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**Rubber compounding ingredients —  
Sulfur — Methods of test**

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

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Published in Switzerland

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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.

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.

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- 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 CS<sub>2</sub>).

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<sub>3</sub>, 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).

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# 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* [http://www.iso.org/iso/iso\\_catalogue/catalogue\\_tc/catalogue\\_detail.htm?csnumber=37075](http://www.iso.org/iso/iso_catalogue/catalogue_tc/catalogue_detail.htm?csnumber=37075)  
[http://www.iso.org/iso/iso\\_catalogue/catalogue\\_tc/catalogue\\_detail.htm?csnumber=37075](http://www.iso.org/iso/iso_catalogue/catalogue_tc/catalogue_detail.htm?csnumber=37075)  
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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 <sub>2</sub> SO <sub>4</sub> , % (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/dm<sup>3</sup>. Dissolve 50 g of anhydrous sodium sulfite in 1 dm<sup>3</sup> 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/dm<sup>3</sup> 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/dm<sup>3</sup> 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 cm<sup>3</sup> of wetting-out solution (4.2.6). Transfer the mixture to a 200 cm<sup>3</sup> flask via a small glass funnel using a stream of the sodium sulfite solution (4.2.1) from a 50 cm<sup>3</sup> 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<sup>3</sup> 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 cm<sup>3</sup> of iodine solution (4.2.2) into a 200 cm<sup>3</sup> conical flask from a burette, and add 10 cm<sup>3</sup> of acetic acid (4.2.5) to it using a pipette.

**4.3.3** Place 10 cm<sup>3</sup> of test solution from the 100 cm<sup>3</sup> volumetric flask in 4.3.1, using a pipette, into a 100 cm<sup>3</sup> beaker. Add 2 cm<sup>3</sup> 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 cm<sup>3</sup> 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<sub>ts</sub>), expressed as a percentage by mass, from the following formula:

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

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 Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub> 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:

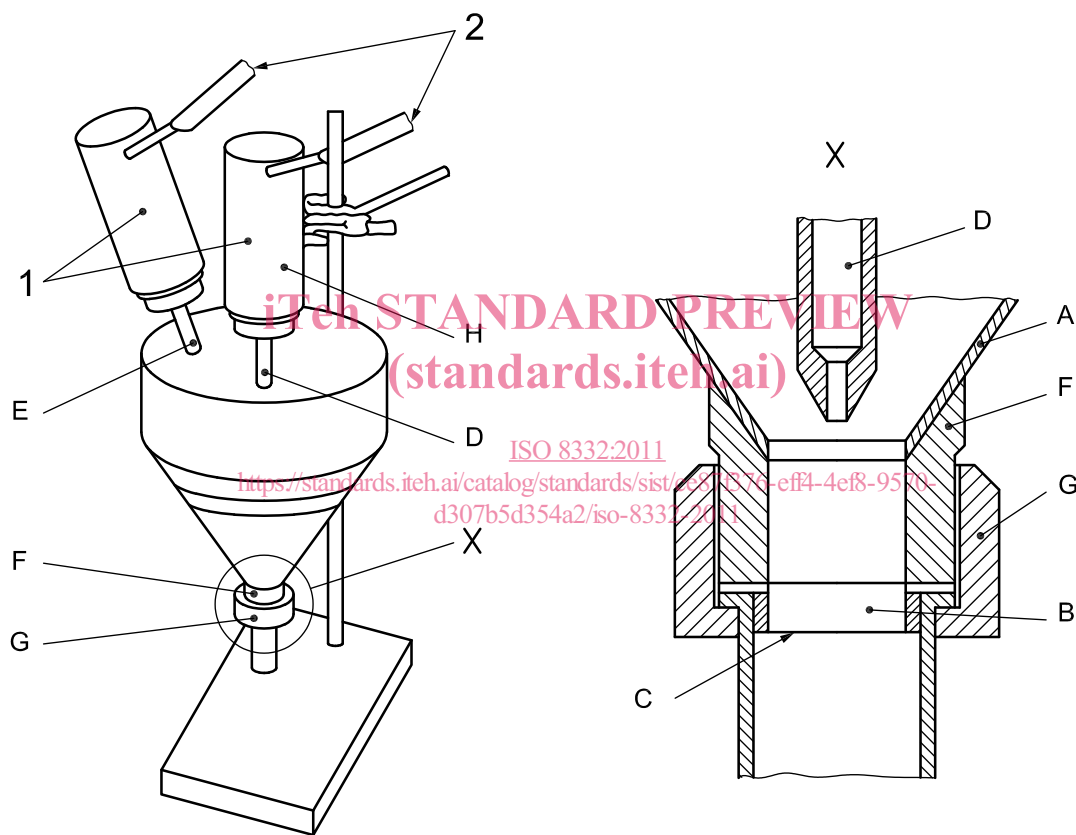
- method A, which uses a sieve in a metal funnel and a high-pressure water jet (see Figure 1);
- method B, in which water is poured over the sulfur on the sieve while the sieve is shaken manually.

