International Standard ISO 6427

INTERNATIONAL ORGANIZATION FOR STANDARDIZATION•МЕЖДУНАРОДНАЯ ОРГАНИЗАЦИЯ ПО СТАНДАРТИЗАЦИИ•ORGANISATION INTERNATIONALE DE NORMALISATION

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

Plastiques — Détermination des matières extractibles avec des solvants organiques (méthodes conventionnelles)

First edition — 1982-12-15

UDC 678.5/.8 : 543.832 **Ref. No. ISO 6427-1982 (E)**

Descriptors : plastics, tests, extraction methods, test equipment, test specimen conditioning.

Price based on 9 pages

Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of developing International Standards is carried out through ISO technical committees. Every member body interested in a subject for which a technical committee has been authorized 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.

Draft International Standards adopted by the technical committees are circulated to the member bodies for approval before their acceptance as International Standards by the ISO Council.

International Standard ISO 6427 was developed by Technical Committee ISO/TC 61, *Plastics*, and was circulated to the member bodies in July 1980.

It has been approved by the member bodies of the following countries :

Australia
Austria
Brazil
Canada
China
Czechoslovakia
Egypt, Arab Rep. of
Finland
Germany, F.R.
Hungary
Iran
Ireland
Italy
Japan
Korea, Rep. of
Mexico
Poland
Romania
South Africa, Rep. of
Spain
Sweden
USA
USSR

The member bodies of the following countries expressed disapproval of the document on technical grounds :

Belgium
France
Netherlands
Switzerland
United Kingdom

Printed in Switzerland

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

0 Introduction

There are several very similar national and International Standards for determination of the percentage of extractable matter with only slight differences in 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.

1 Scope and field of application

1.1 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.

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

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

1.4 This International Standard does not apply for those 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.

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

2 References

ISO 59, *Plastics — Phenolic mouldings — Determination of acetone-soluble matter.* [1]

ISO 308, *Plastics — Phenolic moulding materials — Determination of acetone-soluble matter (apparent resin content of material in the unmoulded state).* [2]

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

ISO 565, *Test sieves — Woven metal wire cloth, perforated plate and electroformed sheet — Nominal sizes of openings.* [3]

ISO 599, *Plastics — Polyamides — Determination of matter extractable by boiling methanol.*

ISO 1773, *Laboratory glassware — Boiling flasks (narrow-necked).*

ISO 1873/1, *Plastics — Polypropylene and propylene-copolymer thermoplastics — Part 1 : Designation.*

ISO 1875, *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 the table).

3.2 Bumping stones.

3.3 Glass wool, pre-extracted.

---

[1] At present at the stage of draft. (Revision of ISO 59-1976.)

[2] At present at the stage of draft. (Revision of ISO 308-1981.)

[3] At present at the stage of draft. (Revision of ISO 565-1972.)

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 to 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.

The extractor shall be of such a design that the crucible or thimble is heated by the rising vapour of the extraction liquid.

4.4.1 Extractor of Soxhlet type as shown in figure 1 with a volume of 100 ml.

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

4.5 Container for test portion to be extracted.

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 those of the thimble (4.5.1).

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

NOTE — The choice of a suitable container for the extraction is very important. The weight of the cellulose thimble (4.5.1) depends on its moisture content and can give variable results in 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 one of the components of the plastic. Difficulties can arise 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 joint to fit the extraction flask (4.4), for example reflux condenser of Dimroth type.

4.7 Appropriate heating device for flasks without an open flame and explosion-proof.

4.8 Balance, accurate to 0,1 mg.

4.9 Desiccator, containing calcium chloride or silica gel.

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 **heating oven with fresh air circulation** and explosion-proof.

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

5 Preparation of test sample

5.1 The plastic material or plastic product shall be free of dust and foreign matter. If the material or product has to be cleaned, an organic solvent shall not be used.

5.2 The plastic sample 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, a pair of scissors or a file for hard materials. The portion of the sample of specified granular size (see the table) 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

The specific details of the procedure to be used depend on the material to be tested and are given in the table. The general procedure is described hereafter.

7.1 Dry the paper thimble (4.5.1), wire basket (4.5.2) or filter crucible (4.5.3) for 1 h in the heating 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 may be necessary to pre-extract the thimble with the extraction liquid (3.1).

Weigh a test portion of the mass given in the table 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 % (*m*/*m*) 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 bumping stones (3.2) may be added. Mount the extractor 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 the table.

