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Rubber, raw styrene-butadiene, emulsion-polymerized — Determination of bound styrene content — Refractive index method

*Caoutchouc butadiène-styrène brut polymérisé en émulsion —
Détermination de la teneur en styrène lié — Méthode par l'indice de
réfraction*



Reference number
ISO 2453:1991(E)

Foreword

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Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

International Standard ISO 2453 was prepared by Technical Committee ISO/TC 45, *Rubber and rubber products*.

This second edition cancels and replaces the first edition (ISO 2453:1975), clauses 1 and 7 and sub-clause 5.3 of which have been technically revised.

Annex A of this International Standard is for information only.

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Rubber, raw styrene-butadiene, emulsion-polymerized — Determination of bound styrene content — Refractive index method

1 Scope

This International Standard specifies a method for determining the bound styrene content of emulsion-polymerized styrene-butadiene (SBR) rubbers by correlation with the measured refractive index of an extracted sample according to a table of refractive indices versus percentages by mass of styrene.

The method is also applicable to extracted oil-extended emulsion-polymerized SBR as long as it produces a film suitable for refractive index measurements. It is not applicable to solution-polymerized SBR.

2 Significance of the test

The bound styrene test is a measure of the bound monomeric composition of the rubber. It is used as a check on the accuracy of monomer charging and also as a guide to the uniformity of the product, since the bound styrene content affects the physical properties.

3 Principle

Extraction of a test piece with ethanol-toluene azeotrope (ETA), followed by drying and pressing between sheets of aluminium foil to provide sheeted rubber having a thickness of not more than 0,50 mm. Calculation of the bound styrene content from the refractive index obtained at 25 °C on this thinly sheeted rubber.

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 Acidified ETA.

Add 10 cm³ of concentrated hydrochloric acid [approximately 35 % (*m/m*)] to a portion of ETA (4.1) and make the solution up to 1 000 cm³ with ETA.

NOTE 1 Acidified ETA is used for alum-coagulated polymers.

4.3 α -Bromonaphthalene.

5 Apparatus

5.1 Spiders, consisting of 13 mm squares of sheet aluminium or stainless steel having a nickel-chromium wire leg about 38 mm long attached to each corner. When the extracting solvent is ETA acidified with hydrochloric acid, the spider and the legs shall be made of tantalum.

5.2 Reflux condenser.

5.3 Abbe-type refractometer, having fourth-decimal-place accuracy and whose refracting prism can be placed in a nearly horizontal position for measurement of the refractive index of solids. An Amici-type compensating prism for achromatization is necessary unless a sodium-vapour lamp is used as a light source.

The refractometer shall be maintained at a temperature of 25 °C \pm 0,1 °C, obtained by the use of a constant temperature room or by circulating constant temperature water through the instrument.