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Plastics — Determination of matter extractable by organic solvents (conventional methods)

Plastiques — Détermination des matières extractibles par des solvants organiques (Méthodes conventionnelles)

Reference number
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Foreword

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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 6427 was prepared by Technical Committee ISO/TC 61, *Plastics*, Subcommittee SC 5, *Physical-chemical properties*.

This third edition cancels and replaces the second edition (ISO 6427:1992), of which it constitutes a minor revision, the main purpose of which was to delete the second sentence in (subclause) 5.1. In addition, the minimum density for which the method is suitable for PE has been indicated directly in footnote “e” to Table 1, rather than referring the reader to ISO 1872-1[1] and what was Table 1 in the 1992 edition has been split into two tables for ease of comprehension.

Introduction

There are several very similar national and international standards for determination of the percentage of extractable matter, with only slight differences in the procedures. To facilitate the work of the laboratory staff which has to carry out these determinations on various plastics products, the generally applicable methods are described in this International Standard.

Plastics — Determination of matter extractable by organic solvents (conventional methods)

1 Scope

This International Standard specifies methods for the determination of components in plastics that can be extracted by hot organic liquids near their boiling points. For one special case, a so-called cold-extraction method is given in Annex B.

The extractable components can be monomers, oligomers, polymers, plasticizers, stabilizers, etc. The kind and percentage of extractable matter influence the properties of plastics.

The recommended extraction liquid depends on the type of plastic and on the purpose of the determination (see Table 1). The extracted amounts of special constituents are often not quantitative in the sense of analytical chemistry.

This International Standard does not apply to plastics that come into contact with food or drinking water. Special regulations for those plastics are established in many countries. In order to test plastics for compliance with these regulations, methods other than those given in this International Standard are used in most cases. The methods of this International Standard are not intended to be used for migration tests.

If this International Standard is used to test plastics other than those mentioned in Table 1, the operating conditions is intended to be agreed upon by the interested parties.

2 Normative references

The following documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 308, *Plastics — Phenolic moulding materials — Determination of acetone-soluble matter (apparent resin content of material in the unmoulded state)*

ISO 383, *Laboratory glassware — Interchangeable conical ground joints*

ISO 565, *Test sieves — Metal wire cloth, perforated metal plate and electroformed sheet — Nominal sizes of openings*

ISO 1773, *Laboratory glassware — Narrow-necked boiling flasks*

ISO 1875, *Plastics — Plasticized cellulose acetate — Determination of matter extractable by diethyl ether*

3 Reagents and materials

3.1 Extraction liquid, of recognized analytical grade, to be selected according to the requirements of the plastic material being tested (see Table 1).

3.2 Anti-bumping granules.

3.3 Glass wool, pre-extracted.

4 Apparatus

4.1 Mill, for reducing the sample to the required grain size.

A mill in which the sample is cut between rotating and stationary blades is preferred. Large pieces can be reduced in size with a pair of shears before they are fed into the mill.

4.2 Set of sieves, complying with the requirements of ISO 565.

4.3 Flat-bottomed flask, of suitable capacity, for example 250 ml, complying with the requirements of ISO 1773, with ground-glass neck complying with the requirements of ISO 383.

4.4 Extraction apparatus, of such a design that the crucible or thimble is heated by the rising vapour of the extraction liquid.

4.4.1 Soxhlet extractor, as shown in Figure 1.

4.4.2 Other extractors, for example that designed by Twisselmann (see Figure 2), may be used, if they give the same results as the Soxhlet extractor.

4.5 Container, for the test portion to be extracted: chosen from one of the following:

4.5.1 Cellulose paper thimble, of suitable size, for example diameter 33 mm and length 94 mm.

4.5.2 Metal wire basket, of the same dimensions as the thimble (4.5.1).

4.5.3 Glass-filter crucible, pore size 40 µm to 100 µm.

NOTE The choice of a suitable container for the extraction is very important. The mass of the cellulose thimble (4.5.1) depends on its moisture content, and this can lead to variable results when weighing. The metal wire basket (4.5.2) cannot be used with a powder sample or if a chemical reaction is possible between the metal and any of the components of the plastic. Difficulties can be caused by penetration of components of the plastic into the pores of the glass-filter crucible (4.5.3) and subsequent swelling.

