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BSI Standards Publication

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

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National foreword

This British Standard is the UK implementation of ISO 16565:2013. It supersedes BS ISO 16565:2008 which is withdrawn.

The UK participation in its preparation was entrusted to Technical Committee PRI/23, Test methods for rubber and non-black compounding ingredients.

A list of organizations represented on this committee can be obtained on request to its secretary.

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INTERNATIONAL STANDARD

ISO 16565

Third 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.

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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INTERNATIONAL STANDARD ISO 16565:2013(E)

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 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.

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3.2.3 For liquid EPDMs, a ring washer of 22 mm outer diameter by 16 mm inner diameter and 400 µm-thick is used as a spacer between salt plates (NaBr, NaCl) to set a fixed path length. The spacer is sized to cover only the outer edge of the salt plates.

3.3 PTFE-coated aluminium moulding sheets, type A, 36 µm thick, or **silicone release sheeting**.

3.4 Specimen-film holders: films may be moulded, cut out and transferred to a film holder. Magnetic-film holders are ideal. Alternatively, a mould sized to fit in the spectrophotometer specimen compartment, with an appropriate-sized opening, may be used to support the film without removal after compressing. A standard salt plate support is used for liquid samples prepared between salt plates.

3.5 Infrared Fourier transform spectrophotometer, capable of measuring absorbances in the range of 4 000 cm^{-1} to 600 cm^{-1} with a transmittance specification (accuracy) of ±1 % T or better. The instrument shall be capable of spectral resolution of 2 cm^{-1}. A deuterated triglycine sulfate (DTGS) or, as an alternative, a mercury cadmium telluride (MCT) detector shall be used.

NOTE A specification for the evaluation of the analysis of infrared spectra is given in Reference.[1]

The instrument shall be capable of spectral accumulation, averaging and subtracting. Water is the primary source of interference in this method. Methods, physical and electronic, which minimize the moisture level and moisture-level variation are required to obtain the highest precision. The preferred method is to use an instrument equipped with a dry-gas purge and specimen shuttle, which permits alternating and repetitive collection of single-beam specimen and background spectra (see Clause 5). Alternatively, should a specimen shuttle be unavailable, careful purging of the specimen compartment with dry nitrogen can yield satisfactory results. High precision of calibration-standard data is indicative of adequate purging. When moisture interference is not removed by purging, spectral subtraction of water vapour may be used. A procedure for further development of the method is described in Annex A.

4 Test specimen preparation

4.1 Primary method

Place 0,20 g ± 0,05 g of the material to be analysed between two sheets (see 3.3) in the mould (see 3.2.1). Place the mould between the press platens heated to 125 °C ± 5 °C and apply 4 MPa pressure for 60 s ± 10 s.

Should the material be highly viscous, the mould may be heated to 175 °C ± 5 °C.

Cool the material to ambient temperature. Cut out a piece of film of approximately 15 mm by 50 mm. Detach the specimen film from the sheets (see 3.3), and position it on the spectrophotometer specimen compartment window.

4.2 Alternative method

When using the thinner mould described in 3.2.2, place a small piece (0,04 g to 0,06 g) of the material to be analysed in the mould opening between two sheets (see 3.3) and compress as in 4.1. Remove the mould from the press, turn it over and compress it again. Then, remove the mould from the press and allow it to cool to ambient temperature. When cool, carefully remove the sheets (see 3.3), allowing the specimen film to remain attached to the mould.

4.3 Alternative method for liquid polymer film preparation

Place a washer (see 3.2.3) on top of a salt plate. Place a small amount (about 0,3 g) of the liquid EPDM polymer to be analysed in the centre of the washer, filling the hole completely. Place a second salt plate on top of the filled washer. Gently place a 1 kg weight on top of the salt plate/washer assembly and allow the weighted assembly to sit for 2 min to 3 min. (For viscous samples, it may be necessary to warm the sample prior to compressing.) Remove the 1 kg weight and, if necessary, allow to cool. Wipe off any excess material that might have been pressed out of the assembly. Hold the assembly up to the light and

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