National foreword

This British Standard is the UK implementation of ISO 24076:2021.

The UK participation in its preparation was entrusted to Technical Committee PRI/82, Thermoplastic materials.

A list of organizations represented on this committee can be obtained on request to its committee manager.

This publication does not purport to include all the necessary provisions of a contract. Users are responsible for its correct application.

This British Standard was published under the authority of the Standards Policy and Strategy Committee on 28 February 2021.

Amendments/corrigenda issued since publication

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Compliance with a British Standard cannot confer immunity from legal obligations.

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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*.

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.

Introduction

The LR-NMR method in this document is a relative method established with ISO 9113 as the absolute method. The method and concept of isotactic index of polypropylene are specified in ISO 9113, that is, the polypropylene sample is separated into the extractable and the unextractable matter by n-heptane extraction, and the percentage of unextractable matter in the sample is calculated as isotactic index.

Isotactic index in ISO 9113 sounds similar to tacticity (isotacticity or stereotacticity) of polymer chain, but the concept and result are different. The result is related to but not equal to the tacticity (isotacticity or stereotacticity) of molecular chain, which can be determined by high resolution carbon-13 nuclear magnetic resonance and infrared method. The result of ISO 9113 is also related to crystallization, molecular weight, chain entanglement of the sample, solvent solubility and other effects.

For solid polymers, extraction always takes a long time for the diffusion of long molecular chain from polymer to solvent. To improve test efficiency, relative methods are developed. This document provides a relative non-destructive method for the determination of isotactic index by low-resolution nuclear magnetic resonance spectrometry through a calibration curve establishing with magnetic signal and isotactic index determined by ISO 9113. No solvent is used, and the determination efficiency is improved during samples measurement procedure except for the calibration part.

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INTERNATIONAL STANDARD

Plastics — Polypropylene (PP) — Determination of isotactic index by low-resolution nuclear magnetic resonance spectrometry

1 Scope

This document specifies a relative method for the determination of polypropylene (PP) isotactic index by low-resolution pulsed nuclear magnetic resonance spectroscopy (LR-NMR).

This method enables the identification and coding of types H propylene (PP-H) plastics according to ISO 19069-1.

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

NOTE   The direct method for the determination of polypropylene isotactic index is specified in ISO 9113.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 9113, *Plastics — Polypropylene (PP) and propylene-copolymer thermoplastics — Determination of isotactic index*

3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

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/

3.1
isotactic index
II
percentage mass fraction of unextractable matter content in polypropylene, which cannot be extracted from polypropylene by boiling n-heptane

4 Principle

The sample is placed in LR-NMR. After electromagnetic equilibration in the static magnetic field of the NMR spectrometer and application of a 90° radio frequency pulse, the magnetization decay signal curve from the protons of the sample is recorded. The signal of the un-extractable matter in the sample drops much faster than that of extractable matter. In the signal curve, initial signal corresponds to the whole sample including the un-extractable and the extractable matter, and later slower dropping signal corresponds to the extractable matter in the sample. So, the magnetization decay signal and the isotactic index of the sample are related.

The isotactic index is either calculated from the ratio between the magnetization decay signal of the un-extractable part and the entire sample detected by a ratio probe (method A ratio method), or from

7.1.2   LR-NMR measurement procedure of reference samples

7.1.2.1   Put the reference sample into a sample tube (5.2), with a depth of between 30 mm and 50 mm.

NOTE   The sample amount can be specified by the spectrometer manufacturer which matches to the probe height.

7.1.2.2   Put the tube in thermostatic apparatus (5.4) for a minimum of 30 min to achieve temperature equilibration.

7.1.2.3   Transfer the tube to LR-NMR probe (5.1) within 15 s. Hold for 15 min to achieve temperature equilibration.

NOTE   Experience shows that it is easy to transfer a tube from thermostatic apparatus to LR-NMR probe within 15 s.

7.1.2.4   Measure the magnetization decay signal. The typical signal range corresponding to the sample is 7 μs to 9 μs, and that of extractable matter is 50 μs to 90 μs.

NOTE   For exact magnetization decay signal time, refer to the spectrometer manufacturer's instructions.

7.1.3   LR-NMR measurement procedure of test samples

Shall be the same as that of reference samples (see 7.1.2) except that the reference sample is replaced by the test sample.

7.2   Calculation

7.2.1   Calibration curve

Prepare calibration curve by plotting II' obtained from extraction method against LR-NMR signal. An example of a calibration curve for method A is shown in Annex A.

