Designation: D3900 – 15

Standard Test Methods for Rubber—Determination of Ethylene Units in Ethylene-Propylene Copolymers (EPM) and in Ethylene-Propylene-Diene Terpolymers (EPDM) by Infrared Spectrometry[1]

This standard is issued under the fixed designation D3900; 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 proportion of ethylene and propylene units in ethylene-propylene copolymers (EPM) and ethylene-propylenediene terpolymers (EPDM) over the range from 35 to 85 mass % ethylene. Four test methods are needed to encompass the variety of commercial polymers that contain additives or polymerized diene units that interfere with the various infrared peaks. Except when interferences are present, all four test methods should give similar results. The test methods appear in the following order:

1.1.1 *Pressed Film Test Methods:*

	Sections
Test Method A—For EPM and EPDM between 35 and 70 mass % ethylene	9 – 14
Test Method B—For EPM and EPDM between 60 and 85 mass % ethylene, except for ethylene/propylene/1,4-hexadiene terpolymers	15 – 19
Test Method C—For all EPM and EPDM polymers between 35 and 85 mass % ethylene, using near infrared	20 – 24

1.1.2 *Cast Film Test Methods:*

Test Method D—For all EPM and EPDM polymers between 35 and 85 mass % ethylene, except for ethylene/propylene/1,4-hexadiene terpolymers	25 – 32

1.2 These test methods are not applicable to oil-extended EPDM unless the oil is first removed in accordance with Test Method D.

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

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

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]

3. Summary of Test Methods

3.1 *Test Method A*—Pressed films are measured for their infrared absorbance ratios at 8.65/13.85 µm (1156/722 cm^{-1}), and mass percent ethylene is read from a calibration obtained from standard polymers.

NOTE 1—Peak typically appears at the wavelength indicated, but peak may shift slightly in its position.

3.2 *Test Method B*—Thin pressed films are measured for their infrared absorbance ratios at or near 7.25/13.85 µm (1379/722 cm^{-1}), and mass percent ethylene is read from a calibration obtained from standard polymers.

3.3 *Test Method C*—Pressed films are measured for their infrared absorbance ratios at or near 8.65/2.35 µm (1156/4255 cm^{-1}) using near infrared, and mass percent ethylene is read from a calibration obtained from standard polymers.

3.4 *Test Method D*—Ultra-thin cast films on a salt plate are measured for their infrared absorbance ratios at or near 7.25/6.85 µm (1379/1460 cm^{-1}), and mass percent ethylene is read from a calibration obtained from standard polymers.

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

Current edition approved June 1, 2015. Published July 2015. Originally approved in 1980. Last previous edition approved in 2010 as D3900 – 05a (2010). DOI: 10.1520/D3900-15.

[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. Significance and Use

4.1 These test methods can be used for determining which EPDM polymers are evaluated in the different compounds in Test Methods D3568.

4.2 Differences in ethylene sequence distribution cause differences in crystallinity and green strength at the same ethylene content. Since these are important variables in EPM and EPDM processability and end-use properties, the ethylene content of the rubber should not be used as the sole measurement to determine the suitability of a particular rubber for an intended purpose.

5. Interferences

5.1 Ethylene/propylene/1,4-hexadiene EPDM has an interference at the 7.25-µm (1379-cm^{-1}) peak and should be measured by Test Method A or C.

5.2 Various commercial polymers have interferences due to additives and stabilizers that prevent or hinder the use of the 8.65-µm (1156-cm^{-1}) peak. Test Methods A and C should be applied carefully, or Test Methods B and D should be used as described in the procedure.

5.3 Extender oil, when present, will interfere with all determinations and must be removed before infrared analysis.

6. Apparatus

6.1 *Hydraulic Press,* capable of 200 MPa (29 000 psi) and 150°C.

6.2 *Infrared Spectrophotometer,* double-beam, having a percent transmission specification of ±1 %, or better, at full scale, capable of recording a spectrum over the 2.5 to 15-µm (4000 to 667-cm^{-1} or 400 000 to 66 700-m^{-1}) region for Test Methods A, B, and D. Test Method C requires an instrument capable of recording a spectrum over the 2.0 to 15-µm (2000 to 667-cm^{-1} or 200 000 to 66 700-m^{-1}) region. Any spectrophotometer complying with these requirements may be used. The equipment shall be operated by an experienced analyst according to the manufacturer's directions for optimum performance. Recommended practices for general techniques of infrared quantitative analysis are given in Practices E168.

6.3 For routine testing, Fourier Transform Infrared (FT-IR) may be used in place of double beam instruments provided the baseline calculation procedures in the Procedures and Calculation Sections of each method are followed. Sample film temperature is lower with FT-IR than double beam instruments, and some partly crystalline polymers may respond differently. Calibration equations (Section 33) may have different mathematical forms with FT-IR.

7. Sampling

7.1 Take precautions to ensure as representative a sample as possible for spectral analysis, since infrared absorption is additive in nature and will be influenced by extraneous materials.

7.2 Where possible, take the sample from a freshly cut surface to avoid testing of a partially oxidized polymer.

8. Precision and Bias[4]

8.1 This precision and bias section has been prepared in accordance with Practice D4483. Refer to Practice D4483 for terminology and other statistical calculation details.

8.2 The precision results in this precision and bias section give an estimate of the precision of these test methods with the materials (rubbers) used in the particular interlaboratory program as described below. The precision parameters should not be used for acceptance or rejection testing of any group of materials without documentation that they are applicable to those particular materials and the specific testing protocols that include these test methods.

8.3 A Type 1 (interlaboratory) precision was evaluated. Both repeatability and reproducibility are short term; a period of a few days separates replicate test results.

8.4 The precision of these test methods was determined from an interlaboratory study of several materials by several laboratories on two days as explained below:

Test Method	Ethylene Range, %	No. of Laboratories	No. of Materials
A	40–60	6	3
A	65–76	5	2
B	60–66	4	2
D (Nonoil—Extended Polymer)	40–70	4	4
D (Oil-Extended Polymer)	72	3	1

A test result is a single determination of percent ethylene.

8.5 The results of the precision calculations for repeatability and reproducibility are given in Table 1 for Test Methods A, B, and D.

