



Designation: D5227 – 21

# Standard Test Method for Measurement of Hexane Extractable Content of Polyolefins<sup>1</sup>

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 ( $\epsilon$ ) indicates an editorial change since the last revision or reapproval.

## 1. 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, health, and environmental 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 to this standard.

1.4 *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:*<sup>2</sup>  
**D883 Terminology Relating to Plastics**

<sup>1</sup> 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.

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<sup>2</sup> 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.

- 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  
E456 Terminology Relating to Quality and Statistics  
E691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method  
E2935 Practice for Evaluating Equivalence of Two Testing Processes  
2.2 *Federal Document:*<sup>3</sup>  
21 CFR 177.1520 Olefin Polymers

## 3. Terminology

- 3.1 The definitions given in Terminology D883, D1600, E131, and E456 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.

## 4. Summary of Test Method

- 4.1 Film samples are extracted with hexane for 2 h at 49.5  $\pm$  0.5°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.

<sup>3</sup> 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

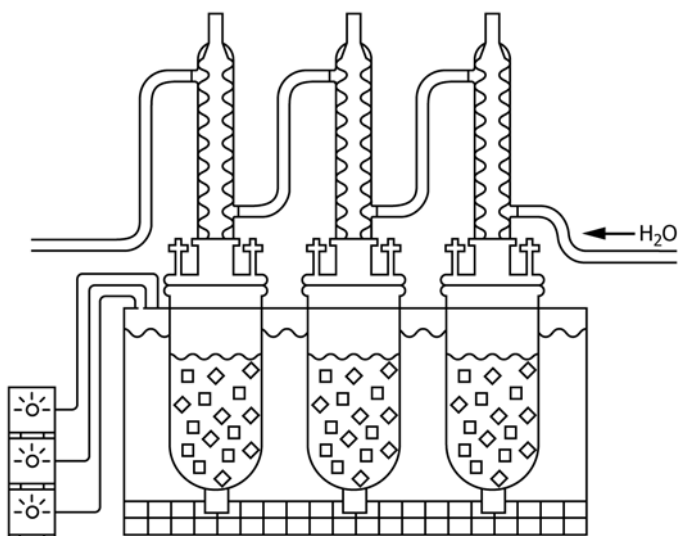


FIG. 1 Resin Kettle Setup

## 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.)
- 9.2 Add 1000 mL of *n*-Hexane to the kettle assembly.

9.3 Stopper the kettle and clamp the assembly into the water bath set at  $49.5 \pm 0.5^\circ\text{C}$ .

NOTE 2—Temperature is a critical factor in this analysis and must not vary more than  $1^\circ\text{C}$ . If the temperature exceeds these limits, the test must be discontinued and restarted. The FDA protocol also states the temperature of the contents must be brought to  $49.5 \pm 0.5^\circ\text{C}$  within 20 to 25 min.

NOTE 3—The level of water in the bath must be kept at least 2 cm above the level of the solvent in the kettle to ensure the temperature equilibrium. Position the kettle so that the center bottle of the kettle is sitting on a submersible stirrer. Start stirring and allow the hexane to heat for 1 h.

9.4 Using gloves and metal tweezers to avoid sample contamination, cut 2.7 g of the prepared film sample (4 mil or less in thickness) into 1-in. squares using clean sharp scissors.

NOTE 4—Care must be exercised when cutting the samples to avoid ragged edges on the specimen. Small shards of film or contamination present at initial weighing can easily be lost during the test, adversely affecting the test results.

9.5 Weigh  $2.5 \pm 0.05$  g of film squares and record the initial film weight to the nearest 0.1 mg. Also record the number of film pieces.

NOTE 5—Forty or more squares will be obtained depending on the film thickness. Some laboratories have found that a basket assembly, as shown in Appendix X1, eliminates the need to count the film pieces before and after the solvent extraction step.

