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Standard Test Method for Rubber—Determination of Residual Unsaturation in Hydrogenated Nitrile Rubber (HNBR) by Infrared Spectrophotometry[1]

This standard is issued under the fixed designation D5670; 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 This test method covers the percentage of residual unsaturation (double bonds in unhydrogenated butadiene) in hydrogenated nitrile rubber and is based on infrared examination of rubber films cast from solution.

1.2 This test method is applicable to all grades of hydrogenated nitrile rubber in the raw state.

1.3 This test method assumes that specimens and infrared spectra are prepared and analyzed by experienced personnel and that the equipment is operated according to the manufacturer's direction for optimum performance. No details for operation of infrared spectrophotometers are included in this test method.

1.4 Since it is customary in infrared spectrophotometry to use wavenumbers (cm^{-1}) rather than Hertz (Hz), the unit for frequency in the SI system, the former is employed throughout this test method.

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

1.6 *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]

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

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

3. Summary of Test Method

3.1 A specimen of the raw rubber is purified by precipitation with methanol from a solution in methyl ethyl ketone or by extraction of the solid rubber with methanol in a Soxhlet apparatus.

3.2 The purified rubber specimen is again dissolved in methyl ethyl ketone and a film is cast on a potassium bromide (KBr) disc.

3.3 The infrared spectrum of the film is obtained with a Fourier Transform Infrared Spectrophotometer (FT-IR) or a dispersive infrared spectrophotometer.

3.4 The corrected absorbance of the specific absorption bands for acrylonitrile (ACN), butadiene (BD), and hydrogenated butadiene (HBD) are determined using the baseline method and the percentage of residual unsaturation (double bonds in unhydrogenated butadiene) is calculated with the aid of absorbance factors from the referenced literature.[3]

4. Significance and Use

4.1 Hydrogenated nitrile rubbers are available at different levels of unsaturation and different acrylonitrile content. Highly saturated grades offer optimum resistance to aging, such as exposure to heat, ozone, and chemicals, and can be effectively cured only with peroxides or high energy radiation.

4.2 Partially unsaturated grades can be cured by sulfur systems in addition to peroxides and high energy radiation.

4.3 This test method provides an infrared technique to determine the unsaturation level of hydrogenated nitrile rubbers in the raw state. It can be used for research and development, quality control, and referee purposes.

[1] This test method is under the jurisdiction of ASTM Committee D11 on Rubber and is 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] Brück, D., Kautschuk, Gummi, *Kunststoffe*, Vol 42, 1989, pp. 107–110 and 194–197.

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5. Apparatus

5.1 *Preparation of Rubber Solution:*

5.1.1 *Erlenmeyer Flask with Ground Glass Stopper,* (50 cm^3).

5.1.2 *Flask Shaker.*

5.2 *Precipitation of Rubber from Solution:*

5.2.1 *Beaker,* (250 cm^3).

5.2.2 *Magnetic Stirrer.*

5.2.3 *Dropping Funnel,* (150 cm^3).

5.3 *Extraction of Rubber:*

5.3.1 *Soxhlet Extraction Apparatus with Flask,* (150 cm^3).

5.3.2 *Extraction Thimbles,* (27 by 100 mm).

5.4 *Kofler Heating Bench or Other Heating Device,* with temperature control.

5.5 *Spectrophotometer,* required resolution capability of 2 cm^{-1}. The instrument must be in good operating condition as defined by the manufacturer and Practices E932 and E1421. The instrument shall be capable of scale expansion along the absorbance or transmittance axis. The spectral region of 2500–600 cm^{-1} is used in this test method.

6. Reagents

6.1 Reagent grade chemicals shall be used in all tests. All reagents shall conform to the specification of the Committee on Analytical Reagents of the American Chemical Society.[4] Other grades may be used, provided they are of sufficiently high purity as not to lessen the accuracy of the determination.

