

Chemical tests for raw and vulcanized rubber —

**Part 23: Methods for determination of
total sulfur content —**

**Section 23.1 Oxygen combustion flask
method**

Committees responsible for this British Standard

The preparation of this British Standard was entrusted by the Plastics and Rubber Standards Policy Committee (PRM/-) to Technical Committee PRM/23, upon which the following bodies were represented:

- Institute of Water and Environmental Management
- Laboratory of the Government Chemist
- Ministry of Defence
- Royal Society of Chemistry
- Rubber and Plastics Research Association

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National foreword

This Section of BS 7164 has been prepared under the direction of the Plastics and Rubber Standards Policy Committee and is identical with ISO 6528-1:1992 *Rubber — Determination of total sulfur content — Part 1: Oxygen combustion flask method*, published by the International Organization for Standardization (ISO).

BS 7164 rationalizes all the methods that deal with chemical analysis of raw, compounded and vulcanized rubber, some of which have previously been published in BS 903, BS 1673 and BS 5923. Relevant Parts of those standards will be withdrawn or deleted by amendment, as appropriate, when superseded by Parts and Sections of BS 7164.

This Section of BS 7164 supersedes Part B6, Section 3 of BS 903-B6:1958 to BS 903-B10:1958, which is deleted by amendment.

A British Standard does not purport to include all the necessary provisions of a contract. Users of British Standards are responsible for their correct application.

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Summary of pages

This document comprises a front cover, an inside front cover, pages i and ii, pages 1 to 8 and a back cover.

This standard has been updated (see copyright date) and may have had amendments incorporated. This will be indicated in the amendment table on the inside front cover.

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 thiorin 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 [$\text{Ba}(\text{ClO}_4)_2$] 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 (Na_2SO_4) 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):

- 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 thiorin solution);