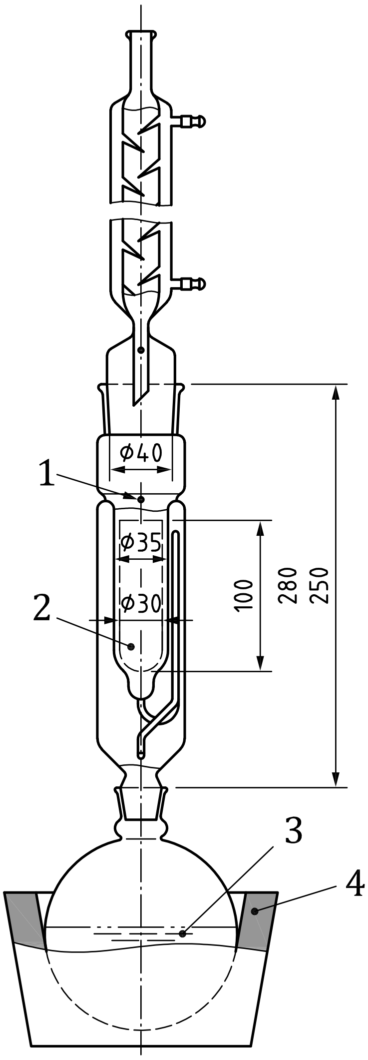
4.6 Reflux condenser, fitted with a ground-glass cone to fit the extraction apparatus (4.4), for example a Dimroth condenser.

4.7 Heating device, which does not use a naked flame and is explosion-proof, suitable for use with the flask (4.3).

4.8 Balance, accurate to 0,1 mg.

4.9 Desiccator, containing calcium chloride or silica gel.

Dimensions in millimetres



Key

1	extractor trap
2	thimble
3	solvent
4	heater

Figure 1 — Extraction apparatus capable of receiving the thimble (4.5.1) or other sample containers

4.10 Distillation equipment.

One of the following devices shall be used:

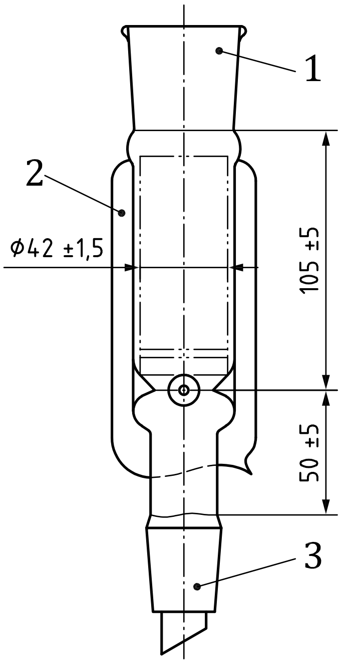
4.10.1 Rotary evaporator.

4.10.2 Distillation apparatus, fitted with a Vigreux or equivalent distillation column of length at least 400 mm.

4.11 Vacuum oven or **oven with fresh air circulation**, explosion-proof, capable of maintaining a temperature of 105 °C.

4.12 Evaporating dish, of suitable capacity, for example 200 ml.

Dimensions in millimetres



Key

1 45/40 ground-glass socket
2 vacuum jacket
3 29/32 ground-glass cone

Figure 2 — Extractor of Twisselmann type with vacuum jacket

5 Preparation of test sample

5.1 The plastics material or plastics product shall be free of dust and foreign matter.

5.2 The sample of plastic shall be reduced to small pieces, for example by grinding in a suitable mill (4.1), but shall not be heated more than necessary. In some cases, it may be necessary to add solid carbon dioxide to prevent heat build-up during grinding. The reduction in size may also be done by the use of a razor blade or a pair of scissors, or a file for hard materials. The portion of the sample of specified granular size (see Table 1) shall be kept in a closed bottle until tested. Films with a thickness of less than 0,5 mm may be cut into small fragments for insertion into the thimble.

6 Number of tests

At least two determinations shall be made.

7 Procedure

7.1 The specific details of the procedure to be used depend on the material to be tested and are given in Tables 1 and 2. The general procedure is as described hereafter.