The Formula (1) is obtained by linear regression method according to the calibration curve.

$$II' = a \times \frac{N'_{1a} - N'_{2a}}{N'_{1a}} + b \tag{1}$$

where

   II' is the isotactic index of the reference sample determined by n-heptane extraction specified in ISO 9113, expressed in percent (%);

   N'_{1a} is the average magnetization decay signal of the reference sample measured by method A;

   N'_{2a} is the average magnetization decay signal of the extractable matter in the reference sample measured by method A;

   a is the slope of the calibration curve;

   b   is the intercept of the calibration curve.

7.2.2   Calculation of isotactic index II_{na}

Isotactic index II_{na} is given by the Formula (2).

$$II_{na} = a \times \frac{N_{1a} - N_{2a}}{N_{1a}} + b \tag{2}$$

where

II_{na} is the isotactic index of the test sample determined by method A, expressed in percent (%);

N_{1a} is the average magnetization decay signal of the test sample;

N_{2a} is the average magnetization decay signal of the extractable matter in the test sample;

a is the slope of the calibration curve;

b is the intercept of the calibration curve.

8   Method B — Absolute method

8.1   Procedure

8.1.1   Measurement of reference samples by n-heptane extraction

See 7.1.1.

8.1.2   LR-NMR measurement procedure of reference samples

8.1.2.1   Weigh approximately 5 g of the reference sample, to the nearest 1 mg, put them into a sample tube (5.2) within the height of LR-NMR probe.

NOTE   The sample amount can be specified by the spectrometer manufacturer which matches to the probe height.

8.1.2.2   See 7.1.2.2.

8.1.2.3   See 7.1.2.3.

8.1.2.4   Measure the magnetization decay signal. The typical signal range corresponding to extractable matter is 100 μs to 120 μs

NOTE   For exact magnetization decay signal time, refer to spectrometer manufacturer's instructions.

8.1.3   LR-NMR measurement procedure of test samples

Shall be the same as that of reference samples (see 8.1.2) except that the reference sample is replaced by the test sample.

8.2   Calculation

8.2.1   Calibration curve

Prepare calibration curve by plotting II' obtained from extraction method against LR-NMR signal. An example of a calibration curve for method B is shown in Annex B.

The Formula (3) is obtained by linear regression method according to the calibration curve.

$$II' = a \times \frac{N'_{2b}}{m'} + b \tag{3}$$

where

    II' is the isotactic index of the reference sample determined by n-heptane extraction specified in ISO 9113, expressed in percent (%);

    N'_{2b} is the average magnetization decay signal of the extractable matter in the reference sample measured by method B;

    m' is the mass of the reference sample, expressed in grams (g);

    a is the slope of the calibration curve;

    b is the intercept of the calibration curve.

8.2.2   Calculation of isotactic index II_{nb}

Isotactic index II_{nb}, is given by the Formula (4):

$$II_{nb} = a \times \frac{N_{2b}}{m} + b \tag{4}$$

where

    II_{nb} is the isotactic index of the test sample determined by method B, expressed in percent (%);

    N_{2b} is the average magnetization decay signal of the extractable matter in the test sample measured by method B;

    m is the mass of the test sample, expressed in grams (g);

    a is the slope of the calibration curve;

    b is the intercept of the calibration curve.

9   Expression of results

Express the result as the arithmetic mean of the two determinations. Report the result to one decimal place. The absolute difference between two determinations shall be within 0,2 %. If this condition is not fulfilled, repeat the test.

10 Test report

The test report shall include the following particulars:

a)   a reference to this document. i.e. ISO 24076:2021;

b)   the method used (method A or method B);

c)   all information necessary for the complete identification of the test sample;

d)   details of the LR-NMR used;

e)   results of calibration: number and isotactic index of reference samples, magnetization decay signals used for calculation, calibration curve and coefficients;

f)   the individual results, their mean value and the absolute difference between two determinations, for more than two individual values the results obtained;

g)   any operation not specified in this document or regarded as an option;

h)   the date of the determination.