NOTE 2—Insufficient data were generated for Test Method C to calculate a precision statement. However, extensive use of Test Method C within one laboratory suggests precision levels similar to the other test methods.

8.6 The precision of these test methods may be expressed in the format of the following statements, which use an *appropriate value* of *r*, *R*, (*r*), or (*R*), to be used in decisions about test results. The appropriate value is that value of *r* or *R* associated with a mean level in Table 1 closest to the mean level under consideration at any given time, for any given material, in routine testing operations.

8.7 *Repeatability*—The repeatability, *r*, 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.

8.8 *Reproducibility*—The reproducibility, *R*, 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.

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[4] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR: D11-1021.

TABLE 1 Type 1 Precision

NOTE 1—The midpoint of the range was used to calculate (*r*) and (*R*).

Test Method	Ethylene Range, %	Within Laboratories[A]			Between Laboratories[A]		
		S_r	*r*	(*r*)	S_R	*R*	(*R*)
A	40 to 60	0.569	1.61	3.22	0.857	2.43	4.86
A	65 to 76	0.471	1.33	1.90	1.74	4.92	7.0
B	60 to 66	0.433	1.23	1.95	0.540	1.53	2.43
D (Non OE)	40 to 70	0.856	2.42	4.40	2.11	5.97	10.9
D (OE)	72	2.12	6.00	8.3	3.55	10.0	13.9

[A] S_r = repeatability, standard deviation, in measurement units.
r = repeatability, in measurement units.
(*r*) = repeatability, (relative) percent.
S_R = reproducibility, standard deviation, in measurement units.
R = reproducibility, in measurement units.
(*R*) = reproducibility, (relative) percent.

8.9 Repeatability and reproducibility expressed as a percent of the mean level, (*r*) and (*R*), have equivalent application statements as above for *r* and *R*. For the (*r*) and (*R*) statements, the difference in the two single test results is expressed as a percent of the arithmetic mean of the two test results.

8.10 *Bias:*

8.10.1 Bias was established with a round-robin test conducted on six EPM standard polymers by the five U.S. EPM/EPDM manufacturers. Each manufacturer made eight determinations on each of the six standards, using his own infrared method and absorbance/ethylene calibration chart. Each individual calibration chart had been established with infrared tests of C_{14}-tagged pilot plant polymers or equivalent, with confirming tests such as NMR used where necessary.[5]

8.10.2 Ethylene contents of the following ten EPM standards were established via 13C NMR by a consortium of European and North American laboratories.

Standard No.	Mass Percent Ethylene[6]
1	40.1
2	52.4
3	58.6
4	66.8
5	70.8
6	78.6
7	44.8
8	52.6
9	69.5
10	77.5

8.10.3 The standards were each produced in a commercial plant and sizable quantities have been set aside for calibration purposes (see 13.2).

8.11 The precision of Test Method A was determined utilizing FTIR spectrometers from an interlaboratory study of five materials by nine laboratories on two days. The results of the precision calculations for repeatability and reproducibility are given in Table 2. The FTIR spectrometers utilized were calibrated using the percent ethylene values established by 13C NMR.

TEST METHOD A—PRESSED FILM METHOD USING THE 8.65/13.85-µm PEAK RATIO

9. Scope

9.1 This test method covers the determination of percent ethylene in EPM and EPDM between approximately 35 and 70 mass %.

9.2 This test method may be used with caution between approximately 60 and 80 mass % ethylene, but is inferior to Test Method B in precision with many instruments.

9.3 Additives in some commercial polymers will cause an additional absorbance on the shoulder of the 8.65-µm (1156-cm^{-1}) peak at approximately 8.93 µm (1120 cm^{-1}), giving difficulty in drawing the baseline. Where this occurs, Test Method B or D must be used.

9.4 This test method is not intended for oil-extended polymers unless the oil is first removed by extraction.

10. Summary of Test Method

10.1 This test method makes use of the ratio of the absorbance of methyl groups from propylene units at 8.65 µm (1156 cm^{-1}) versus the absorbance of methylene sequences from ethylene units at 13.85 µm (722 cm^{-1}). A series of known EPM polymers is used to prepare a calibration of $A_{8.65}/A_{13.85}$ versus mass percent ethylene.

11. Reagents and Materials

11.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available.[7] Other grades may be used, provided it is first ascertained that the reagent is of

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[5] Gardner, I. J., Cozewith, C., Ver Strate, G., "Infrared Determination of Composition of Ethylene-Propylene Copolymers," *Rubber Chemistry and Technology*, Vol 44, September 1971, pp. 1015 –1024.

[6] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR: D11-1065.

[7] *Reagent Chemicals, American Chemical Society Specifications*, American Chemical Society, Washington, DC. For Suggestions on the testing of reagents not listed by the American Chemical Society, see *Annual Standards for Laboratory Chemicals,* BDH Ltd., Poole, Dorset, U.K., and the *United States Pharmacopeia and National Formulary,* U.S. Pharmacopeial Convention, Inc. (USPC), Rockville, MD.

TABLE 2 Type 1 Precision Test Method A[A]

Material	Mean Level, %	Within Laboratories			Between Laboratories		
		S_r	r	(r)	S_R	R	(R)
EPDM–1237	52.4	0.259	0.724	1.38	0.557	1.56	2.98
EPDM–865	56.9	0.225	0.629	1.11	0.688	1.93	3.39
EPDM–2154	58.1	0.197	0.550	0.947	0.766	2.15	3.70
EPDM–306	67.9	0.400	1.12	1.65	0.814	2.28	3.36
EPDM–227	74.4	0.711	1.99	2.67	1.12	3.14	4.22

[A] $p = 9$, $q = 5$, and $m = 2$.

sufficiently high purity to permit its use without lessening the accuracy of the determination.

11.2 Exercise all safety and health precautions while carrying out the subsequent analyses, especially those involving the use of toxic or flammable solvents, or both.

11.3 *Acetone.*

11.4 *Adhesive Labels.*

11.5 *Flat Steel Plates,* 200 by 200 by 1.5 mm (8 by 8 by 0.06 in.).

