Designation: D6047 – 15

Standard Test Methods for Rubber, Raw—Determination of 5-Ethylidenenorbornene (ENB) or Dicyclopentadiene (DCPD) in Ethylene-Propylene-Diene (EPDM) Terpolymers[1]

This standard is issued under the fixed designation D6047; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 These test methods cover the determination of the content of 5-ethylidenenorbornene (ENB) or Dicyclopentadiene (DCPD) in ethylene-propylene-diene (EPDM) terpolymers. They are applicable to diene contents in the 0.1 to 10 mass % range.

1.2 ENB and DCPD are dienes introduced in ethylene/propylene rubbers to generate specific cure properties. Since high precision for diene content determination has become very important, a Fourier Transform Infrared Spectroscopic (FTIR) method was developed. Diene determination was performed in the past by a refractive index technique.

NOTE 1—The procedures for % ENB and % DCPD differ only in the location in the infrared (IR) of the IR peak being quantified.

1.3 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.4 *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 determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D297 Test Methods for Rubber Products—Chemical Analysis

D1416 Test Methods for Rubber from Synthetic Sources—Chemical Analysis (Withdrawn 1996)[3]

D3568 Test Methods for Rubber—Evaluation of EPDM (Ethylene Propylene Diene Terpolymers) Including Mixtures With Oil

D3900 Test Methods for Rubber—Determination of Ethylene Units in Ethylene-Propylene Copolymers (EPM) and in Ethylene-Propylene-Diene Terpolymers (EPDM) by Infrared Spectrometry

D4483 Practice for Evaluating Precision for Test Method Standards in the Rubber and Carbon Black Manufacturing Industries

E168 Practices for General Techniques of Infrared Quantitative Analysis (Withdrawn 2015)[3]

E932 Practice for Describing and Measuring Performance of Dispersive Infrared Spectrometers

E1421 Practice for Describing and Measuring Performance of Fourier Transform Mid-Infrared (FT-MIR) Spectrometers: Level Zero and Level One Tests

2.2 *ANCHA Document:*[4]

Specification for Evaluation of Research Quality Analysis of Infrared Spectra

3. Summary of Test Methods

3.1 The test specimen is molded between two PTFE-coated aluminum or Mylar sheets. The ENB content is determined from its infrared absorbance at 1681–1690 cm^{-1}, a measure of ENB's exocyclic double bond. The DCPD content is determined from its infrared absorbance at 1605–1610 cm^{-1}, a measure of DCPD's monocyclic double bond.

3.2 The second derivative of the absorbance is calculated and ratioed to an internal thickness gage. For ENB the resulting second derivative peak near 1690 cm^{-1} is related to ENB mass fraction by calibrating the instrument with known EPDM standards. For DCPD, the resulting second derivative peak near 1610 cm^{-1} is related to DCPD mass fraction by calibrating the instrument with known EPDM standards.

3.3 Two main steps are performed in this procedure: automatic determination of film thickness and quantification of diene.

[1] These test methods are under the jurisdiction of Committee D11 on Rubber and are the direct responsibility of Subcommittee D11.11 on Chemical Analysis.

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[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

[3] The last approved version of this historical standard is referenced on www.astm.org.

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

3.4 For oil-extended polymers, the oil must be extracted before diene is determined. Test Methods D1416, Sections 67 through 74 and Test Methods D297 can be used for this purpose.

4. Apparatus

4.1 *Press,* Carver-type, capable of pressing films at 150°C and 10 MPa or higher.

4.1.1 *Primary Mold*—A stainless steel (SS) mold, approximately 400 µm thick, with an opening sized appropriately for the specimen film holder described in 4.4 (typically 2 by 2 cm). The mold should have approximately the same dimensions as the press platens.

4.1.2 *Alternate Mold*—A thinner mold may be used; however, precision may be adversely affected at low diene levels. Test precision should be determined when thinner films are utilized. Example: a 127-µm (0.005-in) SS shim with a 15- by 35-mm opening may be used for the simultaneous determination of ethylene (Test Methods D3900) and diene.

