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**NATIONAL STANDARD OF THE
PEOPLE'S REPUBLIC OF CHINA**

GB/T 4497-1984

Replacing GB/T 4497-1984

**Rubber -- Determination of total sulfur content -- Part 1: Oxygen
combustion flask method
(English Translation)**

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Foreword

This standard is drafted in accordance with the rules given in GB/T 1.1-2009.

GB/T 4497 "Determination of the total sulfur content of rubber" is divided into three parts.

Part 1. Oxygen bottle combustion method;

- Part 2. Sodium peroxide melting method;

Part 3. Furnace combustion method.

This part is part 1 of GB/T 4497.

This part of the GB/T 4497-1984 "vulcanized rubber, the determination of total sulfur content of oxygen bottle combustion method." this section is the main change compared with GB/T 4497-1984 as follows.

--- modified the standard number and standard name;

--- Added foreword;

--- added warning words;

- the standard titration solution has been modified to barium chloride into barium perchlorate (see 3.4, 1984 version 2.3.5);

--- the standard absorption solution was modified, the concentration of 1% hydrogen peroxide absorption solution used as a concentration of 2% hydrogen peroxide and peroxide Hydrogen - hydrochloric acid (see 3.1.1 and 3.1.2, 1984, 2.3.1);

- modified alcohol was added to the alcohol before the titration, and the added ethanol was modified to 2-propanol (see 3.4, 2.2.3 in 1984);

- increased sample preparation (see Chapter 5);

--- Added the contents of the test report (see Chapter 9).

ISO 6828-1:1992 "Determination of total sulfur content of rubber Part 1. Oxygen bottle combustion" (English version).

This section is proposed by the China Petroleum and Chemical Industry Association.

This part is under the jurisdiction of the National Committee for the Standardization of Test Methods (SAC/TC35/SC2).

The main drafting unit of this part. Beijing Rubber Industry Research and Design Institute.

1 Scope

This part of GB/T 4497 specifies the method for the determination of total sulfur content in rubber by the oxygen bottle combustion method.

This part is applicable to the determination of total sulfur content in rubber and rubber products containing barium sulfate.

The methods specified in this section apply to the determination of total sulfur content in various rubbers. natural rubber, chloroprene rubber, styrene-butadiene rubber, Rubber, isoprene rubber, butyl rubber, EPDM rubber, nitrile rubber, hard rubber and rubber for the above-mentioned rubber.

The methods specified in this section are generally low on the presence of lead, antimony, zinc, barium and calcium compounds. If calcium is stored as calcium carbonate in the case, a reliable test result can be obtained by changing the absorption liquid.

2 Principle

In a flask containing hydrogen peroxide absorbing liquid and filled with oxygen, the sample is fired, in which the carbon and hydrogen of the organic matter are oxidized and the sulfur oxide is absorbed by hydrogen peroxide solution into sulfuric acid, and sometimes the absorption liquid through the ion exchange column to remove the interference ions.

Absorbent with thorium reagent or Thorium reagent and methine blue as indicator, titrated with standard solution of barium perchlorate.

3 Reagents and materials

Unless otherwise specified, the reagents used are analytical reagents, the experimental water is distilled water or water of the same purity.

Note: Two methods of preparing indicators (3.8) are described. Analysts can choose the most appropriate method based on their experience, experimental equipment, and experimental requirements.

Preparation of indicators.

3.1 Absorbent liquid

3.1.1

Hydrogen peroxide, 2% (mass fraction) Absorbate. 1 volume of 30% hydrogen peroxide was diluted to 15 volumes with distilled water.

Warning. 30% of the hydrogen peroxide solution is very corrosive to the skin, the operation should wear rubber or plastic gloves and goggles.

3.1.2

Hydrogen peroxide - hydrochloric acid mixed solution. 4 volumes of 30% hydrogen peroxide diluted with distilled water to 60 volumes plus 5 volumes of hydrochloric acid ($\rho_{20} = 1.18$ g/mL) were mixed evenly.

3.2 hydrochloric acid solution, $c(\text{HCl}) = 0.5\text{mol/L}$.

3.3 2-propanol.

3.4 barium perchlorate standard titration solution, $c[\text{Ba}(\text{ClO}_4)_2] = 0.01\text{mol/L}$.

3.4.1 Preparation. Dissolve 3.363 g of anhydrous barium perchlorate with 200 mL of distilled water. The pH was adjusted to 3.5 with hydrochloric acid solution (3.2). With 2-Propanol was diluted to 1 L.

3.4.2 Calibration.