INTERNATIONAL STANDARD

ISO 16152

Second edition
2022-06

Plastics — Determination of xylene-soluble matter in polypropylene

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

Reference number
ISO 16152:2022(E)

ISO copyright office
CP 401 • Ch. de Blandonnet 8
CH-1214 Vernier, Geneva
Phone: +41 22 749 01 11
Email: copyright@iso.org
Website: www.iso.org

Published in Switzerland

Contents

Page

Foreword ..... **iv**
Introduction ..... **v**
1 Scope **1**
2 Normative references **1**
3 Terms and definitions **1**
4 Method 1 — Reference method **1**
4.1 Principle 1
4.2 Apparatus 1
4.3 Reagents and materials 2
4.4 Procedure 3
4.4.1 Preparation of the xylene 3
4.4.2 Determination of level of impurities in the xylene (solvent blank) 3
4.4.3 Determination of percentage xylene-soluble matter in the polypropylene 4
4.5 Calculations 6
4.6 Precision and bias 7
5 Method 2 — Automated instrumental method **7**
5.1 Principle 7
5.2 Apparatus 7
5.3 Reagents and materials 8
5.4 Procedure 9
5.4.1 Preparation of the solvent 9
5.4.2 Preparation of the sample 9
5.4.3 Calibration curve 9
5.4.4 Testing 9
5.5 Calculation 10
5.6 Precision and bias 11
6 Test report **11**
Annex A (informative) **Precision data for polypropylene obtained from an intercomparison of xylene-soluble content testing** **12**
Bibliography **13**

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).

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).

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.

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.

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 α-olefin copolymers.

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 in 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 °C ± 5 °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 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.

Table 1 — Size of test portion

Expected soluble-matter content	Test portion mass g
<8,0 % 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

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.

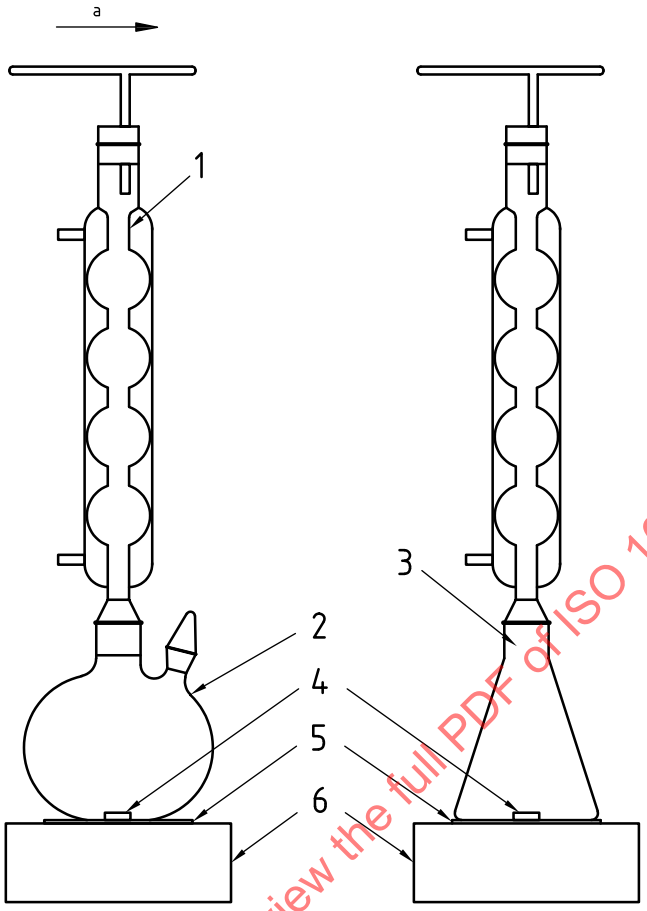
4.4.3.4 Attach the flask to the condenser (4.2.1).

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 °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 in 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.

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.