4.1.3 *Alternative Mold for Low Molecular Weight (Liquid) EPDM Terpolymers*—For liquid EPDM a ring washer 22 mm OD by 16 mm ID (7/8 in OD by 5/8 inch in ID) 400 µm thick is used as a apacer between salt plates (NaBr, NaCl) to set a fixed path length. The spacer is sized to cover only the outer edge of the salt plate.

4.2 *Sheets for Molding,* either PTFE-coated aluminum sheets or Mylar, Type A, 36 µm thick or less.

4.3 *Specimen Film Holders*—Films may be molded, cut, and transferred to a film holder. Magnetic film holders are ideal. Alternatively, a mold sized to fit in the spectrometer specimen compartment, with an appropriate size opening may be used to support the film without removal after pressing.

4.4 *Infrared Fourier Transform Spectrophotometer*—An instrument capable of measuring absorbances in the range 400 to 6000 cm^{-1} with a transmittance specification (accuracy) of ± 1 % *T* or better. The instrument should be capable of spectral resolution of 2 cm^{-1} (see "Specification for Evaluation of Research Quality Analysis of Infrared Spectra"). A deuterated triglycine sulfate (DTGS) detector should be used. The instrument must be operating in accordance with Practices E168. Practices E932 and E1421 are other important references.

4.4.1 The instrument should be capable of spectral accumulation, averaging, and subtracting capabilities. Water is the primary source of interference in this method. Methods, physical and electronic, that minimize moisture level and variation are required to obtain the highest precision. The preferred method is use of an instrument equipped with a dry gas purge and a specimen shuttle, which permits alternating and repetitive collection of single beam specimen and background spectra (see Section 6). Alternatively, should a specimen shuttle be unavailable, careful purging of the sample 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 vapor may be used. A procedure for further method development is described in Appendix X1.

5. Test Specimen Preparation

5.1 *Primary Method*—Place an appropriate amount of the test specimen to fill the mold opening (typically 0.2 to 0.5 g) between two PTFE-coated aluminum or Mylar sheets in the mold. Place the mold between the press platens heated to 125 ± 5°C and apply approximately 10 MPa or higher pressure for 60 ± 10 s.

NOTE 2—High molecular weight polymers may require higher temperatures and pressures to obtain a good test specimen.

5.1.1 Cool the specimen to ambient temperature. Cut, if necessary, a piece of film to the appropriate size to cover the specimen holder window. Detach the specimen film from the aluminum or Mylar and position it on the spectrophotometer sample holder window.

5.2 *Alternate Method*—When using the thinner mold described in 4.1.2, place a small piece (0.04–0.06 g) of test specimen in the mold opening between two Mylar sheets and press as in 5.1. Remove the mold from the press, turn it over and press it again, then remove the mold from the press and cool it to ambient temperature. When cool, carefully remove the Mylar sheets, allowing the specimen film to remain attached to the mold.

5.3 *Alternate Method for Liquid Sample Film Preparation*—Place a washer (described in 4.1.3) on top of a salt plate. Place a small amount (about 0.3 g) of liquid EPDM polymer in the center 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 to 3 min. (For viscous samples it may be necessary to warm the sample prior to pressing.) Remove the weight and allow to cool, if necessary. Wipe off any excess material that may have been pressed out of the assembly. Hold the assembly up to the light and inspect for bubbles or voids, or both. Should there be imperfections, repeat the sample preparation with a larger amount of sample.

6. Spectral Acquisition

6.1 *With a specimen shuttle:*

6.1.1 *Data acquisition parameters:*

6.1.1.1 Resolution: 2 cm^{-1}.

6.1.1.2 Scans/Scan time: Total scan time required, split between specimen and background, is about 90 s.

6.1.2 Place the test specimen in the specimen compartment, allow purge to reestablish, and in alternating fashion, collect single beam specimen (*P*) and "empty specimen compartment" (P_0) spectra. Eight passes of the shuttle should be sufficient (eight specimen and eight empty compartment collections), collecting four scans at each position.