7.2 Depending on the type of plastic extracted (see the table), further process the residue according to 7.3 or the extract according to 7.4. In the case of cellulose esters further process both residue and extract.

7.3 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 the table (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.

7.4 The extraction liquid in the flask may be either distilled to about 20 ml using the rotary evaporator (4.10.1) or distillation column (see 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 the dried and weighed evaporating dish. If there are bumping stones 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 the table. 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* 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.

7.5 The table lists the appropriate extraction liquids and conditions for several types of plastics. It should be realized that the resulting extraction values do not permit the differentiation of the extractable substances according to their type and quantity.

8 Expression of results

8.1 Calculate the extractable matter content by the following formula.

a) For the procedure described in 7.3 the extractable matter content, including volatile substances, expressed as a percentage by mass, is given by the formula

$$\frac{m_0 - m_1}{m_0} \times 100$$

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

$$\frac{m_2}{m_0} \times 100$$

where

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

m_1 is the mass, in grams, of non-extractable matter after extraction in the vessel;

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

8.2 The test shall be repeated if the two individual values differ by more than 5 % in relative value, unless other limits are specified in the table.

9 Test report

The test report shall include the following information :

a) reference to this International Standard;

b) complete identification of the plastic tested;

c) if not specified in the table :

1) the method of preparation of the sample;

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

3) extraction liquid;

4) time of extraction;

5) drying conditions;

d) arithmetic mean and individual values of the percentage of extractable matter content to the nearest 0,05 % (m/m) and the calculation formula used;

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

---

* 1 kPa = 0,01 bar

Dimensions in millimetres

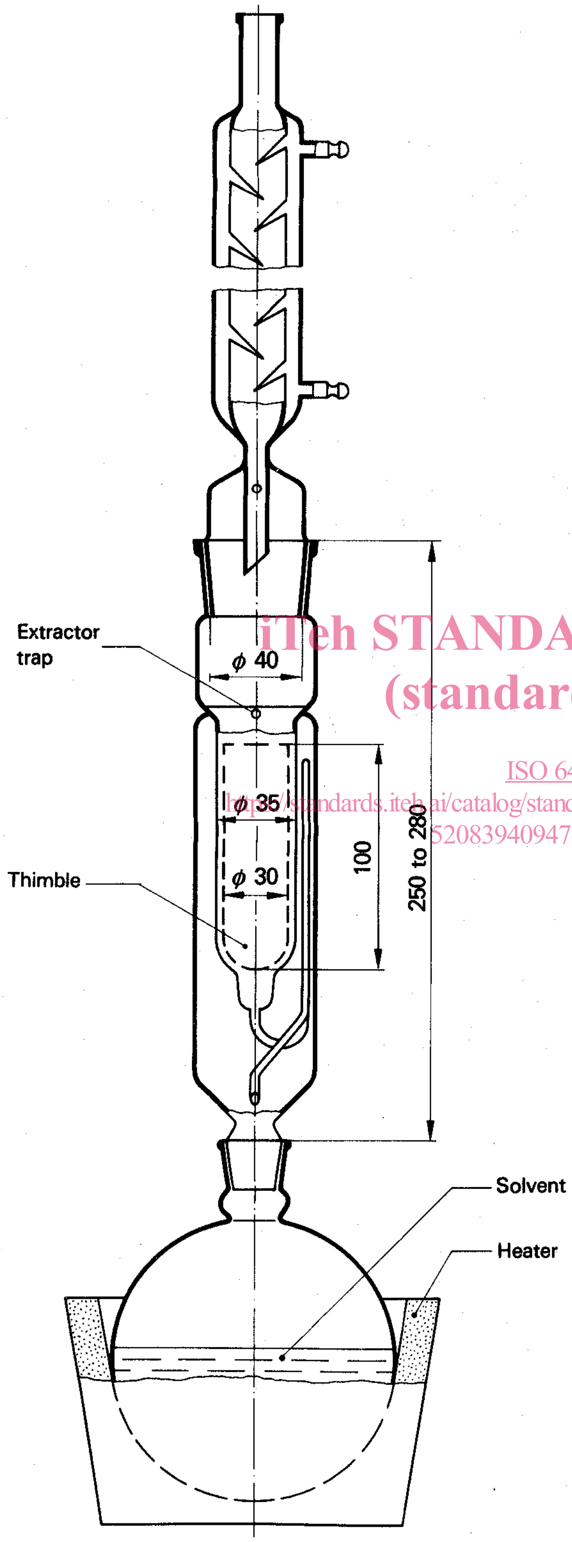

Figure 1 — Extraction apparatus capable of receiving the thimble (4.5.1) or other sample containers