7.2 Dry the paper thimble (4.5.1), wire basket (4.5.2) or filter crucible (4.5.3) for 1 h in the oven (4.11) at the same temperature as used later for the drying of the plastic tested; allow to cool to room temperature in the desiccator (4.9) and weigh in a closed weighing bottle.

NOTE In special cases, it might be necessary to pre-extract the thimble with the extraction liquid (3.1).

Weigh a test portion of the mass given in Table 1 to the nearest 1 mg in the thimble, basket or crucible, cover it with a piece of glass wool (3.3), and put it into the extraction apparatus (4.4). If the expected content of extractable material is below 0,5 % (by mass), increase the mass of the test portion to obtain a residue of at least 25 mg. Pour the appropriate volume of extraction liquid (3.1) into the flask (4.3). One or two anti-bumping granules (3.2) may be added. Mount the extractor (4.4) and the reflux condenser (4.6) on the flask and adjust the heating device (4.7) so that, when a Soxhlet-type extractor (4.4.1) is used, the extraction liquid syphons several times per hour. For the number of syphonings and the extraction time, see Table 1.

7.3 Carry out the extraction. Depending on the type of plastic extracted (see Table 2), further process the residue in accordance with 7.4 and/or the extract in accordance with 7.5.

7.4 When the extraction is finished, take the thimble, basket or crucible out of the extractor, allow it to drain and air-dry, and then dry it under the conditions given in Table 2 (depending on the kind of extraction liquid). Allow it to cool to room temperature in the desiccator (4.9) and weigh the thimble, basket or crucible to the nearest 1 mg. When a thimble is used, weigh the thimble and its contents in a closed weighing bottle.

7.5 The extraction liquid in the flask may be either distilled to about 20 ml, using the rotary evaporator (4.10.1) or distillation apparatus (4.10.2), or the liquid may be placed directly in a pre-dried and weighed evaporating dish (4.12). In the case of distillation of the main amount of the liquid, transfer the remaining contents of the flask into a dried and weighed evaporating dish. If there are anti-bumping granules in the flask, remove these by filtration. Wash the flask three times with 5 ml of the extraction liquid, collecting the washings in the evaporating dish.

Dry the extract under the conditions given in Table 2. If no conditions are specified for the material being tested, place the dish on a water bath and evaporate the extraction liquid completely; dry the dish with the extract in the vacuum oven (4.11) at 40 °C and at a pressure less than or equal to 3 kPa[1] until constant mass is reached. Allow the dish to cool in the desiccator (4.9) to room temperature and weigh to the nearest 0,2 mg.

7.6 Table 1 lists the appropriate extraction liquids and conditions for several types of plastic.

8 Expression of results

8.1 Calculate the extractable matter content using the appropriate one of the following formulae.

a) For the procedure described in 7.4, the extractable matter content, including volatile substances, expressed as a percentage by mass, is given by the formula

$$\frac{m_0 - m_1}{m_0} \times 100 \qquad (1)$$

b) For the procedure described in 7.5, the nonvolatile extractable matter content, expressed as a percentage by mass, is given by the formula

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[1] 1 kPa = 0,01 bar

$$\frac{m_2}{m_0} \times 100 \qquad (2)$$

where

m_0 is the mass, in grams, of the test portion;

m_1 is the mass, in grams, of nonextractable matter remaining in the extraction vessel after extraction;

m_2 is the mass, in grams, of extractable matter in the evaporating dish.

8.2 Repeat the test if the two individual values differ by more than 5 % in relative value, unless other limits are specified.

9 Precision

The precision of the method is not known because interlaboratory data are not available. A single precision statement is not appropriate because of the number of materials involved. However, a precision of about ±5 % could be expected.

10 Test report

The test report shall include the following information:

a) a reference to this International Standard;

b) a complete identification of the plastic tested;

c) where appropriate, if not specified in Table 1 or Table 2:

  1) the method of preparation of the sample;

  2) the thickness of the sample or the size of the sieves used;

  3) the extraction liquid;

  4) the time of extraction;

  5) the drying conditions;

d) the individual values of the extractable matter content, and the arithmetic mean of these values, expressed as a percentage by mass to the nearest 0,05 % (by mass), as well as the calculation formula used;

e) any deviation, by agreement or otherwise, from the test procedure specified.

