INTERNATIONAL STANDARD

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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

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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. www.iso.org/directives

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The committee responsible for this document is ISO/TC 45, *Rubber and rubber products*, Subcommittee SC 2, *Testing and analysis*.

This third edition cancels and replaces the second edition (ISO 16565:2008), of which it constitutes a minor revision. The main changes are: a correction to the Scope concerning the range of termonomer content (either ENB or DCPD) to be determined and the locations of points A and B on Figure 1.

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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 concerns, if any, associated with its use. It is the responsibility of the user of this International 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 10 % 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 the mass fraction of ENB and the mass fraction of DCPD differ only in the location in the infrared (IR) of the 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 shall 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 gives 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.

3.2.2 Alternatively, a thinner mould plate may be used. However, precision can be adversely affected at low diene levels. The precision of the method shall be determined when thinner films are utilized. For example, 127 µm-thick stainless-steel foil with a 15 mm by 35 mm opening may be used for the simultaneous determination of ethylene and diene.