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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 It supersedes BS ISO 16565:2008 which is withdrawn.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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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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Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

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.

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.

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 inspect for bubbles and/or voids. Should there be imperfections, repeat the preparation with a larger amount of material.

5 Acquisition of spectra

5.1 With a specimen shuttle

5.1.1 The data-acquisition parameters shall be:

— resolution: 2 cm^{-1};

— scans/scan time: total scan time, split between specimen scans and background scans, about 90 s.

5.1.2 Place the test specimen in the specimen compartment, allow purge to be re-established and, in alternating fashion, collect single-beam specimen and empty specimen compartment spectra. Eight passes of the shuttle shall be made (eight specimen and eight empty-compartment collections), collecting four scans at each position.

5.1.3 Calculate the specimen absorbance spectrum as $-\log_{10}$ of the ratio of the accumulated single-beam specimen to the empty-compartment spectrum using Formula (1):

$$A = -\log_{10}(P/P_0) \tag{1}$$

where

A is the absorbance of the specimen at a given wavelength;

P_0 is the empty specimen compartment light-beam intensity at that wavelength;

P is the single-beam specimen light-beam intensity at that wavelength.

5.2 Without a specimen shuttle

5.2.1 The data-acquisition parameters shall be:

— resolution: 2 cm^{-1};

— scans/scan time: background scans: 32 scans, 120 s total; specimen scans: 32 scans, 120 s total.

5.2.2 Establish a dry atmosphere inside the empty specimen compartment and collect the empty specimen compartment spectra.

5.2.3 Place the test specimen in the specimen compartment and re-establish a dry atmosphere inside the compartment. Collect the single-beam specimen spectra and calculate the specimen absorption spectrum as described in 5.1.3.

6 Calibration of the spectrophotometer

6.1 Obtain a series of known standards covering the diene content range 0 % to 10 %. Calibration may be based on secondary standards qualified by other laboratories using this method or, more generally, by primary standards whose diene content is well known. Primary standards may be established via use of proton nuclear magnetic resonance (^{1}H-NMR), in conjunction with other techniques. The ENB calibration standards[1] employed in the development of this method were determined by a combination of refractive index and H-NMR (utilizing samples dissolved in deuterated *o*-dichlorobenzene at 120 °C; the ENB assignment was based exclusively on the exocyclic olefinic protons of ENB). The DCPD standards[1] were determined by a similar H-NMR technique involving the cyclic olefinic protons of DCPD. The use of four standards at the (copolymer) 2 %, 5 % and 10 % levels is the minimum recommended.

6.2 Using the procedure in Clause 5, acquire a minimum of five absorbance spectra for each of the calibration standards described in 6.1. Several repetitions on separate specimens of each standard may be averaged to improve the accuracy of the calibration.

6.3 Using the procedure in Clause 7, calculate the ratio of the second-derivative diene peak height to the internal thickness gauge for each of the spectra acquired.

6.4 Calculate a linear calibration line (diene peak ratio versus assigned values of the standards as mass fraction in per cent of diene) by computing a slope and intercept using standard least-squares linear-regression techniques.

7 Diene determination

7.1 Preparation

Prepare the specimen film as described in Clause 4.

7.2 Single absorbance spectrum

Collect a single absorbance spectrum based on the procedure in Clause 5.

7.3 Determination of film thickness

7.3.1 Normalize the spectrum by bringing the lowest point of the spectrum to zero (that is, determine the minimum absorbance in the spectrum and offset the spectrum to bring the minimum absorbance to zero).

7.3.2 To determine the film thickness automatically, subtract the absorbance at 2 750 cm^{-1} from the absorbance at 2 703 cm^{-1}. If the result is positive, the sample belongs to group 1. Otherwise, it belongs to group 2 (see Figure 1).

Group 1: the thickness gauge is the net absorbance difference between 2 708 cm^{-1} (isooptic point) and 2 450 cm^{-1} (anchor point) (see Figure 2).

Group 2: the thickness gauge is the net absorbance difference between 2 668 cm^{-1} (isooptic point) and 2 450 cm^{-1} (anchor point) (see Figure 3).

