Designation: ~~D7210 – 13~~ D7210 – 21

Standard Practice for
Extraction of Additives in Polyolefin Plastics[1]

This standard is issued under the fixed designation D7210; 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 practice covers a variety of extraction techniques for antioxidant and slip additives used in polyolefins. Typically, the extracted additives are separated and quantified using a chromatographic technique such as the procedure explained in Test Method D6953. Chromatographic analysis of the extracts is not included in this practice.

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~~ safety, health, and ~~health~~environmental practices and determine the applicability of regulatory limitations prior to use.*

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

1.3 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]
D883 Terminology Relating to Plastics
D1600 Terminology for Abbreviated Terms Relating to Plastics
D6953 Test Method for Determination of Antioxidants and Erucamide Slip Additives in Polyethylene Using Liquid Chromatography (LC)
E131 Terminology Relating to Molecular Spectroscopy

2.2 *Other Documents:*
Dionex ASE 350 Application Note 331—Accelerated Solvent Extraction of Additives from Polymeric Materials[3]

3. Terminology

3.1 *Definitions:*

3.1.1 For definitions of plastic terms used in this practice, see Terminologies D883 and D1600.

3.2 For units, symbols, and abbreviations used in this practice refer to Terminology E131.

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[1] This practice is under the jurisdiction of ASTM Committee D20 on Plastics and is the direct responsibility of Subcommittee D20.70 on Analytical Methods and Section D20.70.01 on Physical 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.

[3] https://www.thermofisher.com/order/catalog/product/083114#/083114

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

3.3 *Abbreviations:*

3.3.1 *LDPE*—Low-Density Polyethylene

3.3.2 *LLDPE*—Linear Low-Density Polyethylene

3.3.3 *HDPE*—High-Density Polyethylene

3.3.4 *PP*—Polypropylene

3.4 *Definitions: Additives:*

3.4.1 *BHEB*—2,6-di-t-butyl-4-ethyl-phenol or butylated hydroxyethyl benzene, CAS No. 4130-42-1

3.4.2 *BHT*—2,6-di-t-butyl-cresol or butylated hydroxytoluene, CAS No. 128-37-0

3.4.3 *2-benzotriazol-2-yl-4-methylphenol,* CAS No. 2440-22-4

3.4.4 *bis(3,3,5,5-tetramethyl-4-piperidyl)decanedioate,* CAS No. 91450-21-4

3.4.5 *Cis-13-docosenamide or erucamide slip,* CAS No. 112-84-5

3.4.6 *N,N'-diphenylbenzene-1,4-diamine,* CAS No. 74-31-7

3.4.7 *2,2'-ethylidene bis (4,6-di-t-butyl phenol),* CAS No. 112-84-5

3.4.8 *Tetrakis[methylene (3,5-di-t-butyl-4-hydroxy hydrocinnamate)] methane,* CAS No. 98584-37-3

3.4.9 *1,3,5-trimethyl-2,4,6-tris (3,5-di-t-butyl-4-hydroxybenzyl) benzene,* CAS No. 99346-90-4

3.4.10 *Octadecyl-3,5-di-t-butyl-4-hydroxy hydrocinnamate,* CAS No. 2082-79-3

3.4.11 *Sodium Alkyl Sulfonate,* CAS No. 68411-30-3

3.4.12 *Tris(2,4-di-t-butylphenyl) phosphite,* CAS No. 31570-04-4

3.4.13 *Tris(2,4-di-t-butylphenyl) phosphate (oxidized Tris(2,4-di-t-butylphenyl) phosphite)*

3.4.14 *TNPP*—Tris(nonylphenyl) phosphite, CAS No. 26523-78-4

3.4.15 *Hydrolyzed TNPP*—Nonylphenol, CAS No. 104-40-5

3.4.16 *Oxidized TNPP*—Tris(nonylphenol)phosphate CAS No. 26569-53-9

4. Summary of Practice

4.1 The polymer sample is ground to approximately a 0.5-mm (≈40-mesh) or 1-mm (≈20-mesh) particle size and extracted by one of the techniques listed in this practice.

5. Significance and Use

5.1 Separation and identification of additives used in the manufacture of polyolefin plastics is necessary in order to correlate performance properties with polymer composition. This practice provides guidelines for extracting phenolic antioxidants, phosphite antioxidants, UV stabilizers, antistatic agents, and slip additives, from milled polyolefin plastics .

5.2 Water bath shakers and steam-reflux Soxhlet extraction techniques have also been used to successfully remove additives from polyolefin plastics.

6. Interferences

6.1 A major source of interferences can be from solvent impurities. For this reason, quality, high-grade solvents shall be used with this practice. It is recommended that solvents be examined prior to use.

6.1.1 Solvent evaporation after an extraction technique can increase the concentration of impurities.

6.2 The milling or grinding process has the potential to cause a low bias. For example, some erucamide slip is known to be lost during milling. Excessive grinding will cause degradation of some antioxidant additives.

6.3 Excessive fillers (for example, pigments, talc, etc.) in the polymer formulation can mask the sensitivity of polymer additive detection.

7. Apparatus

7.1 *~~Wiley~~Grinding Mill,* or equivalent, equipped with a 20-mesh screen and water-cooled jacket to prevent the thermodegradation of antioxidants such as BHT.

NOTE 2—A ~~SPEX Certiprep, 6750 Freezer Mill~~ freezer mill using liquid nitrogen as a coolant has also been used to successfully prepare milled polyolefin samples.

