Designation: D5492 – 17

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

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

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 Conducting Equivalence Testing in Laboratory Applications

2.2 *ISO Standard:*

ISO 16152 Plastics—Determination of Xylene Solubles of Polypropylene[3]

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 ± 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.

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

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[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] 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 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 ± 0.5°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 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.

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.

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[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,[1] which you may attend.

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 10.2.

9.2.6 Dry the aluminum or stainless steel vessel (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 vessel on the analytical balance to the nearest 0.0001 g.

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

9.2.8 Place the vessel 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 vessel until the residue in the dish is almost dry.

9.2.9 Place the vessel into a vacuum oven at 100 ± 10°C at a pressure less than 13-13.5 kPa for a known suitable time where constant weight is observed.

9.2.10 Cool the vessel 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 polypropylene reactor powder before analysis. If pellets or other large particles are used, drying is not necessary unless it is known that they contain high levels of moisture. If necessary, dry the samples in a vacuum oven at 70 ± 5°C, at a pressure range of 13-13.5 kPa for a minimum of 20 min. Cool the sample in a desiccator to prevent moisture pickup.

NOTE 6—For large pellets or other large particles where there is concern that the polymer sample will not dissolve in a reasonable time frame, grind the pellets or particles to an appropriate size to afford a faster dissolution. Avoid subjecting the sample to excessive mechanical shear, which can lead to an increase in residual solubles level. Ground material shall be dried as specified in 9.3.1.

10. Procedure

10.1 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 the 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.

10.2 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 10.15.

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

10.4 Attach the flask to the condenser.

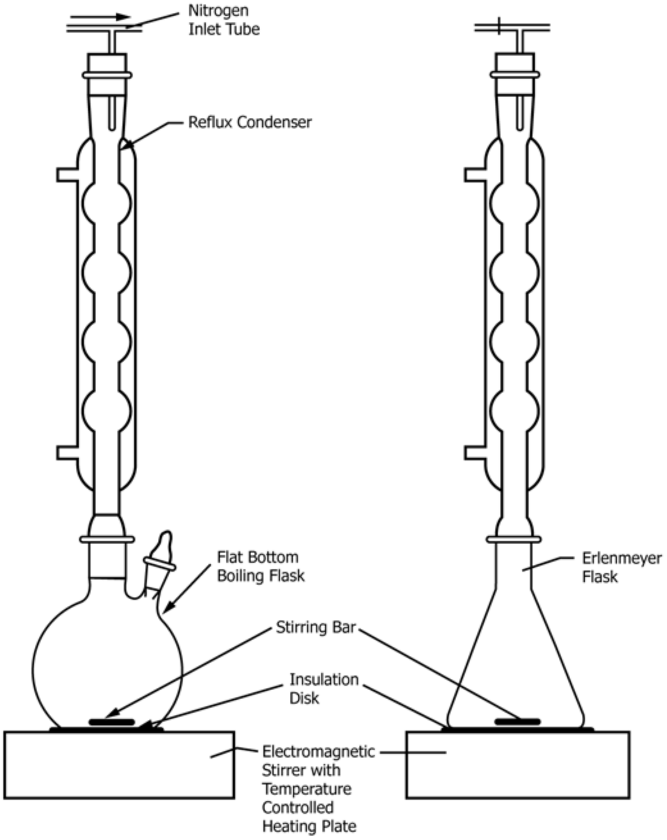

FIG. 1 Equipment Setup

10.5 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, which shows a flat-bottom boiling and Erlenmeyer flask). Position the nitrogen inlet tube in the top of the condenser. Turn on the cooling water to the condenser.

10.6 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. Ensure that nitrogen and water is flowing in the condenser with the aid of bubblers or indicator devices to ensure flow.

10.7 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, splashing, and sticking of the polymer to the flask walls does not occur.

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

NOTE 7—For some polymers the total dissolution time can be longer than 30 min for the liquid to appear water-clear.

10.9 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.

