

This fifth edition cancels and replaces the fourth edition (ISO 8332:2011), which has been technically revised. The main changes are the following:

— Change in the Scope to indicate that some of the properties can be determined by more than one test method.

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

Sulfur appears in three allotropic forms:

a) rhombic;

b) monoclinic;

c) amorphous (insoluble in CS_2).

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 $MgCO_3$, 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 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.

1 Scope

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

Several of these properties can be determined by more than one test method and the user can choose the most appropriate method following the description of the test method principles provided under the corresponding clauses.

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 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 565, *Test sieves — Metal wire cloth, perforated metal plate and electroformed sheet — Nominal sizes of openings*

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 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 https://www.electropedia.org/

4 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 5
Fineness (sieve residue) [% (by mass)]	Clause 6
Oil content [% (by mass)]	Clause 7
Insoluble sulfur [% (by mass)]	Clause 8
Thermal reversion (%)	Clause 9
Loss in mass at 80 °C [% (by mass)]	Clause 10
Ash [% (by mass)]	Clause 11
Acidity [as H_2SO_4, % (by mass)]	Clause 12
Arsenic (mg/kg)	Clause 13

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.

5 Determination of total sulfur content

5.1 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.

5.2 Reagents

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

5.2.1 Sodium sulfite solution, 50 g/dm^3. Dissolve 50 g of anhydrous sodium sulfite in 1 dm^3 of water.

5.2.2 Standard volumetric iodine solution, $c(I_2)$ = 0,05 mol/dm^3, freshly prepared and standardized.

5.2.3 Standard volumetric sodium thiosulfate solution, 0,1 mol/dm^3 solution, freshly prepared and standardized.

5.2.4 Formaldehyde solution, 37 % (by mass).

5.2.5 Glacial acetic acid.

5.2.6 Wetting-out solution, consisting of a 20 g/dm^3 solution of an alkylaryl sulfonate (e.g. sodium dodecylbenzene sulfonate) in water.

5.2.7 Soluble-starch solution, freshly prepared.

5.3 Procedure

5.3.1 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 cm^3 of wetting-out solution (5.2.6). Transfer the mixture to a 200 cm^3 flask via a small glass funnel using a stream of the sodium sulfite solution (5.2.1) from a 50 cm^3

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 cm^3 volumetric flask. Make up to the mark with water.

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

5.3.2 Place exactly 30 cm^3 of iodine solution (5.2.2) into a 200 cm^3 conical flask from a burette, and add 10 cm^3 of acetic acid (5.2.5) to it using a pipette.

5.3.3 Place 10 cm^3 of test solution from the 100 cm^3 volumetric flask in 5.3.1, using a pipette, into a 100 cm^3 beaker. Add 2 cm^3 of formaldehyde solution (5.2.4) and stir using a magnetic stirrer for 2 min. After a 5-min rest, add the solution to the conical flask in 5.3.2. Wash the walls of the beaker and add the washings to the conical flask. While swirling the conical flask, add 1 cm^3 of the starch solution (5.2.7) and titrate with the sodium thiosulfate solution (5.2.3) until the dark-blue colour vanishes. V_1 is the volume, in cubic centimetres, of the sodium thiosulfate solution used for this titration.

5.3.4 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.

5.4 Expression of results

Calculate the total sulfur content (TS_{ts}), expressed as a percentage by mass, from Formula (1):

$$TS_{ts} = \frac{(V_0 - V_1)}{m_0} \times c \times 10 \times 3{,}2 \quad (1)$$

where

V_0 is the volume, in cubic centimetres, of sodium thiosulfate solution (5.2.3) used to titrate the blank (see 5.3.4);

V_1 is the volume, in cubic centimetres, of sodium thiosulfate solution (5.2.3) used to titrate the test portion (see 5.3.3);

c is the exact concentration, in moles of $Na_2S_2O_3$ per cubic decimetre, of the sodium thiosulfate solution (5.2.3);

m_0 is the mass, in grams, of the test portion (see 5.3.1).