7.4 Determination of diene content

7.4.1 Normalize the total spectrum to one optical density (OD) unit by multiplying the total spectrum by $1/A$, where A is the net absorbance at thickness gauge.

7.4.2 Calculate the ENB content, as a mass fraction, in per cent, using Formula (2):

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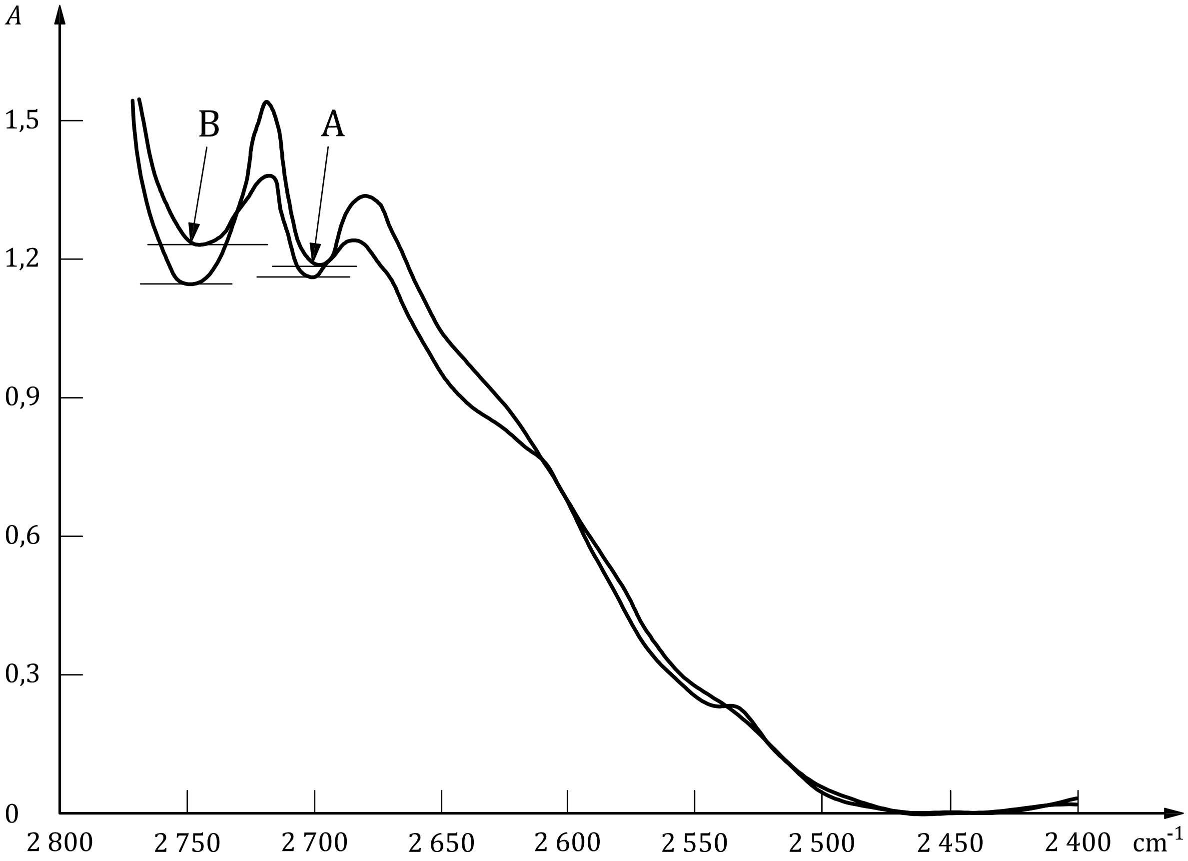
[1] The ENB standards are supplied by Exxon Chemical Polymer Laboratories (the sole source), P.O. Box 5200, Baytown, TX 77522, USA. DCPD standards are supplied by Uniroyal Chemical Company (the sole source), World Headquarters, Chemical Characterization Laboratory, Benson Road, Middlebury, CT 06749, USA. This information is given for the convenience of users of this document and does not constitute an endorsement by ISO of the products/standards named. Equivalent products/standards may be used if they can be shown to lead to the same results.

$$\text{Peak ht} = A_{1\,681} - (0{,}75A_{1\,688} + 0{,}25A_{1\,689}) \qquad (2)$$

This is subsequently referred to as the ENB peak height. Figure 4 shows a typical second-derivative spectrum for EPDM.