11.6 *Polyester Film,* uncoated, 0.04 mm (0.0015 in.) thick.

11.7 *TFE-Fluorocarbon-Coated Stainless Steel or Aluminum Foil.*

12. Procedure

12.1 Obtain/prepare a small piece of polymer about the size of a matchhead (approximately 0.2 g).

12.2 Place the polymer between two sheets of polyester film or TFE-fluorocarbon-coated stainless steel or aluminum foil.

12.3 Insert the sheets of film between the platens of the press, or if the platens are too rough, between the steel plates, and then between the platens of the press.

12.4 Press the sample for 30 to 60 s at a pressure and T sufficient to obtain consistent thickness film (typically 70 MPa, 10 000 psi and 150 ± 10°C).

12.5 Release the pressure, remove from the press, cool, and carefully remove the polymer film from the polyester film or TFE-fluorocarbon-coated foil. Acetone may be used as a release agent from the polyester film for especially sticky or weak films. Mount the film in the sample holder for the spectrophotometer.

12.6 If the film has holes or other imperfections, discard it and mold a new one.

12.7 Some polymers that exhibit ripples and surface designs when hot-molded can be cooled to room temperature under pressure to obtain a smoother film. This may be necessary especially when using TFE-fluorocarbon-coated foil.

12.8 For especially sticky or weak films, use one of the following methods:

12.8.1 Mold the sample between TFE-fluorocarbon-coated foil. Cool the foil, remove one side from the sample, and remove the polymer film with an adhesive label by sticking it to the sample. Mount the sample in the holder for the spectrophotometer.

12.8.2 Use any other accepted spectrographic sample preparation technique to mount difficult films.

12.9 Place the sample in the sample beam of the spectrophotometer with air in the reference beam, and measure the total absorbance at 13.85 µm (722 cm^{-1}). The measured total absorbance should be within the linear range of the instrument, typically in the range of 0.5 to 1.2 A (in certain cases the linearity can be up to 1.5 A; Reference – Handbook of Instrumental Techniques for Analytical Chemistry by Frank Settle; Chapter 14; p. 271). If the total absorbance is not within the linear range, preferably between 0.5 and 1.2 A; that is, within the linear range of the instrument used, mold another sample to the correct thickness.

12.10 Follow the instrument manufacturer's recommendations for quantitative infrared spectroscopy and use the control settings advised. Particularly, control the scan speed, the gain, and the slit width according to the recommendations to give consistent results.

12.11 Measure the spectrum in the range from 7 to 16 µm (1400 to 600 cm^{-1}).

12.12 Draw a baseline for the 8.65-µm peak using shoulders at approximately 8.20 µm and 8.93 µm. The absorbance at 8.20 µm should generally be greater than the absorbance at 8.93 µm. If it is less than at the 8.20-µm area, this indicates a possible interference (perhaps from stabilizers). See Fig. 1. Remove such extraneous materials before infrared examination if possible; preferably, use Test Methods B or D, which do not utilize this peak.

12.13 Measure the absorbance of the peak at ~8.65 µm; ~1156 cm^{-1} (see Fig. 2).

12.14 Draw a baseline for the 13.85-µm peak using shoulders at approximately 12.7 µm and 14.8 µm.

12.15 Measure the absorbance of the peak at approximately 13.85 µm (see Fig. 2). If this peak is relatively large in relation to the 8.65-µm peak (greater than 5×), this indicates a high ethylene content. Test Method B, C, or D should be used, preferably, for better precision.

12.16 For referee testing only, repeat the procedure on a duplicate sample.

13. Calculation

13.1 Divide the absorbance at 8.65 µm by the absorbance at 13.85 µm.

13.2 Prepare a calibration chart between $A_{8.65}$ /$A_{13.85}$ and known EPM standards over the range from 35 to 70 mass %

