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Water quality — Sampling —

Part 27: Guidance on sampling for microplastics in water

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Foreword

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This document was prepared by Technical Committee ISO/TC 147 *Water quality*, Subcommittee SC 6, *Sampling (general methods)*.

A list of all parts in the ISO 5667 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

Microplastic occurrence in the environment is a prominent concern both to the public and to the scientific community. Determining the amount and distribution of microplastics in water bodies and domestic water is therefore a critical task.[1]-[6] However, the methodology for sampling microplastics in water samples is still lacking in precision. Consistent methodology is only starting to emerge, but still no universal protocol exists for the sampling of these contaminants in water.

The presence of small plastic fragments in the ocean was first reported in 1972,[7] but it was in 2004 that the term “microplastics” was proposed for the first time to describe plastic particles of a few micrometres in diameter.[8] Since then, a wealth of information became available on the abundance and type of microplastics in the marine environment, freshwater and estuarine systems. However, the different studies have used diverse techniques to sample, extract, treat and detect microplastic present in water.

There are many reasons why different studies investigating microplastic occurrence in water and wastewater show different results. The disparity between some of the findings (for microplastic type and abundance) can be partially explained by the fact that differing sampling techniques have been used. Variables pertaining to both time of year and time of day, flow rate and volume of water sampled, grab sampling or sieving the water over an extended period, the use of plastic containers or tubing, selection of a few parts of the sample for analysis, or dissimilar devices to capture the microplastic fragments, can be the causes of variation in study results.

While several standards for water sampling and water quality already exist (e.g. ISO 5667 series and, in particular, ISO 5667-17), microplastics as particular determinands pose a specific challenge which requires a more specific approach. For example, microplastics sampling requires the use of very specific materials for collecting, handling and storing to avoid cross-contamination. Also, microplastic buoyancy can vary depending on their composition, size, shape or colonization by microorganisms, and microplastics are not homogeneously distributed in the water column. Therefore, a more targeted and detailed set of sampling protocols is required to account for these differences. To better understand the fate and impact of microplastics in the environment, a more specific standardized sampling approach should be adopted and applied.

Water quality — Sampling —

Part 27: Guidance on sampling for microplastics in 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 and to ensure compliance with any national regulatory conditions.

IMPORTANT — It is essential that tests conducted according to this document be carried out by suitably trained staff.

1 Scope

This document specifies the basic methods for sampling suspended microplastics in water (domestic water, freshwater, seawater, treated wastewater and untreated wastewater), for their subsequent characterization. Suspended particles can also include synthetic or semi-synthetic polymeric materials (such as rubber). This document does not cover chemical analysis, biological (ecotoxicological) methods or physical methods, nor the pre-treatment or digestion methods intrinsic to such analyses.

This document covers general methodologies:

— for grab sampling, sampling using a set of successive filters of different pore sizes (cascade filtration), for water samples with low, medium and high content of suspended solids, and

— for net sampling using, for example, manta, plankton or neuston nets.

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 5667-3, *Water quality — Sampling — Part 3: Preservation and handling of water samples*

3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

ISO and IEC maintain terminology 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
microplastic
solid plastic or synthetic polymer particle insoluble in water with the largest dimension between 1 µm and 5 mm

Note 1 to entry: Microplastics can show various shapes.

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

3.2
plastic
material which contains as an essential ingredient a high polymer and which, at some stage in its processing into finished products, can be shaped by flow

[SOURCE: ISO 472:2013, 2.702, modified — Notes 1 and 2 to entry have been deleted.]

3.3
rubber
family of polymeric materials which are flexible and elastic

[SOURCE: ISO 1382:2020, 3.420, modified — the domain and Notes 1 and 2 to entry have been deleted.]

3.4
suspended solid
solid remaining in suspension in water, which can be removed by sedimentation, filtration or centrifugation

[SOURCE: ISO 6107:2021, 3.554]

3.4.1
high suspended solid content
concentration of solids suspended in water above 500 mg/l

3.4.2
medium suspended solid content
concentration of solids suspended in water ranges between 100 mg/l to 500 mg/l

3.4.3
low suspended solid content
concentration of solids suspended in water ranges between 1 mg/l to 100 mg/l

3.5
grab sample
single discrete sample directly collected from a water body at a specific time and location (and depth when relevant)

3.6
blank
sample identical to the sample of interest, but in the absence of the determinand

3.7
field blank
blank (3.6) to verify possible contamination during sampling

Note 1 to entry: A field blank is prepared in the laboratory using water (filtered before use through an inert filter with a pore size smaller than 1 µm) and sent with the sampling personnel for exposure to the sampling environment.

3.8
domestic water
water either in its original state or after treatment

Note 1 to entry: Domestic water is intended for human use, such as cooking, food preparation, washing, drinking or other domestic purposes.

3.9
limit of detection
smallest true value of the measurand which ensures a specified probability of being detectable by the measurement procedure

Note 1 to entry: For *microplastics* (3.1), the limit of detection is defined as the methods' capability of reliably detecting at least one particle or a defined mass.

