International Standard 6528/2

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Rubber — Determination of total sulfur content — Part 2 : Sodium peroxide fusion method

Caoutchouc — Dosage du soufre total — Partie 2 : Méthode par fusion au peroxyde de sodium

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Foreword

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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.

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It has been approved by the member bodies of the following countries :

Belgium	Hungary	Poland
Canada	India	Romania
China	Indonesia	South Africa, Rep. of
Czechoslovakia	Korea, Rep. of	Spain
Denmark	Malaysia	Sri Lanka
Egypt, Arab Rep. of	Netherlands	Thailand
France	New Zealand	Turkey
Germany, F. R.	Nigeria	USSR

No member body expressed disapproval of the document.

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Rubber — Determination of total sulfur content — Part 2 : Sodium peroxide fusion method

1 Scope and field of application

This part of ISO 6528 specifies a sodium peroxide fusion method for the determination of the total sulfur content of rubber. This method is applicable for the determination of all the sulfur present in a rubber or rubber product, including that contained in barium sulfate which may be used as a filler.

It is applicable to NR, CR, SBR, BR, IR, EPDM, NBR and ebonite.[1)]

2 Principle

Oxidation of a test portion of rubber with sodium peroxide with a few drops of ethylene glycol added, in a suitable bomb reaction vessel. The sulfur is converted to sulfate ions. Gravimetric determination of the sulfate as barium sulfate.

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

Failure to heed the instructions given in this International Standard, or those of the manufacturer of the device used, may result in explosions.

3 Reagents[2)] and materials

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

3.1 Ethylene glycol.

3.2 Sodium peroxide, granulated.

3.3 Sodium carbonate, anhydrous.

3.4 Calcium carbonate, anhydrous.

3.5 Hydrochloric acid, solution.

Add 1 volume of hydrochloric acid (ϱ = 1,18 Mg/m^3) to 4 volumes of water.

3.6 Barium chloride, 50 g/dm^3 solution.

Dissolve 50 g of barium chloride dihydrate ($BaCl_2.2H_2O$) in water, add 0,5 cm^3 of hydrochloric acid (ϱ = 1,18 Mg/m^3) and dilute to 1 dm^3.

3.7 Acetic acid, 4 mol/dm^3 solution.

3.8 Cupferron (*N*-nitroso-*N*-phenylhydroxylamine ammonium salt), 60 g/dm^3 solution.

Dissolve 6 g of cupferron in 100 cm^3 of water.

3.9 Silica gel or **calcium chloride,** for use in the desiccator (4.9).

3.10 pH indicator paper.

3.11 Ashless filter paper, high density.

4 Apparatus

Usual laboratory equipment, and

4.1 Balance, capable of weighing to 0,000 1 g.

4.2 Combustion bomb (Parr or similar), gas- or electrically fired.

WARNING — The bomb must be operated in accordance with the safety instructions given by the manufacturer.

4.3 Nickel crucible with **lid,** to fit into the gas fired bomb (if used). Suitable dimensions are :

diameter 25 mm;
height 40 mm.

NOTE — A fusion cup is used with the electrically fired bomb.

1) See ISO 1629, *Rubbers and latices — Nomenclature.*

2) The term millilitre (ml) is commonly used as a special name for the cubic centimetre (cm^3), in accordance with a decision of the 12th Conférence Générale des Poids et Mesures. The term millilitre is acceptable, in general, for references in International Standards to capacities of volumetric glassware and to liquid volumes.

4.4 Safety oven.

4.5 Muffle furnace, capable of being controlled at 600 ± 25 °C.

4.6 Filtration equipment.

4.7 Filter crucible, of pore size less than 15 µm, or **crucibles** of capacity 30 cm^3.

4.8 Nickel crucible, of capacity 30 cm^3.

4.9 Desiccator.

5 Procedure

Carry out at least two determinations.

WARNING — All hazardous operations must be performed behind a safety shield.

