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

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

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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

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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 third edition cancels and replaces the second edition (ISO 8332:1997), in which the following changes have been made:

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- a) the methods for the determination of total sulfur content and sieve residue have been revised;
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 - b) method B for the determination of sieve residue has been added;
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 - c) the methods for the determination of loss in mass at 80 °C have been added;
 - d) the method for the determination of ash at 850 °C to 900 °C has been added.

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 they 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. Insoluble sulfur can also contain an amount of rhombic sulfur, as insoluble sulfur tends to revert back to the rhombic state on storage.

These considerations are each the subject of the methods of test specified in this International Standard.

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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 565:1990, *Test sieves — Metal wire cloth, perforated metal plate and electroformed sheet — Nominal sizes of openings*

ISO 2590:1973, *General method for the determination of arsenic — Silver diethyldithiocarbamate photometric method*

ISO 3704:1976, *Sulphur for industrial use — Determination of acidity — Titrimetric method*

ISO 3705:1976, *Sulphur for industrial use — Determination of arsenic content — Silver diethyldithiocarbamate photometric method*

ISO 4793:1980, *Laboratory sintered (fritted) filters — Porosity grading, classification and designation*

ISO/TR 9272:2005, *Rubber and rubber products — Determination of precision for test method standards*

ISO 15528:2000, *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	} of this International Standard
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)]	ISO 3704	
Arsenic (mg/kg)	ISO 3705 and ISO 2590	

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 volume).

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 % of the oil treated 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 (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 (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 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 Na₂S₂O₃ per cubic decimetre, of sodium thiosulfate solution (4.2.3);

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

4.5 Precision result

For the evaluation of precision data, three laboratories participated in the interlaboratory test programme. Using two different sulfur samples (soluble sulfur, insoluble sulfur with 20 % oil treated), the total sulfur content was measured. The number of within-laboratory replicates was two, and the time span for repeatability was 7 days. The precision data were calculated according to normal statistical procedures. The precision results for sulfur content are given in Table 1.

Table 1 — Precision data for sulfur content

Sample	Mean level %	Within lab			Between labs		
		s_r	r	(r)	s_R	R	(R)
Soluble sulfur	99,94	0,14	0,38	0,38	0,27	0,77	0,77
Insoluble sulfur, 20 % oil treated	79,92	0,17	0,49	0,61	0,35	1,00	1,25
<p>($p = 3, q = 2, n = 2$) s_r = Within-lab standard deviation s_R = Between-labs standard deviation r = Repeatability, in measurement units (r) = Repeatability, in percent ^a R = Reproducibility, in measurement units (R) = Reproducibility, in percent ^a</p>							
^a These values represent percent relative, i.e. percent of a percent.							

5 Determination of sieve residue

5.1 Principle

A test portion is passed through sieves of specified mesh opening and the residue retained is weighed. Two procedures are specified:

- a) a wet procedure for all oil-treated grades of sulfur;
- b) a dry procedure for all other (i.e. non-oil-treated) grades.

In the case of extremely fine-grade sulfurs, the wet procedure shall be used.