5.5 Precision

See B.3.2.

6 Determination of sieve residue

6.1 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:

a) method A, which uses a sieve in a metal funnel and a high-pressure water jet (see Figure 1);

b) method B, in which water is poured over the sulfur on the sieve while the sieve is shaken manually.

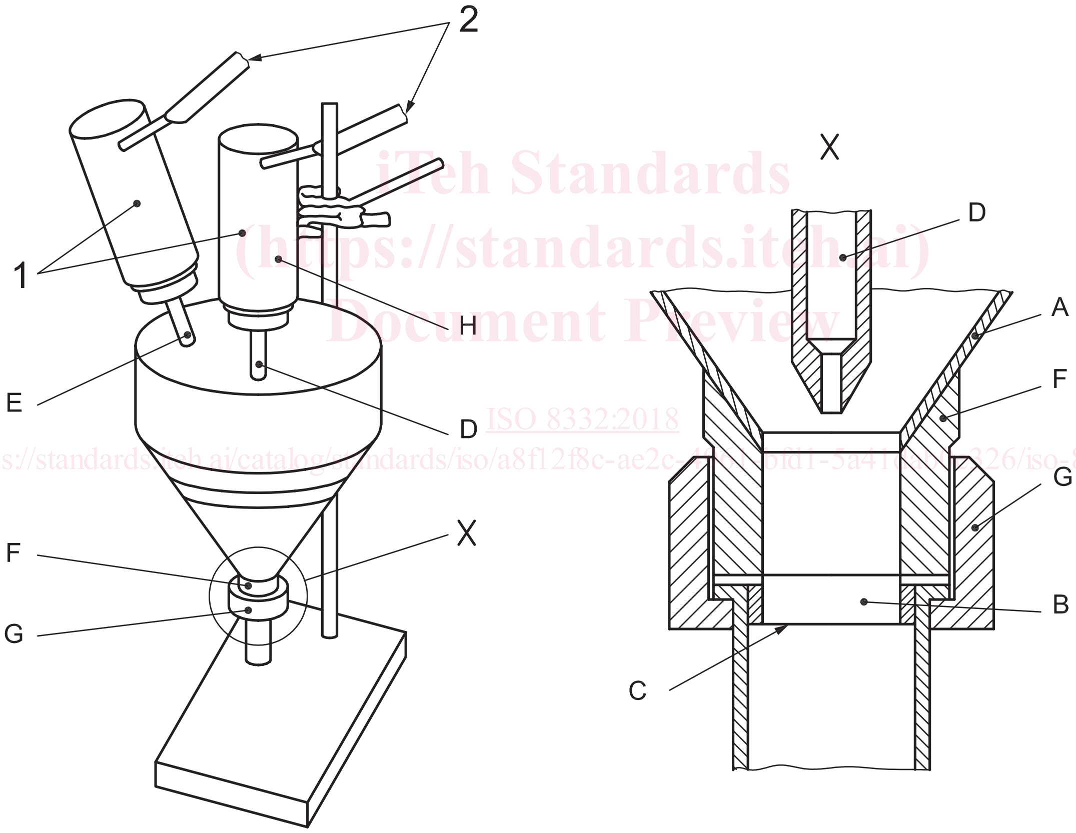
6.2 Method A

6.2.1 Apparatus

Use standard laboratory equipment together with the following.

6.2.1.1 Metal funnel (A), about 200 mm in diameter, terminating at the foot in a short cylindrical outlet (see Figure 1) in which is inserted a shallow removable cup (B), on the bottom of which a wire-cloth sieve (C) (6.2.1.2) is soldered; this is 25 mm in diameter. Water under pressure is supplied by a tube (D) fitted with a nozzle designed to discharge a spreading jet through the sieve; the distance of the orifice from the sieve can be adjusted. The recommended minimum water pressure is 200 kPa. The tube is provided with a filter (H) to remove any solid particles from the water; this filter shall be made from wire cloth at least as fine as the sieve (C), and shall be sufficiently large to prevent undue loss of pressure.