6.1.3 Calculate the specimen absorbance spectrum as minus the $\log_{10}$ of the ratio of the accumulated single beam specimen spectrum to the single beam empty specimen compartment spectrum:

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

6.2 *Without a specimen shuttle:*

6.2.1 *Data acquisition parameters:*

6.2.1.1 Resolution: 2 cm^{-1}.

6.2.1.2 Scans/Scan Time: Background scan: 32 scans, 20 s total; specimen scan: 32 scans, 20 s total.

6.2.2 Establish dry atmosphere inside empty specimen compartment and collect "empty specimen compartment" (P_0) spectra.

6.2.3 Place test specimen in the specimen compartment and re-establish a dry atmosphere inside the specimen compartment. Collect single beam specimen spectra (P) and calculate the specimen absorption spectra (A) as described in 6.1.3.

7. Calibration of the Spectrophotometer

7.1 Obtain a series of known standards covering the 0–10 mass % diene range. 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 ^{1}H Nuclear Magnetic Resonance (NMR), in conjunction with other techniques. The calibration standards employed in the development of this method were determined by a combination of refractive index and ^{1}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 use of four standards at the 0 (copolymer), 2, 5, and 10 mass % levels are the minimum recommended.[5]

7.2 Using the procedures in Section 6, acquire a minimum of five absorbance spectra for each of the calibration standards described in 7.1. Several repetitions on separate specimens of each standard may be averaged to improve the accuracy of the calibration.

7.3 Using the procedures in Section 8, calculate the ratio of the second derivative diene peak height to the internal thickness gage for each of the spectra acquired.

7.4 Calculate a linear calibration line (diene peak ratio versus assigned values of the standards in mass % diene) by computing a slope and intercept using standard least squares linear regression techniques.

8. Diene Determination

8.1 Prepare the specimen film as described in Section 5.

8.2 Collect a single absorbance spectrum based on the procedure in Section 6.

8.3 *Determination of Film Thickness:*

8.3.1 Correct the offset of 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 absorbance to zero).

8.3.2 To determine the film thickness automatically, calculate the difference of the absorbance at the basepoint near 2703 cm^{-1} minus the absorbance at the basepoint near 2750 cm^{-1}. If the net difference is positive, the sample belongs to Group 1. Otherwise, it belongs to Group 2 (see Fig. 1).

8.3.2.1 Group 1: Thickness gage is the net absorbance difference between 2708 cm^{-1} (isooptic point) and 2450 cm^{-1} (anchor point) (see Fig. 2).

8.3.2.2 Group 2: Thickness gage is the net absorbance difference between 2668 cm^{-1} (isooptic point) and 2450 cm^{-1} (anchor point) (see Fig. 3).

8.4 *Diene Quantitation:*

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

8.4.2 *ENB Calculation*—Calculate the peak height (in OD) of the second derivative (the second derivative algorithm should use at least nine point smoothing) between 1681 and 1690 cm^{-1} by applying the following formula:

$$\text{Peak Ht} = A_{1681} - (0.75\,A_{1688} + 0.25\,A_{1689}) \quad (2)$$

This will be called the ENB peak height throughout this procedure. Fig. 4 gives a visual impression of a typical second derivative spectrum of EPDM.

8.4.3 Use the calibration developed in Section 8.4.2 to compute a mass % ENB for the sample, employing the principle that only interpolation (and not extrapolation) is used. If the ENB peak height determined in 8.4.5 is lower than the ENB peak height of the lowest, or higher than the ENB peak height of the highest calibration standard, then the ENB mass % should be reported as "out of range for the calibration employed."

8.4.4 *DCPD Calculation*—Calculate the peak height (in OD) of the second derivative (the second derivative algorithm should use nine point smoothing) between 1601 and 1620 cm^{-1} by applying the following formula:

$$\textit{Peak Ht} = (A_{1601} - A_{1610}) \quad (3)$$

This will be called the DCPD peak height throughout this procedure.

8.4.5 Use the calibration developed in Section 8.4.4 to compute a mass % DCPD for the sample, employing the principle that only interpolation (and not extrapolation) is used. If the DCPD peak height determined in 8.4.5 is lower than the DCPD peak height of the lowest, or higher than the DCPD peak height of the highest calibration standard, then the DCPD mass % should be reported as "out of range for the calibration employed."