5.2 Wet procedure

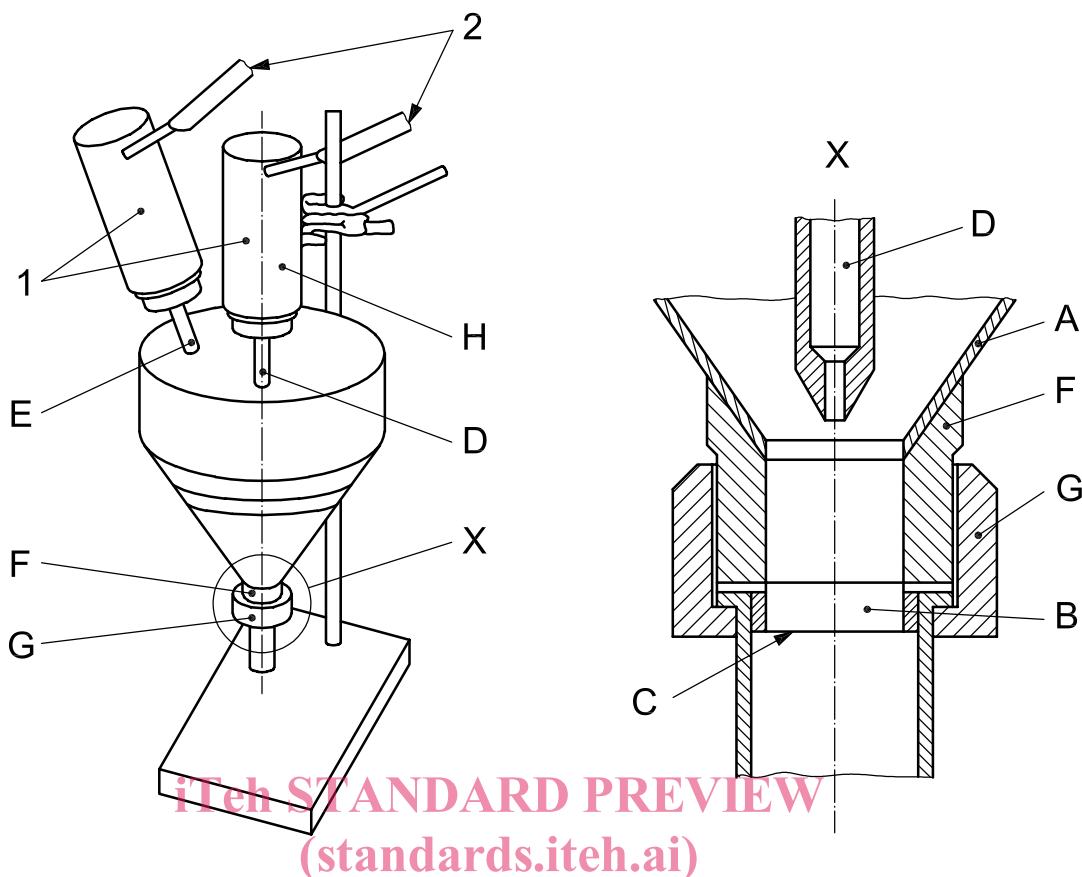
5.2.1 Method A

5.2.1.1 Apparatus

5.2.1.1.1 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.

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**Key**

- 1 filters
2 water inlets

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A to E and H are as described in 5.2.1.1.1.

F and G are described in 5.2.1.2.

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

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

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

5.2.1.2 Determination

Mount a sieve cup (B) with a sieve (C) of the required mesh, 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 to constant mass at $80\text{ }^{\circ}\text{C} \pm 2\text{ }^{\circ}\text{C}$ for 30 min. Then weigh it, to the nearest 0,1 mg, and mount it in the apparatus, holding it in place by union nut G. Weigh out about 25 g (m_1) of the sample accurately to 0,1 mg. 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 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 (5.2.1.1.3), subsequently washing them through the filter. Then wash the remaining grit into the cup.

Unscrew the milled union nut (G), remove the discharge pipe and cup, and wash the contents of the latter with distilled water. Dry the cup and contents at $80\text{ °C} \pm 2\text{ °C}$ for 30 min and weigh to the nearest 0,1 mg. This mass, less the initial mass of the cup, equals the mass of the sieve residue (m_2).

5.2.1.3 Expression of results (wet procedure: method A)

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

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5.2.2 Method B

5.2.2.1 Apparatus

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

5.2.2.1.2 Flat brush, of camel hair or equivalent, approximately 25 mm wide.

5.2.2.1.3 Desiccator.

5.2.2.2 Determination

Dry a sieve with the specified openings in an air oven at the temperature of $80\text{ °C} \pm 2\text{ °C}$, allow it to cool in a desiccator (5.2.2.1.3), and weigh it. Repeat this operation and record the mass of the sieve when the change in mass is reduced to not more than 1 mg through the process of heating for 15 min and cooling to room temperature. From the sample de-agglomerated beforehand, weigh, accurately to 0,1 mg, about 10 g (m_3) in the case of the sieve opening under 100 μm , or about 30 g (m_3) if over 100 μm , and transfer to a 100 ml beaker.

Make the sample wet with a little of a 20 g/dm³ solution of alkylaryl sulfonate in water, then press it slightly with a glass rod to collapse the lumps. After adding about 100 cm³ of water and 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 sample to pass through it. Then put the sieve into an evaporating dish, fill the dish with water up to 10 mm to 15 mm above the screen, and sweep the surface of the screen with a brush (5.2.2.1.2).

Take out the sieve from the dish, drain out the water from the openings of the sieve, and renew the water in the dish. Repeat this operation until the sample is not perceived in the water of the dish. Wash down any solid particles adhering to the brush onto the screen with water then dry the sieve in an air oven at the temperature of $80\text{ °C} \pm 2\text{ °C}$ for 30 min. Keep it in a desiccator (5.2.2.1.3) and weigh it after cooling. Repeat this operation until the change in mass is reduced to not more than 1 mg. The final mass minus the initial mass of the sieve equals the mass of the sieve residue (m_4).