International Standard 7781

INTERNATIONAL ORGANIZATION FOR STANDARDIZATION•МЕЖДУНАРОДНАЯ ОРГАНИЗАЦИЯ ПО СТАНДАРТИЗАЦИИ•ORGANISATION INTERNATIONALE DE NORMALISATION

Rubber, raw styrene-butadiene — Soap and organic acid content — Determination

Caoutchouc butadiène-styrène brut — Détermination de la teneur en savons et acides organiques

First edition — 1986-10-15

UDC 678.746.22-136.22 : 543.85 **Ref. No. ISO 7781-1986 (E)**

Descriptors : rubber, styrene-butadiene rubber, chemical analysis, determination of content, soaps, organic acids.

Price based on 4 pages

Foreword

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International Standard ISO 7781 was prepared by Technical Committee ISO/TC 45, *Rubber and rubber products*.

It cancels and replaces International Standards ISO 2002-1975 and ISO 2003-1975, of which it constitutes a technical revision.

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.

Printed in Switzerland

Rubber, raw styrene-butadiene — Soap and organic acid content — Determination

1 Scope and field of application

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

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

2 References

ISO 248, *Rubber, raw — Determination of volatile matter content.*

ISO 385/1, *Laboratory glassware — Burettes — Part 1: General requirements.*

ISO 648, *Laboratory glassware — One-mark pipettes.*

ISO 1042, *Laboratory glassware — One-mark volumetric flasks.*

3 Principle

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

4 Reagents

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

All recognized health and safety precautions shall be observed when carrying out the procedures specified in this International Standard.

4.1 Ethanol-toluene azeotrope (ETA).

Mix 7 volumes of 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.

4.2 Ethanol-toluene-water mixture.

Mix 95 cm^3* of the ETA (4.1) and 5 cm^3 of water.

4.3 Sodium hydroxide, 0,1 mol/dm^3 solution, accurately standardized.

4.4 Meta-cresol purple indicator.

Dissolve 0,1 g of meta-cresol purple in 100 cm^3 of ethanol or water and bring the solution to the neutral point by adding 2,6 cm^3 of the sodium hydroxide solution (4.3).

4.5 Thymol blue indicator.

Dissolve 0,06 g of thymol blue in 6,45 cm^3 of 0,02 mol/dm^3 sodium hydroxide solution and dilute to 50 cm^3 with water.

4.6 Hydrochloric acid, 0,05 mol/dm^3 solution, accurately standardized.

5 Apparatus

5.1 Balance, accurate to ± 0,1 mg.

5.2 Hot-plate.

* 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.

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

5.4 Volumetric flask, 250 cm^3, complying with the requirements of ISO 1042.

5.5 Reflux condenser.

5.6 Conical flask, 250 cm^3.

NOTE — Alternatively, a Soxhlet extractor may be used instead of 5.5 and 5.6.

5.7 Burette, 25 cm^3, complying with the requirements of ISO 385/1.

5.8 Pipette, 100 cm^3, complying with the requirements of ISO 648.

5.9 Auto-titrator (optional), only if indicator is used.

6 Procedure for determination of soap content

Sheet out about 6 g of the rubber, dried according to ISO 248, 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 weigh to the nearest 0,01 g.

Place a circular filter paper in the bottom of the conical flask (5.3) and add 100 cm^3 of the ETA extracting solvent (4.1) for all rubbers except alum-coagulated rubbers. For alum-coagulated rubbers, use the ethanol-toluene-water mixture (4.2).

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.

Transfer 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.

NOTE — Alternatively, the weighed strips of sample may be wrapped in filter paper and placed in a Soxhlet extractor (see the note to 5.6) and extracted with the ETA (4.1) or the ethanol-toluene-water mixture (4.2) under reflux for a minimum of 4 h.

After thorough mixing, pipette 100 cm^3 of the diluted extract into the conical flask (5.6), add six drops of the meta-cresol purple indicator (4.4) for all rubbers, except alum-coagulated rubbers. For alum-coagulated rubbers, use the thymol blue indicator (4.5).

Titrate the solution with the hydrochloric acid solution (4.6) 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. The determination of change of colour at the end of the titration can be very difficult in the case of oil-extended SBR. In this case, determination of equivalence point by potentiometry is more accurate, and is therefore recommended.

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

Proceed to 8.1 for calculation of the soap content.

7 Procedure for determination of organic acid content

Proceed exactly as in clause 6 for determination of soap content, but titrate the aliquot portion with the sodium hydroxide solution (4.3), using an indicator, for example phenolphthalein.

Proceed to 8.2 for calculation of the organic acid content.

8 Expression of results

8.1 Calculate the soap content using the equation

$$w_s = \frac{2{,}5 \times (V_1 - V_2) \times c_1 \times K_s}{m}$$

where

w_s is the soap content, as a percentage by mass;

V_1 is the volume, in cubic centimetres, of the hydrochloric acid solution used to titrate the rubber extract;

V_2 is the volume, in cubic centimetres, of the hydrochloric acid solution used to titrate the blank;

c_1 is the actual concentration, in moles per cubic decimetre, of the hydrochloric acid solution (4.6);

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

K_s the appropriate factor selected from the following:

3,06 when the soap is to be calculated as sodium stearate,

3,68 when the soap is to be calculated as sodium rosinate,

3,37 when the soap is to be calculated as a 50:50 mixture of sodium stearate and sodium rosinate,