SLOVENSKI STANDARD
SIST EN ISO 11337:2004

01-oktober-2004

Polimerni materiali ï Poliamidi ï Določanje e-kaprolaktama in w-laurolaktama s plinsko kromatografijo (ISO 11337:2004)

Plastics - Polyamides - Determination of e-caprolactam and w-laurolactam by gas chromatography (ISO 11337:2004)

Kunststoffe - Polyamide - Gaschromatographische Bestimmung von e-Caprolactam und w-laurolactam (ISO 11337:2004)

Plastiques - Polyamides - Détermination du e-caprolactame et du w-laurolactam par chromatographie en phase gazeuse (ISO 11337:2004)

Ta slovenski standard je istoveten z: **EN ISO 11337:2004**

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83.080.20	Plastomeri	Thermoplastic materials

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Plastics - Polyamides - Determination of e-caprolactam and w-laurolactam by gas chromatography (ISO 11337:2004)

Plastiques - Polyamides - Détermination du e-caprolactame et du w-laurolactam par chromatographie en phase gazeuse (ISO 11337:2004)

Kunststoffe - Polyamide - Gaschromatographische Bestimmung von e-Caprolactam und w-laurolactam (ISO 11337:2004)

This European Standard was approved by CEN on 13 May 2004.

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EUROPEAN COMMITTEE FOR STANDARDIZATION
COMITÉ EUROPÉEN DE NORMALISATION
EUROPÄISCHES KOMITEE FÜR NORMUNG

Management Centre: rue de Stassart, 36 B-1050 Brussels

Foreword

This document (EN ISO 11337:2004) has been prepared by Technical Committee ISO/TC 61 "Plastics" in collaboration with Technical Committee CEN/TC 249 "Plastics", the secretariat of which is held by IBN.

This European Standard shall be given the status of a national standard, either by publication of an identical text or by endorsement, at the latest by November 2004, and conflicting national standards shall be withdrawn at the latest by November 2004.

According to the CEN/CENELEC Internal Regulations, the national standards organizations of the following countries are bound to implement this European Standard: Austria, Belgium, Cyprus, Czech Republic, Denmark, Estonia, Finland, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Latvia, Lithuania, Luxembourg, Malta, Netherlands, Norway, Poland, Portugal, Slovakia, Slovenia, Spain, Sweden, Switzerland and United Kingdom.

Endorsement notice

The text of ISO 11337:2004 has been approved by CEN as EN ISO 11337:2004 without any modifications.

INTERNATIONAL STANDARD

ISO 11337

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Plastics — Polyamides — Determination of ε-caprolactam and ω-laurolactam by gas chromatography

Plastiques — Polyamides — Détermination du ε-caprolactame et du ω-laurolactame par chromatographie en phase gazeuse

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Case postale 56 • CH-1211 Geneva 20
Tel. + 41 22 749 01 11
Fax + 41 22 749 09 47
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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 11337 was prepared by Technical Committee ISO/TC 61, *Plastics*, Subcommittee SC 5, *Physical-chemical properties*.

Plastics — Polyamides — Determination of ε-caprolactam and ω-laurolactam by gas chromatography

WARNING — This International Standard may involve hazardous chemicals, materials or operations. It does not purport to address all of the safety problems, if any, associated with its use. It is the responsibility of the user to establish appropriate safety and health practices and to determine the applicability of regulatory limitations prior to use.

1 Scope

This International Standard specifies a method for determining ε-caprolactam and ω-laurolactam in polyamides by gas chromatography. It is suitable particularly for the determination of ε-caprolactam in polyamide 6 and ω-laurolactam in polyamide 12. Bearing in mind that gas chromatography offers a wide range of possible conditions, the method specified is that shown to have been suitable in practice.

Two variants of the basic method are specified:

— Method A is an extraction method with boiling methanol, and the extract is injected into a gas chromatograph.

— Method B is a method using a solvent, and the solution is injected into a gas chromatograph.

Method A is suitable for the determination of ε-caprolactam and method B for ε-caprolactam and ω-laurolactam.