Table 1 — Operating conditions

Type of plastic	**Main components of extract**	**Extraction liquid**	**Specific requirements in preparation of test sample**	**Mass of test portion** g	**Extraction**			
					Equipment	**Volume of solvent** ml	**Extraction time**[a] h	**Number of syphonings per hour**
Homopolyamides	Monomers Oligomers Additives (if present)	Methanol	Grind at below 40 °C and sieve to 0,5 mm to 0,7 mm.	5 ± 0,5	Soxhlet extractor with glass-filter crucible or porous ceramic thimble	150	3 h ± 5 min	15 to 25
Copolyamides	Monomers Oligomers	Dichloromethane, in special cases methanol	Grind at below 40 °C. Remove particles smaller than 0,5 mm by sieving.	10	Soxhlet extractor with glass-filter crucible	150	6	15 to 25
Plasticized cellulose esters	Plasticizer	Diethyl ether	Grind and sieve to <1 mm or cast a film of 0,1 mm thickness (see Annex A). Pre-dry for 30 min at 60 °C.	2	Soxhlet extractor with pre-extracted and pre-dried thimble of cellulose paper	200	3 In special cases longer (sometimes 48 h are needed)	15 to 25
Phenolic resin moulding compounds	Phenolic resin Hexamethylene tetramine	Acetone	Grind and sieve to <1,5 mm. Pre-dry for 24 h in a vacuum (2,5 kPa) at room temperature over a desiccant.	3	Soxhlet extractor with pre-extracted and pre-dried thimble of cellulose paper	100	16 ± 0,5	15 to 25
Moulded phenolic resins	Uncured resin Additives	Acetone	Grind and sieve to 0,25 mm to 0,43 mm. Pre-dry for 24 h in a vacuum (2,5 kPa) over a desiccant.	3	Soxhlet extractor with pre-extracted and pre-dried thimble of cellulose paper	150	6	15 to 25
Polypropylene	Atactic and low-molecular isotactic components	*n*-Heptane	Grind and sieve to <0,5 mm. Pre-dry for 2 h at 140 °C under nitrogen vacuum (2,5 kPa).	5	Soxhlet extractor with thimble of glass fibre or cellulose paper	300	16	15 to 25
Vinyl-chloride polymers	Emulsifier	Methanol		12	Soxhlet extractor with cellulose paper thimble	150	10	15 to 25

Table 1 *(continued)*

Type of plastic	Main components of extract	Extraction liquid	Specific requirements in preparation of test sample	Mass of test portion g	Extraction			
					Equipment	Volume of solvent ml	Extraction time[a] h	Number of syphonings per hour
PVC-P plastics	Monomer plasticizer Polymer plasticizer	Diethyl ether Dimethoxymethane[d]	Grind and sieve to <0,5 mm.	3	Soxhlet extractor with cellulose paper thimble or glass-filter crucible	150	8	15 to 25
PE-D[e]	Low-molecular PE	*n*-Heptane	Grind and sieve to <0,5 mm. Pre-dry for 2 h at 110 °C in a vacuum (2,5 kPa).	5	Soxhlet extractor with pre-extracted and pre-dried thimble of cellulose paper	300	16	15 to 25
Cross-linked PE	Uncross-linked PE	Xylene with 1 % 2,6-di-*tert*-butyl-4-methyl phenol	Grind and sieve to <0,5 mm.	1	Soxhlet extractor with glass-filter crucible	150	8	15 to 25

[a] Extraction times other than those listed may be used, provided they give equivalent results.

[b] For high methanol-extractable matter contents, the drying time may be increased if a rotary evaporator is not used.

[c] If a rotary evaporator is used, frothing of the extract occasionally takes place and can lead to a loss of extract. Repeat the determination if frothing occurs.

[d] CH_3-O-CH_2-O-CH_3.

[e] Density greater than or equal to 932 kg/m^3. The method does not give useful results with PE of lower density because of increasing solubility.

