



Designation: ~~D5492-06~~ Designation: D5492 - 10

Standard Test Method for Determination of Xylene Solubles in Propylene Plastics¹

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~~ 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 and health practices and determine the applicability of regulatory limitations prior to use.

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

2. Referenced Documents

2.1 ASTM Standards:²

D883 Terminology Relating to Plastics

D1600 Terminology for Abbreviated Terms Relating to Plastics

E691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method

2.2 ISO Standard:

ISO 16152 Plastics—Determination of Xylene Solubles of Polypropylene³

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 $+25 \pm 0.5^\circ\text{C}$ and held at that temperature for a specified period of time.

4. Summary of Test Method

~~4.1~~ 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 $+25^\circ\text{C}$ 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 ~~ortho-xylene~~ 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~~ 5.1 The results of this test provide a relative measure of the total soluble fraction of polypropylene homopolymers and copolymers. The soluble fraction can be approximately correlated to the amorphous fraction in the polypropylene. Xylene is widely used for determining the soluble fraction in polypropylene. Xylene 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~~ 6.1 It is possible that materials with solubilities similar to the soluble fraction, such as additives, may can interfere with the measurement of solubles. When present in concentrations that are judged to impart a significant error to the

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. Current edition approved May Aug. 1, 2006-2010. Published June 2006-September 2010. Originally approved in 1994. Last previous edition approved in 2003-2006 as D5492-98(2003)-D5492 - 06. DOI: 10.1520/D5492-106.

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

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

soluble-fraction data, the level of interference must be determined and corrections made.

~~6.2 Small-particle fillers and pigments~~ 6.2 It is possible that ~~may pass through the filter~~ small-particle fillers and pigments and insoluble gels present in the polymer may ~~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*, 400 mm, with 24/40 glass joint.
- 7.2 *Flat-Bottom Boiling Flask*, with one or two necks, 400 mL with 24/40 joint, Erlenmeyer flask, or cylindrical bottle.
- 7.3 *Insulation Disk*, made of fiberglass or rock wool.
- 7.4 *Electromagnetic Stirrer Unit*, with temperature-controlled heating plate, thermostatted oil bath, or heater block capable of maintaining 145 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 $+25 \pm 0.5^\circ\text{C}$.
- 7.10 *Electromagnetic Stirrers*.
- 7.11 *Filter Paper*, fluted, Whatman No. 4, No. 541,⁴ 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 Pans*, 300-mL capacity, 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.

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 from lot to lot and the level of ethylbenzene is less than 2 %.

NOTE 3—Grade

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. Procedure

9.1 *Preparation of the Xylene:*

~~9.1.1 Stabilization of the xylene is not required. When testing non-stabilized polypropylene powders or spheres, antioxidants may be added to prevent degradation. This addition is optional if previous testing shows there is no significant change in xylene soluble level.~~

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NOTE 3—When testing non-stabilized polypropylene powders or spheres, antioxidants may 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), and/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~~ 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):*

⁴ 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, which you may attend.

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 should 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.

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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 after three days after being opened, a solvent blank must be run.

NOTE 5—If the xylene is an extra pure grade (minimum 99.5%) and is used immediately after being opened, the determination of the blank may be eliminated. If not used immediately (within three days) a solvent blank must be run. It is recommended that xylene be purchased in glass or glass-lined containers and of a size such that the xylene will be 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.

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

9.2.3 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.

9.2.4 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.

9.2.5 Dry the aluminum pans (or other suitable container) for 30 min in an oven at 200°C. Cool the pans in a desiccator until ready to use. For each sample weigh a clean, dry pan on the analytical balance to the nearest 0.0001 g.

9.2.67 With a Class A pipet, pipet a 100-mL aliquot of the filtered xylene into the weighed pan.

9.2.78 Place the pan on a temperature-controlled heating plate maintained at 145 to 150°C. Allow the aliquot to obtain a rolling boil to prevent splashing. Blanket the pan with a slow stream of nitrogen. Continue heating the pan until the residue in the dish is almost dry.

