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МЕЖДУНАРОДНАЯ ОРГАНИЗАЦИЯ ПО СТАНДАРТИЗАЦИИ

Rubber — Determination of total sulfur content —

Part 3 : Furnace combustion method

Caoutchouc — Dosage du soufre total —

Partie 3 : Méthode par combustion au four

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.

Draft International Standards adopted by the technical committees are circulated to the member bodies for approval before their acceptance as International Standards by the ISO Council. They are approved in accordance with ISO procedures requiring at least 75 % approval by the member bodies voting.

International Standard ISO 6528-3 was prepared by Technical Committee ISO/TC 45, *Rubber and rubber products*.

Users should note that all International Standards undergo revision from time to time and that any reference made herein to any other International Standard implies its latest edition, unless otherwise stated.

Rubber — Determination of total sulfur content —

Part 3 :

Furnace combustion method

0 Introduction

Parts 1 and 2 of ISO 6528 give alternative methods for the determination of the total sulfur content of rubber.

1 Scope and field of application

1.1 This part of ISO 6528 specifies a method for the determination, by combustion in a furnace, of the total sulfur content of raw rubbers, unvulcanized compounded rubbers and vulcanizates, including sulfur present as barium sulfate, calcium sulfate and lithopone.

1.2 It is applicable to chlorinated rubbers, nitrile rubbers and hydrocarbon rubbers (including natural rubber). The determination is not affected by the presence of metal salts which form insoluble sulfates.

2 References

ISO 383, *Laboratory glassware — Interchangeable conical ground joints.*

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

ISO 6528, *Rubber — Determination of total sulfur content*

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

3 Principle

3.1 A test portion is burnt in a stream of oxygen in the presence of a catalyst, the oxides of sulfur are passed into hydrogen peroxide and the resulting sulfuric acid is determined by titration.

3.2 One of two titration methods is used, depending on the type of rubber combusted. An alkalimetric titration is used when no chlorine or nitrogen combustion products are present and a barium perchlorate titration is used when chlorine and/or nitrogen is present.

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

4 Reagents

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

4.1 Reagents for the combustion

4.1.1 Calcium chloride, anhydrous, particle size approximately 800 to 1 400 μm (14 to 22 mesh ASTM).

4.1.2 Catalyst.

Mix together 0,8 g of dry vanadium pentoxide and 0,2 g of zinc oxide for each determination. The vanadium pentoxide shall be previously dried by heating in a shallow dish at 140 to 160 °C for 16 h. It shall be cooled and stored in a desiccator.

4.1.3 Hydrogen peroxide, 3 % (V/V) solution.

Carefully mix 1 volume of 30 % hydrogen peroxide with 9 volumes of water.

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

4.1.4 Magnesium perchlorate, anhydrous, particle size 800 to 1 400 μm (14 to 22 mesh ASTM).

WARNING — Magnesium perchlorate can cause irritation of skin and mucous membranes.

4.1.5 Supply of oxygen, e.g. a cylinder with suitable regulator to give a pressure of not less than 35 kPa.

4.1.6 Carbon dioxide absorbent (sodium hydroxide absorbed on synthetic silicate), particle size 800 to 1 400 μm (14 to 22 mesh ASTM), preferably of the self-indicating type.

4.2 Reagents for titration

4.2.1 Perchloric acid, dilute solution.

Carefully mix 1 volume of 50 % perchloric acid (HClO_4) solution with 1 volume of water.

WARNING — Perchloric acid should be handled with extreme caution. Contact with dust or organic particles may, in many cases, cause ignition. Contact with organic materials must be avoided.

4.2.2 2-Propanol.

Use re-distilled 2-propanol if analytical grade reagent is not available.

4.2.3 Barium perchlorate, standard volumetric solution, $c[\text{Ba}(\text{ClO}_4)_2] = 0,01 \text{ mol/dm}^3$.

4.2.3.1 Preparation

Dissolve 3,363 g of barium perchlorate in 200 cm^3 of water. Adjust the pH to about 3,5 with the dilute perchloric acid (4.2.1). Make up to 1 dm^3 with the 2-propanol (4.2.2).

4.2.3.2 Standardization

Standardize the barium perchlorate by weighing, to $\pm 0,1 \text{ mg}$, approximately 0,10 g anhydrous sodium sulfate and dissolving it in 10 cm^3 of water in a 100 cm^3 one-mark volumetric flask. Make up to the mark with water. Pipette exactly 10 cm^3 of this solution into a small flask or beaker, add 40 cm^3 of the 2-propanol to make the solution 80 % alcohol by volume and titrate to the same end-point as will be used in the analysis of rubbers, using the same indicator system (see 7.2).

Determine the molar concentration, c_1 , of the barium perchlorate solution, expressed in moles of $\text{Ba}(\text{ClO}_4)_2$ per cubic decimetre, from the formula

$$\frac{0,1 \times m_s \times 1\,000}{142,06 \times V_s}$$

where

m_s is the mass, in grams, of sodium sulfate;

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

4.2.3.3 Storage

Preparations of barium perchlorate deteriorate on storage and should be standardized often enough to detect a change in concentration of 0,000 5 mol/dm^3 .

4.2.4 Sodium hydroxide solution, $c(\text{NaOH}) = 0,02 \text{ mol/dm}^3$, accurately standardized or commercial solution.

4.2.5 Indicator systems for barium perchlorate titrations.

Two systems are given, both suitable for use in this part of ISO 6528.

4.2.5.1 Thorin indicator.

Dissolve 0,2 g of Thorin¹⁾ in 100 cm^3 of water.

The reagent shall be a uniform red powder and produce a clear orange aqueous solution.

4.2.5.2 Thorin and methylene blue indicator.

Dissolve 0,5 g of Thorin in 100 cm^3 of water.

Dissolve 0,012 g of solid methylene blue in 100 cm^3 of water.

4.2.6 Indicator system for alkalimetric titration.

Dissolve 0,125 g of methyl red and 0,04 g of methylene blue in 100 cm^3 of methanol.

4.2.7 Hydrochloric acid, $c(\text{HCl}) = 2 \text{ mol/dm}^3$.

Dilute 1 volume of hydrochloric acid ($\rho = 1,18 \text{ Mg/m}^3$) with 5 volumes of water.

4.2.8 Cation exchange resin, strongly acidic, containing nuclear sulfonic acid active groups.²⁾

Before filling the column (5.6) 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 cm^3 of sulfuric acid solution, $c(\text{H}_2\text{SO}_4) = 0,02 \text{ mol/dm}^3$, shall be completely eluted with 15 cm^3 of water; test the last portion of the eluate for the absence of sulfate using Thorin solution (4.2.5.1);
- the ion exchange column shall be capable of removing 0,1 g of zinc, which is the most commonly interfering cation in rubber analyses.

1) Thorin is 2,2-hydroxy-3,6-disulfo-1-naphthyl-azobenzene-arsonic acid.

2) There are many types and particle size ranges of resin available. DOWEX 50W-8X spheres 75 to 150 μm (100 to 200 mesh) from J.T. Baker Company, 222 Red School Lane, Phillipsburg, New Jersey 088 65, USA, has been found satisfactory. This information is given for the convenience of users of this part of ISO 6528 and does not constitute an endorsement by ISO of the product named. Equivalent products may be used if they can be shown to lead to the same results.