9. Report

9.1 Report the following information:

9.1.1 ENB content to two decimal places:

ENB = xx.xx mass %

DCPD = xx.xx mass %

9.1.2 Type of mold utilized,

9.1.3 Whether spectra were acquired with or without a specimen shuttle.

[5] The sole source of supply for the ENB standards known to the committee at this time is ExxonMobil Chemical Polymer Laboratories, P.O. Box 5200, Baytown, TX 77520. The sole source of supply for the DCPD standards known to the committee at this time is UniRoyal Chemical Company, Chemical Characterization Lab, Benson Road, Middlebury, CT 06749. If you are aware of alternative suppliers, please provide this information to ASTM Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee,[1] which you may attend.

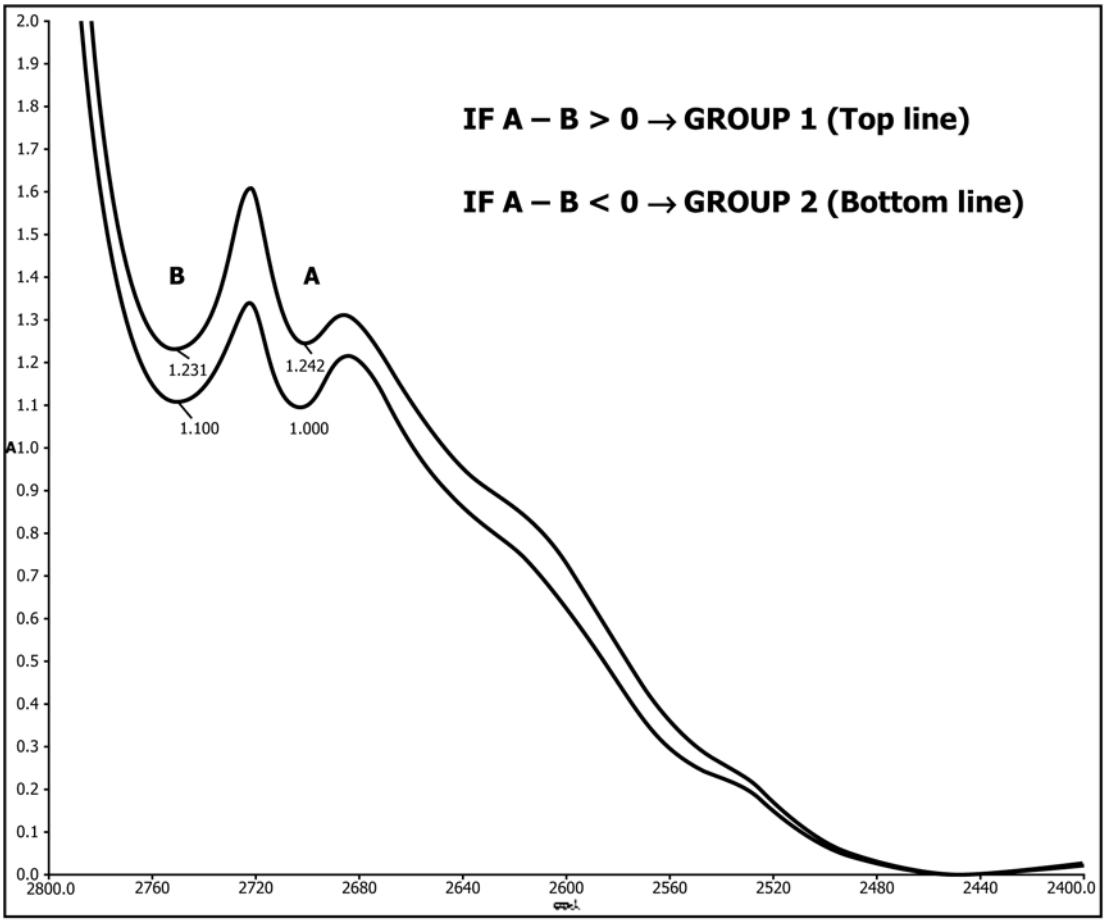

FIG. 1 FT-IR Thickness Gage—Group Determination

10. Precision and Bias for ENB[6]

10.1 This precision and bias section for the primary method has been prepared in accordance with Practice D4483. Please refer to this practice for terminology and other statistical calculation details.