Table 2 — Further processing

Type of plastic	Further processing							Remarks	Relevant International Standard
	Of liquid				Of residue				
	Evaporation	Pressure	Temperature °C	Time h	Pressure	Temperature °C	Time h		
Homopolyamides	Distillation or rotary evaporation, followed by evaporation in a dish	<2,5 kPa	40 ± 2	To constant mass, subsequently cooling the dish in a desiccator[a,b]	<2,5 kPa	40	4 to 6	Allow for water content in calculation.	
Copolyamides	Distillation or rotary evaporation	<2,5 kPa	40 ± 2	4	<2,5 kPa	40	4 to 6	Allow for water content in calculation.	
Plasticized cellulose esters	Rotary evaporation followed by evaporation in a dish	<2,5 kPa	50 ± 2	To constant mass	<2,5 kPa Normal	50	0,5		ISO 1875

Table 2 *(continued)*

Type of plastic	Further processing							Remarks	Relevant International Standard
	Of liquid				Of residue				
	Evaporation	Pressure	Temperature °C	Time h	Pressure	Temperature °C	Time h		
Phenolic resin moulding compounds	No further processing of liquid				<2,5 kPa over a desiccant	Room temperature	24		ISO 308
Plasticized cellulose esters	Evaporation in a dish in a ventilated oven	Normal	50 ± 2	To constant mass	No further processing of residue			The extraction might not be complete. Under fixed conditions, however, comparable results are obtained.	ISO 308
Polypropylene	Rotary evaporation followed by evaporation in a dish	<2,5 kPa	70 ± 2	1 to 2 to constant mass	Nitrogen vacuum (<2,5 kPa)	70	4 to 6	After extraction, wash thimble with residue carefully using acetone.	
Vinyl-chloride polymers	Evaporation in a dish	Normal	105	0,5					
PVC-P plastics	Distillation	Normal	105	2	<2,5 kPa	50	1		
PE-D	Distillation or evaporation	<2,5 kPa	60	2	<2,5 kPa	110	2		
Cross-linked PE	Distillation or rotary evaporation				Normal	140	To constant mass		

[a] For high methanol-extractable matter contents, the drying time may be increased if a rotary evaporator is not used.

[b] If a rotary evaporator is used, frothing of the extract occasionally takes place and can lead to a loss of extract. Repeat the determination if frothing occurs.

Annex A
(normative)

Procedure for casting, drying, and cutting films of cellulose acetate

A.1 General

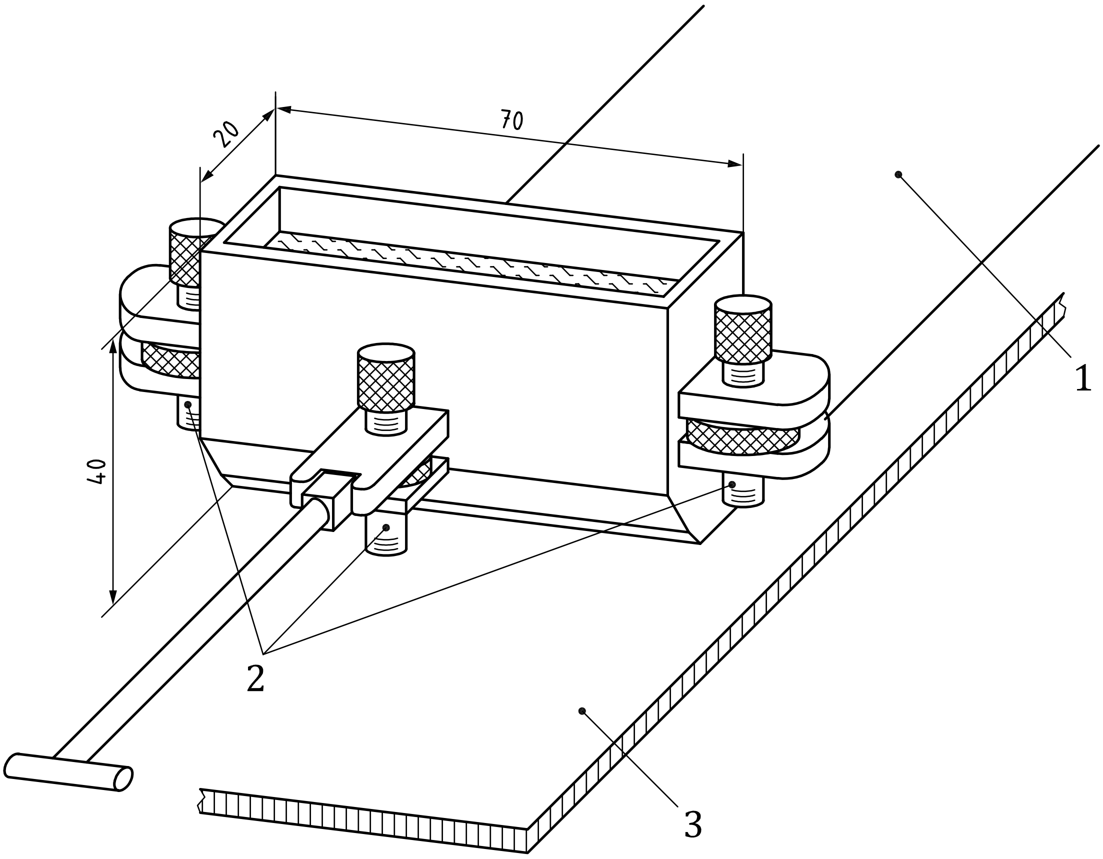
Prepare, at room temperature, a mixture of 90 parts of dichloromethane and 10 parts of methanol by volume.