Use the calibration procedure described in Clause 6 to compute a mass fraction of ENB for the sample, using only interpolation (and not extrapolation). If the ENB peak height is lower than the ENB peak height of the lowest, or higher than the ENB peak height of the highest, calibration standard, then report the ENB content as out of range for the calibration standards employed.



If $A_{\text{Point A}} - A_{\text{Point B}} > 0$, the sample belongs to group 1.

If $A_{\text{Point A}} - A_{\text{Point B}} < 0$, the sample belongs to group 2.

Figure 1 — FT-IR thickness gauge — Determination of group

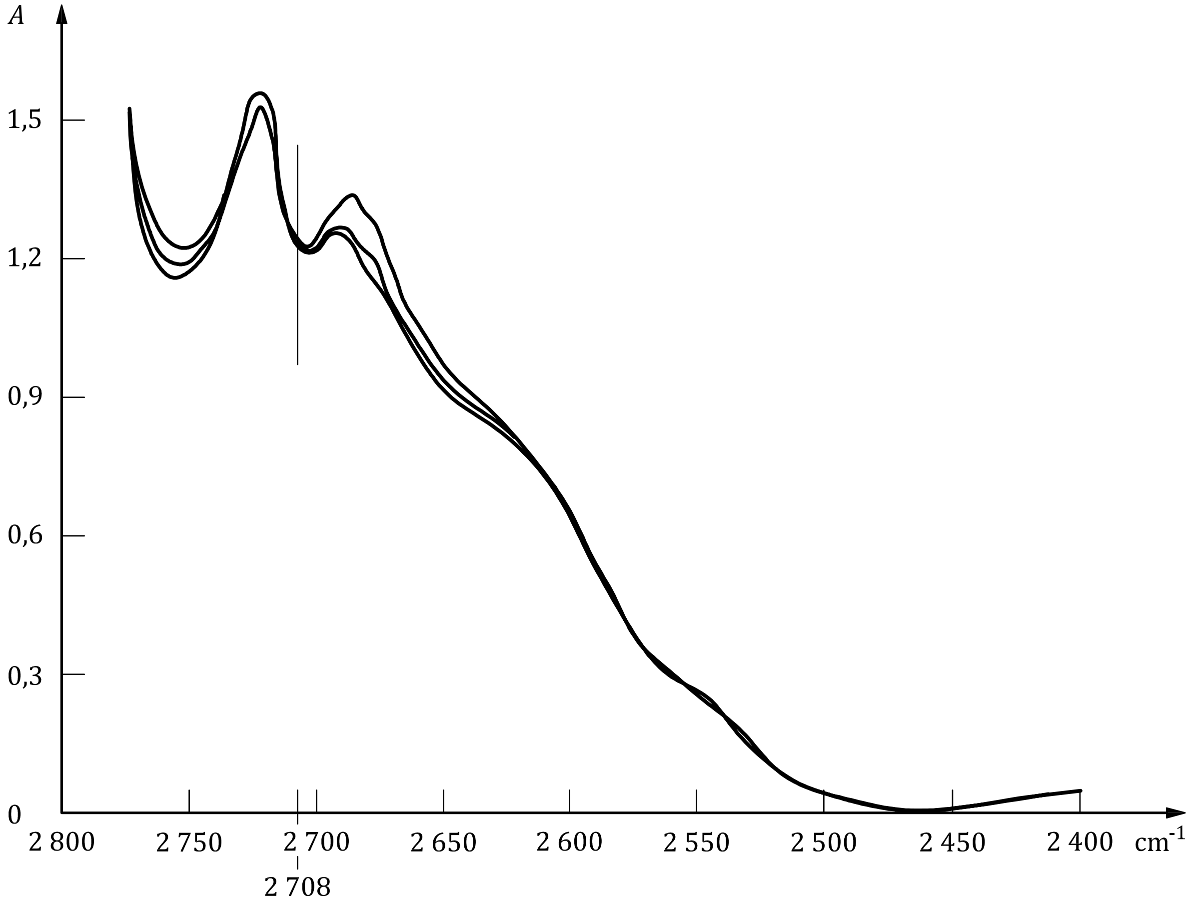


Figure 2 — FT-IR thickness gauge — Group 1

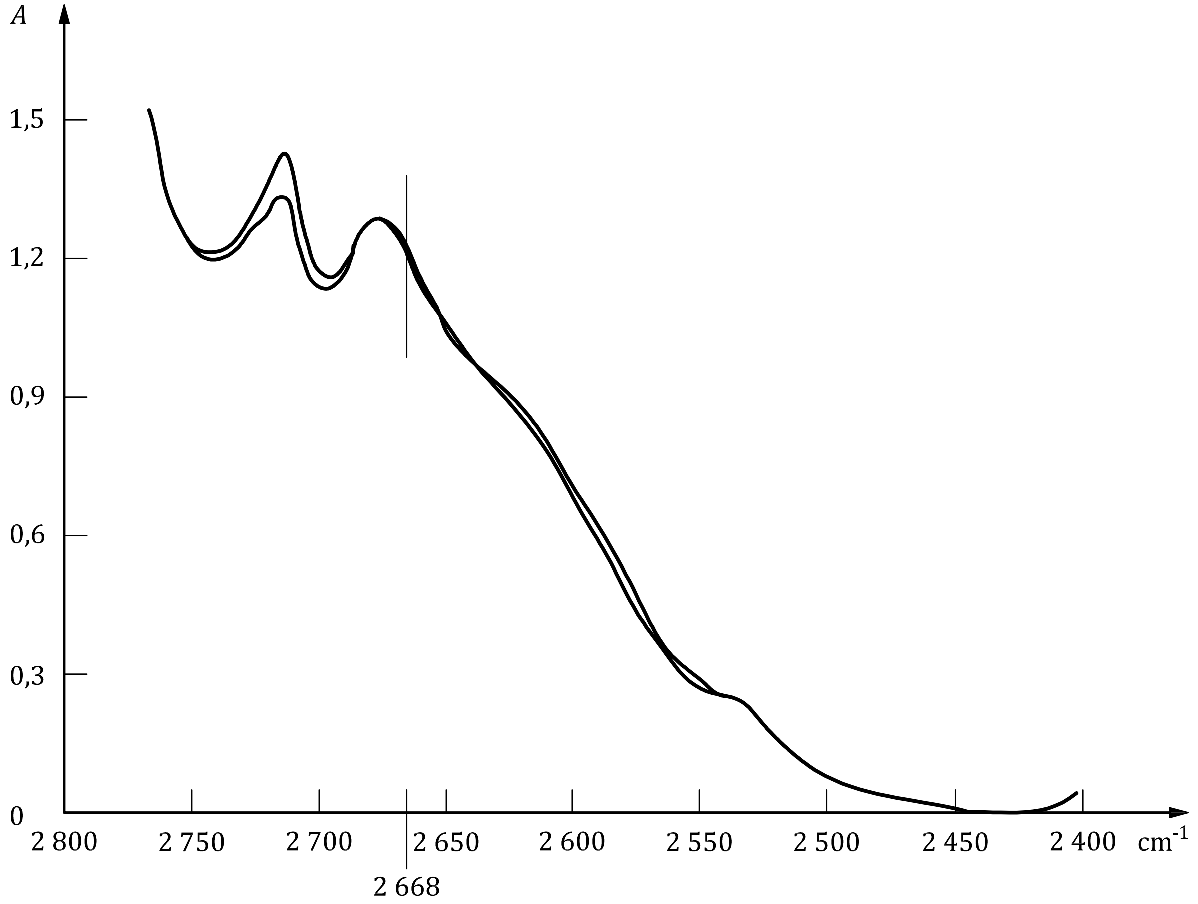
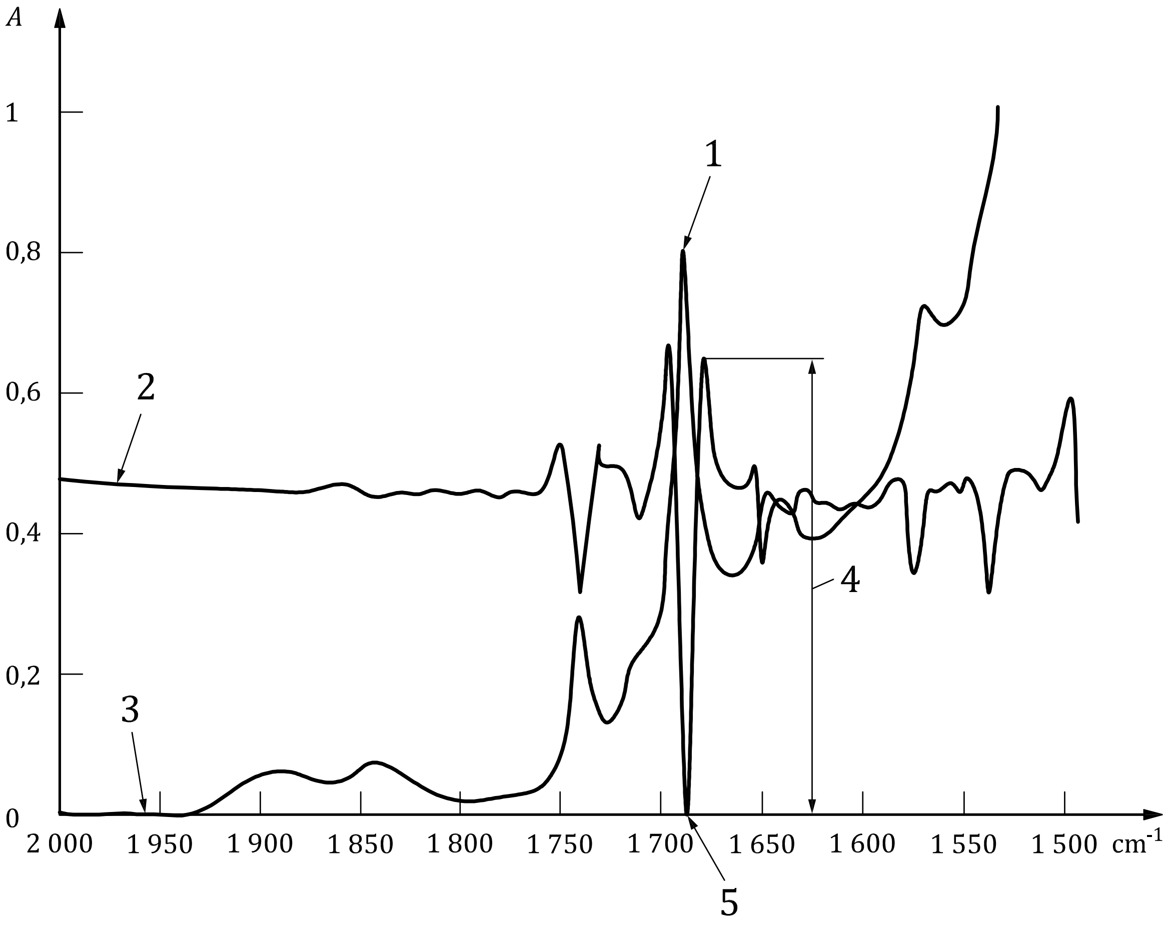


