



Designation: **D4275 – 09 D4275 – 17**

Standard Test Method for Determination of Butylated Hydroxy Toluene (BHT) in Polymers of Ethylene and Ethylene–Vinyl Acetate (EVA) Copolymers By Gas Chromatography¹

This standard is issued under the fixed designation D4275; 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 reappraisal. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reappraisal.

1. Scope-~~Scope~~*

1.1 This test method describes a procedure for the determination of **butylated hydroxy toluene (BHT) (2,6-di-*t*-butyl-4-methyl-hydroxybenzene)** in polymers of ethylene and ethylene–vinyl acetate (EVA) copolymers by solvent extraction followed by gas chromatographic analysis. Detection of the compound is achieved by flame ionization, and quantitative analysis is obtained by use of internal or external standards, as described in Practices **E260**, **E355**, and **E594**.

1.2 The values stated in SI units are to be regarded as the standard. The values given in parentheses are for information only.

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 for this test method.

2. Referenced Documents

2.1 *ASTM Standards:*²

[D883 Terminology Relating to Plastics](#)

[D4968 Practice for Annual Review of Test Methods and Specifications for Plastics](#)

[D7210 Practice for Extraction of Additives in Polyolefin Plastics](#)

[E260 Practice for Packed Column Gas Chromatography](#)

[E355 Practice for Gas Chromatography Terms and Relationships](#)

[E594 Practice for Testing Flame Ionization Detectors Used in Gas or Supercritical Fluid Chromatography](#)

[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 *Definitions*—Units and symbols used in this test method are those recommended in Practice **IEEE/ASTM SI-10**. Chromatographic terms and relationships are as described in Practice **E355**.

3.2 *Abbreviations:*

3.2.1 *BHT*—Butylated hydroxy toluene (2,6-di-*t*-butyl-4-methyl-hydroxybenzene).

3.2.2 *MM*—Methyl myristate.

3.2.3 *EVA*—Ethylene–vinyl acetate copolymers.

3.2.4 *LDPE*—Low-density polyethylene.

3.2.5 *HDPE*—High-density polyethylene.

4. Summary of Test Method

4.1 The BHT from a finely ground polymer sample is extracted by shaking or refluxing with cyclohexane or isopropanol. A known volume of this extract is injected into a gas chromatographic column packed with a liquid-coated solid support. Passing

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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.

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

through this column in a stream of carrier gas, BHT is separated from the extraction solvent and other components. Responses of BHT and any internal standard are measured by a flame ionization detector. This signal is recorded to indicate the relative concentration and retention time of BHT.

5. Significance and Use

5.1 Separation and identification of stabilizers used in the manufacture of polyethylene are necessary in order to correlate performance properties with polymer composition.

5.2 The BHT extraction procedure is made effective by the insolubility of the polymer sample in solvents generally used for gas chromatographic analysis.

6. Interferences

6.1 Any material eluting at or near the BHT or MM retention times will cause erroneous results. Prior to extraction, solvent blanks shall be analyzed to confirm the absence of interfering peaks.

7. Apparatus

7.1 *Reflux Extraction*, consisting of 250-mL round-bottom flask with condenser and heating mantle to fit.

7.2 *Wiley Mill*, with 10 and 20-mesh screens.

7.3 *Wrist-Action Shaker*.

7.4 *Gas Chromatograph*, equipped with a flame ionization detector.

7.5 *Chromatographic Column*, 3.2-mm outside diameter times 1.8 m packed with 20 % UCW-98 on 80/100 mesh Chromosorb P, a similar packed column, or an equivalent capillary column, such as a HP-1 or DB-1.

7.6 *Integrator*, capable of measuring the net peak area on the back side of a solvent peak.

7.7 *Gas Chromatographic Syringe*, 10 μ L.

7.8 *Analytical Balance*, capable of weighing to ± 0.0001 g.

7.9 *Pressure Regulators*, for all required gas cylinders.

7.10 *Filter-Dried Assemblies*, for each required gas cylinder.

7.11 *Soap Film Flowmeter and Stopwatch*, or other means of measuring gas flow rates.

8. Reagents and Materials

8.1 *Cyclohexane*, reagent grade.

8.2 *Isopropyl Alcohol*, reagent grade.

8.3 *Methyl Myristate*, ³99+ %, boiling point 323°C (internal standard).

8.4 *Butylated Hydroxy Toluene*, food grade (2,6-di-tert-butyl-4-methyl-hydroxybenzene).

8.5 *Hydrogen Cylinder*, prepurified.

8.6 *Nitrogen Cylinder*, prepurified, oxygen-free for carrier gas.

NOTE 2—Helium or hydrogen may also be used as the carrier gas.

8.7 *Air*, breathing or water-pumped.

9. Safety Precautions

9.1 Cyclohexane and isopropyl alcohol are flammable. This extraction procedure ~~should~~shall be carried out in a fume hood.

10. Preparation of Gas Chromatograph

10.1 Install the chromatographic column and condition overnight at 200°C with carrier gas flow rate of 35 mL/min. Do not connect the exit end of the column to the detector during this conditioning period. Turn off hydrogen and air flows to the detector while the column is disconnected.

10.2 Connect the exit end of the column to the detector. Set optimum hydrogen and air flow rates for the detector as specified for the chromatograph model in use, or as determined experimentally.

³ The sole source of supply of the methyl myristate known to the committee at this time is Aldrich Chemical Co., P.O. Box 355, Milwaukee, WI 53201. If you are aware of alternative suppliers, please provide this information to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee,¹ which you may attend.