2 Normative references

The following referenced documents are indispensable for the application 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 472, *Plastics — Vocabulary*

ISO 565, *Test sieves — Metal wire cloth, perforated metal plate and electroformed sheet — Nominal sizes of openings*

3 Terms and definitions

For the purposes for this document, the terms and definitions given in ISO 472 apply.

4 Method A: Extraction method

4.1 Principle

A test portion is extracted with boiling methanol and a small volume of the extract injected into a gas chromatograph equipped with a flame-ionization detector to separate and detect the volatile components. The extract contains 1-dodecanol as an internal standard.

4.2 Reagents

During the analysis, use only reagents of recognized analytical grade.

4.2.1 Methanol.

4.2.2 1-Dodecanol.

4.2.3 ε-Caprolactam.

4.3 Apparatus and materials

Ordinary laboratory apparatus, plus the following:

4.3.1 Mill, for reducing the sample to the required grain size.

A mill in which the sample is ground at a low temperature is preferred. Large pieces can be reduced in size with a pair of scissors before they are fed to the mill.

4.3.2 Two sieves, with aperture sizes of 710 µm and 500 µm respectively, complying with the requirements of ISO 565.

4.3.3 Extraction apparatus, that will accommodate an extraction crucible or porous ceramic thimble containing the test portion.

The apparatus shall be of such a design that the crucible or thimble is heated by the rising methanol vapour or the apparatus shall be constructed of an extraction flask with a Soxhlet-type reflux condenser.

Examples of suitable extraction apparatus designed along these lines are:

EXAMPLE 1

- 250 ml extraction flask;
- extraction chamber to accommodate the extraction crucible so that it is enveloped on all sides by the rising methanol vapour and the condensed methanol drips through it continuously;
- glass triangle to support the crucible;
- reflux condenser;
- sintered-glass filter crucible, pore size 40 µm to 50 µm, capacity 30 ml;
- porcelain filter-plate of slightly smaller diameter than the crucible, with holes of diameter 0,4 mm.

EXAMPLE 2

- 250 ml extraction flask;
- jacketed Soxhlet extractor;

— reflux condenser;

— sintered-glass filter crucible, pore size 40 µm to 50 µm, capacity 30 ml, or a porous ceramic thimble of similar capacity (the dimensions shall be such that the crucible or thimble can be satisfactorily accommodated in the Soxhlet apparatus);

— porcelain filter-plate of slightly smaller diameter than the crucible or thimble, as appropriate, with holes of diameter 0,4 mm.

4.3.4 Suitable heating device for extraction apparatus.

4.3.5 Analytical balance, accurate to 0,000 2 g.

4.3.6 Liquid nitrogen or **solid carbon dioxide**, if necessary.

4.3.7 Gas chromatograph, with flame-ionization detector.

a) Column

The following columns are suitable:

— a glass column (3 mm ϕ × 1,6 m), packed with acid-washed Chromosorb W of particle diameter 0,149 mm to 0,177 mm (80 mesh to 100 mesh) coated with 10 % (by mass) poly(ethylene glycol) 20M;

— a fused-silica capillary column (0,31 mm ϕ × 30 m), liquid phase 95 % dimethyl, 5 % diphenyl polysiloxane, film thickness 0,25 µm;

— a megabore carbowax column (0,53 mm ϕ × 15 m) of corresponding separation efficiency.

The method of packing is not specified but shall be such as to obtain satisfactory separation efficiency. The capillary column is preferred.

Other column dimensions are permissible, but only if they have been proved to give the same results.

Suggested operating conditions are shown in Table 1.

Table 1 — Operating conditions for gas chromatograph

Item	Value
Column temperature	200 °C
Injector temperature	250 °C
Detector temperature	250 °C
Carrier gas	Helium or nitrogen
Carrier gas flow rate	20 ml/min

b) Detector

Use a flame-ionization detector in which the hydrogen and air flow rates can be adjusted so that:

— sensitivity is high;

— the relationship between response and concentration is linear over the whole measurement range;

— small changes in flow rate produce only insignificant effects on response and sensitivity.