Weigh 10 g ± 0,2 g of plasticized cellulose acetate and introduce it into a glass bottle. Add 100 ml ± 2 ml of the dichloromethane-methanol mixture. Stopper the glass bottle and shake with a suitable device until dissolution is complete.

Using a film-casting device (see Figure A.1), spread a layer of the solution on a glass sheet, so that, after complete evaporation of the solvent mixture at room temperature, a film of approximately 0,1 mm thickness results.

Remove the dry film and cut it into strips about 5 mm wide and of length suitable for introduction into the extraction thimble.

Dimensions in millimetres



Key

1	film
2	glass sheet
3	screws for adjusting film thickness

Figure A.1 — Suitable film-casting device

Annex B
(normative)

Determination of solubility of polypropylene in cold *p*-xylene

B.1 General

This annex specifies a method of determining the solubility of polypropylene in cold *p*-xylene for the identification and coding of polypropylene types H, B, and R as defined in ISO 1873-1 [H = Homopolymer, B (or R) = Copolymer].[2]

The method is not applicable to the testing of type M (mixture) polypropylene because it is suitable only for base resins and not for mixtures.

B.2 Definition

For the purposes of this annex, the following definition applies.

B.2.1 solubility in cold *p*-xylene: The proportion of polypropylene that remains in solution after a 1 % solution of polypropylene in *p*-xylene, which is clear at the boiling point, is cooled down to room temperature (20 °C or 23 °C).

The *p*-xylene-soluble matter is mainly amorphous. The helix content determined by infrared techniques is less than 15 % (by mass) [usually less than 10 % (by mass)].

B.3 Apparatus

B.3.1 For preparing the solution

B.3.1.1 Flask with three ground-glass necks, of capacity 1 l.

B.3.1.2 Stirring apparatus.

B.3.1.3 Reflux condenser.

B.3.1.4 Nitrogen inlet tube.

B.3.1.5 Thermometer, with ground-glass joint.

B.3.1.6 Mantle heater, rated at 500 W.

B.3.2 For cooling the solution

B.3.2.1 Water bath.

B.3.2.2 Ice chips.

B.3.3 For filtering the solution

B.3.3.1 Funnel.

B.3.3.2 Filter paper, 5 µm average pore diameter, for qualitative analysis.

B.3.4 For drying operation

B.3.4.1 Three aluminium dishes, each of capacity approximately 100 ml.

B.3.4.2 Glass fibre filter paper.

B.3.4.3 Vacuum oven, capable of being maintained at any desired temperature up to 120 °C and at a vacuum of less than 27 kPa, with a nitrogen gas inlet.

B.3.5 For measurements

B.3.5.1 Balance, accurate to 0,1 mg.

B.3.5.2 Measuring cylinder, of capacity 500 ml, graduated in 5 ml.

B.3.5.3 Measuring pipette, of capacity 25 ml, graduated in 0,5 ml.

