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Plastics — Determination of xylene-soluble matter in polypropylene

Plastiques — Détermination des matières présentes dans le polypropylène solubles dans le xylène

ICS: 83.080.01

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Foreword

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The committee responsible for this document is Technical Committee ISO/TC 61, *Plastics*, Subcommittee SC 9, *Thermoplastic materials*.

This second edition cancels and it replaces ISO16152:2005. which has been technically revised by adding an automated instrument method (Method 2)

Any feedback or questions on this document should the directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.htmL.

Plastics — Determination of xylene-soluble matter in polypropylene

1 Scope

This international standard specifies a method of determining the mass fraction of a polypropylene homopolymer of copolymer which is soluble in xylene at 25°C.

Other materials with solubilities similar to that of the xylene-soluble fraction, such as additives, may interfere with the determination.

This international standard included two methods which are “reference method (method 1)”and “automated instrument method (method 2)”, The “reference method” is used to calibrate the method used for the determination of the xylene-soluble matter in polypropylene.

The “automated instrument method” is a method which can be used for the determination if it is calibrated using the reference method described in clause 4 provided it shows a certain permissible repeatability.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 472, *Plastics — Vocabulary*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 472 and the following apply

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

—IEC Electropedial available at http://www.electropedia.org/

—ISO Online browsing platform: available at https://www.iso.org/obp

3.1
Xylene-soluble fraction

w_s

That percentage, by mass, of the polymer that does not precipitate out when a solution of the polymer in xylene is cooled from reflux temperature to 25°Cand help at that temperature for a specified period of time.

4 Method 1 — reference method

4.1 Principle

The polypropylene is dissolved in hot xylene, then cooled under controlled condition down to 25°C,which result in the precipitation of the insoluble fraction. The soluble matter remains in the xylene. The xylene is then evaporated and the residue weighed.

4.2 Apparatus

4.2.1 Reflux condenser, length 400mm.

4.2.2 Flat-bottomed flask, capacity 400ml, with one or two necks, or conical flask or cylindrical bottle of similar capacity.

4.2.3 Insulation disc, made of fiberglass or mineral wool.

4.2.4 Magnetic stirrer, with temperature-controlled hotplate, thermostatted oil bath or heating block capable of maintaining 140°C to 150°C.

4.2.5 Stirrer bar.

4.2.6 Pipette, class A, 200ml or equivalent.

4.2.7 Pipette, class A, 100ml or equivalent.

4.2.8 Glass-stoppered flask, 250ml.

4.2.9 Thermostatically controlled water bath , with sufficient cooling capacity to maintain a constant bath temperature of 25°C±0.5°C while cooling down the flask containing the xylene/polymer solution (see 4.4.3.8 and 4.4.3.9)

4.2.10 Filter paper, fluted, Whatman No. 4, NO.541 or equivalent, at least 125 mm in diameter.

4.2.11 Funnel, 60º, or equivalent, at least 125 mm in diameter.

4.2.12 Vacuum oven

4.2.13 Disposable aluminium pans, 300ml capacity, with smooth sides.

4.2.14 Temperature-controlled hotplate.

4.2.15 Analytical balance, with a minimum weighing sensitivity of 0,1g (a sensitivity of 0,01 mg is preferred).

4.2.16 Desiccator, containing an appropriate desiccant.

4.2.17 Timer, preferably with an alarm, reading in minutes.

4.2.18 Oven, conventional forced-air or gravity-convection type.

4.3 Reagents

4.3.1 Reagent-grade ortho-xylene(*o*-xylene), assay by gas chromatography(GC) 98%min; ethylbenzene content as determined by GC less than 2%; evaporation residue at 140°Cless than 0,002g/100ml; boiling point 144°C.

Or

4.3.2 Reagent-grade para-xylene(*P*-xylene), assay by gas chromatography(GC) 98%min; ethylbenzene content as determined by GC less than 2%; evaporation residue at 140°Cless than 0,002g/100ml; boiling point 138°C.

Reagent-grade ortho-xylene(4.3.1) shall be used as the reference solvent whenever there if s dispute between laboratories on test results, unless the laboratories agree otherwise.

4.4 Procedure

4.4.1 Preparation of the xylene

4.4.1.1 Although stabilization of the xylene is not required, antioxidants shall be added to prevent degradation when testing unstabilized powders or beads.

Butylated hydroxytoluene (BHT), 4,4-thiobis(6-tert-butyl-m-cresol)and tetrakis[methylene(3,5-di-tert-butyl-4-hydroxyhydrocinnamate)methane at an approximate concentration of 0,02g/l of xylene have been found to be effective stabilizers. Agitate with a magnetic stirrer bar and heat for a minimum of one hour a t 80°C to 90°C to ensure thorough mixing of the antioxidants and the xylene. This heating temperature has been chosen as it is suitable for BHT, which is relatively volatile.

4.4.1.2 Degas the xylene every 24h by purging with nitrogen gas for a minimum of 1h.

4.4.2 Determination of level of impurities in the xylene (solvent blank)

4.4.2.1 The purpose of the solvent blank is to determine the evaporation residue, i.e. amount of foreign matter in the xylene.

A blank test should preferably be run on every new batch of xylene. If however the xylene is extra-pure grade (minimum99,5%) and is used immediately after being opened, a blank test is not necessary. If all the xylene in not used immediately (within 3days), a blank test shall be run. It is recommended that xylene be purchased in glass or glass-line containers of a size such that the xylene will be used within 3 days of opining .Containers of larger size may be used if the xylene is used up within a short period of time. The purpose of this time limit is to ensure purity and minimize pick-up of moisture and other contaminants.

Carry out the blank test in triplicate, taking three test portions from each bottle or batch of xylene.