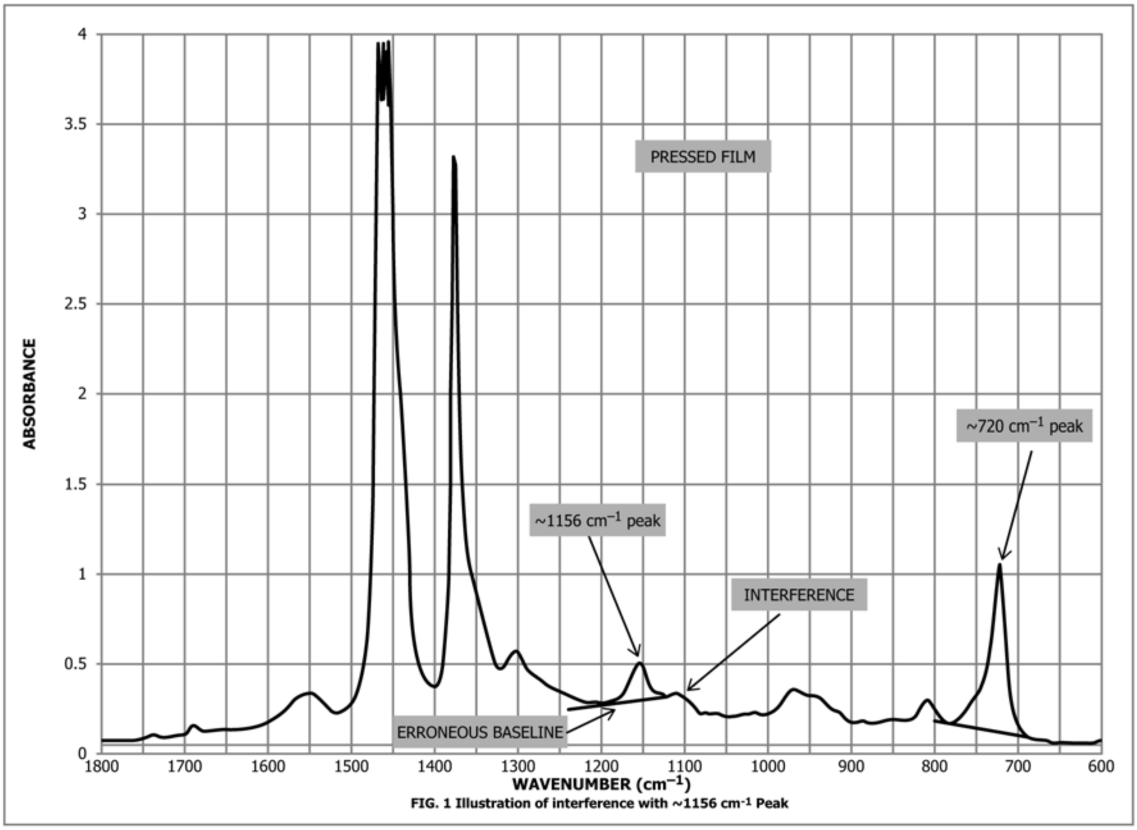

FIG. 1 Illustration of Interference With 8.65-µm Peak

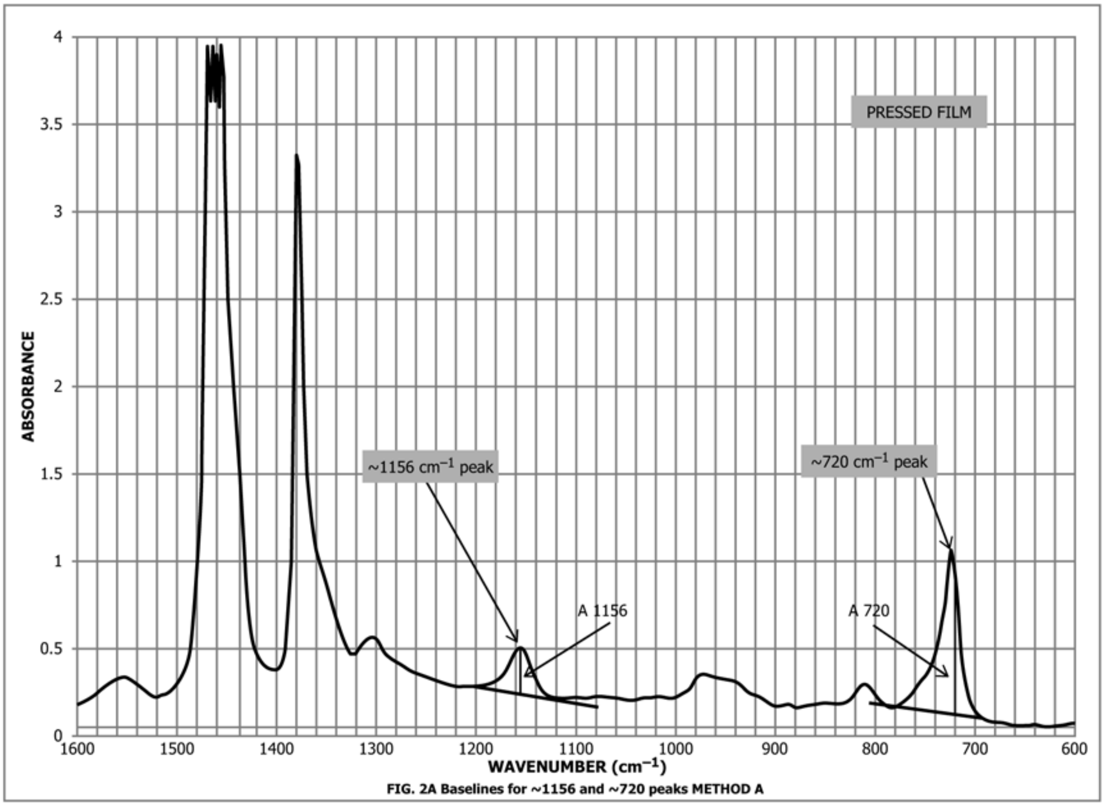

FIG. 2 A: Baselines for 2.35, 8.65, and 13.85 µm

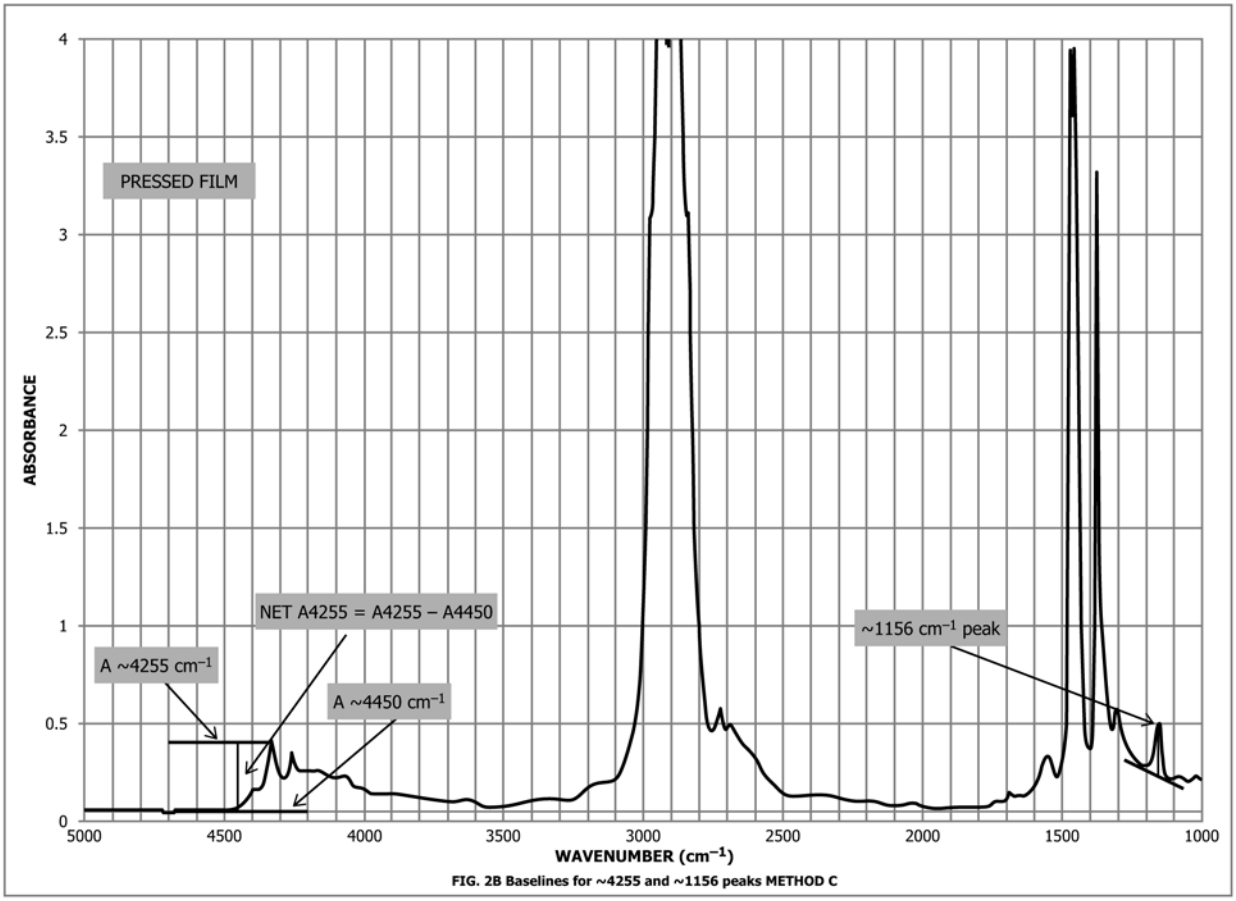

FIG. 2 B: Baselines for 2.35, 8.65, and 13.85 μm *(continued)*

ethylene for the type of polymer being measured. Make multiple determinations on the standards to construct an accurate calibration chart. Obtain such standards from the custodian of the standards used to establish this test method.[8]

13.3 Read the mass percent ethylene for the unknown sample from the calibration chart. See Section 30.

14. Report

14.1 Report the results obtained from an individual determination. For referee purposes, report the results from two individual determinations and their average.

14.2 For EPM, report the result as mass percent ethylene to the nearest tenth.

14.3 For EPDM, report the result as ethylene/propylene mass ratio, uncorrected for diene content, to the nearest tenth.

TEST METHOD B—PRESSED FILM METHOD USING THE 7.25/13.85-μm PEAK RATIO

15. Scope

15.1 This test method covers the determination of percent ethylene in EPM and EPDM between approximately 60 and 85 mass % ethylene.

15.2 This test method may also be used accurately below 60 mass % ethylene, but the required thin pressed films are very difficult to handle in this region.

