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Rubber — Identification — Infrared spectrometric method

Caoutchouc — Identification — Méthode spectrométrique dans l'infrarouge

Reference number
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Contents

Page

Foreword

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ISO 4650 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 4650:1984), which has been technically revised.

Rubber — Identification — Infrared spectrometric method

1 Scope

This International Standard specifies a method for the identification of rubbers, including thermoplastic elastomers, either in the raw state or in the form of vulcanized or unvulcanized mixes. The method is based on infrared spectrometric examination using the transmission technique.

The method comprises examination of polymers by their pyrolysis products (pyrolysates), or by films cast from solution or obtained by moulding (for raw rubbers only).

Typical spectra are given in Annex A.

The principle of the method implies that sample preparation and analysis of the infrared spectra are carried out by experienced personnel and that the equipment used for the production of spectra is operated in accordance with the manufacturer's instructions for optimum performance. Details of the operation of infrared spectrometers are not included in this International Standard.

The method specified is a qualitative method only.

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 18064, *Thermoplastic elastomers — Nomenclature and abbreviated terms*

3 Principle

The extractable material is first extracted from a test sample of the rubber and the rubber then prepared under precise conditions for spectroscopy in the form of raw polymer film, vulcanizate pyrolysate or vulcanizate film. The IR spectrum is recorded and then interpreted by comparison with a set of typical reference spectra.

4 Types of rubber

4.1 General

The method is applicable to rubbers in the raw state and, if compounded, in both the vulcanized and unvulcanized states. It is applicable to the following types of rubber occurring either alone or in a binary mixture when the proportion of the minor component is, in general, not less than 10 % to 20 % by mass of the mixture (see, however, exceptions in 4.2).

4.1.1 M group

4.1.1.1 Acrylic rubber (ACM): Copolymer of ethyl acrylate (or other acrylates) and a small amount of a monomer which facilitates vulcanization.

4.1.1.2 Chloropolyethylene (CM) and chlorosulfonylpolyethylene (CSM): The method will not differentiate CM from CSM, and it will not differentiate between different types of CSM.

4.1.1.3 Ethylene-propylene copolymer (EPM) and ethylene-propylene-diene terpolymer (EPDM): The method will not differentiate between the two types of polymer. However, examination of the spectrum gives some information about the ethylene-to-propylene ratio.

4.1.1.4 Fluorocarbon rubber (FKM): Examination of the pyrolysate may give some information about the different grades of fluorocarbon rubber present.

4.1.2 O group

4.1.2.1 Polychloromethyloxirane (CO): Copolymer of ethylene oxide and chloromethyloxirane (ECO) and terpolymers. Examination of the pyrolysate will not differentiate between different types of CO.

4.1.3 Q group

4.1.3.1 Polydimethylsiloxane (MQ), polymethylphenylsiloxane (PMQ) and polymethylfluorosiloxane (FMQ): Examination of the pyrolysate will differentiate PMQ from MQ.

4.1.4 R group

4.1.4.1 Butadiene rubber (BR): Examination of the pyrolysate will not differentiate between butadiene rubbers having different isomer ratios. However, examination of a raw rubber film gives some information about the isomer ratio.

4.1.4.2 Chloroprene rubber (CR): The method will not differentiate between the different types of CR.

4.1.4.3 Isobutene-isoprene rubber (IIR) and halogenated isobutene-isoprene rubbers (BIIR and CIIR): Under the conditions used for the method, it is not possible to differentiate between IIR, BIIR, CIIR and polyisobutene.

4.1.4.4 Natural rubber (NR) and synthetic isoprene rubber (IR): Natural rubber (1,4-*cis*-polyisoprene), gutta percha, balata (1,4-*trans*-polyisoprene) and synthetic isoprene rubber, whatever their microstructure, (1,4-*cis*, 1,4-*trans* or 3,4-) are included.

4.1.4.4.1 Examination of a rubber film will differentiate between 1,4-*cis*, 1,4-*trans* and 3,4-polyisoprenes; for non-extracted rubbers, it will differentiate natural rubber from 1,4-*cis* synthetic isoprene rubber, and 1,4-*trans* natural polyisoprenes from their synthetic counterparts. Examination of the pyrolysate film obtained from a vulcanizate provides no information on the microstructure of the polyisoprene or its origin, whether natural or synthetic.

4.1.4.5 Acrylonitrile-butadiene rubber (NBR): The method will differentiate carboxylic acrylonitrile-butadiene rubbers (XNBRs) from hydrogenated acrylonitrile-butadiene rubbers (HNBRs). Associations of butadiene copolymers and PVC are included. Examination of the pyrolysate film gives some information about the acrylonitrile content.

4.1.4.6 Styrene-butadiene rubber (SBR): The method will differentiate α-methylstyrene-butadiene rubbers from styrene-butadiene rubbers. Copolymers of styrene and butadiene, as well as of their substituted derivatives (e.g. α-methylstyrene), are included. Examination of a pyrolysate will not differentiate emulsion-polymerized rubbers from solution-polymerized rubbers. However, examination of a spectrum gives some information about the monomer ratio.

4.1.4.7 Polynorbornene.

4.1.5 T group

4.1.5.1 Polysulfide rubbers.

4.1.6 U group

4.1.6.1 Polyester urethane (AU) and polyether urethane (EU): The method covers only millable polyurethanes.

4.1.7 TPE group

4.1.7.1 As defined in ISO 18064.

4.2 Exceptions for blends

4.2.1 Analysis of a blend of ethylene-propylene rubber with other rubbers presents difficulties when its ethylene-propylene content is below 40 %.

4.2.2 The method will not differentiate between blends of ethylene-propylene rubber with chlorinated polyethylene and/or chloro-sulfonated polyethylene.

4.2.3 Analysis of a blend of natural and/or synthetic polyisoprene and chloroprene rubber may present difficulties, and identification of the minor component may only be possible when the content is equal to or greater than 30 % in the blend.

4.2.4 The method will not differentiate NBR from NBR/BR blends or NBR blends, nor will it differentiate SBR from SBR/BR blends or SBR blends.

4.2.5 The presence of high quantities of sulfur in a vulcanizate may affect some characteristic bands.

4.2.6 The method will not differentiate NBR/PVC blends from blends of NBR with other halogenated polymers or additives.

4.3 Reference spectra

Tables of absorption characteristics and reference spectra from 4 000 cm^{-1} to 600 cm^{-1} for typical rubbers are given in Annex A.

5 Reagents

5.1 Nitrogen, in pressurized cylinders.

5.2 Extraction solvents, chosen to achieve maximum extraction (alternative solvents may be used on condition that it can be shown that they do not interfere with the interpretation of the infrared spectrum):

5.2.1 Methanol.

5.2.2 Acetone.

5.3 Solvents for rubber dissolution and film preparation, water-free and free from residues (see ISO 1407):

5.3.1 Chloroform.

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