INTERNATIONAL STANDARD

ISO 16565

Second edition
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Rubber — Determination of 5-ethylidenenorbornene (ENB) or dicyclopentadiene (DCPD) in ethylene-propylene-diene (EPDM) terpolymers

Caoutchouc — Détermination du 5-éthylidènenorbornène (ENB) ou du dicyclopentadiène (DCPD) dans les terpolymers d'éthylène-propylène-diène (EPDM)

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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 16565 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 16965:2002), of which it constitutes a minor revision. The main changes concern corrections to the Y-axes in Figures 1 to 3 and to the X and Y-axes in Figure 4.

Rubber — Determination of 5-ethylidenenorbornene (ENB) or dicyclopentadiene (DCPD) in ethylene-propylene-diene (EPDM) terpolymers

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

1 Scope

This International Standard specifies the methods to be used to determine the content of 5-ethylidenenorbornene (ENB) or dicyclopentadiene (DCPD) in ethylene-propylene-diene (EPDM) terpolymers in the 0,1 % to 15 % range.

ENB and DCPD are dienes introduced into ethylene-propylene rubbers to generate specific cure properties. Since high precision for diene content determination is important, a Fourier transform infrared spectroscopic (FT-IR) method is utilized.

NOTE The procedures for mass fraction of ENB and mass fraction of DCPD differ only in the location in the infrared (IR) peak being quantified.

2 Principle

A test specimen is moulded between two PTFE-coated aluminium or mylar sheets. The ENB content is determined from its infrared absorbance at 1 681 cm^{-1} to 1 690 cm^{-1}, a measure of the exocyclic double bond in ENB. The DCPD content is determined from its infrared absorbance at 1 605 cm^{-1} to 1 610 cm^{-1}, a measure of the monocyclic double bond in DCPD.

The second derivative of the absorbance is calculated and ratioed to an internal standard. For ENB, the resulting second-derivative peak near 1 690 cm^{-1} is related to the ENB mass fraction by calibrating the instrument with known EPDM standards. For DCPD, the resulting second-derivative peak near 1 610 cm^{-1} is related to the DCPD mass fraction in the same way.

For oil-extended polymers, the oil must be extracted before the diene content is determined.

3 Apparatus

3.1 Carver-type press, capable of compressing films at 150 °C and 10 MPa.

3.2 Mould

3.2.1 The mould primarily used is made of a stainless-steel strip 400 µm thick with an opening which will give a specimen of the appropriate size for the specimen-film holder described in 3.4 (2 cm by 2 cm). The mould shall have approximately the same dimensions as the press platens.