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Raw styrene-butadiene rubber (SBR) — Determination of organic acid content

Butadiène-styrène (SBR) brut — Dosage des acides organiques

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FOREWORD

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It has been approved by the Member Bodies of the following countries :

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Raw styrene-butadiene rubber (SBR) — Determination of organic acid content

1 SCOPE AND FIELD OF APPLICATION

This International Standard specifies a method for the determination of the organic acid content of raw styrene-butadiene rubber (SBR). The method depends on the prior extraction of the organic acids and soaps from the rubber by means of a solvent. In practice, therefore, it will often be found convenient to determine both organic acid and soap contents on separate portions of the same solvent extract. Since the organic acids present in the rubber are not single chemical compounds, the method gives only an approximate value for the organic acid content.

The method is applicable to all types of styrene-butadiene rubber, but slight modifications are required for oil-extended rubbers.

2 REFERENCE

ISO 2058, *Raw styrene-butadiene rubber (SBR) — Determination of volatile matter.*

3 PRINCIPLE

Extraction of a weighed test portion of the rubber, in the form of thin strips, by ethanol-toluene azeotrope. After making up to standard volume, withdrawal of a measured portion of the extract followed by titration with standard alkali. With oil-extended rubbers it may be necessary to employ a second aliquot of the diluted extract as a control in order to detect the colour change at the end-point.

4 REAGENTS

4.1 Ethanol-toluene azeotrope (ETA)

Mix 7 volumes of absolute ethanol with 3 volumes of toluene. Alternatively, mix 7 volumes of commercial grade ethanol with 3 volumes of toluene, and boil the mixture with anhydrous calcium oxide (quicklime) under reflux for 4 h. Then distil the azeotrope and collect the fraction with a boiling range not exceeding 1 °C, for use in the test.

4.2 Sodium hydroxide, 0,1 N solution.

4.3 Metacresol purple indicator.

Dissolve 0,1 g of metacresol purple in 100 cm^3 * of ethanol or water and bring the solution to the neutral point by adding 2,6 cm^3 of 0,1 N sodium hydroxide solution.

5 APPARATUS

5.1 Balance.

5.2 Hot-plate.

5.3 Wide-mouthed conical flask, 400 to 500 cm^3 nominal capacity.

5.4 Volumetric flask, 250 cm^3.

5.5 Reflux condenser (optional).

5.6 Conical flask, 250 cm^3.

NOTE — Alternatively, a Soxhlet extractor may be used.

5.7 Burette, 25 cm^3.

5.8 Pipette, 100 cm^3.

* The term millilitre (ml) is commonly used as a special name for the cubic centimetre (cm^3), in accordance with a decision of the Twelfth 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

6 PROCEDURE

Sheet out about 6 g of the rubber, dried according to ISO 2058, on a laboratory mill with a nip setting of 0,25 mm or less and at a roll temperature of about 95 °C. When cool, cut the rubber into strips about 10 mm wide and 50 mm long and then reweigh to the nearest 0,01 g.

Place a circular filter paper on the bottom of the conical flask (5.3) and add 100 cm^3 of the ETA extracting solvent (4.1). For alum-coagulated rubbers, use an ethanol/toluene/water mixture of 95 cm^3 of anhydrous ETA and 5 cm^3 of water. For other types the anhydrous azeotrope will be required.

Introduce the strips of rubber separately into the flask, swirling after each addition so that the strips are thoroughly wetted with solvent and sticking is minimized.

Fit the reflux condenser (5.5) to the flask (or close the mouth of the flask with a cooling device such as an evaporating dish containing cold water) and boil the solvent very gently under reflux for 1 h.

Decant the extract into the volumetric flask (5.4), and treat the rubber with a second 100 cm^3 portion of the extracting solvent under reflux for 1 h. Add this extract also to the volumetric flask. Rinse the strips with three successive 10 cm^3 portions of extracting solvent, add these washings to the volumetric flask and, after cooling to room temperature, adjust the final volume to 250 cm^3 with solvent.

After thorough mixing, pipette 100 cm^3 of the diluted extract into the conical flask (5.6), add six drops of indicator (4.3) and titrate the solution with the sodium hydroxide solution (4.2) to the first colour change. If the solution is so dark in colour that the end-point of the titration is likely to be obscure (as may happen with oil-extended rubbers), pipette a second 100 cm^3 into a similar conical flask, add six drops of indicator and use the solution as a colour reference. In comparison, the slight change in colour at the end-point of the titration of the test solution may be more readily observed.

Carry out a blank titration on 100 cm^3 of extracting solvent taken from the same stock as was used for the test.

7 EXPRESSION OF RESULTS

The organic acid content is given, as a percentage by mass, by the formula :

$$\frac{25 \times (V_1 - V_2) \times T \times C}{m}$$

where

V_1 is the volume, in cubic centimetres, of sodium hydroxide solution used to titrate the test solution;

V_2 is the volume, in cubic centimetres, of sodium hydroxide solution used to titrate the blank;

T is the normality of the sodium hydroxide solution;

m is the mass, in grams, of the test portion;

C is the appropriate factor selected from the following :

2,84 when the acid is to be calculated as stearic acid

3,46 when the acid is to be calculated as rosin acid

3,15 when the acid is to be calculated as a 50 : 50 mixture of stearic acid and rosin acid.

NOTE – Since the organic acids present in the rubber are not single chemical compounds the value assigned to C gives only an approximate value for the organic acid content.

8 TEST REPORT

The test report shall include the following information :

a) all details required for full identification of the sample;

b) reference to this International Standard;

c) the organic acid content;

d) the chemical composition of the acid or of the mixture as given in clause 7;

e) the date of the test.