9.2.8 Place the pan into a vacuum oven at 100 ± 10°C at a pressure less than 13.3 kPa for a known suitable time where constant weight is observed.

9.2.9 Cool the pan to room temperature in a desiccator for a minimum of 15 minutes and weigh the pan to the nearest 0.0001 g. Calculate the average blank-residual mass of the three determinations.

9.3 Determine the Percent Soluble Fraction in the Polymer:

9.3.1 Dry the polypropylene powder or spheres before analysis. It is not necessary to dry the pellets unless it is known that they contain high levels of moisture. If necessary, dry the samples in a vacuum oven at 70 ± 5°C, in a vacuum of 13.3 kPa for a minimum of 20 min. Cool the sample in a desiccator to prevent moisture pickup.

NOTE 6—For large pellets or spheres, where there is concern that the polymer sample will not dissolve in a reasonable time frame, the pellets or spheres may be ground to an appropriate size to afford a faster dissolution. Attention shall be paid not to mechanically shear the polymer and increase the residual solubles level. Ground material shall be dried as specified in 9.3.1.

9.3.2 Weigh out a sample in accordance with Table 1. When the expected solubles level is unknown or referee testing between laboratories is being conducted, a 2.0 ± 0.1 g sample shall be used, unless there is agreement between the laboratories to use a different sample size. Determine mass of the sample to the nearest 0.0001 g. Pour the sample into a flat-bottom boiling flask. Place a magnetic stirring bar in the flask.

NOTE 7—

9.3.3 Table 1 provides a choice of sample mass. Use the largest sample mass possible to minimize variability of the test data, unless from prior experience it is known that the polymer/xylene solution does not filter readily as in 9.3.139.3.15.

9.3.34 Pipet 200 mL of unstabilized or stabilized xylene into the flask.

9.3.45 Attach the flask to the condenser.

9.3.56 Place an insulation disk on top of the electromagnetic stirrer plate to prevent localized heating of the flask. Position the flask and condenser system on top of the insulation disk (see Fig. 1). Position the nitrogen inlet tube in the top of the condenser. Turn on the cooling water to the condenser.

9.3.6 Blanket the contents of the boiling flask with a slow flow of nitrogen directed across the top of the condenser, not directly into the condenser, to minimize possible xylene loss. Nitrogen flow rate shall be approximately 2 L/h.

9.3.78 Heat the polymer/xylene mixture to reflux temperature while vigorously stirring. The stirring shall be vigorous enough to obtain a deep vortex, which keeps the boiling under control and prevents boiling up into the condenser. Ensure that the reflux is gentle so that localized burning and sticking of the polymer to the flask walls does not occur.

TABLE 1 Sample Size

Expected Solubles	Initial Sample Mass, g ^A
<8 % by mass	4.0000 ± 0.1000 or 2.000 ± 0.1000
8.0 to 30.0 % by mass	2.0000 ± 0.1000
>30.0 % by mass	2.0000 ± 0.1000 or 1.0000 ± 0.1000

^A See Note 79.3.3.

