SLOVENSKI STANDARD
oSIST prEN ISO 16094-2:2024

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Kakovost vode - Analiza mikroplastike v vodi - 2. del: Metode vibracijske spektroskopije za vodo z nizko vsebnostjo suspendiranih trdnih delcev, vključno s pitno vodo (ISO/DIS 16094-2:2023)

Water quality - Analysis of microplastic in water - Part 2: Vibrational spectroscopy methods for waters with low content of suspended solids including drinking water (ISO/DIS 16094-2:2023)

Wasserbeschaffenheit - Analyse von Kunststoff in Wasser - Teil 2: Verfahren mittels Vibrationsspektroskopie (ISO/DIS 16094-2:2023)

Qualité de l'eau - Analyse des microplastiques dans l'eau - Partie 2: Méthodes de spectroscopie vibrationnelle pour les eaux à faible teneur en matières en suspension, y compris l'eau potable (ISO/DIS 16094-2:2023)

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Water quality — Analysis of microplastic in water —

Part 2: Vibrational spectroscopy methods for waters with low content of suspended solids including drinking water

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Contents

Page

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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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 147, *Water quality*, Subcommittee SC 2, *Physical, chemical and biochemical methods*.

A list of all parts in the ISO 16094 series can be found on the ISO website

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

Pollution linked to microplastics is recognised as a global phenomenon. The standardization of the sampling, quantification and identification protocols is required to ensure reliability and comparability of the data produced for health and environmental risk assessments.

Microplastics in water can be identified and quantified using various methodological approaches. Depending on the measurement objectives, several complementary approaches shall be used to cover the full spectrum of microplastics (size and chemical nature). Table 1 resumes the characteristics and the information obtained with the spectroscopic technics.

Table 1 — Characteristics of the various analytical techniques and information obtained.

Characteristics and information obtained	Raman microspectroscopy	Infrared microspectroscopy
Type of Sample	Water filtrate residues	
Chemical nature of the polymer	Yes	
Information provided by analytical technique	Functional groups	
Results expression	Polymer type, number of particles, size	
Minimum measurable size of particles	1 µm to 10 µm	15 µm to 20 µm
Minimum mass subject to measurement after preparation	undefined	
Consequences for the test sample after measurement	non-destructive	
Main interferences	Mineral particles, coloured particles, pigments, fluorescence, fatty acids, fatty amides, proteins, surface alterations by biofilms or weathering, carbohydrates, generated by microbes	Mineral particles, proteins, surface alterations by biofilms or weathering, particles loaded with carbon black, presence of water, carbohydrates, generated by microbes

Water quality — Analysis of microplastic in water —

Part 2: Vibrational spectroscopy methods for waters with low content of suspended solids including drinking water

WARNING — Persons using this document should be familiar with normal laboratory practice. This document 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.

IMPORTANT — It is absolutely essential that tests conducted in accordance with this document be carried out by suitably qualified staff.

1 Scope

This document sets out key principles for the investigation of microplastics in drinking water and water with low content of natural suspended solids using a microscopy technique coupled with vibrational spectroscopy.

This method is applicable to:

— determine the size of microplastics [1 µm to 5 000 µm], count them and classify them by size range;

— identify the chemical composition of microplastics, the main ones (most used in industry and most abundant in the environment) being: polyethylene (PE), polypropylene (PP), polyethylene terephthalate (PET), polycarbonate (PC), polystyrene (PS), polytetrafluoroethylene (PTFE), polyvinyl chloride (PVC), polyamide (PA), polymethyl methacrylate (PMMA) and polyurethane (PU);

— identify the nature of other particles that are outside the scope of this document, for example minerals, proteins, cellulose and pigments.

This method is intended to determine and characterize large numbers of particles in the sample in automatic mode.

The method does not apply to the characterization of substances intentionally added to or adsorbed on the surface of microplastics. The method does not apply to the determination of the geometric shape of microplastics.

The method is applicable to water with low quantity of organic matter and other suspended matter as ISO 6107 definition (1-100 mg/l or lower if interfere with the determination), i.e

— ultrapure water;

— water intended for human consumption;

— raw groundwaters.

Given the very low concentrations of microplastics usually present in these waters, special attention shall be paid to potential sources of contamination during the samples preparation.

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 1043-1, *Plastics — Symbols and abbreviated terms — Part 1: Basic polymers and their special characteristics*

ISO/TS 13530, *Water quality — Guidance on analytical quality control for chemical and physicochemical water analysis*

3 Terms, definitions and abbreviations

3.1 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 https://www.electropedia.org/

3.1.1
microplastic
any solid plastic or synthetic polymer particle insoluble in water with the largest dimension between 1 µm and 5 mm

Note 1 to entry: Microplastics may show various shapes.