9.6 Add the film sample to the hexane making sure all squares become immersed in the solvent. (Use tweezers.) Replace the kettle head with condenser column. Extract for 2 h.

9.7 After the extraction period:

9.7.1 Filter the contents of the resin kettle through the fritted porcelain funnel.

9.7.2 Transfer the film squares, using tweezers, to a 200-mL Berzelius beaker and recount the film pieces to verify that none were lost during transfer.

9.7.3 Cover the beaker with a watchglass and place it in a vacuum oven at  $80 \pm 5^\circ\text{C}$  for 2 h.

9.7.4 After 2 h, remove the covered beaker from the vacuum oven and place it in a desiccator to cool to room temperature (estimated 1 h).

9.8 Remove the film squares using tweezers and weigh them to the nearest 0.1 mg.

9.9 Repeat 9.7.3 and 9.7.4 until a constant weight is obtained.

## 10. Calculation

10.1 Calculate the weight percent of extractables present in the original sample as follows:

$$\text{hexane extractables, \%} = \frac{(A - B) \times 100 \times 0.935}{A} \quad (1)$$

where:

- A = weight of original sample film, g,
- B = weight of the film after extraction, g, and
- 0.935 = correlation factor to eliminate the bias between the original FDA technique and this alternate test method.

## 11. Report

11.1 Report the hexane extractables to the nearest 0.01 % as calculated in 10.1.

## 12. Precision and Bias<sup>4</sup>

### 12.1 Hexane Extractable Content of Polyolefins:

12.1.1 The precision of this test method is based on an interlaboratory study of D5227, Hexane Extractable Content of Polyolefins, conducted in 1990. Ten laboratories tested five different materials. Every “test result” represents an individual determination. Each laboratory was asked to submit two replicate test results, from a single operator, for each material. Practice E691 was followed for the design and analysis of the data; the details are given in ASTM Research Report No. RR:D20-1173.

NOTE 6—**Warning:** The data in Table 1 shall not be rigorously applied

<sup>4</sup> Supporting data are available from ASTM Headquarters. Request RR:D20-1173.

**TABLE 1 Hexane Extractable Content of Polyolefins, Weight**

Material	Average	<i>Sr</i>	<i>SR</i>	<i>r</i>	<i>R</i>
HDPE	0.26	0.03	0.05	0.09	0.15
LLDPE	0.88	0.11	0.16	0.31	0.46
LDPE	1.74	0.08	0.15	0.21	0.42
EVA	3.54	0.28	0.33	0.78	0.93
PP	3.80	0.29	0.35	0.81	0.98

to acceptance or rejection of material, as those data are specific to the interlaboratory study and are not necessarily representative of other lots, conditions, materials, or laboratories. Users of this test method shall apply the principles outlined in Practice E691 to generate data specific to their laboratory and materials, or between specific laboratories.

12.1.2 *Bias*—There are no recognized standards by which to estimate bias of this test method.

## 13. Keywords

13.1 ethylene-propylene copolymers; ethylene-vinyl acetate copolymers; extractables; FDA; hexane; plastics; polyethylene; solvent extraction

## APPENDIX

### (Nonmandatory Information)

#### X1. BASKET ASSEMBLY FOR *n*-HEXANE EXTRACTABLES

X1.1 If one uses the basket assembly shown in Fig. X1.1, the following steps should be performed after the 2-h hexane extraction (9.6) Also, the sample and basket must be weighed together and recorded before starting the extraction:

X1.1.1 Rinse the basket and contents by immersing several times in fresh *n*-hexane contained in a small beaker, allowing the basket to drain between rinsings.

X1.1.2 Remove the excess solvent by briefly blowing the basket with a stream of nitrogen or dry air.

X1.1.3 Place the basket in a vacuum oven for 2 h at 80±5°C, then cool to ambient temperature in a desiccator (estimated 1 h).

X1.1.4 Reweigh the basket and its contents to the nearest 0.1 mg.