INTERNATIONAL STANDARD

ISO 6427

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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)

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

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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.

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

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

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

ISO 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 1872-111 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.

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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 <u>Table 1</u>). 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 <u>Table 1</u>, the operating conditions is intended to be agreed upon by the interested parties.

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2 Normative references iteh.ai/catalog/standards/sist/b90ad323-8759-43c2-9428-d364d20420c4/iso-6427-2013

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 <u>Table 1</u>).
- 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.

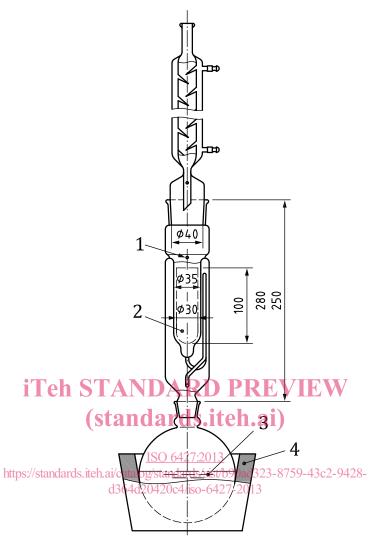
A mill in which the sample is cut between rotating and stationary blades is preferred. Large pieces can be reduced in size with a pair of shears before they are fed into the mill.

- **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 <u>Figure 1</u>.
- **4.4.2 Other extractors**, for example that designed by Twisselmann (see <u>Figure 2</u>), 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. 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 (<u>4.4</u>), 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.

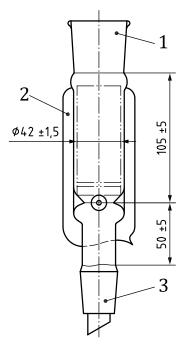
One of the following devices shall be used:

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 $105\,^{\circ}\text{C}$.

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

Dimensions in millimetres



Key

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

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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 <u>Table 1</u>) 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 <u>Tables 1</u> and <u>2</u>. 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 <u>Table 2</u>), further process the residue in accordance with <u>7.4</u> and/or the extract in accordance with <u>7.5</u>.
- **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 three times with 5 ml of the extraction liquid, collecting the washings in the evaporating dish. catalog/standards/sist/b90ad323-8759-43c2-9428-d364d20420c4/iso-6427-2013

Dry the extract under the conditions given in <u>Table 2</u>. 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 kPa¹⁾ 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 <u>7.4</u>, the extractable matter content, including volatile substances, expressed as a percentage by mass, is given by the formula

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

¹⁾ 1 kPa = 0.01 bar