15.3 This test method may not be used for EPDM based on 1,4-hexadiene as the third monomer because of interference with the 7.25-μm peak.

15.4 This test method is not intended for oil-extended polymers unless the oil is first removed by extraction.

16. Summary of Test Method

16.1 This test method makes use of the ratio of the absorbance of methyl groups from propylene units at 7.25 μm versus the absorbance of methylene sequences from ethylene units at 13.85 μm. A series of known EPM polymers is used to prepare a calibration of $A_{7.25}/A_{13.85}$ versus mass % ethylene.

17. Procedure

17.1 See 12.1 – 12.7 for basic sample preparation.

17.2 Films for this test method must be thinner than those for Test Method A in order to keep the 7.25-μm peak on scale. Preparation as in 12.8 at higher mold pressures up to 200 MPa (29 000 psi) will probably be necessary using longer mold time and a smaller sample.

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[8] Available from ExxonMobil Chemical Co., Baytown Technology and Engineering Complex, Ethylene Elastomers Technology Manager, 5200 Bayway Drive, P.O. Box 5200, Baytown, TX 77520.

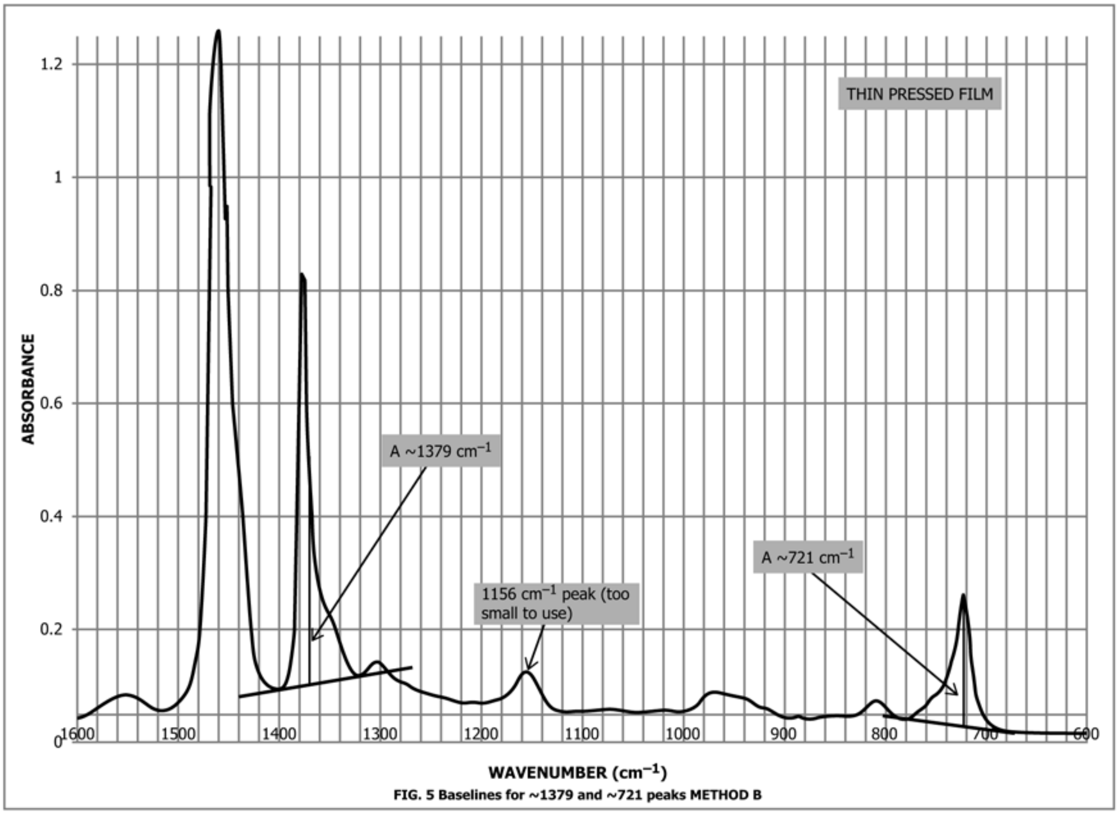


FIG. 3 Baselines for 7.25 and 13.85 µm (Test Method B)

17.3 After molding, place the sample in the sample beam of the spectrophotometer and measure the total absorbance at 7.25 µm. If it is greater than 0.8 A, discard it, and mold and test a thinner sample.

