Designation: D5492 – 17 (Reapproved 2024)

Standard Test Method for Determination of Xylene Solubles in Propylene Plastics[1]

This standard is issued under the fixed designation D5492; 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 is to be used for determining the 25 °C xylene-soluble fraction of polypropylene homopolymers and copolymers.

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.*

NOTE 1—This test method is technically equivalent to ISO 16152.

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D1600 Terminology for Abbreviated Terms Relating to Plastics (Withdrawn 2024)[3]

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 *ISO Standard:*

ISO 16152 Plastics—Determination of Xylene Solubles of Polypropylene[4]

3. Terminology

3.1 *Definitions:*

3.1.1 For definitions of plastic terms see Terminology D883 and for abbreviations see Terminology D1600.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *soluble-fraction (S_S)*—the percentage of the polymer mass that does not precipitate out when the polymer solution is cooled from reflux temperature to +25 °C ± 0.5 °C and held at that temperature for a specified period of time.

4. Summary of Test Method

4.1 A weighed amount of sample is dissolved in xylene under reflux conditions. The solution is cooled under controlled conditions and maintained at a +25 °C equilibrium temperature so that the crystallization of the insoluble fraction takes place. When the solution is cooled the insoluble portion precipitates and is isolated by filtration. The xylene is evaporated from the filtrate, leaving the soluble fraction in the residue. The percentage of this fraction in the plastic is determined gravimetrically.

5. Significance and Use

5.1 The results of this test provide a relative measure of the total soluble fraction of polypropylene homopolymers and copolymers. The soluble fraction approximately correlates to the amorphous fraction in the polypropylene. Xylene is widely used for determining the soluble fraction in polypropylene as it is more specific to the atactic fraction than other solvents. The concentration of a soluble fraction obtained with a specific solvent has been found to relate closely to the performance characteristics of a product in certain applications, for example film and fiber. Data obtained by one solvent and at one precipitation time cannot be compared with data obtained by another solvent or precipitation time, respectively.

6. Interferences

6.1 It is possible that materials with solubilities similar to the soluble fraction, such as additives, can interfere with the measurement of solubles. When present in concentrations that are judged to impart a significant error to the soluble-fraction data, the level of interference must be determined and corrections made.

[1] This test method is under the jurisdiction of ASTM Committee D20 on Plastics and is the direct responsibility of Subcommittee D20.15 on Thermoplastic Materials.

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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] The last approved version of this historical standard is referenced on www.astm.org.

[4] Available from American National Standards Institute (ANSI), 25 W. 43rd St., 4th Floor, New York, NY 10036.

6.2 It is possible that small-particle fillers and pigments and insoluble gels present in the polymer can pass through the filter and cause errors in the measurement.

6.3 The polymer flakes and spheres must be dried before testing to eliminate moisture that can influence the initial weight of sample added to the flask.

7. Apparatus

7.1 *Reflux-Condenser Apparatus,* minimum 400 mL, with 24/40 glass joint.

7.1.1 The use a teflon seal sleeve around the glass joint is an acceptable option provided it has been determined that components from the sleeve or tape are not extracted by the xylene.

7.1.2 The use of silicone greases or other greases shall be avoided.

7.2 *Flat- or Round-Bottom Boiling Flask,* with one or two necks, minimum 400 mL with 24/40 joint, Erlenmeyer flask, or flat-bottomed cylindrical bottle.

7.3 *Insulation Disk,* made of fiberglass or rock wool.

7.4 *Electromagnetic Stirrer Unit,* with temperature-controlled heating plate, oil bath, heater block, or heating mantle capable of maintaining 145 °C to 150 °C.

7.5 *Stirring Bar.*

7.6 *Pipet,* Class A, 200 mL or equivalent.

7.7 *Pipet,* Class A, 100 mL or equivalent.

7.8 *Glass-Stoppered Volumetric Flask,* 250 mL.

7.9 *Thermostatically Controlled Water Bath,* at +25 °C ± 0.5 °C.

7.10 *Electromagnetic Stirrers.*

7.11 *Filter Paper,* fluted, Whatman No. 4, No. 541,[5] or equivalent, at least 125 mm in diameter.