5.2 Method A

5.2.1 Apparatus

5.2.1.1 **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) (5.2.1.2) 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 (2 bar). 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 are as described in 5.2.1.1.  
 F and G are described in 5.2.2.

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

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

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

5.2.1.4 **Desiccator**.

## 5.2.2 Procedure

Mount a sieve cup (B) with a sieve (C) with the required openings (i.e. one of the two sieves specified in 5.2.1.2), 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  $80\text{ °C} \pm 2\text{ °C}$  for 30 min. Allow it to cool in a desiccator (5.2.1.4) 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  $300\text{ cm}^3$  of a  $20\text{ g/dm}^3$  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 (5.2.1.3), 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  $80\text{ °C} \pm 2\text{ °C}$  for 30 min. Allow it to cool in a desiccator (5.2.1.4) 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 5.2.1.2, or with another sieve with openings as agreed between the interested parties.

## 5.2.3 Expression of results

Calculate the sieve residue ( $W_{rA}$ ), expressed as a percentage by mass, using the following formula:

$$W_{rA} = \frac{m_2}{m_1} \times 100$$

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.

## 5.3 Method B

### 5.3.1 Apparatus

**5.3.1.1 Two sieves**, of woven metal-wire cloth, in accordance with ISO 565, having openings of nominal width  $180\text{ }\mu\text{m}$  and  $125\text{ }\mu\text{m}$ , respectively.

**5.3.1.2 Flat brush**, of camel hair or equivalent, 10 mm to 25 mm wide.

**5.3.1.3 Desiccator**.

**5.3.2 Procedure**

Dry one of the sieves specified in 5.3.1.1, or a sieve with openings as agreed between the interested parties, at 80 °C ± 2 °C for 30 min. Allow it to cool in a desiccator (5.3.1.3) 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. From the previously de-agglomerated sample, weigh out, to the nearest 0,1 g, about 10 g ( $m_3$ ) when using a sieve with openings of less than 100 µm or about 30 g ( $m_3$ ) when using a sieve with openings of greater than 100 µm, and transfer to a beaker.

From the test portion, make, in a beaker, a suspension using 100 cm<sup>3</sup> or 300 cm<sup>3</sup> (depending on the size of the test portion) of a 20 g/dm<sup>3</sup> solution of alkylaryl sulfonate in water, pressing it lightly with a glass rod to collapse the lumps. After stirring it sufficiently, pour the suspension onto the screen of the sieve. Shake the sieve gently while pouring in water little by little to allow most of the test portion to pass through it. Then put the sieve into an evaporating dish, fill the dish with water up to a level 10 mm to 15 mm above the screen, and sweep the surface of the screen with a brush (5.3.1.2).

Take the sieve out of the dish, drain out the water from the openings in the sieve, and renew the water in the dish. Repeat this operation until no more of the test portion can be seen in the water in the dish. Wash any solid particles adhering to the brush onto the screen with water, then dry the sieve and contents at 80 °C ± 2 °C for 30 min. Allow it to cool in a desiccator (5.3.1.3) 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 and contents. The final mass minus the initial mass of the sieve equals the mass of the sieve residue ( $m_4$ ).

Repeat the procedure with the other sieve specified in 5.3.1.1, or with another sieve with openings as agreed between the interested parties.

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**5.3.3 Expression of results**

Calculate the sieve residue ( $W_{rB}$ ), expressed as a percentage by mass, using the following formula:

$$W_{rB} = \frac{m_4}{m_3} \times 100$$

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where

- $m_3$  is the mass, in grams, of the test portion;
- $m_4$  is the mass, in grams, of the residue on the sieve.

**5.3.4 Precision**

See B.3.3.

**6 Determination of oil content of oil-treated sulfur**

**6.1 Principle**

Oil is extracted from the sample by using a solvent, and the solvent is then evaporated off and the mass of residual oil determined. The residual sulfur is weighed and the amount of oil is calculated.

Two procedures are specified:

- a) a procedure using hexane for rhombic sulfur;
- b) a procedure using sulfur-saturated hexane for insoluble sulfur.