

INTERNATIONAL STANDARD

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Rubber — Determination of total sulfur content —

Part 1: Oxygen combustion flask method

Caoutchouc — Dosage du soufre total —

Partie 1: Méthode par combustion dans une fiole d'oxygène



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Foreword

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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.

International Standard ISO 6528-1 was prepared by Technical Committee ISO/TC 45, *Rubber and rubber products*.

This second edition cancels and replaces the first edition (ISO 6528-1:1984), of which it constitutes a minor revision.

ISO 6528 consists of the following parts, under the general title *Rubber — Determination of total sulfur content*:

- *Part 1: Oxygen combustion flask method*
- *Part 2: Sodium peroxide fusion method*
- *Part 3: Furnace combustion method*

Annex A of this part of ISO 6528 is for information only.

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Rubber — Determination of total sulfur content —

Part 1:

Oxygen combustion flask method

1 Scope

This part of ISO 6528 specifies an oxygen combustion flask method for the determination of the total sulfur content of rubber.

The method is applicable for the determination of all the sulfur present in a rubber or rubber product, except that contained in barium sulfate.

It is applicable to NR, CR, SBR, BR, IR, IIR, EPDM, NBR and ebonite. (The meanings of these designations are given in ISO 1629:1987, *Rubber and latices — Nomenclature*.)

This method gives unreliable (usually low) results in the presence of lead, antimony, zinc, barium and calcium compounds. If calcium carbonate is the only calcium compound present, good results can be obtained by modifying the absorbing solution.

2 Principle

A test portion is ignited in an atmosphere of oxygen in an oxygen combustion flask containing hydrogen peroxide, the carbon and hydrogen of the organic matter being oxidized and the sulfur being converted to sulfuric acid. If necessary, the resulting solution is passed through a cation-exchange column to remove interfering metals. The eluate is titrated with barium perchlorate using thorin indicator.

WARNING — All recognized health and safety precautions shall be observed when carrying out this method of analysis.

3 Reagents and materials

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

NOTE 1 Two methods are described for the preparation of the indicator (3.8). The analyst may choose that best suited to his experience, equipment and needs.

3.1 Absorbing solution.

3.1.1 Hydrogen peroxide, 2 % (m/m), absorbing solution.

Dilute 1 volume of 30 % (m/m) hydrogen peroxide solution (H_2O_2) to 15 volumes with water.

WARNING — 30 % (m/m) hydrogen peroxide solution is very corrosive to the skin. Wear rubber or plastic gloves and goggles when handling it.

3.1.2 Hydrogen peroxide/hydrochloric acid.

Dilute 4 volumes of 30 % (m/m) hydrogen peroxide solution (H_2O_2) to 60 volumes with water and add 5 volumes of concentrated hydrochloric acid, ($\rho_{20} = 1,18 \text{ Mg/m}^3$), and mix thoroughly.

3.2 Hydrochloric acid, 0,5 mol/dm³ solution.

3.3 2-Propanol.

3.4 Barium perchlorate, 0,01 mol/dm³ solution.**3.4.1 Preparation**

Dissolve 3,363 g of barium perchlorate [Ba(ClO₄)₂] in 200 cm³ of water. Adjust the pH to about 3,5 with hydrochloric acid solution (3.2). Make up to 1 dm³ with 2-propanol (3.3).

3.4.2 Standardization

Standardize the barium perchlorate solution as follows.

Weigh, to the nearest 0,1 mg, 0,10 g of anhydrous sodium sulfate (Na₂SO₄) and dissolve the sodium sulfate in 10 cm³ of water in a 100 cm³ volumetric flask. Dilute to the mark with water.

Pipette exactly 10 cm³ of this solution into a small flask or beaker, add 40 cm³ of 2-propanol (3.3) to make the solution 80 % (V/V) alcoholic and titrate as specified in 7.3.3.

The concentration c of the barium perchlorate solution, expressed in moles per cubic decimetre, is given by the equation

$$c = \frac{0,1 \times m_1 \times 1\,000}{142,06 \times V_s}$$

where

m_1 is the mass, in grams, of sodium sulfate used;

V_s is the volume, in cubic centimetres, of the barium perchlorate solution required for the titration.

3.4.3 Storage

Barium perchlorate solutions deteriorate on storage and should be standardized sufficiently often so as to be able to detect changes in concentration of 0,000 5 mol/dm³.

3.5 Cation exchange resin, strongly acidic, containing nuclear sulfonic acid active groups.

Before filling the column (4.6), allow the resin to stand in a beaker of distilled water to absorb water and become swollen.

Check that the following requirements are fulfilled before use and after regeneration (because of the large capacity of the resin to remove interfering cations, it may be used five to ten times before regeneration becomes necessary):

- a) 10 cm³ of 0,02 mol/dm³ sulfuric acid solution shall be completely eluted with 15 cm³ of water (test the last portion of the eluate for the absence of sulfate using thorin solution);

- b) the ion exchange column shall be capable of removing 0,1 g of zinc, which is the most commonly interfering cation in rubber analyses.

Regenerate the resin by passing 10 cm³ of 2 mol/dm³ hydrochloric acid (3.6) through the resin bed, at the rate of 2 to 3 drops per second, then wash the resin bed with 20 cm³ of water at a faster rate. Test the last few drops of the washings for the absence of sulfate using thorin solution.

3.6 Hydrochloric acid, 2 mol/dm³ solution.**3.7 Oxygen**, compressed, from a cylinder with an outlet suitable for introduction into the combustion flask (4.1).**3.8 Indicator.**

Two types of indicator are described, both of which are suitable for use with the procedure described in this part of ISO 6528, i.e. thorin alone (3.8.1) and thorin plus methylene blue (3.8.2). The thorin used shall be a uniform red powder and shall give a clear, orange, aqueous solution.

3.8.1 Thorin {4-[(2-arsonophenyl)azo]-3-hydroxy-2,7-naphthalenedisulfonic acid, disodium salt}, 0,2 % (m/m) solution.

Dissolve 0,2 g of thorin in 100 cm³ of water.

3.8.2 Thorin/methylene blue mixed indicator.**3.8.2.1 Thorin**, 0,5 % (m/m) solution.

Dissolve 0,5 g of thorin in 100 cm³ of water.

3.8.2.2 Methylene blue [3,7-bis(dimethylamino)-5-phenothiazinium chloride], CI 52015, 0,012 % (m/m) solution.

Dissolve 0,012 g of methylene blue in 100 cm³ of water.

3.9 Potassium nitrate (KNO₃), 0,4 % (m/m) solution.

Dissolve 0,4 g of potassium nitrate in 100 cm³ water.

3.10 Black or white paper, for wrapping the test portions.

NOTE 2 The papers are usually supplied in the form shown in figure 1. For use with smaller platinum baskets, they may be cut as shown in figure 2.

Although most commercial papers are readily combustible, the papers may be cut from regular filter papers and impregnated with the potassium nitrate solution (3.9) to aid combustion of the rubber. Drain the excess solution and dry the papers at