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**Plastics — Determination of xylene-  
soluble matter in polypropylene**

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

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## Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular, the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see [www.iso.org/directives](http://www.iso.org/directives)).

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see [www.iso.org/patents](http://www.iso.org/patents)).

Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

For an explanation of the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT), see [www.iso.org/iso/foreword.html](http://www.iso.org/iso/foreword.html).

This document was prepared by Technical Committee ISO/TC 61, *Plastics*, Subcommittee SC 9, *Thermoplastic materials*.

This second edition cancels and replaces the first edition (ISO 16152:2005), which has been technically revised.

The main changes are as follows:

- the introduction has been updated;
- the mandatory normative references clause has been added and subsequent clauses have been renumbered;
- the pipette with capacity of 200 ml has been deleted from the list of apparatus;
- the symbols  $S_s$ ,  $S_m$  and  $S_c$  have been replaced with  $w_s$ ,  $w_m$  and  $w_c$ , respectively (see [3.1](#) and [4.5](#));
- an automated instrumental method has been added (see [Clause 5](#));
- a new informative [Annex A](#) (Precision data for polypropylene obtained from an intercomparison of xylene-soluble content testing) has been added.

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

## Introduction

With the development of analytical and testing instruments, a new technique of measuring xylene-soluble matter in polypropylene by automatic instrument has been developed. Automation improves the precision, removing human-factor variability, and enhances safety for the laboratory and the operator.

The soluble content represents the fraction of amorphous material and it is important as it has a major influence on the properties of the polypropylene.

The method is used to quantify the soluble/amorphous fraction in polypropylene homopolymers as well as copolymers and can be used with other polyolefin materials, such as homopolymer polyethylene or polyethylene  $\alpha$ -olefin copolymers.

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# Plastics — Determination of xylene-soluble matter in polypropylene

## 1 Scope

This document specifies two methods of determining the mass fraction of a polypropylene homopolymer or copolymer which is amorphous, expressed as soluble in xylene at 25 °C.

- Method 1: Reference method;
- Method 2: Automated instrumental method.

## 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 terminology databases for use in standardization at the following addresses:

- ISO Online browsing platform: available at <https://www.iso.org/obp>
- IEC Electropedia: available at <https://www.electropedia.org/>

### 3.1

#### xylene-soluble fraction

$w_s$

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 °C and held at that temperature for a specified period of time

## 4 Method 1 — Reference method

### 4.1 Principle

The polypropylene is dissolved in xylene under reflux conditions, then cooled under controlled conditions and maintained at 25 °C, to ensure the complete crystallization of the insoluble fraction. The xylene-soluble fraction is then recovered by evaporation of the xylene and determined by weighing the residue.

### 4.2 Apparatus

**4.2.1 Reflux condenser**, length 400 mm.

**4.2.2 Flat-bottomed flask**, capacity 500 ml, 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, 100 ml or equivalent.

**4.2.7 Glass-stoppered flask**, 250 ml.

**4.2.8 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](#)).

**4.2.9 Filter paper**, fluted, at least 125 mm in diameter.

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

**4.2.11 Vacuum oven**.

**4.2.12 Disposable aluminium pans**, 300 ml capacity, with smooth sides.

**4.2.13 Temperature-controlled hotplate**.

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

**4.2.15 Desiccator**, containing an appropriate desiccant.

**4.2.16 Timer**, preferably with an alarm, reading in minutes.

**4.2.17 Oven**, conventional forced-air or gravity-convection type.

### 4.3 Reagents and materials

**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 °C less than 0,002 g/100 ml; boiling point 144 °C.

**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 °C less than 0,002 g/100 ml; boiling point 138 °C.

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

**4.3.3 Antioxidants**, 2,6-Di-tert-butyl-4-methylphenol (BHT), or Pentaerythritol tetrakis(3-(3,5-di-tert-butyl-4-hydroxyphenyl)propionate)(1010).



## 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.

An approximate concentration of 0,02 g/l of xylene have been found to be effective stabilizers. Agitate with a magnetic stirrer bar and heat for a minimum of one hour at 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 24 h by purging with nitrogen gas for a minimum of 1 h.

### 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 (minimum 99,5 %) and is used immediately after being opened, a blank test is not necessary. If all the xylene is not used immediately (within 3 days), 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 opening. 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 200 ml test portion of unstabilized or stabilized xylene into a clean, empty flask (4.2.2).

**4.4.2.3** Place a filter paper or equivalent (4.2.9) in a funnel (4.2.10) in funnel rack over a 250 ml flask (4.2.7).

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

**4.4.2.5** Dry an aluminium pan (4.2.12) for 30 min in an oven (4.2.17) at 200 °C. Allow the pan to cool to room temperature in a desiccator (4.2.15). Weigh the clean, dry pan to the nearest 0,1 mg.

