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Principles for the analysis of microplastics present in the environment

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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/directiveswww.iso.org/directives).

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This document was prepared by Technical Committee ISO/TC 61, *Plastics*, Subcommittee SC 14, *Environmental aspects*, in collaboration with the European Committee for Standardization (CEN) Technical Committee CEN/TC 249, *Plastics*, in accordance with the Agreement on technical cooperation between ISO and CEN (Vienna Agreement).

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Introduction

The analysis of plastics and microplastics is a new field in relation to other areas of environmental analysis. A large number of scientific publications exist, but they do not apply a uniform analysis, which makes it difficult to compare the results.

This document sets out key principles for the investigation of microplastics in the environment, which should be taken into account in the subsequent development of specific procedures for sampling, sample preparation and detection. A large number of the principles described in this document can be applied, analogously, to other matrices and products, including foodstuffs and drinking water. The objective is to present a pool of methods and notes that is as harmonized as possible and to make it available for use in science, businesses and administrations.

What is true for analytics is also true for definitions in the same way. On the one hand, the terms used in this document are based on existing definitions in the subject area, but on the other hand, analytical requirements are also taken into account. This applies, for example, to the term "large microplastics". The particle size to be investigated is closely related to the detection method to be selected. In the course of future specific work, it can be necessary to modify existing definitions slightly and adapt them to new knowledge and requirements.

With regard to the definitions, including the ideas of size classes, it is pointed out, that the discussion is ongoing in various technical committees in ISO and other standardization bodies. The definitions in this document show the status in ISO TC 61/SC 14. The definitions chosen in this document are adapted from the ISO report on plastics. The basis of the classification is based on the metric sizes and the associated designations. Microplastics is thus derived from micrometres.

NOTE Microplastics can also stem from different sources not specifically mentioned in this document, such as textiles, paints and tyres.

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Principles for the analysis of microplastics present in the environment

1 Scope

This document describes the principles to be followed in the analysis of microplastics in various environmental matrices. This includes the unique particle size classification of plastics, the use of certain apparatus with regard to sampling, sample preparation, and the determination of representative sample quantities.

The purpose of this document is to specify minimum requirements until specific standards for the different case situations are available. This is important to ensure that the development of the specific standards is done on a consistent basis to ensure that comparison or correlation of results is possible.

This document does not include requirements for monitoring actions.

2 Normative references

There are no normative references in this document.

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. Foundated references, the latest edition of the referenced document (including any amendments) applies.

ISO 472, Plastics — Vocabulary, iteh.ai/eatalog/standards/sist/ffae5791-fa2d-4626

3 Terms and definitions

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

large microplastic

any solid plastic particle insoluble in water with any dimension between 1- $\underline{\text{mm}}$ and 5- $\underline{\text{mm}}$

Note 1 to entry: Microplastics may show various shapes.

Note 2 to entry: Typically, a large microplastics object represents an item consisting of plastics or a part of an enduser product or a fragment of the respective item.

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 $[SOURCE: ISO/TR\ 21960: 2020,\ 3.10,\ modified\ --\ term\ number\ in\ Note\ 1\ to\ entry\ was\ removed.]$

3.2

microplastic

any solid plastic particle insoluble in water with dimension between 1- μ m and 1 000 μ m (= 1 mm)

Note 1 to entry: Primary microplastics object represents a particle intentionally added to end-user products for example cosmetic means, coatings, paints etc. Secondary microplastics object can also result as a fragment of the respective item.

Note 2 to entry: Microplastics have regular and irregular shapes (see ISO 9276-6:20172008).

Note 3 to entry: The defined dimension is related to the longest length of the particle.

[SOURCE: ISO/TR 21960:2020, 3.9, modified — Note 1 to entry was removed, all other Notes to entry were changed.]

3.3

additives

substances which are used to process plastics or to modify end use properties of plastics

Note 1 to entry: Important additives such as fillers/reinforced materials, softeners and flame retardants are referenced according to ISO 1043-2 to ISO 1043-4.

4 General aspects

Microplastics is a term that comes along with different physical and chemical properties, such as shape, size (range), type of polymer(s), presence of additives, presence of fillers, state of degradation and so on. The amount of microplastics in a given matrix can be measured in different ways, i.e. as number (of particles) or mass content/fraction in relation to the sample's quantity, which itself can be based on various units (volume, weight, etc.). Hence, before selecting a suitable (set of) method(s), the question(s) to be answered and properties to be measured need to be specified carefully. This applies not only to detection methods but also to the sampling and processing/preparation methods associated with them, right up to the statistical evaluation of results.

A schematic representation of the interdependencies of microplastics analysis is shown in Figure 1. As a rule, the objective or objectives of a measurement or a measurement program is/ are based on a clear question/task or on an evaluation concept involving necessary assessment parameters, respectively (for example integration into an overall ecological context, thresholds for monitoring). A suitable detection method is then selected, which generates the desired result parameters (such as polymer type, mass content, number, shape, size, degradation status).