B.4 Procedure

CAUTION — For safety reasons, the entire procedure should be carried out in a hood.

B.4.1 Pour 500 ml of *p*-xylene (analytical grade) into the three-neck flask (B.3.1.1) fitted with the stirrer (B.3.1.2), the reflux condenser (B.3.1.3) and the nitrogen inlet tube (B.3.1.4).

Weigh, to the nearest 1 mg, approximately 5 g of the polypropylene sample (mass m_0) and add it to the flask containing the *p*-xylene.

B.4.2 Heat the flask with gentle stirring of the contents under a nitrogen blanket and allow the dissolution to continue at the boiling point for about 30 min until all the polypropylene is dissolved.

B.4.3 Remove the heater and allow the solution to stand for about 3 min. Then immerse the flask in the water bath (B.3.2.1) kept at about 20 °C and replace the nitrogen inlet tube with the thermometer (B.3.1.5).

Gradually add ice chips (B.3.2.2) to the water bath to cool the vigorously stirred solution at such a rate that the temperature is lowered to 20 °C (or 23 °C) in about 30 min.

The water bath temperature shall not fall below 18 °C.

B.4.4 Filter the solution (now containing polypropylene swollen to gel form) at room temperature through a folded filter paper. If the filtration does not proceed quickly enough, the filter paper may be renewed.

Generally, 350 ml or more of filtrate is obtained.

B.4.5 Using the graduated pipette (B.3.5.3), transfer one portion of exactly 20 ml of the filtrate into one of the aluminium dishes (B.3.4.1), containing a glass fibre filter paper (B.3.4.2) (or, optionally, a glass fibre wool pad — see the Note), the dish plus filter paper (or wool pad) having been previously dried to constant mass and weighed to the nearest 0,1 mg (mass m_3).

NOTE The purpose of the glass fibre pad is to increase the evaporating surface when it is completely wetted with xylene solution.

B.4.6 Repeat the procedure described in B.4.5, transferring another 20 ml portion of the filtrate into a second tared evaporation dish of mass m_4.

B.4.7 Place both tared dishes in the vacuum oven (B.3.4.3) maintained at 105 °C ± 2 °C at a pressure of about 27 kPa or lower in a nitrogen atmosphere.

After 1 h to 2 h, depending on the oven efficiency (see the Note), constant mass should be achieved. To check this, leave one of the two tared dishes 30 min longer than the other in the oven. Then allow them both to cool to room temperature in a desiccator and weigh (masses m_5 and m_6). There shall be no significant difference between the two results for xylene-soluble matter [see the Note to (Clause) B.5].

NOTE The oven efficiency can be checked by measuring the time needed to dry to constant mass 20 ml of a 1 % xylene solution of high-molecular-mass polyisobutylene.

B.5 Expression of results

The solubility of the polypropylene in cold *p*-xylene, expressed as a percentage by mass, is given by the formula

$$\frac{(m_\mathrm{f} - m_\mathrm{i}) \times 500 \times 100}{m_0 \times 20} = \frac{2\,500 \times (m_\mathrm{f} - m_\mathrm{i})}{m_0} \qquad \text{(B.1)}$$

where

m_f is the arithmetic mean of the masses (m_5 and m_6), in grams, of the two tared dishes and contents after evaporation of the 20 ml of *p*-xylene solution;

m_i is the arithmetic mean of the masses (m_3 and m_4), in grams, of the two dishes used for testing;

m_0 is the mass, in grams, of the polypropylene test portion;

500 is the volume, in millilitres, of *p*-xylene poured into the flask;

20 is the volume, in millilitres, of filtrate evaporated in each test.

NOTE The arithmetic mean calculated as above is meaningful only if the individual values of *p*-xylene-soluble matter [(m_5 − m_3) and (m_6 − m_4)] do not differ by more than 10 % from each other.

Report the result to two significant figures.

B.6 Test report

See Clause 10.

Bibliography

[1] ISO 1872-1, *Plastics — Polyethylene (PE) moulding and extrusion materials — Part 1: Designation system and basis for specifications*

[2] ISO 1873-1, *Plastics — Polypropylene (PP) moulding and extrusion materials — Part 1: Designation system and basis for specifications*

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