Figure 3 — FT-IR thickness gauge — Group 2



Key

1	peak for 1 690 cm^{-1} double bond
2	SD of normal spectrum
3	V 5600 normal spectrum
4	SD peak height
5	SD = 0 at peak

Figure 4 — Second-derivative (SD) peak height calculation

7.4.3 Calculate the DCPD content, as a mass fraction, as the peak height (in OD units) of the second derivative (the second-derivative algorithm shall use nine-point smoothing) between 1 601 cm^{-1} and 1 620 cm^{-1}, using Formula (3):

$$\text{Peak ht} = A_{1\,601} - A_{1\,610} \tag{3}$$

This is subsequently referred to as the DCPD peak height.

Use the calibration procedure described in Clause 6 to compute a mass fraction of DCPD for the sample, using only interpolation (and not extrapolation). If the DCPD peak height is lower than the DCPD peak height of the lowest, or higher than the DCPD peak height of the highest, calibration standard, then report the DCPD content as out of range for the calibration standards employed.

8 Precision

See Annex B.

9 Test report

The test report shall include the following information:

a) a reference to this International Standard, i.e. ISO 16565;

b) the ENB content and the DCPB content, as a mass fraction in per cent, giving the result to two decimal places in each case;

c) the type of mould utilized;

d) whether spectra were acquired with or without a specimen shuttle;

e) the date of the analysis.

Annex A
(normative)

Procedure for spectral subtraction of water vapour

A.1 Background

A.1.1 Water vapour and perhaps other ambient-air contaminants have the potential to affect the results of the ENB analysis. Even with a dry-gas purge (using air or nitrogen) of the specimen compartment, some residual water vapour can remain and affect the collected spectra. The procedure, as written, removes most or all of this effect by the use of a specimen shuttle, which permits single-beam specimen and background (empty specimen compartment) spectra to be acquired in alternating fashion, while maintaining the specimen compartment closed throughout the process. When the specimen absorbance spectrum is computed, the effect of background contaminants will be negligible, because they have been present in virtually identical amounts in the specimen and background spectra, thereby cancelling out in the calculation.

A.1.2 This method of removing the interference of background contaminants from the absorbance spectrum cannot be exactly duplicated in an FT-IR spectrophotometer that is not equipped with a specimen shuttle. It is necessary to open the specimen compartment to introduce or remove the specimen from the beam, which allows varying amounts of background contaminants to intrude each time the specimen compartment is open.

A.1.3 Fortunately, water vapour is the only contaminant present in sufficient quantity, and with significant light absorbance near the applicable wavelengths, to affect the analysis. Thus, without the specimen shuttle, it is possible to remove the contribution of water to specimen or background spectra and to use a sorted background spectrum with one or more specimen spectra. The following procedure outlines the steps required to implement such a modification.

A.2 Acquisition of water vapour absorbance spectrum

A.2.1 Acquire a single-beam background spectrum, P_0, with the system fully purged. This will be called the single-beam empty-compartment spectrum.

A.2.2 Acquire a single-beam spectrum, P_w, immediately after opening and closing the specimen compartment cover (still with no polymer film introduced). This will be called the single-beam water spectrum.

A.2.3 Calculate the water absorbance spectrum as given by Formula (A.1):

$$A_\mathrm{w} = -\log_{10}(P_\mathrm{w} / P_0) \tag{A.1}$$

A.3 Use of the single-beam empty-compartment spectrum

A.3.1 Ideally, a single-beam empty-compartment spectrum (see A.2.1) should be acquired before analysing each polymer specimen and stored. In practice, a single stored single-beam empty-compartment spectrum may be used for a short period of time (typically < 4 h) if FT-IR spectrophotometer operation is stable.

A.3.2 When introducing a polymer specimen film into the specimen compartment, impose a waiting period to allow the compartment purge to be re-established. This will minimize the amount of water remaining in the compartment. Determine this waiting time empirically (it could be up to 15 min).

A.3.3 Collect the single-beam spectrum for the polymer specimen and compute the absorbance spectrum, *A*, using the stored single-beam empty-compartment spectrum (see A.3.1).

A.3.4 Analyse the polymer absorbance spectrum for the presence of water. If detected, either as positive peaks (more water when polymer scanned) or negative peaks (more water when background scanned), perform a spectral subtraction or addition to remove the water vapour peaks from the spectrum before the ENB content is calculated. Do this as follows.

a) Determine a band or set of bands for the water vapour that can be used to calculate the scaling for the spectral subtraction. Use peaks that are sufficiently strong but well removed from polymer absorptions. (Note that such peaks have not been determined in the development of this procedure.)

b) Calculate the heights of these peaks in the polymer spectrum and ratio them to the heights of the peaks in the stored water absorbance spectrum (see A.2.3) to calculate a scaling factor *S*.

c) Calculate the water-vapour-free spectrum A^* as:

$$A^* = A(1 - SA_{\mathrm{w}}) \tag{A.2}$$

Use this modified absorbance spectrum, A^*, in place of the polymer absorbance spectrum, *A*, described in 6.2 in all subsequent calculations.