10.10 Transfer the flask to a thermostatically controlled water bath at 25 ± 0.5°C. Do not shake the flask to break up any precipitate before immersing the flask in the bath.

10.11 The water bath shall have sufficient cooling capacity to maintain a constant temperature of 25 ± 0.5°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.

10.12 *Precipitation Time:*

10.13 Without stirring, cool the solution for 30 min in the temperature-controlled bath at 25 ± 0.5°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—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—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.

10.14 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.

10.15 For each sample, pour the contents from the flask into a funnel and allow the filtrate to drip into the second flask.

NOTE 12—With some materials that may have been compounded with filler or rubber, it may be extremely difficult to do the filtration because of clogging of the filter paper. In this case, a filter cloth may be used to remove the large particles of the filler or the precipitate.

10.16 Continue the filtration until all the filtrate has been collected. If the filtered solution is not completely clear it will be necessary to repeat the filtration.

10.17 Dry the aluminum pans (or other suitable container (refer to 7.14)) for 30 min in an oven at 200°C. Cool the pans in a desiccator until ready for use. For each sample weigh a clean, dry pan on the analytical balance to the nearest 0.0001 g.

NOTE 13—It has been shown that other types of containers, including those made with stainless steel can be used by a laboratory.

10.18 Aluminum pans shall be used in the case of referee testing.

10.19 With the clean Class A pipet, pipet a 100-mL aliquot of the soluble filtrate into the weighed pan.

NOTE 14—Smaller aliquots can be used within the laboratory with the understanding that the repeatability may be different.

10.20 A 100-mL aliquot shall be used in the case of referee testing.

10.21 Place the pan on a temperature-controlled hot plate maintained at 140 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. Do not dry completely as this can result in degradation of the residue.

NOTE 15—Degradation or charring of the sample can lead to inaccurate results.

NOTE 16—Other methods of drying can be used if it has been shown that they provide equivalent results to the hot plate method (for example, steam bath drying or vacuum oven).

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

10.23 Cool the pan to room temperature in a desiccator and weigh the pan to the nearest 0.0001 g.

10.24 Obtain the mass of the soluble fraction by subtracting the mass of the pan from the residue-and-pan mass. Adjust the final percent-soluble-fraction data to account for the percent residual contamination from xylene.

11. Calculation

11.1 Calculate the mass percent of the soluble fraction by use of the following equation:

$$S_s = ((V_{bo}/V_{b1} \times (W_2 - W_1) - V_{bo}/V_{b2} \times B)/W_o) \times 100$$

where:

S_S = soluble fraction of sample, %,
V_{bo} = original volume of solvent, mL,
V_{b1} = volume of aliquot used for solubles determination, mL,
V_{b2} = volume of aliquot used for blank residuals, mL,
W_2 = mass of pan and solubles, g,
W_1 = mass of pan, g,
W_0 = mass of original sample, g, and
B = average blank residual, g.

11.2 Materials, such as additives, with solubilities similar to the soluble-polymer fraction interfere in the measurement of percent solubility. Corrections are made to the percent soluble fraction only if the material is present in concentrations that are judged to impart a significant error on the soluble-fraction data. The material(s) must be 100 % soluble and the mass percent of the material present in the polymer must be known for the following correction to be made:

$$S_c = S_s - S_m$$

where:

S_c = corrected soluble fraction of sample, %,
S_s = soluble fraction of sample, %, and

S_m = contamination from materials added to polymer, %, where S_m is the sum of the percent for each of the additives in the material formulation known to be 100 % soluble.

12. Report

12.1 Report the following information:

12.1.1 Sample identification and other pertinent information,

12.1.2 Type and grade of xylene,

12.1.3 Initial sample mass added to flask,

12.1.4 Volume of aliquot used, and

12.1.5 Percent-soluble fraction. (If the data is adjusted for soluble additives, report corrected-percent-solubles fraction, percent-solubles fraction, and percent contamination. Report values to three significant figures.)