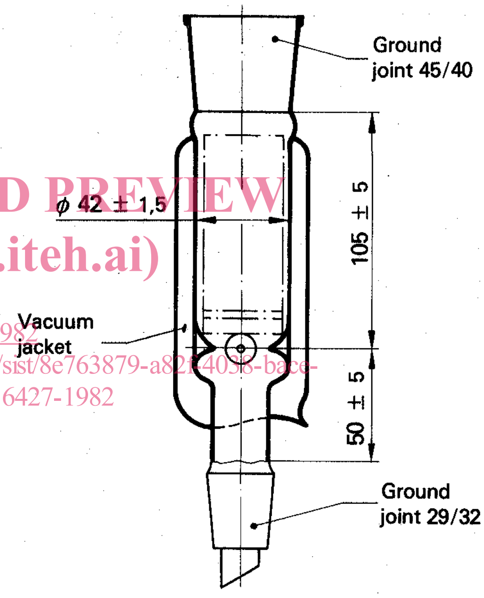

Figure 2 — Extractor of Twisselmann type with vacuum jacket

Table —

Type of plastic	Main components of extract	Extraction liquid	Specific requirements in preparation of test sample	Mass of the test portion g	Extraction			
					Equipment	Volume of solvent ml	Extraction time h	Number of syphonings per hour
Homopolyamides	Monomers Oligomers Additives (if present)	Methanol	Grind below 40 °C or mill in small portions to keep the mill cool. Solid carbon dioxide may be ground with the polymer to prevent heat build-up. Granule size : 0,5 to 0,7 mm	5 ± 0,5	Soxhlet extractor with glass filter crucible or porous ceramic thimble	50	3 ± 5min	5 to 8
Copolyamides	Monomers Oligomers	Dichloromethane In special cases methanol	Grind below 40 °C. Remove particles smaller than 0,5 mm by sieving.	10	Soxhlet extractor with glass filter crucible	150	6	5 to 8
Plasticized cellulose esters	Plasticizer	Diethylether	Grind and sieve to particles smaller than 1 mm or cast a film of 0,1 mm thickness, dry and cut into strips 5 mm wide. Pre-dry sample 30 min at 60 °C. For further details of film casting see annex A.	2	Soxhlet extractor with cellulose paper thimble pre-extracted with diethylether	200	3 In special cases longer (sometimes 48 h are needed)	10
Phenolic resin moulding compounds	Phenolic resin Hexamethylenetetramine	Acetone	Preformed material has to be reduced in size < 1,5 mm. The sample shall be pre-dried in vacuum at room temperature over concentrated sulphuric acid or other desiccant for 24 h.	3	Soxhlet extractor with pre-extracted and pre-dried thimble of cellulose paper	100	16 ± 0,5	15 to 30
Moulded phenolic resins	Uncured resin Additives	Acetone	Grind and sieve the sample to a granule size 0,25 to 0,43 mm. Pre-dry 24 h in a vacuum desiccator at 2 kPa.	3	Soxhlet extractor with thimble of cellulose paper	150	6	20 to 30
Polypropylene	Atactic and low molecular isotactic components. Percentage = "isotactic index"	*n*-Heptane	Grinding or cutting granular size or thickness ⩽ 0,5 mm. Predried 2 h at 140 °C in nitrogen vacuum 25 kPa.	5	Soxhlet extractor with thimble of glass fibre or cellulose paper. The empty thimble and the thimble with the sample are dried and annealed to constant mass (usually 2 h) at 140 °C under 25 kPa nitrogen vacuum or less before weighing	300	> 24	15 to 25

NOTES

1 For high methanol-extractable matter contents, the drying time may be prolonged if a rotating evaporator is not used.

2 When the rotating evaporator is used, frothing of the extract occasionally takes place and can lead to a loss of extract. Repeat the determination if frothing oc

