METHODS OF TESTING VULCANIZED RUBBER

PART B16. DETERMINATION OF ANTIMONY

BS 903 : Part B16 : 1967

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BRITISH STANDARDS INSTITUTION

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The following BSI references relate to the work on this standard:
Committee references RUC/10 and RUC/10/5
Draft for comment D65/12440

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The Rubber Industry Standards Committee, under whose supervision this British Standard was prepared, consists of representatives from the following Government department and scientific and industrial organizations:

*Federation of British Rubber and Allied Manufacturers
*Institution of the Rubber Industry
*Ministry of Technology
Natural Rubber Bureau
*Natural Rubber Producers' Research Association
*Rubber and Plastics Research Association of Great Britain
Rubber Growers' Association
*Society of Motor Manufacturers and Traders Limited

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Ministry of Defence, Navy Department
Ministry of Housing and Local Government
National College of Rubber Technology
Post Office
Royal Institute of Chemistry
Society of Analytical Chemistry

BRITISH STANDARD

METHODS OF TESTING VULCANIZED RUBBER

Part B16. Determination of Antimony

FOREWORD

This British Standard has been published under the authority of the Rubber Industry Standards Committee. In deciding to issue a revision of the 1950 edition of BS 903, it has also been considered desirable to publish it in separate parts; the present part replaces a section of Part 8 of the 1950 edition.

The letter B indicates the parts concerned with chemical analyses.

The main changes introduced are in the method of wet oxidation of the rubber and in the titration procedure; there is also no need in this revised method for special treatment when iron or chromium is present.

METHOD OF TEST

1. PRINCIPLE

The sample is decomposed with sulphuric and nitric acid. Antimony is separated as the sulphide and determined iodometrically.

2. REAGENTS

All reagents shall be of a recognized analytical reagent quality unless otherwise specified. Solutions shall be freshly prepared, and where necessary filtered.

Hydrochloric acid, $d = 1{\cdot}16$.

Hydrochloric acid, 10% v/v.

Hydrogen sulphide acid wash solution. Saturate 500 ml of sulphuric acid (10% v/v) with hydrogen sulphide.

Nitric acid, $d = 1{\cdot}42$.

Potassium iodide, 10% w/v aqueous solution.

Sodium thiosulphate, 0·05N.

Starch, 1% w/v. Make a suspension of 1 g of starch in 10 ml of cold water and pour into 90 ml of boiling water. Cool before use.

Sulphuric acid, $d = 1{\cdot}84$.

Sulphuric acid, 10% v/v.

Potassium chlorate.

3. PROCEDURE

Accurately weigh about 0·5 g of the sample and transfer it to a 450 ml conical beaker with cover glass, add 10 ml of nitric acid ($d = 1{\cdot}42$) and warm gently until the initial reaction is complete. Cool, carefully add 20 ml of sulphuric acid ($d = 1{\cdot}84$) and evaporate to fuming. Make dropwise additions carefully of nitric acid ($d = 1{\cdot}42$) and evaporate to fuming and continue this dropwise addition until the solution is colourless or pale yellow.

Cool, remove the cover glass, add 20 ml of water and evaporate to fuming; cool, add a further 20 ml of water and evaporate to fuming. Cool, add 100 ml of water and bring to the boil. Allow to stand at room temperature for one hour.

If there is an appreciable residue of insoluble material, remove it by filtration through a paper pulp pad which has been previously washed with sulphuric acid (10% w/v). Wash six times with sulphuric acid (10% w/v) collecting the filtrate and washings in a 650 ml conical beaker. Discard the precipitate.

Dilute the filtrate or the original acid solution to about 400 ml, heat to about 70°C and pass hydrogen sulphide through the solution for twenty minutes. Allow to stand at 70°C for about thirty minutes.

Using suction, filter through a Gooch crucible containing an asbestos pad. Wash the pad six times with the hydrogen sulphide acid wash solution. Discard the filtrate and washings.

Add 5 ml of hydrochloric acid ($d = 1{\cdot}16$) to the precipitate on the asbestos pad, cover with a cover glass and, after standing for a few minutes, filter with suction into a clean Büchner flask. Repeat the procedure with a second portion of hydrochloric acid ($d = 1{\cdot}16$). Then place about 1 g of potassium chlorate on the pad, add another 5 ml portion of hydrochloric acid, and filter after standing for a few minutes. Repeat this treatment with potassium chlorate and hydrochloric acid until dissolution of the antimony sulphide is complete.

Wash the asbestos pad six times with minimal amounts of hot water. Transfer the collected solution and washings to a 450 ml conical beaker and evaporate to a volume of about 5 ml. Add 100 ml of hydrochloric acid (10%), warm until all soluble salts are in solution and cool to room temperature.

Add 5 ml of potassium iodide (10%) and allow to stand for five minutes. Titrate with 0·05N sodium thiosulphate until the brown colour is almost discharged. Add 5 ml of starch solution and continue the titration until the blue colour is just discharged.

4. EXPRESSION OF RESULT

$$\text{Antimony \% (calculated as } Sb_2O_3) = \frac{V \times 0{\cdot}003\,64 \times 100}{W}$$

where V = volume of 0·05N sodium thiosulphate in millilitres
and W = weight of sample in grammes.

BRITISH STANDARDS INSTITUTION

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