INTERNATIONAL STANDARD

ISO 16152

First edition
2005-07-01

Plastics — Determination of xylene-soluble matter in polypropylene

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

Reference number
ISO 16152:2005(E)

PDF disclaimer

This PDF file may contain embedded typefaces. In accordance with Adobe's licensing policy, this file may be printed or viewed but shall not be edited unless the typefaces which are embedded are licensed to and installed on the computer performing the editing. In downloading this file, parties accept therein the responsibility of not infringing Adobe's licensing policy. The ISO Central Secretariat accepts no liability in this area.

Adobe is a trademark of Adobe Systems Incorporated.

Details of the software products used to create this PDF file can be found in the General Info relative to the file; the PDF-creation parameters were optimized for printing. Every care has been taken to ensure that the file is suitable for use by ISO member bodies. In the unlikely event that a problem relating to it is found, please inform the Central Secretariat at the address given below.

© ISO 2005

All rights reserved. Unless otherwise specified, no part of this publication may be reproduced or utilized in any form or by any means, electronic or mechanical, including photocopying and microfilm, without permission in writing from either ISO at the address below or ISO's member body in the country of the requester.

ISO copyright office
Case postale 56 • CH-1211 Geneva 20
Tel. + 41 22 749 01 11
Fax + 41 22 749 09 47
E-mail copyright@iso.org
Web www.iso.org

Published in Switzerland

Copyright International Organization for Standardization
Reproduced by IHS under license with ISO
No reproduction or networking permitted without license from IHS
Not for Resale

Contents

Page

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.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

The main task of technical committees is to prepare International Standards. Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

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.

ISO 16152 was prepared by Technical Committee ISO/TC 61, *Plastics*, Subcommittee SC 9, *Thermoplastic materials*.

It replaces ISO 6427:1992, Annex B, which has been technically revised. The revised method tightens the physical parameters of the test to provide improved repeatability and reproducibility.

Introduction

This International Standard specifies a method for the quantitative determination of those components of polypropylene that are soluble in xylene. This new method defines more precisely the factors that have the greatest influence on the repeatability and reproducibility of the determination. The polypropylene is dissolved in hot xylene, then cooled under controlled conditions down to 25 °C, which results in the precipitation of the insoluble fraction. The soluble matter remains in the xylene. The xylene is then evaporated and the residue weighed. The solubles content of polypropylene is important as it has a major influence on the properties of the polypropylene.

Plastics — Determination of xylene-soluble matter in polypropylene

1 Scope

1.1 This International Standard specifies a method for determining the mass fraction of a polypropylene homopolymer or copolymer which is soluble in xylene at 25 °C.

1.2 A weighed amount of dried sample is dissolved in xylene under reflux conditions, then cooled under controlled conditions and maintained at 25 °C to ensure controlled crystallization of the insoluble fraction.

1.3 The xylene-soluble fraction is then recovered by evaporation of the xylene and determined by weighing the residue.

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

2 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

2.1
xylene-soluble fraction
S_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 $+25$ °C and held at that temperature for a specified period of time

3 Apparatus

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

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

3.3 **Insulating disc**, made of fibreglass or mineral wool.

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

3.5 **Stirrer bar**.

3.6 **Pipette**, class A, 200 ml or equivalent.

3.7 **Pipette**, class A, 100 ml or equivalent.

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

3.9 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 5.3.8 and 5.3.9).

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

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

3.12 Vacuum oven.

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

3.14 Temperature-controlled hotplate.

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

3.16 Desiccator, containing an appropriate desiccant.

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

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

4 Reagents

4.1 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.

or

4.2 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 (4.1) shall be used as the reference solvent whenever there is a dispute between laboratories on test results, unless the laboratories agree otherwise.

5 Procedure

5.1 Preparation of the xylene

5.1.1 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 0,02 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.

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

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

5.2.1 The purpose of the solvent blank is to determine the evaporation residue, i.e. the 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-lined 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.

