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

*Plastiques — Polypropylène (PP) — Détermination de l'indice
isotactique par spectrométrie de résonance magnétique nucléaire à
basse résolution*



This document is a preview generated by EKO



COPYRIGHT PROTECTED DOCUMENT

© ISO 2021

All rights reserved. Unless otherwise specified, or required in the context of its implementation, no part of this publication may be reproduced or utilized otherwise in any form or by any means, electronic or mechanical, including photocopying, or posting on the internet or an intranet, without prior written permission. Permission can be requested from either ISO at the address below or ISO's member body in the country of the requester.

ISO copyright office
CP 401 • Ch. de Blandonnet 8
CH-1214 Vernier, Geneva
Phone: +41 22 749 01 11
Email: copyright@iso.org
Website: www.iso.org

Published in Switzerland

Contents

Page

Foreword	iv
Introduction	v
1 Scope	1
2 Normative references	1
3 Terms and definitions	1
4 Principle	1
5 Apparatus	2
6 Preparation of sample	2
6.1 General	2
6.2 Reference samples	2
7 Method A — Ratio method	2
7.1 Procedure	2
7.1.1 Measurement of reference samples by n-heptane extraction	2
7.1.2 LR-NMR measurement procedure of reference samples	3
7.1.3 LR-NMR measurement procedure of test samples	3
7.2 Calculation	3
7.2.1 Calibration curve	3
7.2.2 Calculation of isotactic index II_{na}	4
8 Method B — Absolute method	4
8.1 Procedure	4
8.1.1 Measurement of reference samples by n-heptane extraction	4
8.1.2 LR-NMR measurement procedure of reference samples	4
8.1.3 LR-NMR measurement procedure of test samples	4
8.2 Calculation	4
8.2.1 Calibration curve	4
8.2.2 Calculation of isotactic index II_{nb}	5
9 Expression of results	5
10 Test report	5
Annex A (informative) Example of a calibration curve for method A	7
Annex B (informative) Example of a calibration curve for method B	8
Bibliography	9

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.

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).

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. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see www.iso.org/patents).

Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

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.

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