



Standard Test Method for Determination of Antioxidants and Erucamide Slip Additives in Polyethylene Using Liquid Chromatography (LC)¹

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1. Scope*

1.1 This test method covers a liquid-chromatographic procedure for the separation of primary and secondary antioxidant and slip additives currently used in polyethylene plastics. These additives are extracted with either isopropanol (resin densities $< 0.94 \text{ g/cm}^3$) or cyclohexane (resin densities $> 0.94 \text{ g/cm}^3$) prior to liquid-chromatographic separation. The ultraviolet absorbance of the eluting compound(s) is measured and quantitation is performed using external calibration.

1.2 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this 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 precautionary statements are given in Section 9.

NOTE 1—There is no known ISO equivalent to this standard.

2. Referenced Documents

2.1 ASTM Standards:²

D883 Terminology Relating to Plastics

D1600 Terminology for Abbreviated Terms Relating to Plastics

D4697 Guide for Maintaining Test Methods in the User's Laboratory (Withdrawn 2009)³

E131 Terminology Relating to Molecular Spectroscopy

E169 Practices for General Techniques of Ultraviolet-Visible Quantitative Analysis

E275 Practice for Describing and Measuring Performance of

Ultraviolet and Visible Spectrophotometers

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

E1657 Practice for Testing Variable-Wavelength Photometric Detectors Used in Liquid Chromatography

IEEE/ASTM SI 10 Standard for Use of the International System of Units (SI): The Modern Metric System

3. Terminology

3.1 Definitions:

3.1.1 For definitions of plastic terms and detector terminology used in this test method, see Terminologies D883, D1600, and E1657.

3.1.2 For units and symbols used in this test method, refer to Terminology E131 or IEEE/ASTM SI 10.

4. Summary of Test Method

4.1 The polyethylene sample is ground to a 1-mm (~20 mesh) or 0.5-mm (~40 mesh) particle size and extracted by refluxing with either isopropanol or cyclohexane.

4.2 The solvent extract is analyzed by liquid chromatography.

4.3 Additive concentrations are determined from external calibration curves using reverse phase chromatography (C-8 or C-18 column) with ultraviolet (UV) detection at wavelengths corresponding to the wavelengths of an absorption apex of each additive (except erucamide which does not have an absorption maximum in the accessible UV region).

5. Significance and Use

5.1 Separation and identification of stabilizers used in the manufacture of polyethylene resins are necessary in order to correlate performance properties with polymer composition. This test method provides a means to determine the polymer additives listed in Table 1 in polyethylene samples. This test method is capable of the determination of other antioxidants, but the stability of these during extraction has not been investigated.

5.2 The additive extraction procedure is made effective by the relatively low solubility of the polymer sample in solvents generally used for liquid chromatographic analysis. In this

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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.