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

5.2.3 Place a filter paper or equivalent (3.10) in a funnel (3.11) in a funnel rack over a 250 ml flask (3.8).

5.2.4 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.

5.2.5 Dry an aluminum pan (3.13) for 30 min in an oven (3.18) at 200 °C. Allow the pan to cool to room temperature in a desiccator (3.16). Weigh the clean, dry pan to the nearest 0,1 mg.

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

5.2.7 Place the pan on a hotplate (3.14) 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.

5.2.8 Place the pan in a vacuum oven (3.12) at 100 °C ± 10 °C at a pressure less than 13,3 kPa for a length of time known to be sufficient to dry the residue to constant mass.

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

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

5.3 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 3.4) and a flat-bottomed flask (see 3.2).

5.3.1 If necessary, dry the sample before analysis. For drying, use a vacuum oven at 70 °C ± 5 °C at a pressure less than 13,3 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.

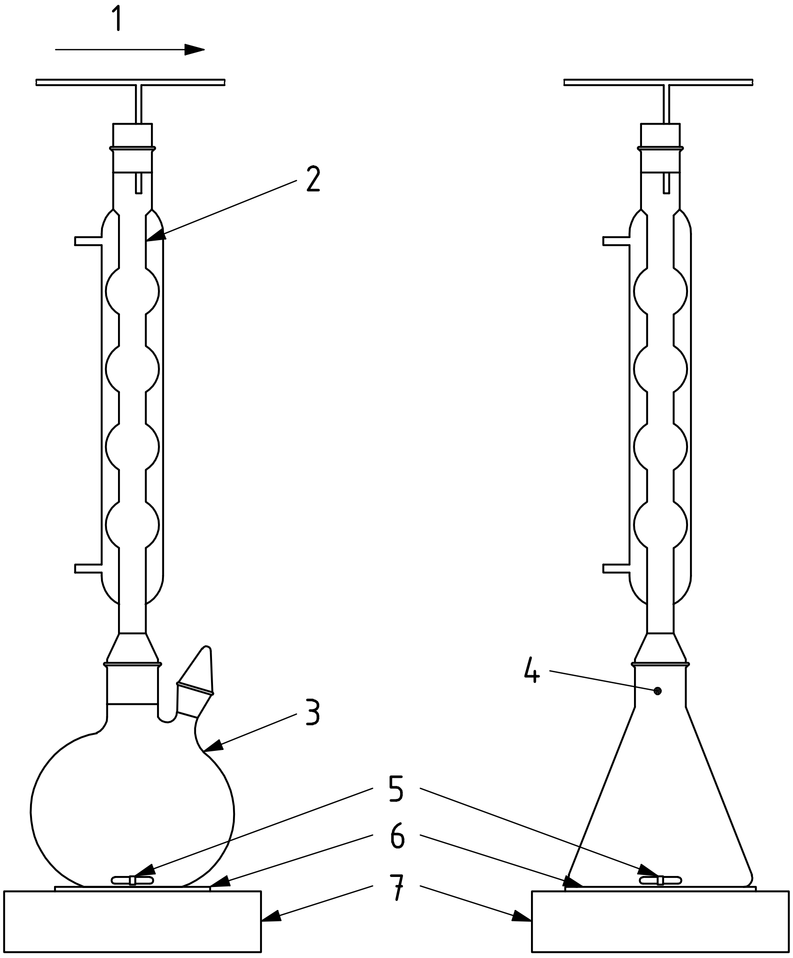
5.3.2 Weigh out a test portion of the size given in Table 1, except when the expected soluble-matter content is unknown or referee testing between laboratories is being conducted. In such cases, use a test portion of 2,0 g ± 0,1 g. Weigh the test portion to the nearest 0,1 mg. Pour the test portion into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

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

NOTE Larger quantities may 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.

5.3.4 Attach the flask to the condenser.

5.3.5 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 Figure 1). 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 — Examples of equipment set-up

5.3.6 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.

5.3.7 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 should preferably be used at a temperature setting of approximately 30 °C above the boiling point of xylene.

