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Plastics — Determination of caprolactam and its cyclic and linear oligomers by HPLC

Plastiques — Détermination du caprolactame et de ses oligomères cycliques et linéaires par CLHP

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

For an explanation on 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 the following URL: www.iso.org/iso/foreword.html.

This document was prepared by Technical Committee ISO/TC 61, *Plastics*, Subcommittee SC 5, *Physical-chemical properties*.

This third edition cancels and replaces the second edition (ISO 15033:2007), which has been technically revised to add method B.

Introduction

The method A specified in this document can be used for HPLC determination of the cyclic oligomers of caprolactam (n = 1) up to and including the hexamer (n = 6), using UV detection. If desired, after post-column reaction of the primary amine with 1,2-phthalic dicarboxaldehyde, online determination of the linear oligomers up to and including the hexamer can also be carried out.

The determination is not quantitative for oligomers higher than the hexamer (n > 6). In the determination of cyclic oligomers the sensitivity for the tetramer and higher oligomers is constant, which means that calibration should take place up to and including the tetramer (n = 4).

The linear oligomers are determined by the fluorescence of the iso-indole group, which is a product of the reaction between the primary amino group, 1,2-phthalic dicarboxaldehyde and 3 mercaptopropionic acid. The calibration with the linear oligomers should be carried out up to and including the hexamer (n = 6).

The method B included in this document is intended for the determination of caprolactam and its cyclic dimer only, following the same principle and using the same equipment as method A, but significantly faster.

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SAFETY STATEMENT — Persons using this document should be familiar with normal laboratory practice, if applicable. This document does not purport to address all of the safety concerns, 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 any other restrictions.

1 Scope

This document describes a high-performance liquid chromatography (HPLC) method for determining the concentrations of cyclic oligomers of caprolactam, from 0,01 % by mass upwards, and linear oligomers of caprolactam, from 5 mg/kg upwards, both up to and including the hexamer of caprolactam (n = 6), in samples of polyamide 6, caprolactam and mixtures of rearrangement products in water.

A second, significantly faster, HPLC method is included for determination of caprolactam and its cyclic dimer, based on the same principle and using the same equipment as the first method.

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 472, *Plastics — Vocabulary*

3 Terms and definitions

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

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

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

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

4 Principle

A test sample is dissolved in, or diluted with, formic acid and the oligomers separated in the presence of a low-pH mobile phase using a column filled with reversed-phase packing material. The cyclic oligomers are detected by UV absorption at 200 nm. If desired, the linear oligomers can be detected by fluorescence after post-column reaction of the primary amino group with 1,2-phthalic dicarboxaldehyde and 3-mercaptopropionic acid. The concentrations are calculated by comparison of the measured values with those of calibration solutions.

5 Reagents

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

5.1 Water, ultrapure or double-distilled.

5.2 Phosphoric acid, 85 % by mass.