10.2 The precision results in this precision and bias section give an estimate of the precision of these test methods with the materials (rubbers, etc.) used in the particular interlaboratory program as described in 10.5 through 10.8. The precision parameters should 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 protocols of the test methods.

10.3 A Type 1 interlaboratory test program (ITP) 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 percent of ENB.

10.4 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 program, testing on successive days does not add a significant source of variation above that inherent in duplicate testing on one day. The repeatability and reproducibility results as generated by this program therefore apply to short-term replicate testing.

10.5 The precision results are given in Table 1; see footnotes at the bottom of the table. Precision may be expressed in the format 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.

10.6 *Repeatability*—The repeatability, r, (in mass % ENB) of these test methods has been established as the appropriate value tabulated in Table 1. Two single test results, obtained under normal test method procedures, that differ by more than this tabulated r (for any given level) must be considered as derived from different or nonidentical sample populations.

[6] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D11-1081.

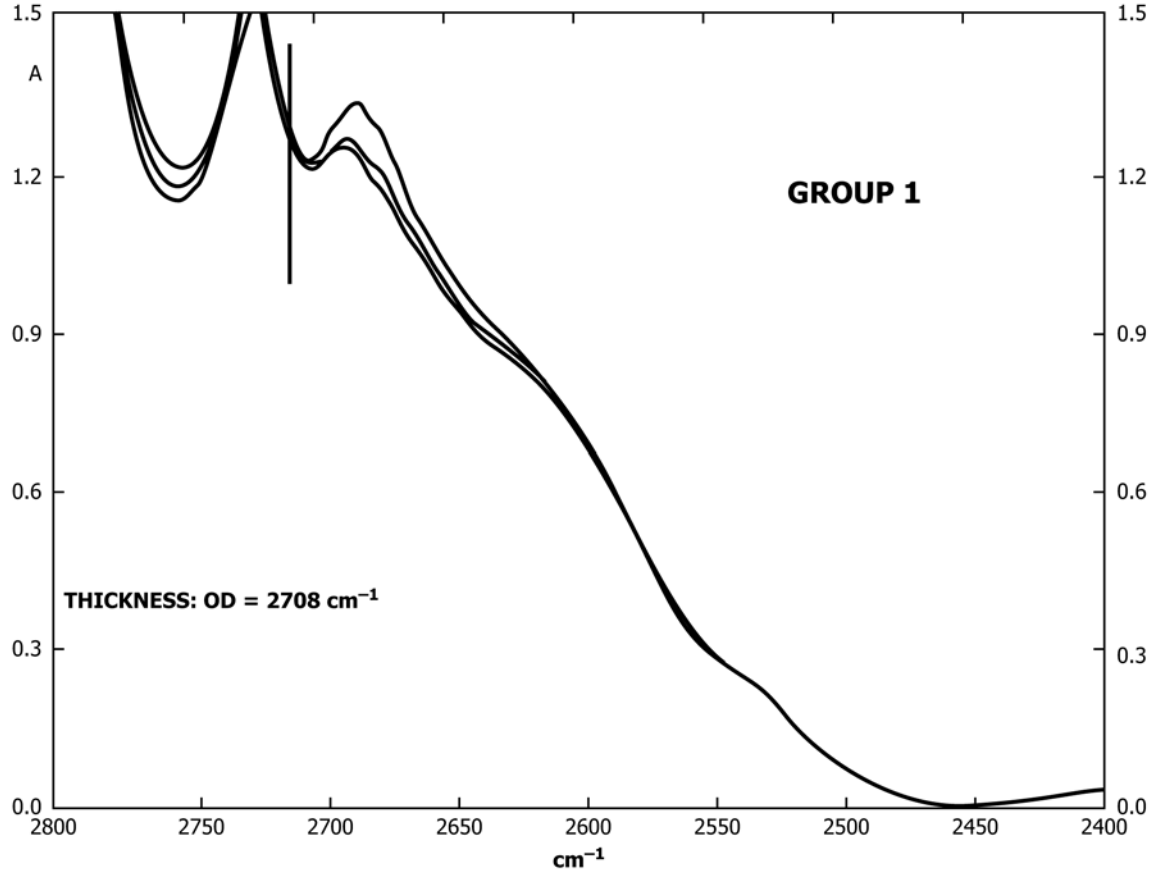

FIG. 2 FT-IR Thickness Gage—Group 1

10.7 *Reproducibility*—The reproducibility, *R*, (in mass % ENB) of these test methods has been established as the appropriate value tabulated in Table 1. Two single test results obtained in two different laboratories, under normal test method procedures, that differ by more than the tabulated *R* (for any given level) must be considered to have come from different or nonidentical sample populations.