17.4 Follow the instrument manufacturer's recommendations for quantitative infrared spectroscopy in accordance with 12.10.

17.5 Measure the spectrum from 6.50 to 17 µm.

17.6 Draw a baseline for the 7.25-µm peak using shoulders at approximately 7.1 µm and 7.6 µm.

17.7 Measure the absorbance of the peak at approximately 7.25 µm (see Fig. 3).

17.8 Draw a baseline for the 13.85-µm peak using shoulders at approximately 12.75 µm and 14.8 µm.

17.9 Measure the absorbance of the peak at approximately 13.85 µm (see Fig. 2).

17.10 For testing referee only, repeat the procedure on a duplicate sample.

18. Calculation

18.1 Divide the absorbance at 7.25 µm by the absorbance at 13.85 µm.

18.2 Prepare a calibration chart between $A_{7.25}$ /$A_{13.85}$ and known EPM standards over the range from 60 to 85 mass % ethylene for the type of polymer being measured. See 13.2 for standards.

18.3 Read the mass percent ethylene for the unknown sample from the calibration chart. See Section 33.

19. Report

19.1 Report the results obtained from an individual determination. For referee purposes, report the results from two individual determinations and their average.

19.2 For EPM, report the result as mass percent ethylene to the nearest whole number.

19.3 For EPDM, report the result as ethylene/propylene mass ratio, uncorrected for diene, to the nearest whole number.

TEST METHOD C—PRESSED FILM METHOD USING THE 8.65/2.35-µm PEAK RATIO

20. Scope

20.1 This test method covers the determination of percent ethylene over the range from 35 to 85 mass % in EPDM containing 1,4-hexadiene as the third monomer.

20.2 This test method may also be used on other EPDM and EPM, subject to the interferences described in 5.2, 9.3, and 12.12 for the 8.65-µm peak. Where these interferences cannot be eliminated, Test Method B or D must be used.

20.3 This test method is limited to those having near infrared measurement capability.

21. Summary of Test Method

21.1 This test method makes use of the ratio of the absorbance of methyl groups from propylene units at 8.65 µm versus the absorbance of C-H bonds at 2.35 µm in the near infrared for internal thickness reference.

22. Procedure

22.1 See 12.1 – 12.7 for sample preparation. Film thickness should be approximately 0.1 to 0.2 mm.

22.2 After molding, place the sample in the sample beam of the spectrophotometer with air in the reference beam, and measure the absorbance at 2.35 µm. If it is less than 0.15 A or greater than 0.2 A, discard it, and mold and test a new sample of the proper thickness. In cases of samples containing unusually high amounts of ethylene, the films may be somewhat thicker to allow practical heights of the 8.65-µm peak.

22.3 Follow the instrument manufacturer's recommendations for quantitative infrared spectroscopy in accordance with 12.10.

22.4 Measure the spectrum from 2.0 to 10.0 µm on the same instrument.

22.5 Measure the absorbances of the peak at approximately 2.35 µm and the shoulder at approximately 2.20 µm. Subtract $A_{2.20}$ from $A_{2.35}$ to give net $A_{2.35}$ (see Fig. 2).

22.6 Draw a baseline for the 8.65-µm peak using shoulders at approximately 8.20 µm and 8.93 µm. The absorbance at 8.20 µm should generally be greater than the absorbance at 8.93 µm. If it is less than at the 8.93-µm area, this indicates a possible interference from stabilizers (see Fig. 1). Remove such extraneous materials before infrared examination if possible; preferably, Test Method B or D should be used, which do not utilize this peak.

22.7 Measure the absorbance of the peak at approximately 8.65 µm (see Fig. 2).

22.8 For referee testing only, repeat the procedure on a duplicate sample.

23. Calculation

23.1 Divide the absorbance at 8.65 µm by the absorbance at 2.35 µm.

23.2 Prepare a calibration chart between $A_{8.65}/A_{2.35}$ and known EPM standards over the range from 35 to 85 mass % ethylene for the type of polymer being measured. See 13.2 for standards.

23.3 Read the mass percent ethylene for the unknown sample from the calibration chart. See Section 33.

24. Report

24.1 Report the results obtained from an individual determination. For referee purposes, report the results from two individual determinations and their average.

24.2 For EPM, report the result as mass percent ethylene to the nearest tenth.

24.3 For EPDM, report the result as ethylene/propylene mass ratio, uncorrected for diene content, to the nearest whole number.

TEST METHOD D—CAST FILM METHOD USING THE 7.25/6.85-µm PEAK RATIO

25. Scope

25.1 This test method covers the determination of percent ethylene in EPM and EPDM between approximately 35 and 85 mass % ethylene, but should not be used for EPDM containing 1,4-hexadiene because of interference in the 7.25-µm peak.

25.2 This test method is applicable to oil-extended polymers after extender oil has been removed.

25.3 The sample preparation technique in this test method may also be used to prepare extracted samples for pressed film Test Methods A, B, and C.

26. Summary of Test Method

26.1 After extraction by a solvent, a polymer film is cast on a salt plate and examined by infrared. This test method makes use of the ratio of the absorbance of methyl groups from propylene units at 7.25 µm versus the absorbance of C—H bonds at 6.85 μm^{-1} for internal thickness. A series of known EPM polymers is used to prepare a calibration of $A_{7.25}/A_{6.85}$ versus mass percent ethylene.

27. Apparatus

27.1 *Extraction Apparatus,* as described in Fig. 1 or 2 of Test Methods D297.