Table 1 — Size of test portion

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

5.3.8 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).

5.3.9 Transfer the flask to a thermostatically controlled water bath 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.

5.3.10 Without stirring, cool the solution for 30 min in the bath at 25 °C ± 0,5 °C. Use a timer, 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.

NOTE The rate of cooling affects the crystallite size and the rate of crystallization. Thus, control of time and temperature during cooling is critical to the final test result.

5.3.11 Place a filter paper (3.10) in a funnel (3.11) in a funnel rack over a 250 ml flask (3.8).

NOTE With materials that 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 filler or precipitate.

5.3.12 Pour the contents of the 400 ml flat-bottomed flask into the funnel and allow the filtrate to drip into the 250 ml flask. Continue the filtration until all the filtrate has been collected.

5.3.13 If the filtered solution is not completely clear, repeat the filtration.

5.3.14 Dry an aluminum pan (3.13) for 30 min in an oven (3.18) at 200 °C. Allow the pan to cool to room temperature in a desiccator (3.16). Weigh the clean, dry pan to the nearest 0,1 mg.

5.3.15 Using a clean pipette, transfer a 100,0 ml aliquot of the filtrate into the weighed aluminum pan.

5.3.16 Place the pan on a hotplate (3.14) 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. Do not dry completely as this can result in degradation of the residue.

5.3.17 Place the pan in a vacuum oven (3.12) at 100 °C ± 10 °C at a pressure less than 13,3 kPa for a length of time known to be sufficient to dry the residue to constant mass.

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

5.3.19 Obtain the mass of the xylene-soluble matter by subtracting the mass of the pan from the mass of the pan plus residue. This will be corrected (see 6.1) to allow for any impurities in the xylene.

6 Calculations

6.1 Calculate the xylene-soluble matter content S_s, as a percentage by mass, using the following equation:

$$S_\mathrm{s} = \frac{\dfrac{V_\mathrm{b0}}{V_\mathrm{b1}} \times \left(m_2 - m_1\right) - \dfrac{V_\mathrm{b0}}{V_\mathrm{b2}} \times B}{m_0} \times 100 \qquad (1)$$

where

V_b0 is the original volume of solvent taken in 5.3.3, in ml;

V_b1 is the volume of the aliquot taken in 5.3.15 for the determination, in ml (= 100 ml);

V_b2 is the volume of the aliquot taken in 5.2.6 for the blank determination, in ml (= 100 ml);

m_0 is the mass of the test portion taken in 5.3.2, in g;

m_1 is the mass of the pan (see 5.3.14), in g;

m_2 is the mass of the pan plus residue (see 5.3.18), in g;

B is the average blank value obtained in 5.2.10, in g.

6.2 Materials, such as additives, with solubilities similar to that of the xylene-soluble matter interfere in the determination. Corrections are made to the result only if the material is present in concentrations that are judged to impart a significant error to the result. The material(s) shall be 100 % soluble in xylene and the mass percent of the material present in the polymer must be known for the correction to be made, as follows:

$$S_\mathrm{c} = S_\mathrm{s} - S_\mathrm{m} \qquad (2)$$

where

S_s is the xylene-soluble matter content determined for a particular sample;

S_m is the total xylene-soluble additives content (i.e. the sum of the percentage for each of the additives in the material formulation known to be 100 % soluble), in mass %;

S_c is the corrected xylene-soluble matter content, in mass %.

6.3 Report the result to three significant figures.

7 Precision and bias

Single-country trials are being conducted to determine the repeatability of the method. A complete study of precision and bias will be completed at a later date.

8 Test report

Report the following information:

a) all details necessary for identification of the sample tested and any other pertinent information;

b) the type and grade of xylene used;

c) the mass of the test portion used;

d) the results of the test (report the xylene-soluble matter content actually determined and the corrected xylene-soluble matter content, plus the total xylene-soluble additives content if the result was corrected for soluble additives);

e) the date of the test.

ICS 83.080.01

Price based on 7 pages