6.2 *Methyl Ethyl Ketone.*

6.3 *Methanol.*

6.4 *Dry Compressed Nitrogen,* (in cylinders or provided as a laboratory service).

6.5 *Potassium Bromide (KBr) Discs.*

7. Procedure

7.1 *Specimen Preparation:*

7.1.1 *Specimen Purification by Precipitation (see Note 1):*

7.1.1.1 Weigh 1.0 g of the finely divided rubber specimen and dissolve in 20 cm^3 methyl ethyl ketone using a 50-cm^3 Erlenmeyer flask with ground glass stopper on a flask shaker.

7.1.1.2 Transfer the solution into a 250-cm^3 beaker and precipitate the rubber by slowly dropping 150 cm^3 methanol from a dropping funnel into the solution under agitation with a magnetic stirrer.

7.1.1.3 Decant the solvent, wash the precipitated rubber with 50 cm^3 methanol and redissolve in 20 cm^3 methyl ethyl ketone.

7.1.2 *Specimen Purification by Extraction (see Note 1):*

7.1.2.1 Extraction is an alternative to the precipitation method.

7.1.2.2 Weigh 1.0 g of the finely divided rubber specimen into an extraction thimble and extract for 6 h in a Soxhlet apparatus (150-cm^3 flask) with 120 cm^3 methanol.

7.1.2.3 Remove the extracted rubber sample from the thimble and dissolve in 20 cm^3 methyl ethyl ketone as described in 7.1.1.1.

7.1.3 *Preparation of Cast Rubber Films:*

7.1.3.1 Cast a smooth rubber film from the solution in methyl ethyl ketone onto a potassium bromide (KBr) disc.

7.1.3.2 Evaporate the solvent on a Kofler bench or similar heating device at a maximum temperature of 100°C under a stream of nitrogen.

7.1.3.3 The thickness of the rubber film must be chosen so that the strongest band to be evaluated from the infrared spectrum has an absorbance of less than 0.8.

7.2 *Infrared Spectrum:*

7.2.1 Obtain a 50-scan spectrum with an FT-IR instrument with 2 cm^{-1} resolution or with a dispersive infrared spectrophotometer and appropriate scan parameters.

7.2.2 In case the repeatability and standard deviation of the absorbance must be determined, test up to six different films of the same specimen.

7.2.3 If only a transmittance mode operation is possible with the instrument being used, the spectrum shall be scanned in that mode.

7.3 *Calculation of Results:*

7.3.1 Calculate the corrected absorbance $A(i)$ by subtracting the baseline absorbance from the peak absorbance at the point below the peak (see Fig. X1.1).

7.3.2 When the transmittance mode is used, calculate the corrected absorbance $A(i)$ by taking the log10 of the quotient of percent transmittance of the baseline at the point above the peak and the percent transmittance of the peak (see Fig. X1.2).

7.3.3 Draw baselines to approximately the following wavenumbers (all values are in cm^{-1}):

ACN: 2280–2200 for the peak 2236 (see Note 2)
BD: 1010–910 for the peak 970
HBD: 840–670 for the peak 723

7.3.4 If repeatability and standard deviation $s(i)$ must be determined, use the normalized absorbance ratios

$$\bar{A}(970) = A(970)/A(2236), \text{ and} \quad (1)$$

$$\bar{A}(723) = A(723)/A(2236) \quad (2)$$

from spectra of different films produced from each specimen (see Note 2).

7.3.5 Calculate the molar concentrations $c(i)$ using absorbance factors from the referenced literature[3] and the measured normalized absorbance ratios $\bar{A}(970)$ and $\bar{A}(723)$ (see Eq 1 and Eq 2).

7.3.6 The absorbance factors from the referenced literature[3] are given below:

$$k(2236) = 1;\ k(970) = 2.3 \pm 0.03;\ k(723) = 0.255 \pm 0.002 \quad (3)$$

7.3.7 Calculate the molar concentrations according to the following equations:

[4] *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.

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