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Plastics — Polypropylene (PP) and propylene-copolymer thermoplastics — Determination of isotactic index

Plastiques — Thermoplastiques à base de polypropylène (PP) et de copolymères de propylène — Détermination de l'indice d'isotacticité

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Contents

Page

Foreword

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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).

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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 9113:1986), of which it constitutes a minor revision. The changes compared to the previous edition are as follows:

— the normative references have been updated;

— the mandatory Clause 3 has been added and the subsequent clauses have been renumbered;

— minor editorial changes have been applied.

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.

Plastics — Polypropylene (PP) and propylene-copolymer thermoplastics — Determination of isotactic index

1 Scope

This document specifies a method for determining the percentage of matter which can be extracted from crystalline propylene plastics by boiling n-heptane under standard conditions of testing. Isotactic index is determined by conventional chemical extraction as an absolute method.

This method provides for the identification and coding of types H, B and R propylene plastics according to ISO 19069-1[1].

This method is suitable only for base polymers and is not applicable for mixtures.

This method starts with solid propylene plastics in the form of particles of specified fineness.

2 Normative references

There are no normative references in this document.

3 Terms and definitions

No terms and definitions are listed in this document.

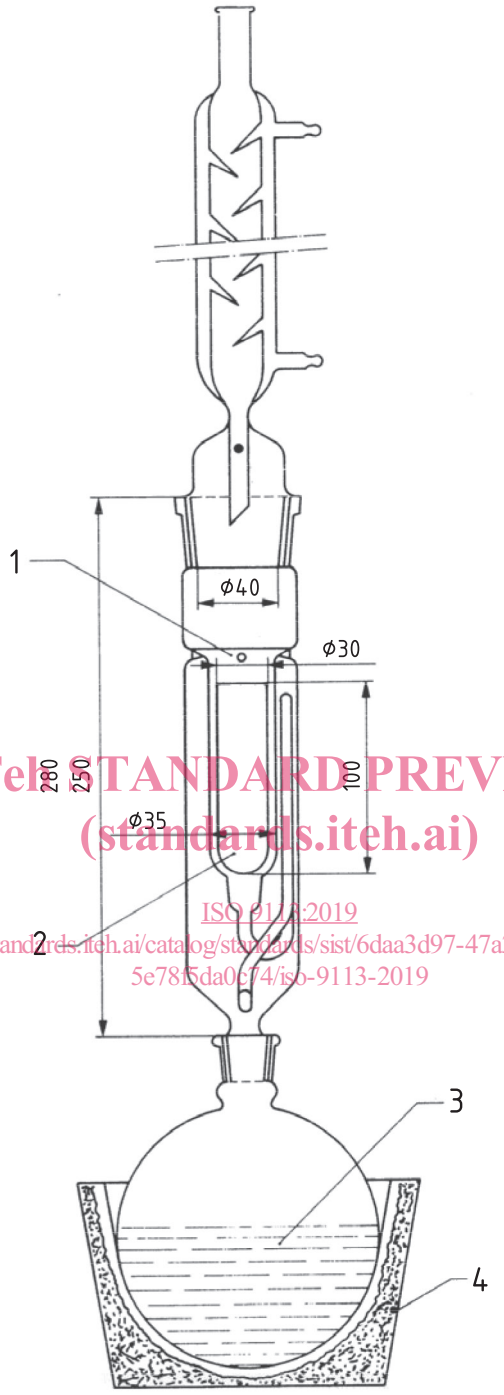
ISO and IEC maintain terminological databases for use in standardization at the following addresses:

— ISO Online browsing platform: available at https://www.iso.org/obp

— IEC Electropedia: available at http://www.electropedia.org/

4 Apparatus

4.1 Extractor, a type of which is shown in Figure 1. Any other type of extractor giving the same results may be used. This extractor shall be suitable for use at the boiling point of *n*-heptane.



Key

1 extractor trap
2 cartridge
3 *n*-heptane
4 heater

Figure 1 — Extraction apparatus capable of receiving the cartridges described in 4.2

4.2 Glass fibre or paper cartridges (thimbles), with a diameter of 30 mm ± 3 mm and length 100 mm ± 10 mm.

4.3 Drying ovens, shall be capable of being maintained at temperatures of 70 °C ± 2 °C and 140 °C ± 2 °C, respectively, both of them provided with vacuum lines of less than 25 kPa.