Annex B
(informative)

Precision and bias

B.1 The precision and bias data given in this annex were obtained for the primary method (see 4.1) in accordance with ISO/TR 9272. This gives terminology and other statistical calculation details.

B.2 The precision results presented in this annex give an estimate of the precision of the test method with the materials (rubbers, etc.) used in the particular interlaboratory programme described in B.3 and B.4. The precision parameters shall not be used for acceptance or rejection testing of any group of materials without documentation that the parameters are applicable to the particular group of materials and the specific testing protocol of the test method used.

B.3 A type 1 interlaboratory test programme was conducted in 1996. Five materials (polymers) with different levels of ENB were supplied to three laboratories. In each laboratory, two replicate tests were conducted on each of two successive test days. A test result is defined as a single measurement of the mass percentage of ENB.

B.4 A preliminary analysis to determine if the combined between-replicate/between-day variance was significantly greater than the between-replicate (on one day) variance showed that the between-replicate/between-day variance was not greater than the between-replicate variance. This demonstrates that, in this programme, testing on successive days did not add a significant source of variation over and above that inherent in duplicate testing on one day. The repeatability and reproducibility results generated by the programme therefore apply to short-term replicate testing.

B.5 The precision results for the ENB grades tested are given in Table B.1 (see also the footnotes to the table).

NOTE At the time of publication of this International Standard, precision and bias testing for DCPD is being undertaken.

Precision can be expressed in the form of the following statements that use an appropriate value of *r* and *R* or (*r*) and (*R*) associated with a mean level or material in the table closest to the mean level under consideration for any material in routine testing operations.

B.6 The **repeatability limit** *r* (expressed as a mass fraction in per cent) of this test method has been established as the appropriate value tabulated in Table B.1. Two single test results, obtained under normal test method procedures that differ by more than the tabulated value of *r* (for any given level) shall be considered as derived from different or non-identical sample populations.

B.7 The **reproducibility limit** *R* (expressed as a mass fraction in per cent) of this test method has been established as the appropriate value tabulated in Table B.1. Two single test results, obtained in two different laboratories under normal test method procedures that differ by more than the tabulated value of *R* (for any given level) shall be considered to have come from different or non-identical sample populations.

B.8 The relative repeatability, *r*, and the relative reproducibility, *R*, are approximately equal over the range 3,0 % to 11,0 % (actual) ENB, at 2,6 % (relative) and 11,0 % (relative), respectively (see pooled values in Table B.1).

B.9 In test method terminology, bias is the difference between an average test value and a reference (or true) test property value. Reference values do not exist for this test method since the value (of the test property) is exclusively defined by the test method. Bias, therefore, cannot be determined.

Table B.1 — Type 1 precision results for mass fraction of ENB

Material	Average ENB content	s_r	r	(r)	s_R	R	(R)
ENB003	0,08	0,110	0,311	—[a]	0,137	0,388	—[a]
ENB005	3,70	0,030	0,083	2,26	0,167	0,473	12,77
ENB002	4,99	0,059	0,168	3,36	0,171	0,484	9,70
ENB004	6,78	0,062	0,177	2,60	0,259	0,733	10,81
ENB001	10,92	0,066	0,186	1,70	0,404	1,143	10,47
Pooled values	—[b]	—	0,159	2,60	—	0,758	11,00

where

s_r is the repeatability standard deviation;
s_R is the reproducibility standard deviation;
r is the repeatability limit, in measurement units;
R is the reproducibility limit, in measurement units;
(r) is the repeatability (relative), in per cent;
(R) is the reproducibility (relative), in per cent.

[a] Relative precision is not meaningful for very low ENB contents.

[b] ENB003 is excluded.

Bibliography

[1] Specification for Evaluation of Research Quality Analysis of Infrared Spectra. *Analytical Chemistry, ANCHA*, 47, No. 11, p. 94a

[2] ISO/TR 9272, *Rubber and rubber products — Determination of precision for test method standards*

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