27.2 *Explosion-Proof, High-Speed Blender and Small Container.*

27.3 *Vacuum Oven.*

28. Reagents and Materials

28.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available.[7] Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

28.2 *Carbon Tetrachloride,* spectroscopic grade or equivalent.

28.3 *Methanol,* spectroscopic grade or equivalent.

28.4 *Toluene,* spectroscopic grade or equivalent.

28.5 *Cloth or Fine-Mesh Wire Screen.*

28.6 *Eyedropper.*

28.7 *Ointment Tin or Other Sealed Container.*

28.8 *Salt Plate.*

28.9 *Stainless Steel Screen,* with openings of approximately 0.177 mm (80-mesh, 5.2-mil wire diameter).

29. Sample Preparation

29.1 Remove extender oils, if present, from the polymer by one of the following methods before infrared analysis.

29.1.1 Extract a polymer film with acetone in accordance with Test Methods D297 to remove extender oil.

NOTE 3—For polymer types that tend to mass by the prepared strips sticking together during extraction and for polymer types containing oil that is difficult or slow to extract, prepare the sample for extraction by pressing it onto a wire screen, giving both support and a large surface area for extraction.

Using the hydraulic press, press approximately 1 g of the polymer into a 50 by 50-mm (2 by 2-in.) stainless steel screen having openings of approximately 0.177 mm (80-mesh, 5.2-mil wire diameter). After extraction, remove the polymer for percent ethylene testing. After the test is completed, the remaining polymer can be burned off within a muffle furnace and the screen can be reused.

29.1.2 As an alternative to 29.1.1, dissolve about 2 g of the polymer in 50 cm^3 of carbon tetrachloride. Some polymers may not dissolve completely, but a final dispersion can be achieved by using refluxing carbon tetrachloride or an explosion-proof high-speed blender and small container, or both.

29.1.2.1 Pour the carbon tetrachloride solution slowly into the vortex caused by rapidly stirring about 250 cm^3 of hot methanol in a 400-cm^3 beaker. Heat is required to keep some heavy extender oils in solution.

29.1.2.2 Bring to a boil until the sample is flocculated thoroughly.

29.1.2.3 Cool and filter the polymer through cloth or a fine-mesh wire screen.

29.1.2.4 Squeeze the polymer between paper to remove as much of the solvents as possible.

29.1.2.5 Repeat the solution and flocculation steps if it is suspected that all the oil is not removed.

29.2 At this point, the polymer from 29.1.1 or 29.1.2.4 may be dried in a vacuum oven or in a hot press to remove the solvent and used for pressed film Test Method A, B, or C. However, both methods 29.1.1 and 29.1.2 will also remove small amounts of low-molecular weight or high-ethylene content EPM or EPDM, or both, which may change the ethylene content of the bulk material. Where this is significant, this sample preparation may not be used.

29.3 Dissolve about 2 g of the sample from 29.1.1 or 29.1.2.4 in approximately 50 cm^3 of toluene or carbon tetrachloride. If the sample does not dissolve completely, it may be dispersed by refluxing or treatment with the explosion-proof high-speed blender, or both.

29.4 For non-oil-extended samples or those which do not need extraction, dissolve the sample from 7.2 as in 29.3 for use with this test method.

30. Calibration

30.1 The following best-fit regression equation forms for calibration curves have been found for most infrared instruments:

Test Methods A, B, and C

$$\text{Mass\,\%\ Ethylene} = a - b\ln(\text{Absorbance Ratio})$$

Test Method D

$$\text{Mass\,\%\ Ethylene} = a - b\,(\text{Absorbance Ratio})$$

30.2 Regression coefficients (*a* and *b* above) are expected to vary with individual infrared instruments and with time on each instrument. The equation form (linear, logarithmic, etc.) established for each test method on each instrument is not expected to change with time.

30.3 A sample calibration curve showing typical scatter for one double beam instrument on one test method (A) is shown in Fig. 4.

31. Procedure

31.1 On a level salt plate, deposit from an eyedropper a thick pool of cement about 1 to 2 mm thick. Cover the salt plate with the inverted ointment tin or other sealed container to retard evaporation.

31.2 After the film has dried, test the absorbance at 6.85 µm. Total absorbance should be between 0.35 and 0.75 A to be acceptable. Since polymer films retain solvent tenaciously, the analyst should satisfy himself that no residual solvent remains in the dried film.

31.3 Measure the spectrum in the range from 5.9 to 8.35 µm.

31.4 Draw a baseline for the 6.85-µm peak using shoulders at approximately 6.37 and 7.85 µm.

31.5 Measure the absorbance of the peak at approximately 6.85 µm (see Fig. 5).

31.6 Draw a baseline for the 7.25-µm peak using shoulders at approximately 7.14 µm and 7.58 µm.

31.7 Measure the absorbance of the peak at approximately 7.25 µm (see Fig. 5).

31.8 For referee testing only, repeat the procedure on a duplicate sample.

32. Calculation

32.1 Divide the absorbance at 7.25 µm by the absorbance at 6.85 µm.

32.2 Prepare a calibration chart between $A_{7.25}$ /$A_{6.85}$ and known EPM standards over the range from 35 to 85 mass % ethylene. See 13.2 for standards.

