INTERNATIONAL STANDARD

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Styrene-butadiene rubber (SBR) — Determination of the microstructure of solution-polymerized SBR

Caoutchouc styrène-butadiène (SBR) — Détermination de la microstructure du SBR polymérisé en solution

Reference number
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Contents

Page

Foreword

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The main task of technical committees is to prepare International Standards. 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.

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ISO 21561 was prepared by Technical Committee ISO/TC 45, *Rubber and rubber products*, Subcommittee SC 2, *Testing and analysis*.

Styrene-butadiene rubber (SBR) — Determination of the microstructure of solution-polymerized SBR

WARNING — Persons using this International Standard should be familiar with normal laboratory practice. This standard does not purport to address all the safety problems, if any, associated with its use. It is the responsibility of the user to establish appropriate safety and health practices and to ensure compliance with any national regulatory conditions.

1 Scope

This International Standard specifies procedures for the quantitative determination of the microstructure of the butadiene units and the content of styrene units in solution-polymerized SBR (S-SBR) by ^{1}H-NMR spectrometry as an absolute method and by IR spectrometry as a relative method. The styrene content is expressed in mass % relative to the whole polymer. The 1,4-trans, 1,4-cis and 1,2-vinyl contents are expressed in mol % relative to the butadiene units.

NOTE IR spectrometry can also give absolute values of microstructure by calibration with S-SBRs of known absolute microstructure obtained by ^{1}H-NMR spectrometry.

2 Normative references

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 1407, *Rubber — Determination of solvent extract*

ISO 1795, *Rubber, raw natural and raw synthetic — Sampling and further preparative procedures*

3 NMR method (absolute method)

3.1 Principle

3.1.1 A small quantity of an extracted S-SBR is dissolved in deuterated-chloroform.

3.1.2 A ^{1}H-NMR spectrum of the sample solution is measured at a 15 ppm sweep width. The peak areas of the 1,4-bond (the sum of the 1,4-trans bond and 1,4-cis bond) and the 1,2-vinyl bond of the butadiene portion are determined along with the peak area of styrene. The microstructure of the butadiene portion and the styrene content are then calculated using theoretical formulae.

3.2 Reagents

3.2.1 Deuterated chloroform, $CDCl_3$, containing 0,03 % of tetramethyl silane (TMS) as internal standard. The purity of the $CDCl_3$ itself shall be $>$ 99,8 %.

3.2.2 Anhydrous ethanol-toluene azeotrope (ETA).