4.4.3.11 Place a filter paper (4.2.9) in a funnel (4.2.10) in a funnel rack over a 250 ml flask (4.2.7).

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

4.4.3.12 Pour the contents of the flat-bottomed flask into the funnel and allow the filtrate to drip into the 250 ml flask (4.2.7). Continue the filtration until all the filtrate has been collected.

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

4.4.3.14 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.

4.4.3.15 Using a clean pipette, transfer a 100 ml aliquot of the filtrate into the weighed aluminium pan.

4.4.3.16 Place the pan on 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. Do not dry completely as this can result in degradation of the residue.

4.4.3.17 Place the pan in a vacuum oven (4.2.11) 100 °C ± 10 °C under the pressure less than 13,3 kPa for a length of time known to be sufficient to dry the residue to constant mass.

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

4.4.3.19 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 4.5.1) to allow for any impurities in the xylene.

4.5 Calculations

4.5.1 Calculate the xylene soluble fraction, w_s, as a percentage by mass, from Formula (1):

$$w_s = \frac{V_{b0}/V_{b1} \times (m_2 - m_1) - V_{b0}/V_{b2} \times B}{m_0} \times 100 \quad (1)$$

where

V_{b0} is the original volume of solvent taken in 4.4.3.3, in ml;

V_{b1} is the volume of the aliquot taken in 4.4.3.15 for the determination, in ml (=100 ml);

m_2 is the mass of the pan plus residue in 4.4.3.18, in g;

m_1 is the mass of the pan in 4.4.3.14, in g;

V_{b2} is the volume of the aliquot taken in 4.4.2.6 for the blank determination, in ml (=100 ml);

B is the average blank value obtained in 4.4.2.10, in g;

m_0 is the mass of the test portion taken in 4.4.3.2, in g.

4.5.2 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 Formula (2):

$$w_c = w_s - w_m \quad (2)$$

where

w_c is the corrected xylene-soluble matter content, in mass %;

w_s is the xylene-soluble matter content determined for a particular sample, in mass %;

w_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 %.

4.5.3 Report the result to three significant figures.

4.6 Precision and bias

The interlaboratory test programme was completed in 2021, involving seven or eight laboratories and six PP samples with a different xylene-soluble content. The precision data presented in Table A.1 of Annex A.

5 Method 2 — Automated instrumental method

5.1 Principle

The polypropylene sample is dissolved in 1,2 - dichlorobezene or 1,2,4 - trichlorobenzene at high temperature (any temperature between 145 °C to 170 °C can be used) until complete dissolution is achieved. The polymer solution is then cooled to 30 °C to 40 °C and kept at that temperature for 30 min to 40 min to enable complete separation between soluble fraction (SF) and crystalline fraction (CF). The aliquots of the whole material and each fraction (SF and CF) are eluted to the detector. Concentration or the respective polymer mass of the whole material and soluble/crystalline fractions are quantified by a concentration detector (IR preferred) properly calibrated to provide accurate mass of polymer.

5.2 Apparatus

5.2.1 Automated instrument. The main components of the instrument are solvent delivery system, dissolution system, separation system, detector, solvent and waste container, data acquisition and data processing system. A schematic diagram of the instrument is shown in Figure 2.

All connecting lines, valves and other hardware used for the injection should be kept at the high operating temperature to prevent polymer precipitation.