5.1 Test portion

Weigh 0,05 to 0,30 g of finely cut or ground test sample.

5.2 Oxidation

Carry out the oxidation using either a gas fired bomb (see 5.2.1) or an electrically fired bomb (see 5.2.2).

5.2.1 Gas fired bomb

5.2.1.1 Place 7 to 7,5 g of sodium peroxide (3.2) in the nickel crucible (4.3). Add the test portion (5.1) and 8 drops (about 150 mg) of the ethylene glycol (3.1). Add a further 7 to 7,5 g of the sodium peroxide so that the crucible is filled to about 2 mm from the top. Mix by stirring with a glass rod. Place the crucible in the bomb (4.2).

5.2.1.2 Place the lid on the bomb and tighten it by hand.

5.2.1.3 Adjust the flame in the safety oven (4.4), prior to firing the bomb containing the test portion, by placing an empty bomb in the oven and adjusting the flame so that it is a few millimetres from the base of the bomb. Remove the empty bomb and replace it with the bomb containing the test portion, ensuring that the flame is at the same height.

5.2.1.4 Heat the bomb for 3 min after ignition, which usually starts about 10 to 30 s after placing the bomb above the gas flame, and which can be detected by a cracking sound and the appearance of a red glow on the bottom of the bomb.

5.2.2 Electrically fired bomb

5.2.2.1 Place 7 to 7,5 g of sodium peroxide (3.2) in the fusion cup. Add the test portion (5.1) and 8 drops (about 150 mg) of the ethylene glycol (3.1). Add a further 7 to 7,5 g of the sodium peroxide so that the cup is filled to about 2 mm from the top. Mix by stirring with a glass rod.

WARNING — The sodium peroxide must be free from water. If white particles are present, the reagent may ignite on contact with air. This may be prevented by covering the sodium peroxide with a 1 mm layer of the sodium carbonate (3.3).

5.2.2.2 Assemble the bomb and tap it gently to settle the contents.

5.2.3.3 Carry out the oxidation following the manufacturer's instructions for safe operation.

5.3 Determination

5.3.1 In the case of a gas fired bomb, cool it, open and remove the crucible.

WARNING — If the bomb is cooled in water, take care that water does not reach the joint between the plug and the bomb.

Alternatively, dismantle the electrically fired bomb.

5.3.2 Place the crucible and lid, or the contents of the electrically fired bomb together with the head and fusion cup, in 100 cm^3 of water in a 600 cm^3 beaker and immediately cover the beaker with a watch glass.

WARNING — If doubt exists as to whether the bomb has been successfully fired, do not place the contents of the bomb in water, but spread them on dry sand and spray with water from a safe distance. Wash with copious amounts of water. FAILURE TO FOLLOW THESE PRECAUTIONS MAY RESULT IN AN EXPLOSION DUE TO THE VIOLENT REACTION OF SODIUM PEROXIDE WITH WATER.

5.3.3 When the reaction is complete, heat the beaker and its contents, then cool. Rinse the crucible and lid, or the fusion cup etc., with water as it is removed from the beaker.

5.3.4 Slowly add the acetic acid solution (3.7) until the pH is about 4, checking by means of the pH indicator paper (3.10). Heat to boiling and filter into another 600 cm^3 beaker. Wash the filter paper with 20 cm^3 of water acidified with 1 cm^3 of the acetic acid solution, collecting the washings in the beaker containing the filtrate. If there is no residue proceed directly to 5.3.7.

5.3.5 If there is a residue, transfer it to the nickel crucible (4.8), add 0,5 g of the sodium carbonate (3.3) and 0,5 g of the calcium carbonate (3.4) and heat to a clear melt. Cool the crucible and place it in 100 cm^3 of water in a 200 cm^3 beaker. Heat to about 50 °C for 10 min, then filter. Wash the residue with water. Acidify the filtrate with the hydrochloric acid solution (3.5) until the pH is about 4.