



Standard Test Method for Determination of Linear Low Density Polyethylene (LLDPE) Composition by Carbon-13 Nuclear Magnetic Resonance¹

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^{ε1} NOTE—Reapproved with editorial changes in April 2009.

1. Scope

1.1 This test method determines the molar composition of copolymers prepared from ethylene (ethene) and a second alkene-1 monomer. This second monomer can include propene, butene-1, hexene-1, octene-1, and 4-methylpentene-1.

1.2 Calculations of this test method are valid for products containing units EEXEE, EXEXE, EXXE, EXXXE, and of course EEE where E equals ethene and X equals alkene-1. Copolymers containing a considerable number of alkene-1 blocks (such as, longer blocks than XXX) are outside the scope of this test method.

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.* See Section 8 for a specific hazard statement.

NOTE 1—There is no equivalent ISO standard.

2. Referenced Documents

2.1 ASTM Standards:²

E177 Practice for Use of the Terms Precision and Bias in ASTM Test Methods

E386 Practice for Data Presentation Relating to High-Resolution Nuclear Magnetic Resonance (NMR) Spectroscopy

E691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method

IEEE/ASTM SI-10 Standard for Use of the International

¹ This test method is under the jurisdiction of ASTM Committee D20 on Plastics and is the direct responsibility of Subcommittee D20.70 on Analytical Methods.

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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.

System of Units (SI): The Modern System³

3. Terminology

3.1 Some units, symbols, and abbreviations used in this test method are summarized in IEEE/ASTM SI-10 and Practice E386. Other abbreviations are listed as follows:

3.2 Abbreviations:

3.2.1 ¹³C—carbon 13,

3.2.2 LLDPE—linear low-density polyethylene,

3.2.3 T1—relaxation time, and

3.2.4 TR—pulse repetition time.

3.3 Definitions of Terms Specific to This Standard:

3.3.1 With a few modifications, terms used to designate different carbon types were suggested by Carman.⁴ Methine carbons are identified by CH and branch carbons are labeled according to branch type as summarized in Table 1. Branch carbons are numbered starting with the methyl as number one.

3.3.2 Backbone methylene carbons are designated by a pair of Greek letters that specify the location of the nearest methine carbon in each direction. For example, α,α-methylene carbon is between two methine carbons or an α,δ⁺ methylene carbon has one immediate methine neighbor and the second methine carbon is located at least four carbons away.

4. Summary of Test Method

4.1 Polymer samples are dispersed in hot solvent and analyzed at high temperatures using Carbon-13 nuclear magnetic resonance (NMR) spectroscopy.

4.2 Spectra are recorded under conditions such that the response of each chemically different carbon is identical. Integrated responses for carbons originated from the different comonomers are used for calculation of the copolymer composition.

³ Available from ASTM International Headquarters, 100 Barr Harbor Drive, C700, West Conshohocken, PA 19428.

⁴ Carman, C. J., Harrington, R. A., and Wilkes, C. E., *Macromolecules* 1977, Vol 10, p. 536.