

# INTERNATIONAL STANDARD

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**6427**

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

**iTeh STANDARD PREVIEW**  
*Plastiques — Détermination des matières extractibles par des solvants  
organiques (méthodes conventionnelles)*  
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ISO 6427:1992

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Reference number  
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## Foreword

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

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.

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

This second edition ~~replaces the first edition~~ (ISO 6427:1982), of which it constitutes a technical revision concerning principally the table.

Annexes A and B form an integral part of this International Standard.

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## 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.

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

## 1 Scope

**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 table 1). The extracted amounts of special constituents are often not quantitative in the sense of analytical chemistry.

**1.4** 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.

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

## 2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this International Standard. At the time of publi-

cation, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 59:1976, *Plastics — Phenolic mouldings — Determination of acetone-soluble matter.*

ISO 308:1981, *Plastics — Phenolic moulding materials — Determination of acetone-soluble matter (apparent resin content of material in the un moulded state).*

ISO 383:1976, *Laboratory glassware — Interchangeable conical ground joints.*

ISO 565:1990, *Test sieves — Metal wire cloth, perforated metal plate and electroformed sheet — Nominal sizes of openings.*

ISO 1773:1976, *Laboratory glassware — Boiling flasks (narrow-necked).*

ISO 1872-1:1986, *Plastics — Polyethylene (PE) and ethylene copolymer thermoplastics — Part 1: Designation.*

ISO 1875:1982, *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.

Dimensions in millimetres

## 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.

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## 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 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 the thimble (4.5.1).

4.5.3 Glass-filter crucible, pore size 40  $\mu\text{m}$  to 100  $\mu\text{m}$ .

NOTE 1 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 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.