10.8 *Relative Precision*—The relative repeatability (*r*) and the relative reproducibility (*R*) are approximately equal over the range of 3.0 to 11.0 actual percent ENB, at 2.6 and 11.0 (relative) percent, respectively. (See pooled values in Table 1.)

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

10.10 Precision and bias testing for DCPD is presently being established.

11. Precision and Bias for DCPD[7]

11.1 This precision and bias section has been prepared in accordance with Practice D4483. Please refer to this practice for terminology and other statistical calculation details.

11.2 The precision results in this precision and bias section give an estimate of the precision of this test method with the materials (rubbers, etc.) used in the particular interlaboratory test program (ITP) as described below. The precision parameters should 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 protocols of the test method.

11.3 A Type 1 interlaboratory test program was conducted in 1998 on three materials. (A, B, C) with different levels of DCPD. Seven laboratories participated in the ITP conducting duplicate tests on each of 2 successive test days. A test result is the average of two measurements. Prior to the standard D4483 precision analysis, a preliminary spreadsheet analysis was conducted to determine if the variance between duplicate tests on the same day was significantly different (smaller) than the variance for the difference between Day 1 vs Day 2. It was found that these two variances are equal at the 95% confidence level.

11.4 *Repeatability*—The repeatability, *r*, of this test method has been established as the value tabulated in Table 2 for each material. Two single test results, obtained under normal test method procedures, that differ by more than this tabulated *r* (for any given level) must be considered as derived from different or nonidentical sample populations.

11.5 *Reproducibility*—The reproducibility, *R*, of this test method has been established as the value tabulated in Table 2

[7] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D11-1088.

