INTERNATIONAL STANDARD

ISO 14558

Second edition
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Rubber — Determination of residual unsaturation of hydrogenated nitrile butadiene rubber (HNBR) by infrared spectroscopy

Caoutchouc — Détermination de la non-saturation résiduelle du caoutchouc nitrile butadiène hydrogéné (HNBR) par spectroscopie à infrarouge

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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.

The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

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Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

For an explanation on the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the WTO principles in the Technical Barriers to Trade (TBT) see the following URL: Foreword - Supplementary information.

The committee responsible for this document is ISO/TC 45, *Rubber and rubber products*, Subcommittee SC 3, *Raw materials (including latex) for use in the rubber industry*.

This second edition cancels and replaces the first edition (ISO 14558:2000), of which it constitutes a minor revision where the normative references were updated and the precision data were moved to an informative Annex B.

Rubber — Determination of residual unsaturation of hydrogenated nitrile butadiene rubber (HNBR) by infrared spectroscopy

WARNING — Persons using this International Standard should be familiar with normal laboratory practice. This International Standard 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.

1 Scope

This International Standard specifies a method for determining the residual unsaturation in hydrogenated nitrile rubber (HNBR) by measuring the infrared (IR) absorbance of HNBR films cast from solution.

This International Standard assumes that samples and IR spectra are prepared and analysed by experienced personnel and that equipment is operated in accordance with the manufacturer's instructions. Details for operating an IR spectrometer are not included in this method.

2 Normative references

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

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

3 Principle

Raw, unvulcanized HNBR is purified by precipitation with methanol from a solution in methyl ethyl ketone (MEK) or by extraction of the solid HNBR with methanol in a Soxhlet apparatus.

The purified sample is dissolved in MEK and a film is cast on a potassium bromide (KBr) disc.

The IR spectrum of the film is obtained with a Fourier-transform (FT) or dispersive IR spectrometer.

The "corrected absorbance" of the specific absorbance bands for acrylonitrile (AN), butadiene (BD) and hydrogenated butadiene (HBD) are determined using the baseline method and the percentage of residual unsaturation (double bonds in unhydrogenated butadiene) is calculated with the aid of absorbance factors from the literature (see 8.5).

4 Reagents

Reagent grade chemicals should preferably be used in all determinations. Other grades may be used provided that they are of sufficiently high purity not to lessen the accuracy of the determination.

4.1 Methyl ethyl ketone (MEK).

4.2 Methanol.

4.3 Dry, compressed nitrogen.

4.4 Potassium bromide discs.

5 Sampling

Sample the raw rubber in accordance with ISO 1795.

6 Apparatus

Ordinary laboratory equipment and the following.

6.1 Conical flask, 50 cm^3, with ground-glass stopper.

6.2 Flask shaker.

6.3 Beaker, 250 cm^3.

6.4 Magnetic stirrer.

6.5 Soxhlet extraction apparatus, with 150 cm^3 flask.

6.6 Extraction thimbles, 25 mm × 100 mm.

6.7 Koffler heating bench, or other heating device, with temperature control to ±2 °C.

6.8 Fourier-transform IR (FTIR) spectrometer, with 2 cm^{-1} resolution or a dispersive IR spectrometer capable of equivalent spectral resolution. The instrument shall be capable of scale expansion along the absorbance or transmittance axis over the special region of 2 500 cm^{-1} to 600 cm^{-1}.

7 Procedure

7.1 Sample preparation

7.1.1 Purification by precipitation

7.1.1.1 Transfer 1 g of the finely divided HNBR rubber sample into a 50 cm^3 conical flask. Add 20 cm^3 of MEK to the flask. Tightly stopper the flask and place it on a flask shaker and shake until the sample has completely dissolved.

7.1.1.2 Precipitate the rubber by slowly pouring the MEK solution into a 250 cm^3 beaker containing 150 cm^3 of methanol, while rapidly stirring the methanol with a magnetic stirrer.

7.1.1.3 Decant the solvent and wash the precipitated rubber with 50 cm^3 of methanol. Decant the methanol washings and redissolve the precipitated rubber in 20 cm^3 of MEK.

7.1.2 Purification by extraction

Transfer 1 g of finely divided rubber into an extraction thimble and extract for 6 h in a Soxhlet apparatus with 100 cm^3 of methanol.

Remove the extracted sample from the thimble and dissolve in 20 cm^3 of MEK.