Note 2 to entry: This definition encompasses the ISO/TR 21960 definitions of large microplastics and microplastics

Note 3 to entry: The term “microplastics” covers the sum of several individual microplastic particles.

3.1.2
microparticle
solid particle insoluble in water, with the largest dimension between 1 µm and 5 mm

3.1.3
Raman spectroscopy
spectroscopy in which the Raman effect is used to investigate molecular energy levels

[SOURCE: ISO 18115-2:2013, 5.129]

3.1.4
Raman effect
scattered light, associated with molecules illuminated with monochromatic light, characterised by an energy loss or gain arising from rotational or vibrational excitations

[SOURCE: ISO 18115-2:2013, 5.128]

3.1.5
infrared spectroscopy
analytical technique based on

— the absorption of infrared radiation by functional groups, used to identify and quantify the absorbing functional groups,

— the Fourier transform or use of a laser source to obtain an IR spectrum

[SOURCE: Adapted from: ISO/TS 14101:2012, 3.3]

3.1.6
particle-free water
Water not containing microplastics or with the lowest possible concentration of microplastics.

3.1.7
Feret diameter
distance between two parallel lines which are tangent to the perimeter of a particle

[SOURCE: ISO 10788:2014, 2.1.4]

3.1.8
maximum Feret diameter
maximum length of an object whatever its orientation

[SOURCE: ISO/TR 945-2:2011, 2.1]

3.1.9
reporting limit
smallest number of objects (microplastics) that the laboratory can measure reliably by type of polymer and size class under routine laboratory operating conditions

Note 1 to entry: A different reporting limit may be associated with each filter reading model.

Note 2 to entry: For determination of the reporting limit see 10.4.

3.1.10
correlation index
hit quality index
(HQI)
index or spectral correlation coefficient (typically between 0 and 1) or percentage allowing to calculate the similarity between two spectral signatures

3.2 Abbreviations

ATR	Attenuated total reflection (internal)
FTIR	Fourier-transform infrared spectroscopy
HEPA	High-Efficiency Particulate Air filter
HQI	Hit quality index
IR	Infrared spectroscopy
PA	Polyamide
PC	Polycarbonate
PE	Polyethylene
PET	Poly(ethylene terephthalate)
PMMA	Poly(methyl methacrylate)
PP	Polypropylene
PS	Polystyrene
PTFE	Polytetrafluoroethylene
PU	Polyurethane
PVC	Poly(vinyl chloride)
RL	Reporting limit
SDS	Sodium Dodecyl Sulfate
USAF	United States Air Force

µFTIR	Microscopy coupled with FTIR spectroscopy
µRaman	Microscopy coupled with Raman spectroscopy

4 Principle

Counting and identification are based on filtering a specified volume of a water or prepared water sample through a filtering membrane with a pore size able to retain the microplastics of interest (based on the microplastics size class claimed to be analysable by the laboratory). The filter is subsequently analysed by microscopy coupled with at least one of the following two techniques:

1) Infrared spectroscopy is used to characterise the molecular composition and the structure of a material. It is based on the absorption of infrared radiation by the analysed material. Absorption bands are obtained, resulting from intramolecular vibrational modes that absorb the infrared radiation of characteristic wavenumbers, enabling the identification of organic or mineral materials.

2) Raman spectroscopy is used to characterise the molecular composition and the structure of a material. A beam of monochromatic light is transmitted onto the sample to be studied and the scattered light is analysed having been collected by a lens sent into the instrument's detection unit to measure the intensity of light in the covered wavenumber range.

Both techniques coupled with optical microscopy can be used to obtain the size distribution and number of the microplastics and identify the type of polymer.

5 Interferences

One of the main problems is to correctly detect and identify microplastic particles in real samples when they are mixed with other solid particles naturally present in the concerned waters (i.e. minerals, natural organic matter).

Given the very low concentrations of microplastics usually present in the matrices of interest, special attention should be paid to possible sources of external contamination during sampling and sample preparation. Recommendations for avoiding external contamination by microplastics are given in paragraphs 7.1, 7.3, 7.4, 8.

It should be noted that spectral interference between microplastics and other naturally occurring particles in the water may lead to false negative or false positive results in terms of microplastic identification. Some common cases and recommendations for the content of the spectral database are given in paragraph 9.10 and in Annexes B - E.

6 Reagents, consumables and reference materials

Check water and all chemicals for the presence of microplastics in advance and remove them by filtration before use (for example, through the cellulose filter or another non-polymeric membrane with a pore size of 0,2 µm).

6.1 Particle-free water of known quality

Water not containing microplastics or with the lowest possible concentration of microplastics, estimated by calculating the mean value and regularly checked via the test of analytical control blanks.

This water may be prepared by the laboratory using ultrapure water previously filtered using a filter made of inorganic materials and a 0,2 µm cellulose filter, for example. This water may also be purchased.