A similar arrangement is provided for another tube (E) used to supply a gentle stream of water for wetting the powder and keeping the volume of the liquid in the funnel constant during the test.



Key

1	filters
2	water inlets
A to E and H	A to E and H are as described in 6.2.1.1.
F and G	F and G are as described in 6.2.2.

Figure 1 — Apparatus for determination of sieve residue (wet procedure: method A)

6.2.1.2 Two sieves, of woven metal-wire cloth, in accordance with ISO 565, having openings of nominal width 180 µm and 125 µm, respectively.

6.2.1.3 Flat brush, of camel hair or equivalent, approximately 7 mm wide.

6.2.1.4 Desiccator.

6.2.2 Procedure

Mount a sieve cup (B) with a sieve (C) with the required openings (i.e. one of the two sieves specified in 6.2.1.2), or with openings as agreed between the interested parties, in the apparatus. Allow water to flow through it for 3 min from tubes D and E. Examine the sieve for particles; if none are observed, the apparatus is ready for use. Dry the sieve cup at 80 °C ± 2 °C for 30 min. Allow it to cool in a desiccator (6.2.1.4) and then weigh it to the nearest 0,1 mg. Repeat this operation until the change in mass is reduced to not more than 1 mg, record the mass of the sieve cup and mount it in the apparatus, holding it in place by the union nut (G). Weigh out, to the nearest 0,1 g, about 25 g (m_1) of the sample. Make into a slurry with 300 cm^3 of a 20 g/dm^3 solution of alkylaryl sulfonate in water in a beaker, and transfer quantitatively to the funnel (A).

About half-fill the funnel with water from tube E. Slowly turn the high-pressure jet (D) on full. Adjust the position of the nozzle under the liquid, in relation to the sieve, until there is little or no disturbance on the surface and the level is, if anything, sinking. Now use the supply from tube E to maintain the level constant and wash down any solid matter adhering to the sides.

NOTE When the best operating conditions have been so attained, the discharge pipe (F) below the sieve is completely filled with water; increasing the length of this column of liquid by adding a length of rubber tubing improves the efficiency of the apparatus.

Take a sample of the issuing water from time to time during the test and, when this is free from suspended matter, reduce the rate of flow from the jet. Wash down any residual material adhering to the sides of the funnel into the lower portion of the apparatus, and then adjust the jet to a rate that keeps the cylindrical portion full of violently agitated water. When the residual matter has been thoroughly washed in this way until the effluent runs clear (usually within 3 min to 4 min), turn off the jet and wash the grit down into the cup. Inspect the remaining sulfur and break up any loose agglomerates by brushing lightly with the camel hair brush (6.2.1.3), subsequently washing them through the sieve. Then wash the remaining grit into the cup.

Unscrew the milled union nut (G), remove the discharge pipe and sieve cup, and wash the contents of the latter with distilled water. Dry the sieve cup and contents at 80 °C ± 2 °C for 30 min. Allow it to cool in a desiccator (6.2.1.4) and then weigh it to the nearest 0,1 mg. Repeat this operation until the change in mass is reduced to not more than 1 mg and record the mass of the sieve cup and contents. This mass, less the initial mass of the cup, equals the mass of the sieve residue (m_2).

Repeat the procedure with the other sieve specified in 6.2.1.2, or with another sieve with openings as agreed between the interested parties.

6.2.3 Expression of results

Calculate the sieve residue (W_{rA}), expressed as a percentage by mass, using Formula (2):

$$W_{\mathrm{rA}} = \frac{m_2}{m_1} \times 100 \qquad (2)$$

where

m_1 is the mass, in grams, of the test portion;

m_2 is the mass, in grams, of the residue on the sieve.