



Standard Test Method for Determination of Phenolic Antioxidants and Erucamide Slip Additives in Polypropylene Homopolymer Formulations Using Liquid Chromatography (LC)¹

This standard is issued under the fixed designation D6042; 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 a liquid-chromatographic procedure for the separation of some additives currently used in polypropylene. These additives are extracted with a cyclohexane:methylene chloride mixture using either reflux or ultrasonic bath prior to liquid-chromatographic separation. The ultraviolet absorbance (200 nm) of the compound(s) is measured, and quantitation is performed using the internal standard method.

1.2 *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 test method.

2. Referenced Documents

2.1 ASTM Standards:²

D883 Terminology Relating to Plastics

D1600 Terminology for Abbreviated Terms Relating to Plastics

E131 Terminology Relating to Molecular Spectroscopy

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

IEEE/ASTM SI-10 Practice for Use of the International System of Units (SI) (the Modernized Metric System)

3. Terminology

3.1 For definitions of plastic terms used in this test method, see Terminologies D883 and D1600.

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 (D20.70.02).

Current edition approved Sept. 1, 2016. Published September 2016. Originally approved in 1996. Last previous edition approved in 2009 as D6042 - 09. DOI: 10.1520/D6042-09R16.

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.

3.2 For the units, symbols, and abbreviations used in this test method, refer to Terminology E131 or Practice IEEE/ASTM SI-10.

3.3 Abbreviations:

3.3.1 LC—liquid chromatography.

3.3.2 PP—polypropylene.

3.4 Trade Names:

3.5 Vitamin E—α-Tocopherol, or 3,4-dihydro-2,5,7,8-tetramethyl-2-(4,8,12-trimethyltridecyl)-2H-1-benzopyran-6-ol.

3.6 Irgafos 168—Tris(2,4 di-tert-butylphenyl) phosphite.

3.7 Irganox 3114—Tris(3,5-di-t-butyl-4-hydroxybenzyl) isocyanurate.

3.8 Kemamide-E—cis-13-docosenamide or erucamide.

3.9 Irganox 1010—tetrakis[methylene(3,5-di-t-butyl-4-hydroxy hydrocinnamate)]methane.

3.10 Irganox 1076—octadecyl-3,5-di-t-butyl-4-hydroxy hydrocinnamate.

3.11 Tinuvin P—2(2'-hydroxy-5'-methyl phenyl)benzotriazole.

4. Summary of Test Method

4.1 The PP sample is ground to a 20-mesh particle size (850 microns) and extracted by refluxing with a mixture of 75:25 methylene chloride:cyclohexane or placing in an ultrasonic bath with the same mixture.

4.2 The solvent extract is examined by liquid chromatography.

4.3 Additive concentrations are determined relative to an internal standard (contained in the solvent) using reverse-phase chromatography (C-18 column) with ultraviolet (UV) detection at 200 nm.

5. Significance and Use

5.1 Separation and identification of stabilizers used in the manufacture of polypropylene is necessary in order to correlate performance properties with polymer composition. This test method provides a means to determine erucamide slip, Vitamin

*A Summary of Changes section appears at the end of this standard