4.4.2.2 Pipette a 200ml test portion of unstabilized or stabilized xylene into a clean, empty flask.

4.4.2.3 Place a filter paper or equivalent (4.2.10) in a funnel (4.2.11) in funnel rack over a 250 ml flask (4.2.8).

4.4.2.4 Pour the 200ml of xylene into the funnel and allow the filtrate to drip into the flack. Continue the filtration until all the filtrate has been collected.

4.4.2.5 Dry an aluminum pan (4.2.13)for 30min in an oven(4.2.18)at 200°C.Allow the pan to cool to room temperature in a desiccator (4.2.16).Weigh the clean, dry pan to the nearest 0,1mg.

4.4.2.6 Using a pipette, transfer a 100ml aliquot of the filtered xylene into the weighed aluminum pan.

4.4.2.7 Place the pan in a hotplate(4.2.14) maintained at 140°C to 150°C. Allow the xylene to boil gently to avoid splashing. Blanket the pan with a slow stream of nitrogen. Continue heating until the residue in the pan is almost dry.

4.4.2.8 Place the pan in a vacuum oven (4.2.12) at 100°C±10°C at a pressure less than 13,3KPa for a length of time known to be sufficient to dry the residue to constant mass.

4.4.2.9 Allow the pan to cool to room temperature in the desiccator, then weigh to the nearest 0,1mg.

4.4.2.10 Calculate the average of the results of the three determinations.

4.4.3 Determination of percentage xylene-soluble matter in the polypropylene

NOTE The procedure is described for the particular case of a magnetic stirrer with a hotplate (see 4.2.4) and a flat-bottomed flask (see 4.2.2).

4.4.3.1 If necessary, dry the sample before analysis. For drying, use a vacuum oven at 70°C±5°C at a pressure less than 13,3kPa for a minimum of 20 min. Allow the sample to cool in a desiccator to prevent moisture pick-up.

NOTE Polymer flakes and beads are dried before testing to eliminate moisture that can influence the initial mass of the test portion.

With large pellets or beads, where there is concern that the test portion will not dissolve in a reasonable time frame, the pellets or beads may be ground to an appropriate size to ensure faster dissolution .Care shall be taken not to mechanically shear the polymer chains or to increase the level of xylene-soluble matter. Dry the ground material as specified above in this subclause.

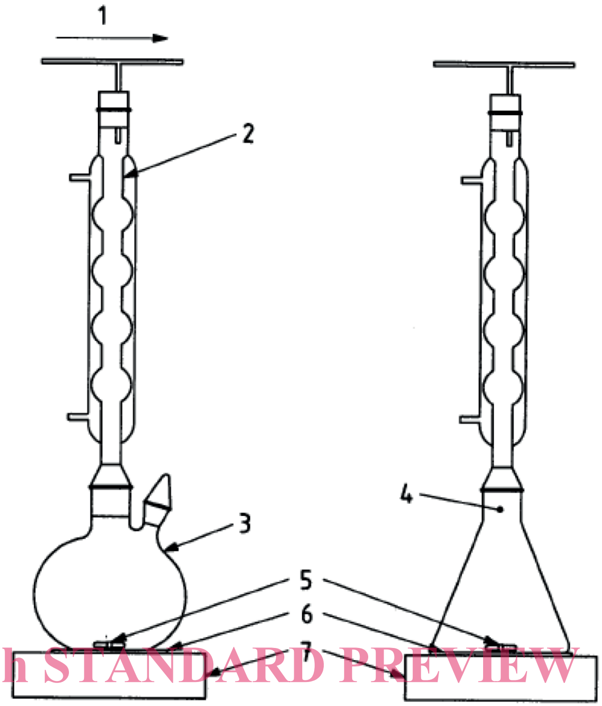
4.4.3.2 Weigh out a test portion of the size given in Table 1,except when the expected soluble-matter content in unknown or referee testing between laboratories is being conducted, in such cases, use a test portion of 2,0g±0,1g.Weigh the test portion to the nearest 0,1mg.Pour the test portion into a flat-bottomed flask. Place a magnetic stirrer bar in the flask.

4.4.3.3 Pipette 200ml of unstabilized or stabilized xylene into the flask.

NOTE Larger quantities may be used in the case lf test portions that are difficult to dissolve or filter and/or when a larger amount of filtrate is needed for further analysis.

4.4.3.4 Attach the flask to the condenser.

4.4.3.5 Place an insulating disc on top of the magnetic-stirrer hotplate to prevent localized heating of the flask Position the flask and condenser on top of the insulating disc (see figure1). Insert the nitrogen supply tube in the top of the condenser. Turn on the cooling water to the condenser.



Key

1 nitrogen flow
2 reflux condenser
3 flat-bottomed flask
4 conical flask
5 stirrer bar
6 insulating disc
7 magnetic stirrer with temperature-controlled hotplate

Figure 1 — Example of equipment set-up

4.4.3.6 Blanket the contents of the flask with a slow flow of nitrogen directed across the top of the condenser, not directly into the condenser, to minimize possible xylene loss. The nitrogen flow rate should preferably be approximately 2L/h.

4.4.3.7 Heat the polymer/xylene mixture to the reflux temperature while stirring .Stirring shall be vigorous enough to obtain a deep vortex. Which keeps the boiling under control and prevents the mixture boiling up into the condenser. Once the reflux temperature is reached, continue to stir for 30 min. The liquid should be perfectly clear. Ensure that refluxing is gentle so that no localized burning or sticking of the polymer to the flack walls occurs.

NOTE If a hotplate is used, it should preferably be used at a temperature setting of approximately 30°Cabove the boiling point of xylene.