



Standard Test Methods for Rubber, Raw—Determination of 5-Ethylidenenorbornene (ENB) or Dicyclopentadiene (DCPD) in Ethylene-Propylene-Diene (EPDM) Terpolymers¹

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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D1416 Test Methods for Rubber from Synthetic Sources—Chemical Analysis (Withdrawn 1996)³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E168 Practices for General Techniques of Infrared Quantitative Analysis (Withdrawn 2015)³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.