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International Standard ISO 9113

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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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Foreword

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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. They are approved in accordance with ISO procedures requiring at least 75 % approval by the member bodies voting.

International Standard ISO 9113 was prepared by Technical Committee ISO/TC 61, *Plastics.*

It cancels and replaces the annex to International Standard ISO 1873/1-1980, of which it constitutes a minor revision.

Users should note that all International Standards undergo revision from time to time and that any reference made herein to any other International Standard implies its latest edition, unless otherwise stated.

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

1 Scope and field of application

1.1 This International Standard 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.

1.2 This method provides for the identification and coding of types H, B and R propylene plastics referred to in ISO 1873/1.

1.3 This method is not applicable to the testing of type Q propylene plastics referred to in ISO 1873/1 because it is intended and is suitable only for base polymers and not for mixtures such as those covered by type Q.

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

2 Reference

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

3 Apparatus

3.1 Extractor, of the type shown in the figure, or any other type giving the same results. This extractor shall be suitable for use at the boiling point of *n*-heptane.

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

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

3.4 Balance, accurate to the nearest 0,000 1 g.

3.5 Grinding mill or equivalent machine.

3.6 Sieve, of mesh size not greater than 1 mm. A mesh size of 0,5 ± 0,1 mm is recommended.

4 Procedure

4.1 Preparation of sample

4.1.1 Reduce the propylene plastic solid matter to particles fine enough to pass through the sieve (3.6). 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.

4.1.2 Grind the sample as follows.

Mix at least 10 g of the sample with solid carbon dioxide or liquid nitrogen in excess, place in the grinding mill (3.5) and crush until reduced to small particles.

After screening, collect the matter which passes through the sieve (3.6) and allow to stand at room temperature until it is tested.

4.2 Determination

4.2.1 Weigh the glass fibre or paper cartridge (3.2), 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 note 1 in 4.2.4).

Then fill it with approximately 5 g of the sample and place it in the oven (3.3) controlled at 140 ± 2 °C, under 25 kPa nitrogen vacuum or less. This means that the existing residual pressure of nitrogen in the apparatus must be 25 kPa or lower (see note 2 in 4.2.4).

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.

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* 25 kPa = 250 mbar

4.2.2 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 the figure) and extract with boiling *n*-heptane (analytical grade, free from aromatic components) for 24 h (see notes 1 and 3 in 4.2.4).

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

4.2.4 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 (3.3) controlled at 70 ± 2 °C, to constant mass, under 25 kPa nitrogen vacuum or less.

Usually 4 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 note 1).

NOTES

1 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.

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

3 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.

5 Calculation and expression of results

5.1 The *n*-heptane extractable matter, S_i, expressed as a percentage by mass, is given by the formula

$$\frac{m_2 - m_3}{m_2 - m_1} \times 100$$

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.

5.2 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, that is if

$$\frac{S_1 - S_2}{S} < 0{,}15$$

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.

6 Calculation of isotactic index (II)

From the definition of isotactic index in ISO 1873/1 it follows that

Isotactic index (II) = 100 − S

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.

7 Test report

The test report shall include the following particulars:

a) a reference to this International Standard;

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 International Standard or regarded as optional.

Dimensions in millimetres

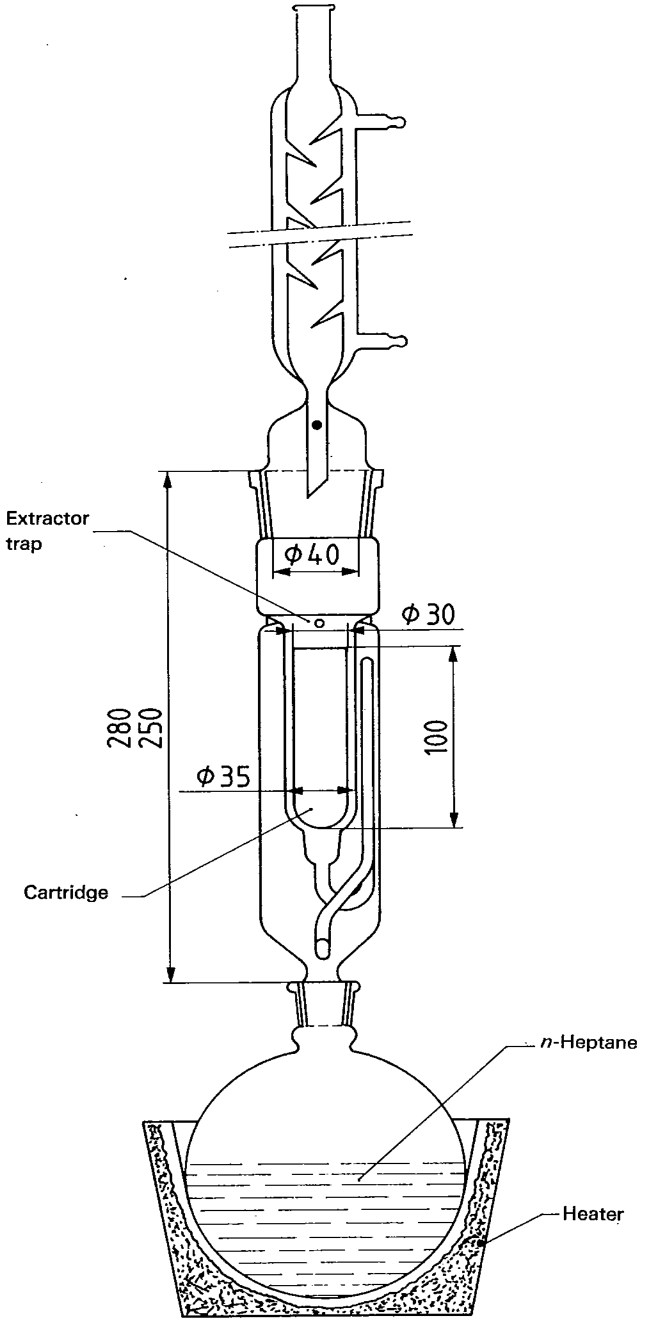

Figure — Extraction apparatus capable of receiving the cartridges described in 3.2