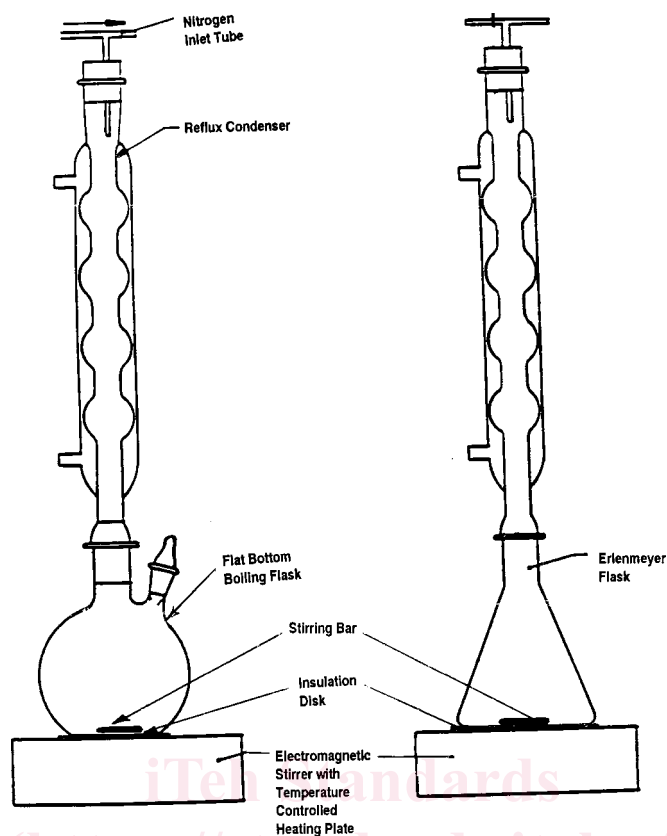


FIG. 1 Equipment Setup

9.3.89 Once the reflux temperature is reached, stir the solution for an additional 30 min. The liquid will appear water-clear.

9.3.910 Remove the heating plate from beneath the flask. Detach the flask from the condenser and lightly stopper the flask. Cool the solution temperature from reflux temperature to below 100°C by cooling the flask in air for 12 to 14 min. A timer, preferably with an alarm, must be used to ensure that the solution is cooled to within a narrow range of temperature from run to run.

9.3.10 Transfer 9.3.11 Transfer the flask to a thermostatically controlled water bath at $25 \pm 0.5^\circ\text{C}$. Do not shake the flask to break up any precipitate before immersing the flask in the bath.

9.3.12 The water bath shall have sufficient cooling capacity to maintain a constant temperature of $25 \pm 0.5^\circ\text{C}$ during cooling.

NOTE 8—Do not shake the flask while the xylene-polymer solution is still hot as this may create a safety hazard.

NOTE 9—Because the rate of cooling affects the crystal size and the rate of crystallization, the time and temperature during the cooling steps are very critical to the final test result. 8—Because the rate of cooling affects the crystal size and the rate of crystallization, the time and temperature during the cooling steps are very critical to the final test result.

9.3.13 Precipitation Time

9.3.13.1 Without stirring, cool the solution for 30 min in the temperature-controlled bath at $25 \pm 0.5^\circ\text{C}$. Using a timer, preferably with an alarm, to ensure that the solution-precipitation time ranges from 30 to 32 min. Remove the flask from the temperature-controlled bath. Stir or gently shake the flask to break up any precipitated-polymer gel.

NOTE 10—The water bath must have sufficient cooling capacity to maintain a constant temperature of $25 \pm 0.5^\circ\text{C}$ during cooling.

9.3.11 Precipitation Time

9.3.11.1 Without stirring, cool the solution for 30 min in the temperature-controlled bath at $25 \pm 0.5^\circ\text{C}$. Using a timer, preferably with an alarm, to ensure that the solution-precipitation time ranges from 30 to 32 min. Remove the flask from the temperature-controlled bath. Stir or gently shake the flask to break up any precipitated-polymer gel. 9—Shorter precipitation times (for example, comparing 30 minutes versus 60 minutes or longer) lead to higher values of soluble content for propylene plastics. Users of this method should be warned that product specification ranges may need to be adjusted in order to compensate for these differences.

NOTE 11—Shorter precipitation times (for example, comparing 30 minutes versus 60 minutes or longer) lead to higher values of soluble content for propylene plastics. Users of this method should be warned that product specification ranges may need to be adjusted in order to compensate for these differences. 10—With the shorter 30 minute precipitation time, a true equilibrium will not be reached. Consequently, it is very critical that both the precipitation time and cooling temperature be controlled very precisely to minimize variance.

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