INTERNATIONAL STANDARD

ISO 5478

Second edition 1990-11-15

Rubber — Determination of styrene content — Nitration method

Caoutchouc — Détermination de la teneur en styrène — Méthode par nitration



Reference number ISO 5478: 1990 (E)

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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

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 5478 was prepared by Technical Committee ISO/TC 45, Rubber and rubber products.

This second edition cancels and replaces the first edition (ISO 5478: 1980), of which it constitutes a technical revision.

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International Organization for Standardization
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Rubber — Determination of styrene content — Nitration method

1 Scope

- 1.1 This International Standard specifies a method for the determination of the styrene content of all types of styrenebutadiene rubber (SBR), including oil-extended types.
- **1.2** The method is applicable to styrene-butadiene rubbers reinforced with styrene homopolymer, to give the total styrene content.
- **1.3** The method is also applicable for determining the styrene in block copolymers containing up to 50 % (m/m) of styrene.
- 1.4 It is applicable to vulcanizates of mixtures of SBR with other polymers (NR, BR, IR and CR) and is considered a method for estimating the SBR content of mixtures, if the styrene content of the SBR is known and provided that no other styrene-containing materials are present.
- 1.5 Any other non-extractable aromatic material which absorbs in the specified spectral region will interfere with this method.
- **1.6** The procedure may give low results when the content of mineral fillers insoluble in nitric acid exceeds 5 % (m/m).

2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this International Standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 383: 1976, Laboratory glassware — Interchangeable conical ground joints.

ISO 1407: 1976, Rubber - Determination of solvent extract.

ISO 1795: 1974, Raw rubber in bales - Sampling.

ISO 1796: 1982, Rubber, raw — Sample preparation.

ISO 4655: 1985, Rubber — Reinforced styrene-butadiene latex — Determination of total bound styrene content.

ISO 4661-2: 1987, Rubber vulcanized — Preparation of samples and test pieces — Part 2: Chemical tests.

3 Principle

- **3.1** A test portion is extracted with acetone and digested with nitric acid to oxidize the styrene to *p*-nitrobenzoic acid.
- **3.2** The p-nitrobenzoic acid is extracted with diethyl ether.
- **3.3** The *p*-nitrobenzoic acid is extracted from the ether with sodium hydroxide solution and measured spectrometrically in the ultraviolet spectral region.

4 Reagents and materials

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

WARNING — Because of possible health and safety hazards inherent in following this procedure, recognized health and safety precautions shall be observed with the use of ether and acids. Extractions shall be carried out in a properly ventilated fume hood and safety glasses shall be worn during these extractions.

4.1 Nitric acid, ϱ 1,43 g/cm³.

The nitric acid shall be fresh. Old batches may give inferior nitration.

4.2 Sodium hydroxide, solution, $c(NaOH) = 5 \text{ mol/dm}^3$.

Dissolve 200 g of sodium hydroxide in water and dilute to 1 000 cm³.

4.3 Sodium hydroxide, solution, $c(NaOH) = 0.1 \text{ mol/dm}^3$.

Dissolve 4 g of sodium hydroxide in water and dilute to 1 000 cm³.

- 4.4 Diethyl ether, free from peroxides.
- 4.5 Sodium chloride, saturated solution.
- 4.6 Sodium sulfate, anhydrous.