



Standard Test Method for Vinyl and Trans Unsaturation in Polyethylene by Infrared Spectrophotometry¹

This standard is issued under the fixed designation D 6248; 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 most types of polyethylene, those ethylene plastics consisting of ethylene and α -olefin comonomers longer than propylene, and blends of the above in any ratio.

1.2 The values stated in SI units are to be regarded as the standard.

1.3 *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.* Specific hazards statements are given in Section 8.

NOTE 1—There is no similar or equivalent ISO standard.

2. Referenced Documents

2.1 ASTM Standards:

D 792 Test Method for Density and Specific Gravity (Relative Density) of Plastics by Displacement²

D 1505 Test Method for Density of Plastics by the Density-Gradient Technique²

D 1898 Practice for Sampling of Plastics²

D 3124 Test Method for Vinylidene Unsaturation in Polyethylene by Infrared Spectrophotometry³

E 131 Terminology Relating to Molecular Spectroscopy⁴

E 168 Practices for General Techniques of Infrared Quantitative Analysis⁴

E 177 Practice for Use of the Terms Precision and Bias in ASTM Test Methods⁵

E 275 Practice for Describing and Measuring Performance of Ultraviolet, Visible, and Near Infrared Spectrophotometers⁴

E 380 Practice for Use of the International System of Unit (SI)⁵

Proposed Methods for the Evaluation of Spectrophotometers

3. Terminology

3.1 *General*—The units, symbols, and abbreviations used in this test method appear in Terminology E 131 or Practice E 380.

4. Summary of Test Method

4.1 The band at 965 cm^{-1} is characteristic of the trans-vinylene substituted group. The band at 908 cm^{-1} is characteristic of the terminal vinyl group.

4.2 These bands are not seriously overlapped by interfering groups from the polymer. The vinyl absorbance at 908 cm^{-1} is close to the absorbance band from terminal methyl groups on chains longer than ethyl, at 895 cm^{-1} . This interference is minimal for most products. Inclusion of the bromination and spectral subtraction step from Test Method D 3124 may improve the results for very low density products.

4.3 Integrated absorbance is used in this test method. Integrated absorbance is found by integrating the spectrum over the absorbance band when the spectrum is plotted as absorbance versus frequency, in cm^{-1} . Most spectral manipulation software contains algorithms for adequately determining baseline corrected integrated absorbencies.

4.4 Calibration is performed using a solution of 1-octene for normal vinyl groups, and trans-3-hexene for trans-vinylene groups.

5. Significance and Use

5.1 There are three types of olefinic groups present in sufficient concentrations to warrant consideration, one or more of that can normally be found in any polyethylene. The three types are trans-vinylene, R-CH=CH-R' , sometimes referred to as trans-internal unsaturation; vinylidene or pendent methylene, RR'C-CH_2 ; and vinyl unsaturation, R-CH=CH_2 , also referred to as terminal unsaturation.

¹ This test method is under the jurisdiction of ASTM Committee D-20 on Plastics and is the direct responsibility of Subcommittee D20.70 on Analytical Methods. Current edition approved April 10, 1998. Published February 1999.

² *Annual Book of ASTM Standards*, Vol 08.01.

³ *Annual Book of ASTM Standards*, Vol 08.02.

⁴ *Annual Book of ASTM Standards*, Vol 03.06.

⁵ *Annual Book of ASTM Standards*, Vol 14.02.