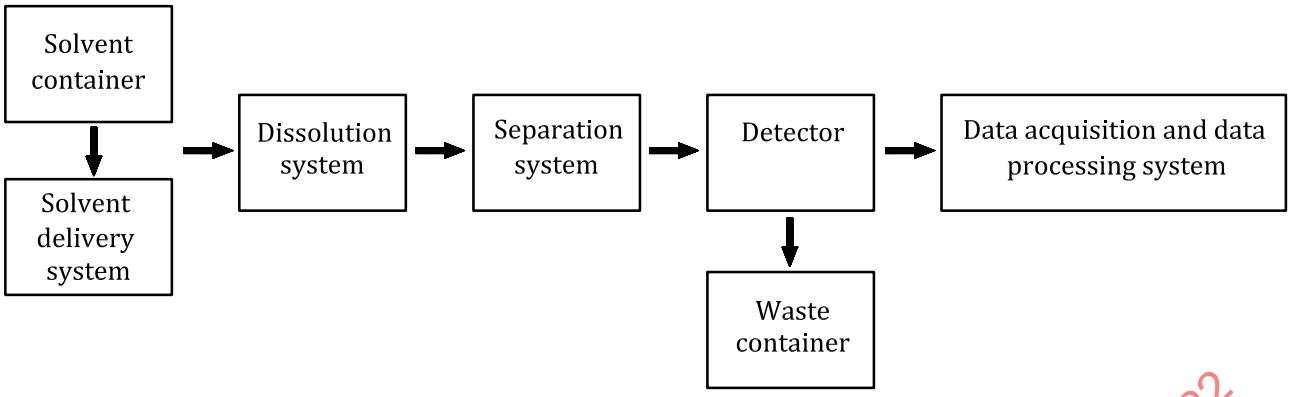


Figure 2 — Schematic diagram of the automated instrument

5.2.2 Solvent and waste container, containers (bottle, vessel, etc.) shall hold sufficient volume of solvent and waste to ensure continuity and uniformity of the solvent delivery during the test. The container material shall be inert to the solvent; glass containers are preferred.

5.2.3 Solvent delivery system, for i.e. dispenser or pump, with capability to provide stable flow rate in the range of 0,1 ml/min to 5,0 ml/min.

5.2.4 Dissolution system, comprising means for heating a container (100 ml to 200 ml) for the polymer and solvent, with capability for mixing of the solution (hotplate with stirring, shaked heating block, oven containing closed stirred vessels, etc.) and able to maintain elevated temperature in the range of 145 °C to 170 °C to ensure full polymer dissolution.

NOTE A high temperature autosampler with analytical vials at least 10 ml capacity can be used when a smaller sample size is considered representative of the material under analysis (range 100 mg to 200 mg).

The dissolution container should be purged with nitrogen to minimize oxidative degradation of the sample during dissolution.

5.2.5 Separation system, positioned in a temperature-controlled oven with controlled cooling possibility and temperature range of 30 °C to 170 °C with minimum accuracy of 0,5 °C. Separation system can be a stainless steel or glass vessel, stainless steel column filled with inert material or similar.

5.2.6 Detector, IR detector with wavenumber range 2 800cm^{-1} to 3 000 cm^{-1}. The total -CH absorbance signal can be calibrated to polymer concentration or mass units. The detector calibration being preferably linear up to 1 absorbance unit (a.u.).

5.2.7 Data acquisition and processing system, able to collect the IR detector absorbance signal and separation system temperature for each fraction and to perform required calculations.

5.2.8 Analytical balance, with an accuracy of 0,1 mg.

5.3 Reagents and materials

5.3.1 Reagents, 1,2,4-trichlorobenzene(TCB), ≥99 % purity or 1,2-dichlorobenzene(ODCB), ≥99 % purity.

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

5.4 Procedure

5.4.1 Preparation of the solvent

Add 300 mg of antioxidant (5.3.2) to 1 000 ml of solvent (5.3.1) and shake before using, if necessary heat at 80 °C to 90 °C, ensure thorough mixing of the antioxidants and the solvent.

The solvent shall be kept in a closed, preferably glass container to be protected from the exposure of the humidity in the air and the formation of hydrochloric acid. It is recommended to blanket the solvent with an atmosphere of inert gas like nitrogen or argon.

5.4.2 Preparation of the sample

If necessary, the details of grinding samples are specified in 4.4.3.1.

5.4.3 Calibration curve

The purpose of this calibration is to translate the values of soluble fraction content (w_{sf}) obtained by the automated instrumental method (method 2) to values equivalent to the reference method (method 1). It accounts for small differences in the observed solubles amounts due to the organic solvent used and the separation temperature in the automated instrumental method compared to the reference values in xylene at 25 °C.

