INTERNATIONAL STANDARD

ISO 7270-2

Second edition
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Rubber — Analysis by pyrolytic gas-chromatographic methods —

Part 2: Determination of styrene/butadiene/isoprene ratio

Caoutchouc — Méthodes d'analyse par pyrolyse et chromatographie en phase gazeuse —

Partie 2: Détermination du rapport styrène/butadiène/isoprène

Reference number
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ISO copyright office
Case postale 56 • CH-1211 Geneva 20
Tel. + 41 22 749 01 11
Fax + 41 22 749 09 47
E-mail copyright@iso.org
Web www.iso.org

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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.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

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.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

ISO 7270-2 was prepared by Technical Committee ISO/TC 45, *Rubber and rubber products*, Subcommittee SC 2, *Testing and analysis*.

This second edition cancels and replaces the first edition (ISO 7270-2:2005), which has been technically revised.

ISO 7270 consists of the following parts, under the general title *Rubber — Analysis by pyrolytic gas-chromatographic methods*:

— *Part 1: Identification of polymers (single polymers and polymer blends)*

— *Part 2: Determination of styrene/butadiene/isoprene ratio*

Rubber — Analysis by pyrolytic gas-chromatographic methods —

Part 2: Determination of styrene/butadiene/isoprene ratio

WARNING — Persons using this part of ISO 7270 should be familiar with normal laboratory practice. This part of ISO 7270 does not purport to address all of 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.

CAUTION — Certain procedures specified in this part of ISO 7270 might involve the use or generation of substances, or the generation of waste, that could constitute a local environmental hazard. Reference should be made to appropriate documentation on safe handling and disposal after use.

1 Scope

This part of ISO 7270 specifies a pyrolytic gas-chromatographic method for the determination of the styrene/butadiene/isoprene ratio in copolymers, or blends of homopolymers and/or copolymers, in raw rubbers or in unvulcanized or vulcanized compounds. It is applicable to copolymers/terpolymers consisting of styrene, butadiene and isoprene, and blends of these polymers.

NOTE 1 The use of this part of ISO 7270 pre-supposes sufficient working knowledge of the principles and techniques of gas chromatography for the analyst to perform the operations described and interpret the results correctly.

NOTE 2 The styrene/butadiene/isoprene ratio determined by this test method is affected by the presence of resin and by a high level of sulfur.

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 1407:2011, *Rubber — Determination of solvent extract*

3 Principle

Calibration curves are first prepared by pyrolysing samples with known styrene/butadiene/isoprene ratios and analysing the pyrolysis products by gas chromatography to determine the percentage content of each component (styrene, butadiene and isoprene), relative to the total of the three components.

Samples of unknown composition are then pyrolysed, and the pyrolysis products are analysed under the same conditions. The styrene/butadiene/isoprene ratio in these samples is determined from the calibration curves.

4 Reagents

All reagents shall be of analytical grade.

4.1 Solvents for extraction purposes.

The following solvents are suitable:

— acetone;

— methanol;

— methylethylketone.

4.2 Carrier gas:

— nitrogen;

or

— helium.

4.3 Gas for flame-ionization detector: hydrogen plus purified compressed air.

5 Apparatus

5.1 Extraction apparatus

As specified in ISO 1407.

5.2 Pyrolysis/gas chromatography system

5.2.1 General

The apparatus utilized to obtain pyrograms consists of four parts: the pyrolysis device, the gas chromatograph, the gas-chromatographic column and the data-handling equipment.

5.2.2 Pyrolysis device

The following types of pyrolysis device are suitable:

— micro-furnace pyrolyser;

— Curie-point pyrolyser;

— platinum-filament pyrolyser.

5.2.3 Gas chromatograph

A wide variety of gas chromatographs using either a flame-ionization detector (FID) or a thermal-conductivity detector (TCD) are suitable for use in this part of ISO 7270. An FID is preferable for use with capillary columns.

5.2.4 Chromatographic columns

A variety of column materials, column lengths, column diameters and stationary and liquid phases are suitable for use in this part of ISO 7270, the main requirement being high resolution of the volatile pyrolysis products styrene, butadiene and isoprene.

NOTE 1 Capillary columns, which have a separation efficiency which is better than that of packed columns, are suitable, but not essential.

NOTE 2 Capillary columns containing non-polar polydimethylsiloxanes and partially modified (diphenyl-, cyanopropylphenyl- or other) semi-polar silicones are suitable.

NOTE 3 When using an ordinary (30 m) capillary column, it is difficult to separate isobutene from butadiene completely. The reason is that, in pyrolytic gas-chromatographic methods, isobutene, which is a decomposition product of rubber containing the isoprene unit, is detected close to the retention time of butadiene. It is possible, however, to discriminate between isoprene and butadiene, even without complete separation, by means of the calibration curves.