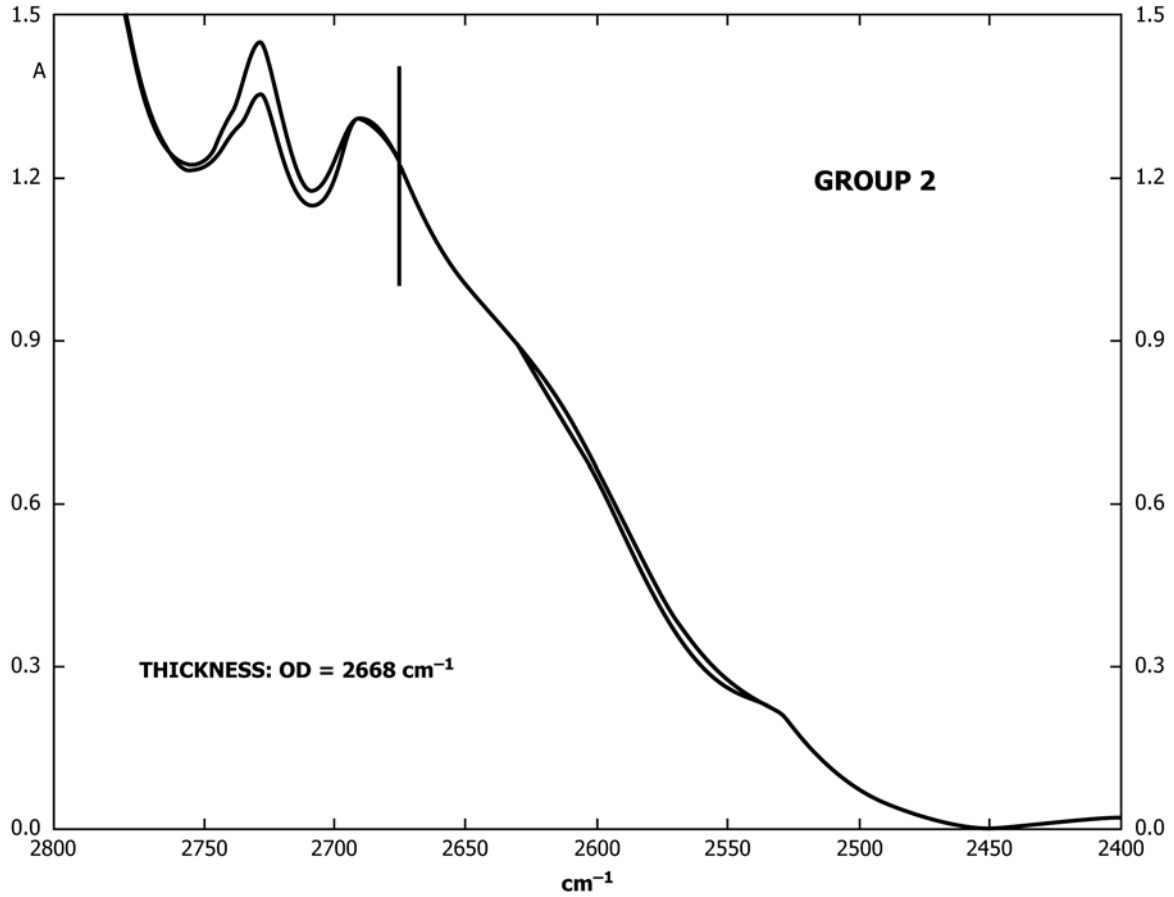

FIG. 3 FT-IR Thickness Gage—Group 2

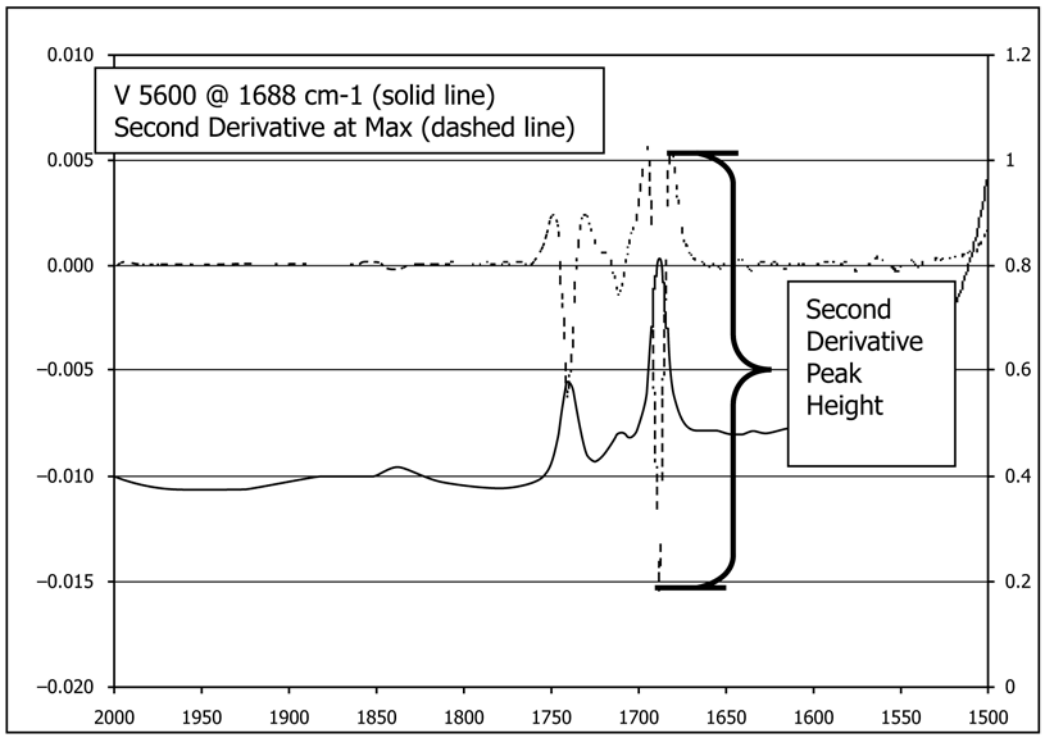

FIG. 4 Second Derivative Peak Height Calculation

TABLE 1 Type 1 Precision Results for Mass % ENB

NOTE 1—Only three laboratories participated in the ITP.