For the calibration, at least five polypropylene materials with xylene-soluble fraction that cover the routine testing range should be used. The nominal values for the xylene- soluble fraction (w_s) of these calibration materials should be determined, preferably as average result of repeated measurements by the reference method.

Calibration materials should be tested by the automated method under the same testing conditions (the same solvent and separation temperature) as the test samples as described in 5.4.4. Calibration curve is built by plotting the nominal xylene- soluble fraction (w_s) results obtained by reference method versus soluble fraction content (w_{sf}) results obtained by automated instrument method. A linear calibration curve shall be achieved.

5.4.4 Testing

5.4.4.1 Weigh out an amount of test portion. Introduce it into the dissolution system (5.2.4).

Add an amount of solvent (5.4.1) to the dissolution container. The concentration of the sample solution is generally suitable for the measurement technology of the instrument in the range of 2 mg/ml to 10 mg/ml.

It is not required accurate weighing because quantification is achieved by an online detector .The target concentration shall be sufficient to ensure reliable quantification by the detector, over the detector sensitivity.

5.4.4.2 The sample is dissolved into the dissolution system (5.2.4) with constant mixing, at high temperature (145 °C to 170 °C) for at least 60 min or until complete dissolution is achieved.

Blanketing of a polymer solution with an atmosphere of nitrogen during the dissolution process is recommended to avoid sample degradation.

5.4.4.3 After full dissolution, the separation process takes place according to the crystallizability separation technique employed.

a) Crystallization in a container with no support (i.e. stirred vessel): The initial concentration, C_I (high temperature, full dissolution) and the soluble concentration, C_{sol} (separation temperature, after crystallization of insoluble fraction) are determined.

b) Separation of soluble / insoluble fractions in a column packed with inert material: an aliquot of the polymer solution is loaded at elevated temperature into the column and then the temperature is decreased down to the separation temperature to allow crystallization of the insoluble fraction. After a full crystallization time, the soluble material is flushed out of the column to the detector to determine the soluble fraction mass (M_{sol}). The column temperature is raised again to 145 °C to 170 °C for re-dissolution of the crystalline fraction, which is moved to the detector and measured as insoluble fraction mass (M_{insol}).

In either case, the temperature at which the soluble fraction is obtained (separation temperature) has an important impact on the obtained results, it must be controlled accurately and has to be included in the report.

An appropriate separation temperature for the solubles determination shall be set in the range 30 °C to 40 °C. The whole crystallization process (cooling and stabilization) shall be minimum 40 min long to achieve complete separation between soluble and crystalline fraction. During the entire cooling process, any flow through the separation system should be stopped.

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

5.5 Calculation

5.5.1 Calculate the soluble fraction content, $w_{sf,}$ expressed as a percentage by mass, is given by the Formulae (3) or (4):

$$w_{sf} = 100 \times \frac{C_{sol}}{C_I} \tag{3}$$

or

$$w_{sf} = 100 \times \frac{M_{sol}}{M_{sol} + M_{insol}} \tag{4}$$

where

C_{sol} is the concentration of solution at low temperature, after crystallization of insoluble fraction, in 5.4.4.3 a);

C_I is the concentration of initial solution at high temperature, full dissolution, in 5.4.4.3 a);

M_{sol} is the soluble fraction mass, in 5.4.4.3 b);

M_{insol} is the insoluble fraction mass, in 5.4.4.3 b).

Translate w_{sf} measured in TCB or ODCB to $w_{s,}$ as given by the Formula (5):

$$w_s = k \times w_{sf} \tag{5}$$

where k is coefficient from the calibration curve obtained as described in 5.4.3.

NOTE For further characterization of the soluble fraction additional detectors can be attached to the automated instrumental setup, to determine the ethylene weight fraction (by online IR) and the molar mass or intrinsic viscosity (online capillary viscometer).

5.5.2 Report the result to three significant figures.