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

**4.4.2.7** Place the pan in a hotplate (4.2.13) 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 (4.2.11) at 100 °C ± 10 °C under the pressure of less than 13,3 kPa 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 0,1 mg.

**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

**4.4.3.1** If necessary, dry the sample before analysis. For drying, use a vacuum oven at  $70\text{ °C} \pm 5\text{ °C}$  under the pressure of less than 13,3 kPa 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 timeframe, 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 is unknown or referee testing between laboratories is being conducted, In such cases, use a test portion of  $2,0\text{ g} \pm 0,1\text{ g}$ . Weigh the test portion to the nearest 0,1 mg. Pour the test portion into a flat-bottomed flask. Place a magnetic stirrer bar in the flask.

**Table 1 — Size of test portion**

Expected soluble-matter content	Test portion mass
	g
<8,0 % by mass	$4,0 \pm 0,1$ or $2,0 \pm 0,1$
8,0 % to 30,0 % by mass	$2,0 \pm 0,1$
>30,0 % by mass	$2,0 \pm 0,1$ or $1,0 \pm 0,1$

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

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

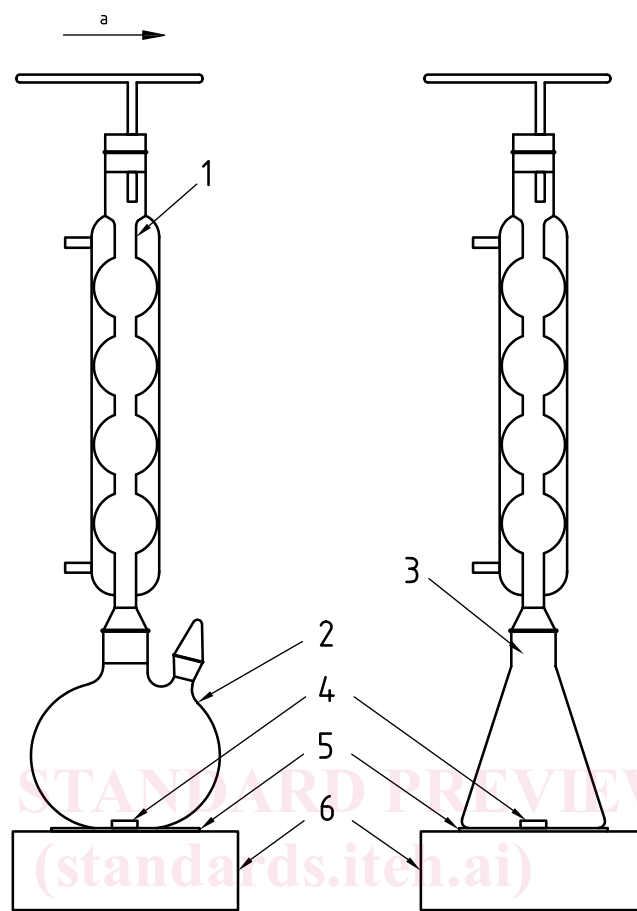
**4.4.3.4** Attach the flask to the condenser ([4.2.1](#)).

**4.4.3.5** Place an insulating disc ([4.2.3](#)) 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 [Figure 1](#)). Insert the nitrogen supply tube in the top of the condenser. Turn on the cooling water to the condenser.

**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 2 l/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 flask walls occurs.

NOTE If a hotplate is used, it can be used at a temperature setting of approximately  $30\text{ °C}$  above the boiling point of xylene.

**Key**

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

**Figure 1 — Example of equipment set-up**

**4.4.3.8** Remove the hotplate from beneath the flask. Detach the flask from the condenser and cover. Allow the solution in the flask to cool in air from the reflux temperature to below 100 °C (this will normally take 12 min to 14 min).

**4.4.3.9** Transfer the flask to a thermostatically controlled water bath (4.2.8) at 25 °C ± 0,5 °C. Do not shake the flask to break up any precipitate before immersing the flask in the bath.

Do not shake the flask while the polymer solution is still hot as this may create a safety hazard.

**4.4.3.10** Without stirring, cool the solution for 30 min in the bath at 25 °C ± 0,5 °C. Use a timer (4.2.16), preferably with an alarm, to ensure that the time allowed for precipitation of dissolved polymer from the solution is between 30 min and 32 min. Remove the flask from the bath. Stir or gently shake the flask to break up any precipitated-polymer gel.