



Designation: **D5227 – 01 (Reapproved 2008)^{ε1} D5227 – 13**

Standard Test Method for Measurement of Hexane Extractable Content of Polyolefins¹

This standard is issued under the fixed designation D5227; 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.

^{ε1} NOTE—Reapproved with editorial changes in November 2008.

1. Scope Scope*

1.1 This test method describes an extraction/gravimetric procedure for determination of the amount of hexane soluble low molecular weight material present in polyethylene, polypropylene, ethylene-propylene copolymers, and ethylene-vinyl acetate copolymers. This test method is a modification of the Food and Drug Administration (FDA) procedure for determining hexane extractables of polyolefins. This test method is based upon the presumption that the weight of the residue extract present in the solvent is equal to the amount extracted from the film sample and could therefore be quantified by measuring the weight loss of the extracted film, eliminating the complex and time-consuming evaporation process described in 21 CFR 177.1520.

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.*

1.3 *The values stated in SI units are to be regarded as the standard.* Units used in 21 CFR 177.1520 are also used in this test method. Units are in conformance with Federal Code 21 CFR 177.1520, from which this test method is derived.

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

2. Referenced Documents

2.1 *ASTM Standards:*²

D883 Terminology Relating to Plastics

D1239 Test Method for Resistance of Plastic Films to Extraction by Chemicals

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

2.2 *Federal Document:*³

21 CFR 177.1520

3. Terminology

3.1 The definitions given in Terminology D883, D1600, and E131 are applicable to this test method.

3.2 *Abbreviations:*

3.2.1 *EVA*—ethylene-vinyl acetate copolymer.

3.2.2 *LDPE*—low-density polyethylene.

3.2.3 *HDPE*—high-density polyethylene.

3.2.4 *LLDPE*—linear low-density polyethylene.

3.2.5 *FDA*—Food and Drug Administration.

3.2.6 *PP*—polypropylene.

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. Current edition approved Nov. 1, 2008; June 1, 2013. Published March 2009; July 2013. Originally approved in 1992. Last previous edition approved in 2004 as D5227 – 01; D5227 – 01(2008)^{ε1}. DOI: 10.1520/D5227-01R08E01-10.1520/D5227-13.

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 Available from Standardization Documents Order Desk, DODSSP, Bldg. 4, Section D, 700 Robbins Ave., Philadelphia, PA 19111-5098, http://www.dodssp.daps.mil.

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

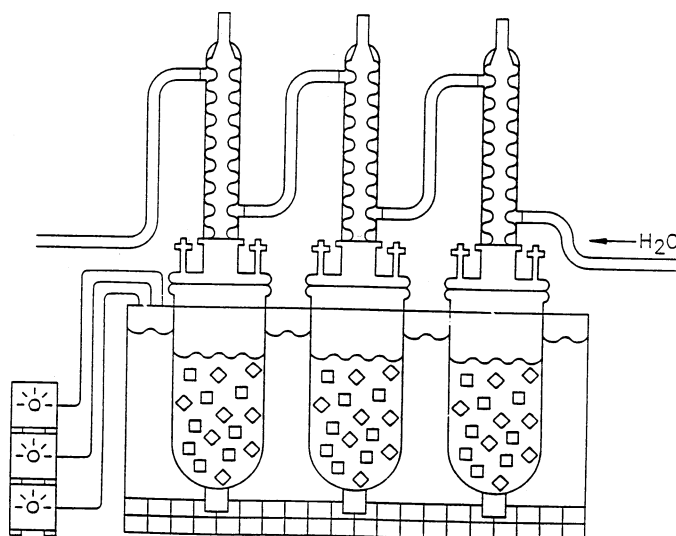


FIG. 1 Resin Kettle Setup

4. Summary of Test Method

4.1 Film samples are extracted with hexane for 2 h at $49.5 \pm 0.5^\circ\text{C}$, dried, and weighed.

4.2 The loss in weight of the film is presumed to be equal to the extractable content determined by solvent evaporation in the FDA protocol.

5. Significance and Use

5.1 FDA requirements for maximum extractables are specified for resin and uses. This test method provides a means to determine the amount of hexane-soluble low molecular weight material present in polyolefins. It is applicable to resins containing greater than 0.20 % extractables.

6. Apparatus

6.1 *Water Bath*, maintained at $49.5 \pm 0.5^\circ\text{C}$.

6.2 *Resin Kettle*, 1500-mL.

6.3 *Kettle Head*, 3-neck, with one 45/50 and two 24/40 female joints, and appropriate stoppers.

6.4 *Clamp*.

6.5 *Allihn Condenser*, Size C, with 45/50 male joint.

6.6 *Plastic Sleeves*, tetrafluoroethylene (TFE), to fit Allihn condenser 45/50 male joint.

6.7 *Vacuum Oven*, capable of maintaining $80 \pm 5^\circ\text{C}$ and a minimum of 25-in. Hg pressure.

6.8 *Magnetic Stirring Bar*, egg-shaped, TFE-coated, $1\frac{1}{2}$ by $\frac{5}{8}$ in.

6.9 *Submersible Magnetic Stirring Motor*, with power supply.

6.10 *Analytical Balance*, capable of weighing to 0.1 mg.

7. Reagents and Materials

7.1 *n-Hexane*, aromatic free (<1 mg/L), minimum 85 % *n*-Hexane-reagent grade or equivalent. The solvent must be free of aromatic compounds that would significantly increase the solubility of the resin. The solvent grade specified represents the minimum required purity.

8. Materials

8.1 *Blown Film*, compression molded films, or cast films are suitable for testing.

8.2 *Film*, approximately 2.5 g, with a thickness not exceeding 4 mil is required for a single determination.

9. Procedure

9.1 Assemble the resin kettle setup with glass stopper, clamp, and magnetic stirring bar. (See Fig. 1.)