[SOURCE: ISO/TR 22930-1:2020, 3.6, modified — the term has been changed from "detection limit" to "limit of detection", Note 1 to entry has been replaced and Note 2 to entry has been deleted.]

4 Principles and general considerations

4.1 Methodologies

4.1.1 General

The microplastic sampling approaches described in this document can be grouped mainly as grab sampling and volume-reduced sampling.

Grab sampling (also known as spot sampling) consists of collecting a given volume of water (without reducing it during this procedure). Subsequent separation, pre-treatment (when required) and analysis of the microplastics are done in a laboratory. In volume-reduced sampling, the microplastics are collected while reducing the volume of water during the sampling process, by passing the water through nets or sieves. The collected microplastics are preserved for further processing in a laboratory.

4.1.2 Grab sampling

The methodology for the sampling of microplastics via grab sampling entails filling non-plastic containers (or made of materials which are less present in the environment or not part of the measurement concept) with domestic water, freshwater, treated or untreated wastewater, or surface, near surface or deep-water samples, for the subsequent pre-treatment and analysis in a laboratory. The container material must be suitable for the sample. The sampling date, time, location, depth (when relevant) and collected volume are recorded.

The main advantage of grab sampling is that all the microplastics present in the sampled water are collected without size limitation, in contrast to volume-reduced sampling, where the selected mesh size determines the smallest size of sampled particles. In grab sampling, risks of contamination are reduced when compared to volume-reducing sampling in an open system (e.g. using manta trawls, plankton or neuston nets), because handling of the sample and time of exposure to the surrounding environment are shorter. However, the main disadvantage of grab sampling is the limited volume of sample that can be collected, stored and processed.[9]

4.1.3 Volume-reduced sampling

In volume-reduced sampling, the microplastics are extracted and aggregated from the medium before analysis. This can be done via cascade filtration, where a volume of water passes through a series of filters with decreasing mesh sizes; or by using manta trawls, plankton or neuston nets. While volume-reducing methods allow for a greater area and/or volume of water to be sampled, their main disadvantages are the limits on the minimum microplastic size that can be collected. This is determined by the sizes of the filters, sieves used and mesh selectivity,[10][11] alongside a higher risk of contamination because the time of exposure to the surrounding environment is long.

The methodology of cascade filtration allows for the sampling of domestic, treated and untreated wastewater, surface and subsurface waters with low, medium or high suspended solid content, and consists of passing the water to be sampled through a series of filters of different pore sizes, for the retention of the microplastics in the different filters. The sampling date, time, location, mesh pore size and the flow rate of the water passing through the filters over the sampling time interval are recorded, in order to express the results in terms of collected particles per unit volume, or mass per unit volume.[1)]

1) Cascade filtration systems can be limited by the minimum pore size of the sieves used or volume of water sampled (see Table 1). The use of sedimentation boxes or continuous flow centrifuges have recently been proposed as potential alternatives of volume-reduced sampling of microplastics from large volumes of water, with initial studies showing the capability to capture the smallest microplastics (<30 µm) from river waters. While these techniques are currently outside the scope of this document, References [12] to [16] can be consulted for further information of these recent proposals. General information on sedimentation boxes and continuous flow centrifuges can be found in ISO 5667-17 (not related to microplastic sampling).

For sampling surface waters (up to about 100 cm below surface), the use of manta trawls, plankton and neuston nets in dynamic and stationary methods are described. Other types of nets are also used for subsurface water sampling. The sampling date, time, location, mesh pore size and water passing through the nets over the sampling time should be recorded. Additionally, for dynamic sampling, recording of log speeds of boats or vessels, tow duration, area covered, wind velocities and significant wave heights is recommended.[6]

4.2 Selecting the most appropriate sampling method

Sampling strategies can differ depending on the targeted environmental compartment that needs to be monitored and depending on the target question (see Table 1).

Grab sampling can be used not only to collect microplastics from the water surface but also from the water column, by using a container or a submersible water pump. Grab sampling is applicable to all categories of water samples (domestic water, freshwater, seawater, treated wastewater and untreated wastewater) and is preferred over net tows for smaller microplastics, which typically cannot be collected by the larger mesh sizes. However, given the more limited volume that can practically be collected and stored, the detection limit for grab samples can be higher, particularly when sampling water bodies such as rivers, lakes or sea water. Replicates or combined spot samples can compensate for this deficiency.

Column waters can also be sampled by pumping, followed by a set of sieves to isolate microplastics of different size ranges (cascade filtration). This method is applicable to domestic water, freshwater, treated wastewater and untreated wastewater. Cascade filtration systems are also limited by the minimum pore size of the filters or sieves used. While mesh sizes can be down to 1 µm, samples such as untreated wastewater require larger pore sizes to avoid clogging. Cascade filtration systems allow more significant capture of smaller microplastics when compared to nets.