7.12 *Funnel,* 60°, or equivalent, at least 125 mm in diameter.

7.13 *Heated Vacuum Oven.*

7.14 *Aluminum or stainless steel pans or beaker at a minimum 125 mL capacity, but not larger than 300 mL*, with smooth sides or other suitable container of similar design.

7.15 *Temperature-Controlled Heating Plate.*

7.16 *Analytical Balance,* with minimum weighing sensitivity to 0.0001 g (a sensitivity of 0.00001 g is preferred).

7.17 *Desiccator,* containing appropriate desiccant.

7.18 *Timer,* preferably with an alarm, in minutes.

7.19 *Oven,* conventional forced air or gravity.

[5] The sole sources of supply (EU/U.S.) of the apparatus known to the committee at this time are Whatman Int'l. Ltd., Maidstone, England or from Fisher Scientific, 711 Forbes Ave., Pittsburgh, PA 15219. 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.

8. Reagents

8.1 *Reagent-Grade Ortho-Xylene (o-Xylene)*—Assay gas chromatography (GC) = 98 % min; less than 2 % ethylbenzene as established by GC; evaporation residue at 140 °C less than 0.002 g⁄100 mL; boiling point 144 °C.

8.2 *Reagent-Grade Para-Xylene (p-Xylene)*—Assay gas chromatography (GC) = 98 % min; less than 2 % ethylbenzene as established by GC, evaporation residue at 140 °C less than 0.002 g⁄100 mL; boiling point 138 °C.

NOTE 2—Mixed xylene may be used within a laboratory if the ratio of para-xylene to ortho-xylene remains constant and the level of ethylbenzene is less than 2 %.

8.3 Reagent grade ortho-xylene shall be used as the reference solvent whenever there is a dispute between laboratories on test results, unless the laboratories agree otherwise.

9. Reagent and Specimen Preparation

9.1 *Preparation of the Xylene:*

9.1.1 Stabilization of the xylene is not required.

NOTE 3—When testing non-stabilized polypropylene powders, (that is, unstabilized reactor products) it is recommended that antioxidants be added to prevent degradation. This addition is optional if previous testing has shown there is no significant change in xylene soluble level.

NOTE 4—Butylated hydroxyl toluene (BHT), 4,4 thiobis (6-tert-butyl-m-cresol), or tetrakis (3,5-di-tert-butyl-4-hydroxy-hydrocinnamate) methane at an approximate concentration of 0.02 g⁄L of xylene have been found to be effective stabilizers. Agitate with a magnetic stirring bar and heat for a minimum of one hour at 80 °C to 90 °C to ensure the thorough mixing of the antioxidants and the xylene. This is a suitable heating temperature for BHT, which is highly volatile.

9.1.2 Degas the xylene. Using nitrogen gas, purge the xylene for a minimum of 1 h every 24 h.

9.2 *Determine the Level of Contamination in the Xylene (Solvent Blank):*

9.2.1 The purpose of the solvent blank is to determine whether the xylene to be used contains significant amounts of evaporation residue or foreign components. A solvent-blank test for residue shall be run on every new lot of xylene. Test and average the solvent-blank results, for three aliquots per bottle or lot of xylene. Each aliquot shall be 200 mL.

9.2.2 If the xylene is an extra pure grade (minimum 99.5 %) and is used within three days after being opened, the determination of the blank is not required. If used more than three days after being opened, a solvent blank must be run.

NOTE 5—It is recommended that xylene be purchased in glass or glass-lined containers and of a size such that the xylene is used within three days, once opened. Containers of larger size may be used if the xylene is used up within a short period of time. The purpose of the short time period is to ensure purity and minimize moisture pickup and other contaminants.

9.2.3 Pipet 200 mL of unstabilized or stabilized xylene into a clean empty flask.

9.2.4 Place a 125 mm diameter or larger No. 4 filter paper or equivalent in a 125 mm diameter of larger funnel in a funnel rack over a 250 mL glass-stoppered flask.

9.2.5 For each sample blank, pour the contents from the flask into a funnel and allow the filtrate to drip into a second flask. Continue the filtration until all the filtrate has been collected.