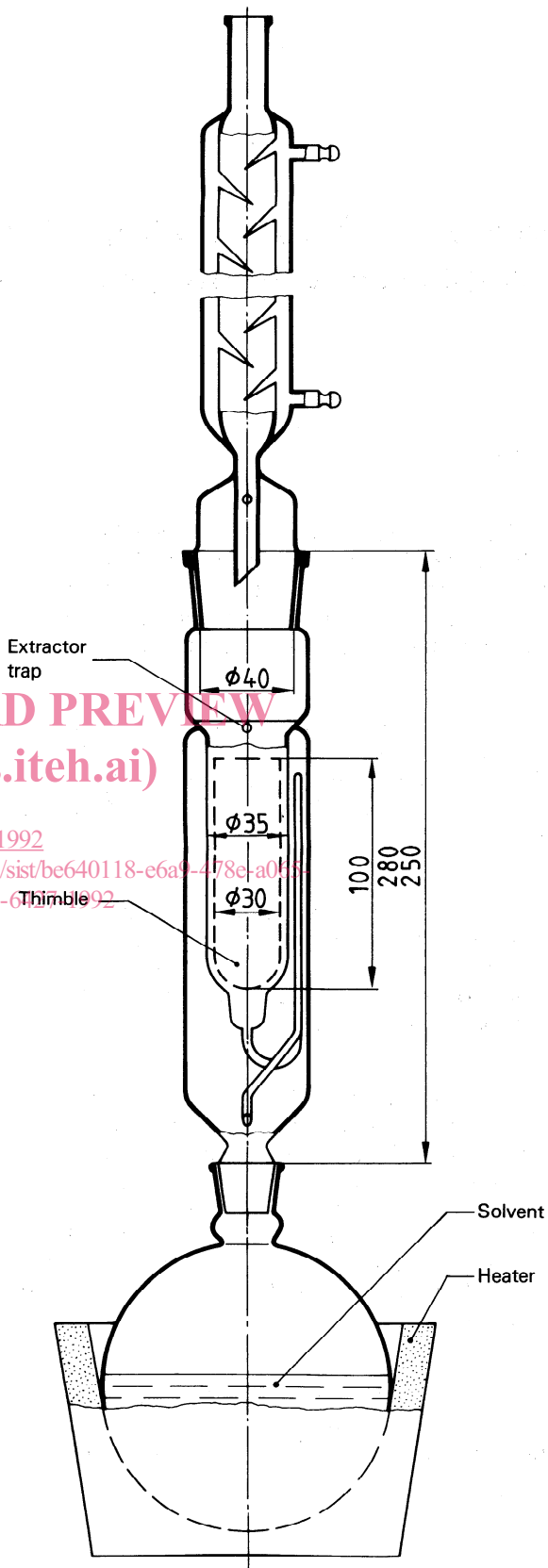


Figure 1 — Extraction apparatus capable of receiving the thimble (4.5.1) or other sample containers

**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.

**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.

## 5 Preparation of test sample

**5.1** The plastics material or plastics product shall be free of dust and foreign matter. If the material or product has to be cleaned, organic solvent shall be used only at room temperature.

**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.

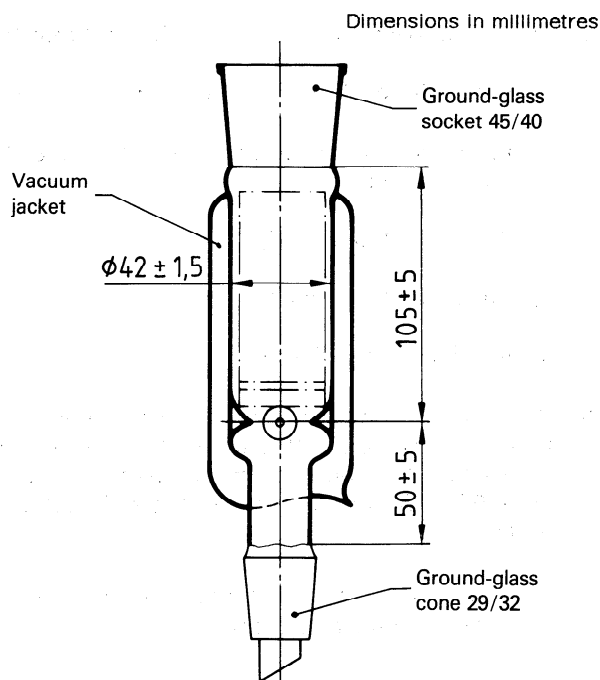
## 7 Procedure

The specific details of the procedure to be used depend on the material to be tested and are given in table 1. 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 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 2 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 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 % (*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 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.



**Figure 2 — Extractor of Twisselmann type with vacuum jacket**

**7.2** Carry out the extraction. Depending on the type of plastic extracted (see table 1), further process the residue in accordance with 7.3 and/or the extract in accordance with 7.4.

**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 table 1 (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 apparatus (4.10.2), or the liquid may be placed directly in a predried 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 1. 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<sup>1)</sup> 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** 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 7.3 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;
- $m_1$  is the mass, in grams, of non-extractable 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 in table 1.

## 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  $\pm 5$  % 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:
  - 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 % ( $m/m$ ), as well as the calculation formula used;
- e) any deviation, by agreement or otherwise, from the test procedure specified.

1) 1 kPa = 0,01 bar



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 <sup>1)</sup> h	Number of syphonings per hour
Homo-polyamides	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
Copoly-amides	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 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	<i>n</i> -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
PVC-P plastics	Monomer plasticizer Polymer plasticizer	Diethyl ether, dimethoxy-methane <sup>4)</sup>	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 <sup>5)</sup>	Low molecular PE	<i>n</i> -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- <i>tert</i> -butyl-4-methyl phenol	Grind and sieve to < 0,5 mm.	1	Soxhlet extractor with glass-filter crucible.	150	8	15 to 25

1) Extraction times other than those listed may be used, provided they give equivalent results.

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

3) 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.

4)  $\text{CH}_3 - \text{O} - \text{CH}_2 - \text{O} - \text{CH}_3$

5) Density code, as defined in ISO 1872-1, greater than or equal to 35. The method does not give useful results with PE of lower density because of increasing solubility.