Table — Operating conditions

cific requirements reparation of test sample	Mass of the test portion g	Extraction				Further processing			
						Of liquid			
		Equipment	Volume of solvent ml	Extraction time h	Number of syphonings per hour	Evaporation	Pressure	Temperature °C	Time h
below 40 °C or mill all portions to keep ll cool. Solid carbon e may be ground he polymer to pre- eat build-up. le size : 0,7 mm	5 ± 0,5	Soxhlet extractor with glass filter crucible or porous ceramic thimble	50	3 ± 5 min	5 to 8	Distillation in Vigreux column or rotary evaporation, and evaporation in a dish	Vacuum	40 ± 2	4 After the drying co the dish in the des cator for 30 to 40 m and weigh it to t nearest 0,2 mg. Co tinue drying until t difference betwe two successive weig ings is less than 2 of the mass of t extract. (See notes and 2.)
below 40 °C. ve particles smaller ,5 mm by sieving.	10	Soxhlet extractor with glass filter crucible	150	6	5 to 8	Distillation or rotary evaporation	Vacuum < 2,5 kPa	40 ± 2	4
and sieve to par- smaller than 1 mm t a film of 0,1 mm ess, dry and cut strips 5 mm wide. y sample 30 min at rther details of film g see annex A.	2	Soxhlet extractor with cellulose paper thimble pre-extracted with diethylether	200	3 In special cases longer (sometimes 48 h are needed)	10	Rotary evaporation and evaporation in a dish	Vacuum	50 ± 2	To constant mass
med material has to educed in size mm. ample shall be pre- n vacuum at room rature over concen- sulphuric acid or desiccant for 24 h.	3	Soxhlet extractor with pre-extracted and predried thimble of cellulose paper	100	16 ± 0,5	15 to 30	No further processing of liquid			
and sieve the e to a granule size 0,43 mm. y 24 h in a vacuum ator at 2 kPa.	3	Soxhlet extractor with thimble of cellulose paper	150	6	20 to 30	Evaporation in a dish in a ventilated oven	Normal	50 ± 2	To constant mas First weighing aft 30 min
ng or cutting ar size or thickness mm. d 2 h at 140 °C in n vacuum 25 kPa.	5	Soxhlet extractor with thimble of glass fibre or cellulose paper. The empty thimble and the thimble with the sample are dried and annealed to constant mass (usually 2 h) at 140 °C under 25 kPa nitrogen vacuum or less before weighing	300	> 24	15 to 25	No further processing of the *n*-heptane-solution			

drying time may be prolonged if a rotating evaporator is not used.

xtract occasionally takes place and can lead to a loss of extract. Repeat the determination if frothing occurs.

erating conditions

	Further processing							
	Of liquid			Of residue				
Evaporation	**Pressure**	**Temperature °C**	**Time h**	**Pressure**	**Temperature °C**	**Time h**	**Remarks**	**Relevant International Standard**
Distillation in Vigreux column or rotary evaporation, and evaporation in a dish	Vacuum	40 ± 2	4 After the drying cool the dish in the desiccator for 30 to 40 min and weigh it to the nearest 0,2 mg. Continue drying until the difference between two successive weighings is less than 2 % of the mass of the extract. (See notes 1 and 2.)	No further processing of the residue			Polyamides may contain a small percentage of water, forming part of the mass of the test portion. Unless the water content is 3 % or greater, it is not taken into account in the calculation of the percentage of extractable matter since its effect is small compared with the variance of the determination. If the results of the two determinations differ by more than 0,3 % in absolute value, carry out another duplicate determination.	ISO 599
Distillation or rotary evaporation	Vacuum < 2,5 kPa	40 ± 2	4	No further processing of the residue			If water content is 3 % or greater, consider it in the calculation.	
Rotary evaporation and evaporation in a dish	Vacuum	50 ± 2	To constant mass	Vacuum Normal	50 ± 2 followed by 105	0,5 3		ISO 1875
No further processing of liquid				Vacuum over a desiccant (concentrated sulphuric acid or other)	Room temperature	24		ISO 308
Evaporation in a dish in a ventilated oven	Normal	50 ± 2	To constant mass First weighing after 30 min	No further processing of the residue			The extraction may not be complete. Under fixed conditions, comparable results are obtained.	ISO 59
No further processing of the *n*-heptane-solution				Nitrogen-vacuum 25 kPa	70 ± 2	4 to 6 to constant mass	The thimble with the residue after extraction shall be carefully washed with acetone before drying.	ISO 1873/1, annex