NOTE 2—
Sr = repeatability standard deviation.
SR = reproducibility standard deviation.
r = repeatability, in measurement units.
R = reproducibility, in measurement units.
(r) = repeatability (relative) in percent.
(R) = reproducibility (relative) in percent.

Material	Average	*Sr*	*r*	*(r)*	*SR*	*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	2.6	...	0.758	11.0

[A] Relative precision is not meaningful for very low % ENB.
[B] ENB003 excluded.

for each material. Two single test results obtained in two different laboratories, under normal test method procedures, that differ by more than the tabulated *R* must be considered to have come from different or nonidentical sample populations.

11.6 The relative repeatability and reproducibility, (*r*) and (*R*), are also given in Table 2. These precision parameters have the same applicability statements as given in 10.6 and 10.7.

TABLE 2 Precision (Type 1) for percent DCPD

NOTE 1—Only three laboratories participated in the ITP.

NOTE 2—
Sr = repeatability standard deviation.
SR = reproducibility standard deviation.
r = repeatability, in measurement units.
R = reproducibility, in measurement units.
(r) = repeatability (relative) in percent.
(R) = reproducibility (relative) in percent.

		WIthin Labs		Between Labs	
Material	Mean	*Sr*	*r*	*SR*	*R*
B	2.67	0.012	0.033	0.076	0.213
A	3.20	0.034	0.096	0.102	0.288
C	9.57	0.046	0.129	0.063	0.178

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

12. Keywords

12.1 Dicyclopentadiene (DCPD) (DCP); ethylene-propylene-diene-monomer (EPDM); ethylidenenorbornene (ENB)

APPENDIX

(Nonmandatory Information)

X1. DEVELOPMENTAL PROCEDURE FOR SPECTRAL SUBTRACTION OF WATER VAPOR

X1.1 Water vapor and perhaps other ambient air "contaminants" have the potential to affect the results of the diene analysis. Even with a dry gas purge (air or nitrogen) of the specimen compartment, some residual water vapor can remain and affect the collected spectra. As written, D6047 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 taken in alternating fashion, while maintaining the specimen compartment closed throughout the process. When the specimen absorbance spectrum is computed, the effect of background contaminants should be negligible, because they have been present in virtually identical amounts in the specimen and background spectra, thereby cancelling out in the calculation.

X1.2 This method of removing the interference of background contaminants from the absorbance spectrum cannot be exactly duplicated in an FTIR 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.

X1.3 Fortunately, water vapor 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 should be 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 development required to implement such a modification.

X1.3.1 Collect a water vapor absorbance spectrum.

X1.3.1.1 Collect a single-beam background spectrum (P_0) with the system fully purged. This will be called the single beam empty compartment spectrum.

X1.3.1.2 Collect 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.

X1.3.1.3 Calculate the water absorbance spectrum as:

$$A_w = -log_{10}(P_w/P_0) \quad \text{(X1.1)}$$

X1.4 Ideally, an "empty" single beam spectrum should be collected (see X1.3.1.1) before analyzing each polymer specimen and stored. In practice, a single stored "empty" spectrum

may be used for a short period of time (typically <4 hours) if the FTIR spectrophotometer operation is stable.

X1.5 When introducing a polymer specimen film into the sample compartment, impose a waiting period to allow the compartment purge to re-establish. This should minimize water remaining in the compartment. Determine this waiting time empirically. (It could require up to 15 minutes.)

X1.6 Collect the single beam spectrum of the polymer specimen and compute the absorbance spectrum (A), using the stored "empty" single beam spectrum (X1.4).

X1.7 Analyze 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 vapor peaks from the spectrum before the diene content is calculated. Do this as follows:

X1.7.1 Determine a band or set of bands for the water vapor that could 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.)

X1.7.2 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 (Paragraph X1.3.1.3) to calculate a scaling factor S.

X1.7.3 Calculate the "water-vapor-free" spectrum A^* as:

$$A^* = A\left(1 - S\,A_w\right) \quad \text{(X1.2)}$$

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