4.4 Balance, shall be accurate to the nearest 0,000 1 g.

4.5 Grinding mill, or equivalent machine.

4.6 Sieve, mesh size of the sieve shall not be greater than 1 mm. A mesh size of 0,5 mm ± 0,1 mm is recommended.

5 Procedure

5.1 Preparation of the sample

5.1.1 Reduce the propylene plastic solid matter to particles fine enough to pass through the sieve (4.6). For powder, flakes, fibres or films, grinding and screening are unnecessary if at least one dimension is less than 0,6 mm. Films shall be cut into small fragments or changed to a crushable form by melting to the shape of ribbons or small plaques.

5.1.2 Grind the sample as follows.

Mix at least 10 g of the sample with solid carbon dioxide or liquid nitrogen in excess, place it in the grinding mill (4.5) and crush until reduced to small particles.

After screening, collect the matter which passes through the sieve (4.6) and allow standing at room temperature until it is tested.

5.2 Determination

5.2.1 Weigh the glass fibre or paper cartridge (4.2), dried to constant mass at 140 °C and cooled to room temperature in a desiccator, to the nearest 0,000 1 g (mass m_1) (see 5.2.5).

Then, fill it with approximately 5 g of the sample and place it in the oven (4.3) controlled at 140 °C ± 2 °C, under 25 kPa nitrogen vacuum or less. This means that the existing residual pressure of nitrogen in the apparatus shall be 25 kPa or lower (see 5.2.6).

Usually, 2 h are sufficient for complete drying and sample annealing, but calibration of the oven efficiency is recommended to determine the minimum time required to achieve constant mass of the samples.

5.2.2 Cool the cartridge containing the test portion to room temperature in a desiccator and weigh again to the nearest 0,000 1 g (mass m_2). Record the mass of the propylene plastic test portion by difference ($m_2 - m_1$). Then place the cartridge in the extractor trap (see Figure 1) and extract with boiling *n*-heptane (analytical grade, free from aromatic components) for 24 h (see 5.2.5 and 5.2.7).

5.2.3 Regulate the solvent boiling rate to give 15 to 25 extractions per hour, using 300 ml of *n*-heptane in the flask.

5.2.4 After 24 h of extraction, cool the cartridge containing residual polymer to room temperature, carefully wash it with acetone (analytical grade) and dry it in the oven (4.3) controlled at 70 °C ± 2 °C, to constant mass, under 25 kPa nitrogen vacuum or less.

Usually, 4 h to 6 h are sufficient to reach constant mass, but it is recommended that the oven drying efficiency be checked by a preliminary test.

Weigh the dried cartridge containing the residual polymer to the nearest 0,000 1 g after cooling to room temperature in a desiccator (mass m_3) (see 5.2.5).

5.2.5 If paper cartridges are used, they shall be placed in a glass weighing bottle before weighing, in order to avoid the influence of atmospheric humidity absorbed by the paper walls.

5.2.6 In order to improve the reproducibility of extraction tests, it is recommended that ground propylene plastic particles be annealed in oven for 2 h at 140 °C under a nitrogen atmosphere to equalize the degree of crystallinity.

5.2.7 If powder or crushed particles of type H propylene plastic pass through 0,5 mm mesh sieves, 6 h extraction time is allowed (for practical purposes), provided that the relevant results are consistent with those of 24 h extraction. The standard 24 h extraction is mandatory in the following cases:

a) for type B propylene plastics;

b) in case of dispute concerning the results.

6 Calculation and expression of results

6.1 The *n*-heptane extractable matter S_i, expressed as a percentage by mass, is given by the Formula (1):

$$\frac{m_2 - m_3}{m_2 - m_1} \times 100 \qquad (1)$$

where

m_1 is the mass, in grams, of the cartridge;

m_2 is the mass, in grams, of the cartridge plus test portion before extraction;

m_3 is the mass, in grams, of the cartridge plus test portion after extraction.

6.2 Record the value of extractable matter (S) as the arithmetic mean (quoted to two significant figures) of the observed values in a duplicate test, provided that they are within ±7,5 % of the average, see Formula (2):

$$\frac{S_1 - S_2}{S} \leq 0{,}15 \qquad (2)$$

where

S_1 and S_2 are the observed individual values of S_i;

S is the arithmetic mean of these two values.

If this condition is not fulfilled, repeat the test.

7 Calculation of isotactic index (II)

Calculate the Isotactic index II as shown in Formula (3):

$$\text{Isotactic index (II)} = 100 - S \qquad (3)$$

Round off the value of 100 − S to the nearest unit and report it as the isotactic index. If the first decimal digit is 5, round off to the next lower unit.

8 Test report

The test report shall include the following information:

a) a reference to this document, i.e. ISO 9113:2019;

b) complete identification of the product tested;

c) the individual results and the arithmetic mean for *n*-heptane extractable matter, and the isotactic index;

d) any unusual features noted during the determination;

e) any operation not specified in this document or regarded as optional.