

SLOVENSKI STANDARD
oSIST prEN ISO 10927:2017
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Polimerni materiali - Ugotavljanje molekularne mase in porazdelitev molekularne mase z masno spektrometrijo po laserski desorpciji/ionizaciji i(MALDI-TOF-MS) (ISO/DIS 10927:2017)

Plastics - Determination of the molecular mass and molecular mass distribution of polymer species by matrix-assisted laser desorption/ionization time-of-flight mass spectrometry (MALDI-TOF-MS) (ISO/DIS 10927:2017)

Kunststoffe - Bestimmung der Molmasse und Molmassenverteilung von polymeren Species durch matrixunterstützte Laser-Desorptions/Ionisations-Flugzeit-Massenspektrometrie (MALDI-TOF-MS) (ISO/DIS 10927:2017)

Plastiques - Détermination de la masse moléculaire et de la distribution des masses moléculaires des polymères par spectrométrie de masse, à temps de vol, après désorption/ionisation laser assistée par matrice (SM-MALDI-TOF) (ISO/DIS 10927:2017)

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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.

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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The committee responsible for this document is ISO/TC 61, *Plastics*, Subcommittee SC 5, *Physical-chemical properties*.

This second edition cancels and replaces the first edition (ISO 10927:2011), which has been technically revised.

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Introduction

The molecular mass and molecular mass distribution of a synthetic polymer are fundamental characteristics that result from the polymerization process. They may be used for a wide variety of correlations for fundamental studies and for processing and product applications. Determination of the molecular mass and molecular mass distribution is used for quality control of polymers and for specification purposes in the commerce of polymers. The comparability of MALDI-TOF-MS results obtained in different laboratories can be ensured by using standardized conditions of measurement, identical samples and identical matrix preparation methods. The classification of MALDI-TOF-MS as an equitable (standardized) method compared with other established methods of polymer characterization could result in a significant increase in the use of MALDI-TOF-MS.

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Plastics — Determination of the molecular mass and molecular mass distribution of polymer species by matrix-assisted laser desorption/ionization time-of-flight mass spectrometry (MALDI-TOF-MS)

1 Scope

This International Standard specifies a general method for determining the average molecular mass and molecular mass distribution of polymers (see Reference^[1]) from $2\,000\text{ g} \cdot \text{mol}^{-1}$ to $20\,000\text{ g} \cdot \text{mol}^{-1}$ by matrix-assisted laser desorption/ionization time-of-flight mass spectrometry (MALDI-TOF-MS).

The average molecular masses and molecular mass distributions are calculated from a calibration curve constructed using synthetic-polymer and/or biopolymer standards. This method is therefore classified as a relative method.

The method is not applicable to polyolefins or to polymers with a polydispersity $> 1,2$.

2 Normative references

The following documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. 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 and the following apply.

3.1 matrix-assisted laser desorption/ionization time-of-flight-mass spectrometry MALDI-TOF-MS

mass-spectrometric technique in which the separation is based on different flight times in a field free flight tube depending on the mass of formed polymer ions after ionization by a laser, desorption and acceleration by high voltage

3.2 molecular mass

M

sum of the masses of the atoms making up a molecule

Note 1 to entry: Molecular weight is also used for molecular mass.

3.3 average molecular mass

three possible types of average molecular mass are defined by the following equations, where

N_i is the number of molecules of species i of molecular mass M_i ;

m_i is the mass of the i th species (i.e. $m_i = N_i M_i$);

$z_i = m_i M_i / \sum m_i$.

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3.3.1

number-average molecular mass

 M_n

$$M_n = \frac{\sum_{i=1}^{\infty} (N_i \times M_i)}{\sum_{i=1}^{\infty} N_i} \quad (1)$$

3.3.2

mass-average molecular mass

 M_w

$$M_w = \frac{\sum_{i=1}^{\infty} (N_i \times M_i^2)}{\sum_{i=1}^{\infty} (N_i \times M_i)} = \frac{\sum_{i=1}^{\infty} m_i \times M_i}{\sum_{i=1}^{\infty} m_i} \quad (2)$$

3.3.3

z-average molecular mass

 M_z

$$M_z = \frac{\sum_{i=1}^{\infty} (N_i \times M_i^3)}{\sum_{i=1}^{\infty} (N_i \times M_i^2)} = \frac{\sum_{i=1}^{\infty} (m_i \times M_i^2)}{\sum_{i=1}^{\infty} m_i \times M_i} = \frac{\sum_{i=1}^{\infty} z_i \times M_i}{\sum_{i=1}^{\infty} z_i} \quad (3)$$

4 Principle

The MALDI process involves the desorption and the ionization of an analyte dispersed in an organic small-molecule matrix. The matrix must be able to absorb the laser energy. A metal salt may be added to cationize the analyte. A polymer is co-crystallized or co-mixed with the matrix molecule and deposited on the target. A short-duration UV laser pulse is used to desorb the matrix and the analyte. The laser energy is transferred to the matrix molecules, causing them to vaporize. Analyte and matrix molecules leave the target surface in a plume. Due to the very short desorption time, polymer molecules do not degrade. The polymer in the desorption plume gains a cation and is accelerated by a high voltage, drifts down the field-free flight tube and is detected at the end of the flight tube. The time of flight of the species is a measure of its mass. From the distribution of arrival times and the calibration of the arrival times with known mass standards, the mass distribution of the polymer is determined.

5 Reagents

5.1 Matrices

2,5-dihydroxybenzoic acid (gentisic acid, DHB), trans-2-[3-(4-tert-Butylphenyl)-2-methyl-2-propenylidene]malononitrile (DCTB), and 1,8,9-trihydroxyanthracene (dithranol) are the recommended matrices for this method. All of these materials shall be at least 97 % pure. They shall be stored in a freezer and warmed to room temperature immediately before use.

5.2 Solvents

The recommended solvent is tetrahydrofuran (THF). THF with an antioxidant, such as 2,6-di-tert-butyl-4-methylphenol (dibutylated hydroxytoluene, BHT) at a concentration of 0,025 % to 0,1 % (m/V), shall be stored in an amber container. If THF without an antioxidant is used, it shall be stored in an amber container under an inert gas. Otherwise, it will react with oxygen to form peroxides which are hazardous on evaporative concentration.

Depending on the solubility of the polymer being investigated, toluene, methanol and acetone may also be used.

High-purity solvents are recommended.