13. Precision and Bias

13.1 Tables are based on a round robin[5] tests conducted in 1996 in accordance with Practice E691, involving five materials tested by seven laboratories. One laboratory distributed the five commercial polypropylene formulations chosen for the evaluation. The individual specimens were prepared at the laboratories that tested them. All pellet samples were extracted using a 60-minute precipitation time. Each test result was an individual determination. Each laboratory obtained two test results for each material. Results for the 60-minute precipitation time are listed in Table 2. These results shall only be used as a guideline until a new round robin study can be completed using the 30-minute precipitation time. (**Warning—**The following explanation for *r* and *R* (13.2 through 13.2.3) only are intended to present a meaningful way of considering the approximate precision of this test method. Do not apply the data in Table 2 to acceptance or rejection of material, as these data apply only to materials tested in the round robin and are unlikely to be rigorously representative of the other lots, formulations, conditions, material, or laboratories. Users of this test method shall apply the principles outlined in Practice E691 to generate data specific to their materials and laboratory, or between specific laboratories. The principles of 13.2 through 13.2.3 then would be valid for such data.)

TABLE 2 Xylene Solubles by the 60-Minute Precipitation Method for Seven Laboratories and Five Materials

Material	Values Expressed in %				
	Average	S_r[A]	S_R[B]	r[C]	R[D]
PP	3.58	0.202	0.408	0.565	1.14
PP	5.62	0.490	0.729	1.37	2.04
PP	6.87	0.230	0.715	0.644	2.00
PP	15.0	0.354	0.799	0.993	2.24
PP	22.3	1.22	1.63	3.42	4.56

[A] S_r is the within-laboratory standard deviation for the indicated material. It is obtained by pooling the within-laboratory standard deviations of the test results from all of the participating laboratories.

[B] $S_r = [[s_1)^2 + (s_2)^2 +. . . . . + (s_n)\ ^2]/n]^{1/2}$

S_R is the between-laboratories reproducibility, expressed as standard deviation.

$S_R = [S_r^2 + S_L{}^2]^{1/2}$, where$S_L$ is the standard deviation of laboratory means.

[C] r is the within-laboratory critical interval between two test results = 2.8 × S_r .

[D] R is the between-laboratories critical interval between two test results = 2.8 × S_R .

13.2 *Concept of "r" and "R" in* Table 2—If S_r and S_R have been calculated from a large enough body of data, and for test results from testing one specimen for each test result, then perform the test as follows:

13.2.1 *Repeatability—"r"* the value below which the absolute difference between two individual test results obtained under repeatability conditions are expected to occur with a probability of approximately 0.95 (95 %).

13.2.2 *Reproducibility—"R"* the value below which the absolute difference between two individual test results obtained under reproducibility conditions are to be expected to occur with a probability of approximately 0.95 (95 %).

13.2.3 Inferences about equivalence from applying estimates based only on variability are likely conservative and can lead to incorrect decisions being made. Equivalence testing in ASTM standards is covered by ASTM E2935. These terms and definitions come from E456 on statistical terminology. Alternatively, it's permissible to use an appropriate test for two means.

13.3 There are no recognized standards by which to estimate bias of this test method.

14. Keywords

14.1 plastics; polypropylene; xylene solubles

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[5] Supporting data are available from ASTM Headquarters. Request RR:D20-1199.

SUMMARY OF CHANGES

Committee D20 has identified the location of selected changes to this standard since the last issue, D5492 - 10, that may impact the use of this standard. (August 1, 2017)

(1) Technical additions incorporated and modifications made to Sections 7 (Apparatus), 9 (Reagent and sample preparation-modified), 10 (Procedure-modified).
(2) Editorial and permissive language changes.
(3) Subsections 7.1 and 7.2 revised to expand and explain incorporation of the optional Teflon sleeves and the non-use of silicon grease.
(4) Subsections 13.2.1 and 13.2.2 revised definitions of repeatability and reproducibility.
(5) Notes 8 and 16 added.
(6) Added reference of ASTM E456 and E2935 into the referenced document section.

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