



Designation: D 5492 – 98

Standard Test Method for Determination of Xylene Solubles in Propylene Plastics¹

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1. Scope

1.1 This test method is to be used for determining the 25°C ortho-xylene-soluble fraction of polypropylene and propylene-ethylene 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 standard is similar to ISO 6427-1982 in title only. The technical content is significantly different.

2. Referenced Documents

2.1 ASTM Standards:

D 883 Terminology Relating to Plastics²

D 1600 Terminology of Abbreviated Terms Relating to Plastics²

2.2 ISO Standard:

ISO 6427-1982 Plastics—Determination of Matter Extracted by Organic Solvents (Conventional Methods) Annex B Standard Method of Test for Determination of Polypropylene Solubility in Cold Xylene³

3. Terminology

3.1 For definitions of plastic terms see Terminology D 883 and for abbreviations see Terminology D 1600.

3.2 There are no terms in this test method that require new or other-than-dictionary definitions.

4. Summary of Test Method

4.1 A weighed amount of sample is dissolved in orthoxylene 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. One of two precipitation-time periods can be used, although the longer precipitation time should be used for homopolymers and copolymers with solubles less than 12 mass

%. When the solution is cooled the insoluble portion precipitates and is isolated by filtration. The orthoxylene 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 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. 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. Xylene is more specific to the atactic fraction than other solvents.

6. Interferences

6.1 Materials with solubilities similar to the polymer fraction, such as additives, may 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.

6.2 Small-particle fillers and pigments that may pass through the filter and insoluble gels present in the polymer may 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*, with temperature-controlled heating plate, thermostated oil bath, or heater block capable of maintaining 145 to 150°C.

7.5 *Stirring Bar*.

7.6 *Pipette*, Class A, 200 mL.

¹ This test method is under the jurisdiction of ASTM Committee D-20 on Plastics and is the direct responsibility of Subcommittee D20.12 on Olefin Plastics.

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² *Annual Book of ASTM Standards*, Vol 08.01.

³ Available from American National Standards Institute, 11 W. 42nd St., 13th Floor, New York, 10036.

- 7.7 Pipette, Class A, 100 mL.
- 7.8 Glass-Stoppered Volumetric Flask, 250 mL.
- 7.9 Thermostatically Controlled Water Bath, at 25°C.
- 7.10 Electromagnetic Stirrers.
- 7.11 Filter Paper, fluted, Whatman No. 4, No. 541,⁴ or equivalent, 200 mm.
- 7.12 Funnel, 60°, 200 mm.
- 7.13 Heated Vacuum Oven.
- 7.14 Disposable Aluminum Pans, 300-mL capacity, with smooth sides.
- 7.15 Temperature-Controlled Heating Plate.
- 7.16 Analytical Balance, with minimum weighing sensitivity to 0.0001 g (a sensitivity of 0.00001 is preferred).
- 7.17 Desiccator, containing appropriate desiccant.
- 7.18 Timer with Alarms, in minutes.

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.

9. Procedure

9.1 Preparation of the o-Xylene:

9.1.1 It is not necessary to stabilize the o-xylene before using, but it may be stabilized if desired.

NOTE 2—BHT,⁵ Irganox 1010,⁶ and Santonox R⁷ have been found to be effective stabilizers for o-xylene.

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

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

9.2.1 The purpose of the solvent blank is to determine whether the o-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 o-xylene. Run and average the solvent-blank results, for three aliquots per bottle or lot of o-xylene. Each aliquot shall be 200 mL.

NOTE 3—It is recommended that o-xylene be purchased in glass or glass-lined containers and of a size such that the o-xylene will be used within three days, once opened. Containers of larger size may be used if the o-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.

9.2.2 Pipet 200 mL of unstabilized or stabilized o-xylene into a clean empty flask.

9.2.3 Place a 200-mm No. 4 filter paper or equivalent in a 200-mm 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 for 30 min in an oven at 150°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.

9.2.6 With a Class A pipette, pipet a 100-mL aliquot of the filtered o-xylene into the weighed aluminum pan.

9.2.7 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 ± 5°C for about an hour at a pressure less than 13.3 kPa.

9.2.9 Cool the pan to room temperature in a desiccator and weigh the pan to the nearest 0.0001 g. Repeat the drying, cooling, and weighing steps until two consecutive weighings agree within 0.0002 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 pellets or spheres before analysis. 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 4—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. Ground material should 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 use a 2.0 ± 0.1 g sample. For referee testing between laboratories a sample 2.0 ± 0.1 g 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 5—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/o-xylene solution does not filter readily as in 9.3.11.

9.3.3 Pipet 200 mL of unstabilized or stabilized o-xylene into the flask.

9.3.4 Attach the flask to the condenser.

9.3.5 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.

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 1.

⁴ Available from Whatman Intn. Ltd., Maidstone, England or from Fisher Scientific, 711 Forbes Ave., Pittsburgh, PA 15219.

⁵ Available from Uniroyal Chemical Co., Inc., Specialty Chemicals Division, World Headquarters, Benson Rd., Middlebury, CT 06749.

⁶ Available from CIBA-GEIGY Corp., Additive Division, Seven Skyline Drive, Hawthorne, NY 10532.

⁷ Available from Monsanto Co., Chemical Group, 800 N. Lindberg Blvd., St. Louis, MO 63167.