Annex A
(informative)

Example of a calibration curve for method A

An example of a calibration curve obtained from six reference samples for method A is given below.

a) Number of reference samples: 6

b) Isotactic index of reference samples (tested in accordance with ISO 9113): 96,2 %, 96,8 %, 97,2 %, 98,4 %, 98,9 %, 99,2 %;

c) Magnetization decay signals range used for calculation (refer to spectrometer manufacturer's instructions): 7,5 µs to 9,5 µs for N_{1a}, 47,5 µs, to 87,5 µs for N_{2a};

d) Calibration curve and coefficients: Prepared by plotting the isotactic index II' versus $(N'_{1a} - N'_{2a})/N'_{1a}$ as shown in Figure A.1, intercept –5,97, slope 1,14, linear correlation coefficient 0,998.
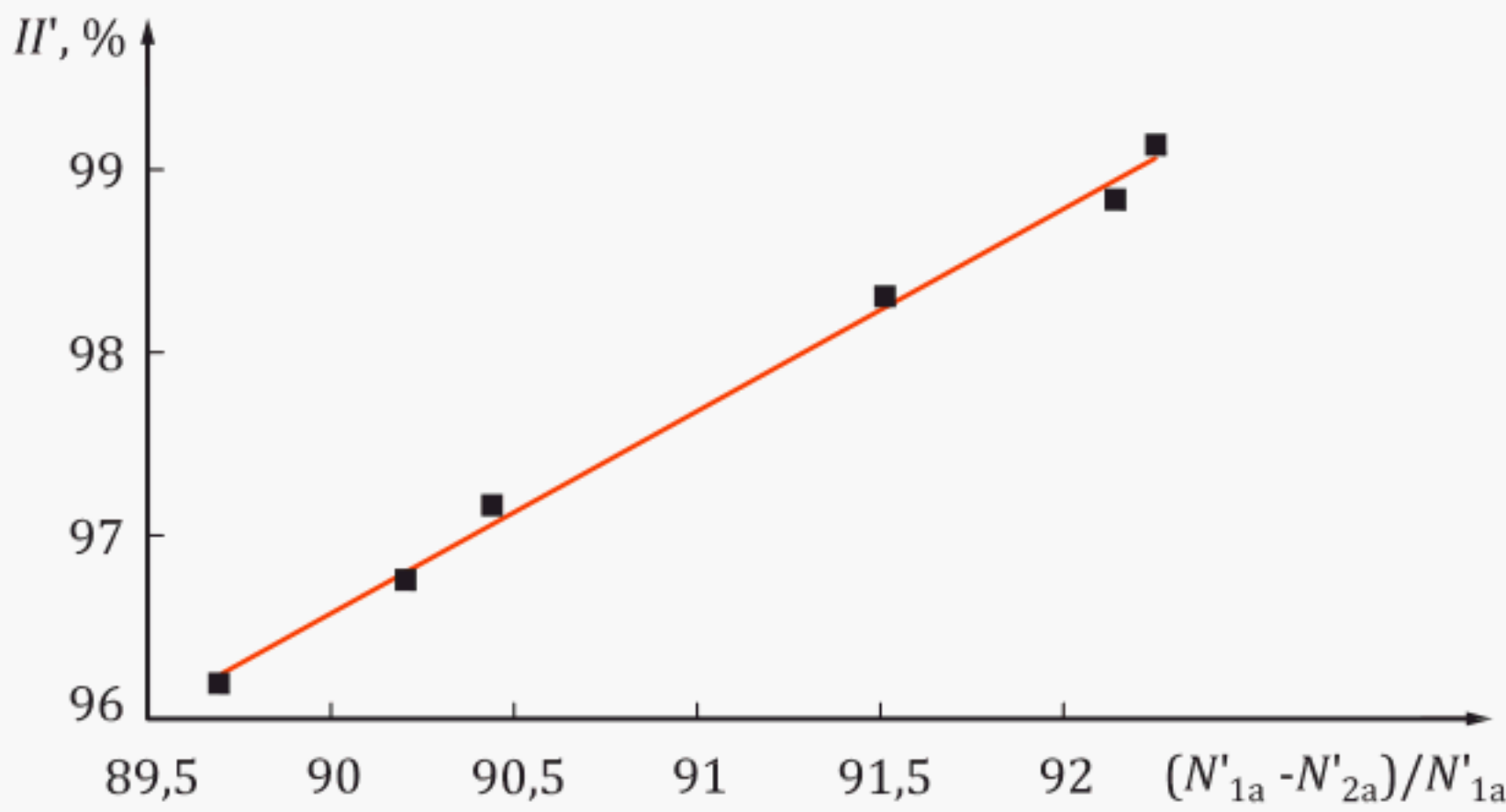
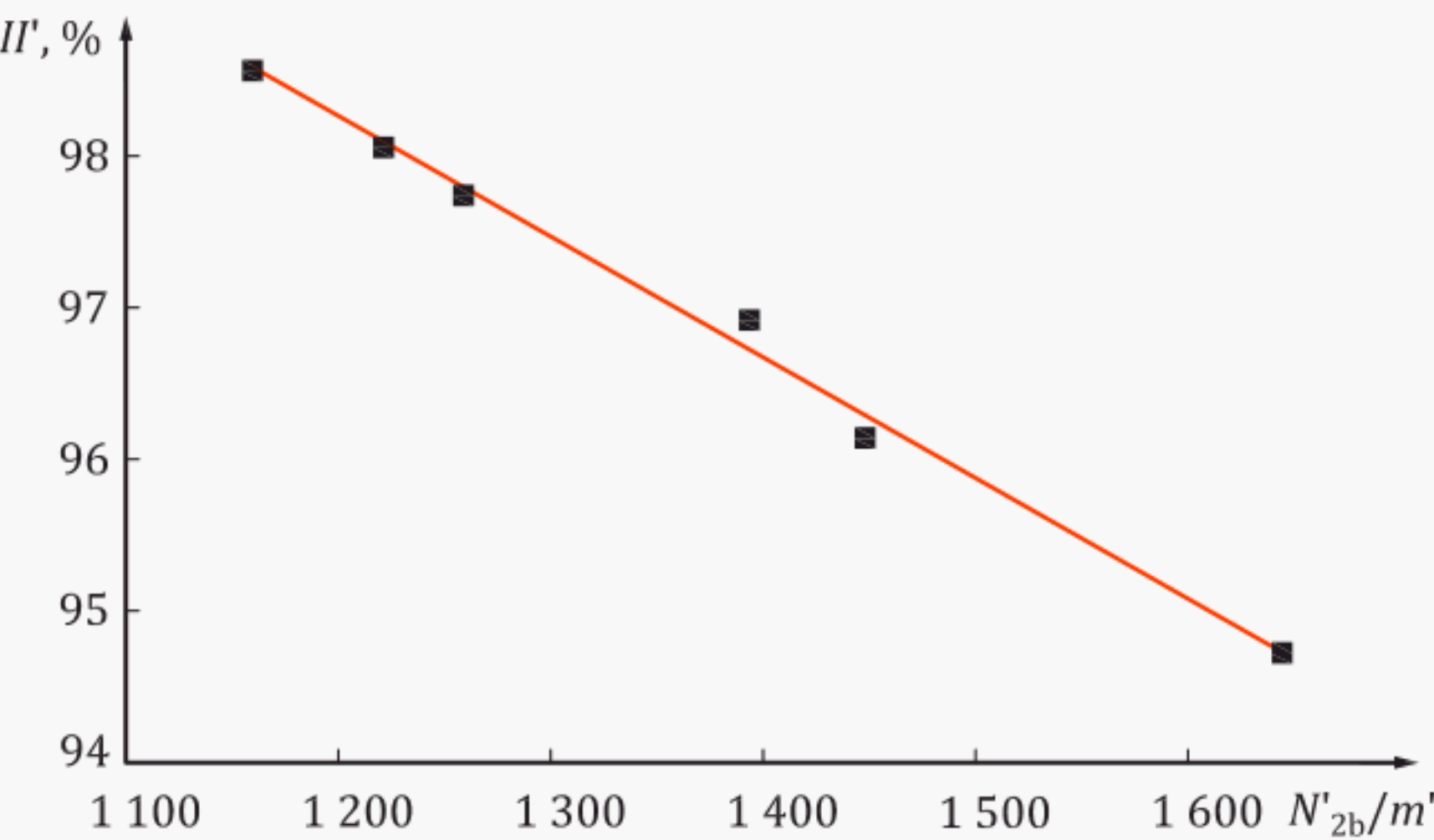
Figure A.1 — Example of a calibration curve for method A

Annex B
(informative)

Example of a calibration curve for method B

An example of a calibration curve obtained from seven reference samples for method B is given below.

a) Number of reference samples: 7

b) Isotactic index of reference samples (tested in accordance with ISO 9113): 94,7 %, 95,3 %, 96,1 %, 96,9 %, 97,7 %, 98,0 %, 98,5 %;

c) Magnetization decay signals range used for calculation (refer to spectrometer manufacturer's instructions): 100 μs, to 120 μs for N_{2b};

d) Calibration curve and coefficients: Prepared by plotting the isotactic index II' versus N'_{2b}/m' as shown in Figure B.1, intercept 107, slope −0,0076, linear correlation coefficient −0,996.

Figure B.1 — Example of a calibration curve for method B

Bibliography

[1]   ISO 19069-1, *Plastics — Polypropylene (PP) moulding and extrusion materials — Part 1: Designation system and basis for specifications*

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