For sampling the water surface and water column in rivers, lakes and sea, the most common method is the net tow, using neuston or plankton nets or manta trawls.[2][6][17]-[19] Manta trawls and neuston nets are mainly used for microplastics sampling in the ocean, while plankton nets are used for sampling in rivers. Net mesh sizes vary between 50 µm and 500 µm, with 330 µm the most commonly used.[2][6][18][20] Plankton nets have the smallest mesh pore sizes (between 50 µm to 100 µm) and they usually need to be towed at lower speeds in order to reduce clogging.[9] Microplastics smaller than the minimum mesh size are not collected, resulting in a potential underestimation of their total abundance in the sampled water. It is important to note that the frequency of occurrence of dispersed microplastics is likely to vary inversely with size (i.e. there will be fewer larger items)[2][21] and needs to be taken into account when planning the sampling method and sampling strategy.

In net tow, the collection of microplastics can be performed by a dynamic or stationary sampling method.

In dynamic sampling, trawls are towed by a boat with a rope, keeping the nets outside the waves from the vessel to prevent disturbance or dispersion of the particles to be collected. It is recommended to position the net on the side of the boat, by a suitable pole installed.[6]-[9]

For streams, creeks and smaller rivers with variable water regime not entirely navigable, stationary sampling is recommended. For this, the floating nets need to be fixed on the banks. The nets filter water using the stream current with their mouths skimming the surface, with a weight used to maintain a continuous and consistent submersion depth. The nets are collocated in the opposite direction of the water flow.[9] Plankton nets are more commonly used for stationary sampling in rivers.

Table 1 — Comparison of different methodologies for sampling microplastics in water

Method	Overview and application	Advantages	Limitations
Grab sampling	Discrete sample collection with non-plastic containers (or made of materials which are less present in the environment or not part of the measurement concept). Applicable to domestic water, freshwater, treated wastewater and untreated wastewater. Applicable for surface waters and water column.	Relatively quick and straightforward method. Reduced risk of contamination. No limitation of microplastic size. It can sample surface and water column.	Sampling smaller volumes of water. It requires transportation of large or multiple containers to the lab. Less representative. If sampling in deeper waters such as sea, middle of a lake or river, a boat or ship can be needed.
Cascade filtration	Volume-reduced sampling. Applicable to domestic water, freshwater, seawater, treated wastewater and untreated wastewater. Applicable for surface waters and water column. Cascade filters using non-plastic sieves (or made of materials which are less present in the environment or not part of the measurement concept).	Mesh sizes can vary between 1 µm to 5 mm, allowing for a more efficient and wider range of particle sizes when compared to manta or neuston nets. Cascade filtering using finer filters can collect smaller-sized particles more efficiently than the use of manta, plankton or neuston nets. It allows sampling large volumes of water. It permits size fractionation directly in the field. It can sample surface and water column.	The minimum mesh pore size and mesh selectivity determine the minimum particle size to be captured. It requires electric energy (unless sampling from a pressurized system or if a manual or hand-operated pump is used). It involves more equipment than grab sampling. Filters can be clogged, particularly when using smaller mesh sizes or when sampling water with high-suspended solid contents. Higher risk of sample contamination from apparatus and due to manipulation. If sampling in deeper waters such as sea, middle of a lake or river, a boat or ship can be needed.
Net sampling: Dynamic sampling	Volume-reduced sampling. Widely used for sampling water in navigable rivers, lakes and sea. Applicable for surface waters and water column. Meshes are towed by a boat with a rope. The position of the net should be away from the wake from the vessel. Ideally, mesh materials should be made of compounds which are less present in environment or not part of the measurement concept. The mesh pore size determines the minimum particle size to be captured. The mesh opening must be reported. For multiple depth collection at the water subsurface, nets can be tethered at predetermined increments. Multiple opening or closing nets can also be used for water column sampling (e.g. vertical multiple-opening plankton sampler, VMPS or multiple opening and closing net with environmental sensing system, MOCNESS).	It allows sampling large volumes of water and covers large areas.	Most available nets have mesh sizes between 500 and 50 µm, with 330 µm being the most common one. This limits the minimum microplastic size that can be captured. Clogging problems. It requires a boat or ship. Risk of sample loss or contamination due to manipulation and transfer from the nets.
Net sampling: Stationary sampling	Volume-reduced sampling. Widely used for sampling surface waters. Recommended for streams, creeks and smaller rivers with variable water regime not entirely navigable. The nets are collocated in the opposite direction of the water flow, with a weight used to maintain a continuous and consistent submersion depth, and fixed on the banks. Ideally, mesh materials should be made of compounds which are less present in environment or not part of the measurement concept, The mesh pore size determines the minimum particle size to be captured. The mesh opening must be reported.	It allows sampling large volumes of water and sampling over longer periods of time.	Most available nets have mesh sizes between 500 and 50 µm, with 330 µm being the most common one. This can limit the minimum microplastic size that can be captured, with potential underestimation of the real quantity of microplastics in the sampled water. Clogging problems. Anchoring the nets to the riverbed can be difficult. Mostly for surface sampling (up to about 100 cm below surface). Not usually applicable for deep water column. Risk of sample loss or contamination due to manipulation and transfer from the nets.