32.3 Read the mass percent ethylene for the unknown sample from the calibration chart. See Section 30.

33. Report

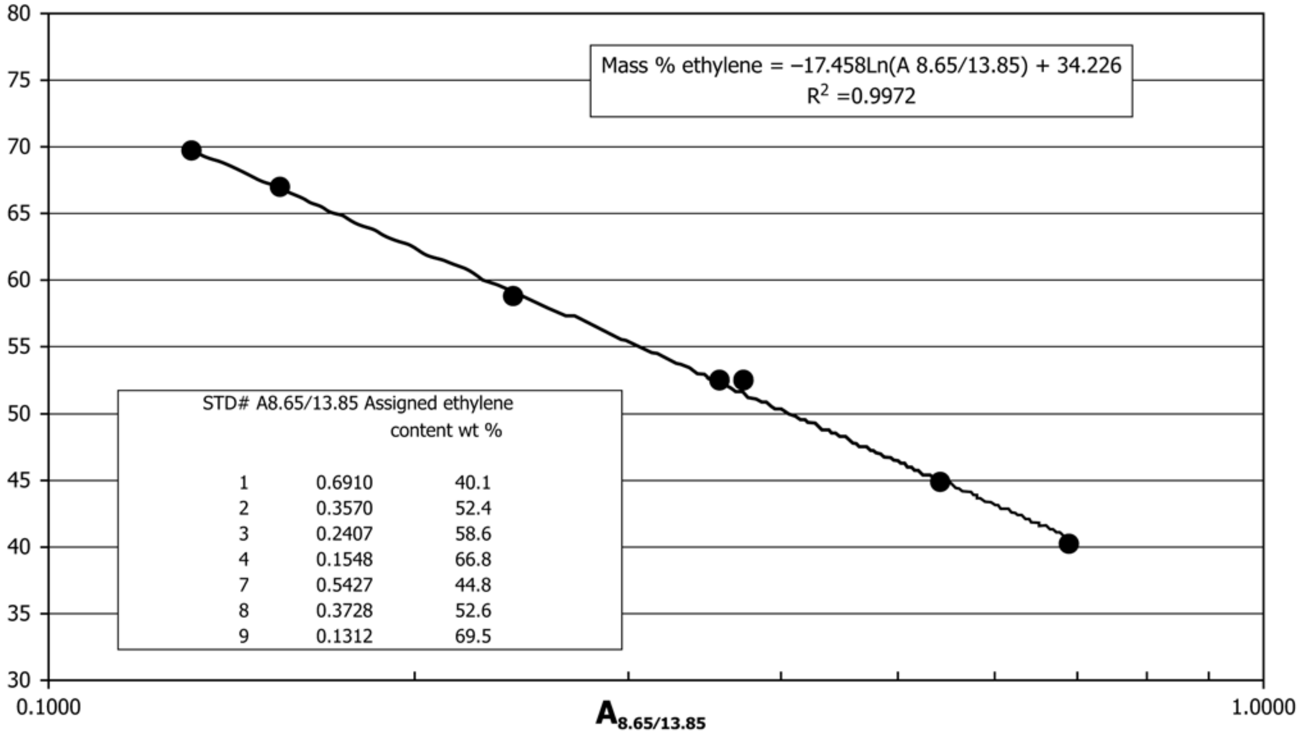
33.1 Report the results obtained from an individual determination. For referee purposes, report the results from two individual determinations and their average.

33.2 For EPM, report the result as mass percent ethylene to the nearest whole number.

33.3 For EPDM, report the result as ethylene/propylene mass ratio, uncorrected for diene content, to the nearest whole number.

34. Keywords

34.1 ethylene–propylene copolymers; ethylene–propylene–diene terpolymers; infrared



STD#	A8.65/13.85	Assigned ethylene content wt %
1	0.6910	40.1
2	0.3570	52.4
3	0.2407	58.6
4	0.1548	66.8
7	0.5427	44.8
8	0.3728	52.6
9	0.1312	69.5

FIG. 4 Sample Calibration—Test Method A

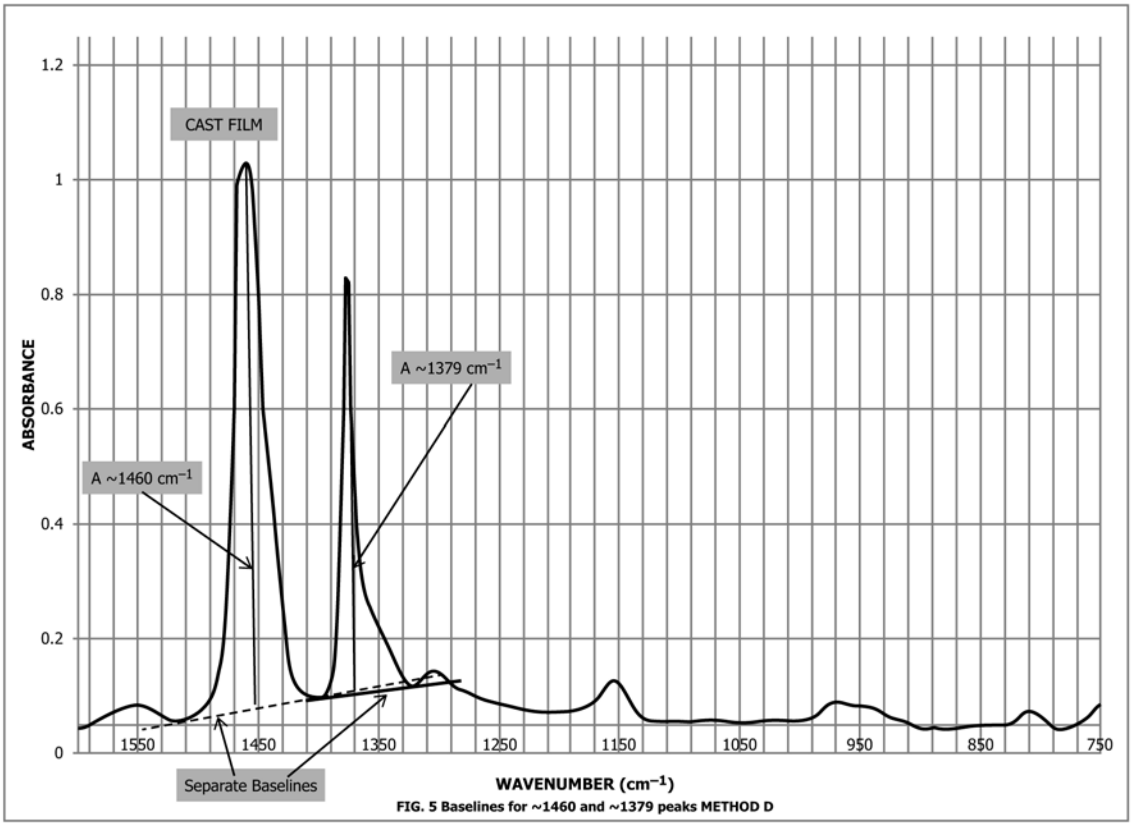


FIG. 5 Baseline for 6.85 and 7.25 µm (Test Method D)

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