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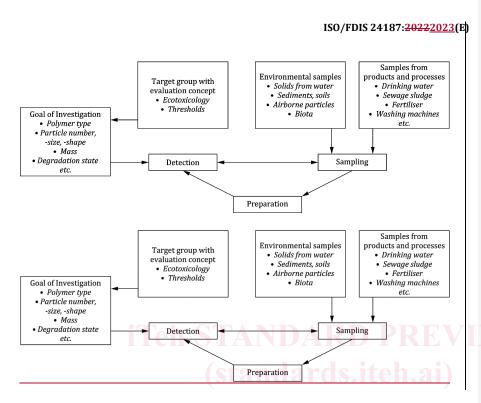


Figure 1 — Schematic representation of interdependencies during microplastics analysis in environmental and related matrices 18.24187

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5 General requirements for all analytical steps

All analytical steps (sampling, sample preparation, detection) shall be undertaken in plastics-free or low-plastics working conditions. These include the avoidance of standard plastics products (for example tubes, vessels). Contamination, especially cross-contamination shall be avoided, the user should avoid using plastics equipment wherever possible. Instead, alternatives made of metal, glass or ceramics should be used. As an exception and after it was proved by experiments (for example by characterizing the container), types of plastics that are not to be detected or evaluated can be used as well. Care should be taken that also-personal protective equipment (e.g. lab coats, gloves) are also made of non-synthetic material or material that does not interfere with the analyses. Recovery tests should be performed for each analytical step.

If feasible, samples should be handled in laminar flow boxes in the laboratory or clean rooms (class 3 according to ISO 14644-1, especially during the preparation process of samples and during the determination of particle numbers.

It shall be determined beforehand, whether—a hygienization of samples is necessary. Sterilization is a standard recommendation for the analysis of dry samples from wastewater, sewage sludge and organic

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wastes. Various methods can be applied, but each of them has specific impact on the integrity of microplastics particles in the sample $\frac{1}{2}$.

- a) Steam sterilization: risk of melting microplastics (for example PE, PP).
- b) Radiation sterilization (gamma, beta radiation, UV radiation): risk that the polymer structure is degraded (cleavage of polymer chains and oxidation).
- c) Chemical sterilization: risk that polymer structure or the particles' surface is chemically modified.

Relevant information about the measurement conditions and control processes (quality assessment and quality control/QAQC) shall be recorded, including all analytical steps. For general quality control measures in laboratories, see ISO/IEC 17025:2017. For intercomparison tests, see ISO 13528.

Blank value determination for the applied detection methods is essential, since contamination (for example by airborne particles) during sampling, preparation and detection can easily occur. Determination of blank values is essential; the number of blanks depends on the concrete method to be applied. More specific requirements have to be given in upcoming standards.

A classification of microplastics into size classes according to Table 1 is recommended. Small particles that occur in higher quantities are grouped into narrower classification classes than the larger particles, which are more relevant in terms of mass and classified into wider classes. This also enables a higher methodological feasibility of processes (including feasibility of filtration, detection limits in analytics) and a better integration of particle quantities/masses in impact analyses (i.e. for environmental assessments). The proposed size classes are given in Table 1. The maximum dimension/-diameter-/length of a particle defines the size class

Table 1 — Particle size classification

ation	Microplastics					Large microplastics	
humps:	1 to < 5	5 to < 10	10 to < 50	50 to < 100	100 to < 500	500 to < 1 000	1 000 to 5 000
μm	3	7,5	30	75	300	750	3 000
mg	1,4 × 10 ⁻⁸	2,2 × 10 ⁻⁷	1,4 × 10 ⁻⁵	2,2 × 10 ⁻⁴	0,014	0,22	14
number	1,0 × 10 ⁹	6,4 × 10 ⁷	1,0 × 10 ⁶	6,4 × 10 ⁴	1 000	64	1
	μm μm mg	μm 3 mg 1,4 × 10-8	μm 3 7,5 mg 1,4 × 10 ⁻⁸ 2,2 × 10 ⁻⁷	μm 3 7,5 30 mg 1,4 × 10 ⁻⁸ 2,2 × 10 ⁻⁷ 1,4 × 10 ⁻⁵	μm 3 7,5 30 75 mg 1,4 × 10 ⁻⁸ 2,2 × 10 ⁻⁷ 1,4 × 10 ⁻⁵ 2,2 × 10 ⁻⁴	μm 1 to < 5 5 to < 10 10 to < 50 50 to < 100 100 to < 500 μm 3 7,5 30 75 300 mg 1,4 × 10 ⁻⁸ 2,2 × 10 ⁻⁷ 1,4 × 10 ⁻⁵ 2,2 × 10 ⁻⁴ 0,014	μm 1 to < 5 5 to < 10 10 to < 50 50 to < 100 100 to < 500 to < 1000 750

6 Identification of appropriate detection methods

6.1 General

The selection of one or more quantitative or qualitative detection method(s) depends specifically on the objectives and tasks of a project or an existing requirement. The various detection methods differ regarding the generated result per measurement. These include identification of the polymer (type of polymer) and other qualitative properties (i.e. presence of additives, chemical composition, molecular

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