7.2 *Extraction Apparatus*

7.2.1 *Reflux-Extraction Apparatus,* consisting of a condenser (24/40 ground-glass joint), a flat bottom 125-mL flask having a 24/40 ground glass joint, and a hot plate with magnetic stirrer.

7.2.2 *Soxhlet Apparatus,* consisting of a Soxhlet extraction tube (24/40 ground-glass joint at bottom and a 34/45 ground-glass joint at top), an Allihn condenser (34/45 ground-glass joint), a flat bottom 125-mL flask having a 24/40 ground-glass joint, and a hot plate with magnetic stirrer. Automated Soxtec Solvent extraction systems or equivalent devices are also acceptable for use.

7.2.3 *Accelerated Solvent Extractor (ASE) Apparatus*[4,5,6], equipped with 11-mL extraction cells and a solvent controller (optional) or equivalent.

7.2.4 *Microwave Extraction Apparatus ~~(MAE)~~(MAE),* ~~,~~ equipped with 100-mL extraction vessels or equivalent.

7.2.5 *Sonication Extraction Apparatus,* consisting of a suitable ultrasonic ~~cleaner such as an Aquasonic Model 75D Ultrasonic Cleaner or equivalent.~~cleaner.

7.3 *Filter System,* for nonaqueous solutions (pore size of 0.22 µm to 0.5µm).

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

8. Reagents and Materials

8.1 *Cellulose Extraction Thimble*—16 mm by 60 mm, single thickness for use with Soxhlet apparatus.

8.2 *Cellulose Extraction Thimble*—17 by 19 by 37 mm, for use in the ASE system.

8.3 *Microwave Extraction Vessels*—100-mL extraction vessel.

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[4] The Accelerated Solvent Extractor (ASE) Apparatus is a registered trademark of Dionex Corporation.

[5] The sole source of supply of the apparatus ~~(ASE 200)~~ (ASE) known to the committee at this time is Thermo Fisher/Dionex Corporation, 1228 Titan Way, P.O. Box 3603, Sunnyvale, CA 94088-3603, USA; www.Dionex.com.

[6] 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.

NOTE 3—11-mL extraction cells were used to evaluate the ASE portion of this practice and 100-mL extraction vessels were used to evaluate the MAE portion of this practice.

8.4 *Cyclohexane,* optima grade, spectroquality, or chromatography quality reagent.

8.5 *Isobutanol,* optima grade, spectroquality, or chromatography quality reagent.

8.6 *2-Propanol or Isopropanol,* optima grade, spectroquality, or chromatography quality reagent.

8.7 *Methylene Chloride,* optima grade, spectroquality or chromatography quality reagent.

9. Safety Precautions

9.1 Isopropanol, isobutanol, and cyclohexane are flammable. The extraction cells used in the instruments shall be free of leaks.

9.2 Accelerated solvent and microwave extraction systems operate at elevated temperatures and pressures. Follow all safety precautions supplied by the instrument manufacturer.

10. Sample Preparation

10.1 Mill the sample to a particle size of 1-mm or smaller using a cooled ~~Wiley Mill~~grinding mill or another suitable grinding apparatus.

10.2 Weigh 0.5 ± 0.01 to 10.0 ± 0.01 g of sample into the appropriate vessel (that is, flat-bottom flask for reflux extraction procedures, cellulose extraction thimble for Soxhlet or ASE extraction, or microwave vessel for MAE extraction, 8 oz glass jar for ultrasonic bath extraction). The required sample size will vary from laboratory to laboratory and will depend on the sensitivity of the analytical determinative method employed.

10.3 Depending on the extraction procedure chosen and the type of polyolefin, or additive, or both, to be extracted, prepare the extraction solvent as follows:

10.3.1 *Reflux or Soxhlet Extraction*

10.3.1.1 LDPE add 5.0 ± 0.01 g of sample into the flask, add a stirring bar, accurately add 50.0 mL of a suitable solvent such as 2-propanol and boil for 1 h (with stirring).

10.3.1.2 LLDPE add 5.0 ± 0.01 g of sample into the flask, add a stirring bar, accurately add 50.0 mL of a suitable solvent such as either isobutanol or 2-propanol and boil for 90 min (with stirring).

10.3.1.3 HDPE add 5.0 ± 0.01 g of sample into the flask, add a stirring bar, accurately add 50.0 mL of a suitable solvent such as cyclohexane and boil for 1 h (with stirring).

10.3.1.4 PP add 5.0 ± 0.01 g of sample into the flask, add a stirring bar, accurately add 50.0 mL of a suitable solvent such as methylene chloride: cyclohexane (75+25) and boil for 90 min (with stirring).

NOTE 4—If internal standard calibration is used with reflux or Soxhlet extraction, add the internal standard to the extraction solvent at the start of the extraction procedure. If internal standard calibration is used with accelerated solvent or microwave assisted extraction, add the internal standard to the extraction vessel prior to initiating the extraction.

NOTE 5—If Soxhlet extraction is used, add the sample to the cellulose extraction thimble and place the thimble in the Soxhlet extraction tube before initiating the extraction.

NOTE 6—A suitable solvent is one that results in a quantitative extraction of the additive(s) of interest.

10.3.1.5 Cool the solution to room temperature by raising the flask off the hot plate while it is still attached to the condenser.

10.3.1.6 Analyze the extract using an appropriate chromatographic technique.