



SLOVENSKI STANDARD
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Water quality - Sampling - Part 23: Determination of priority pollutants in surface water using passive sampling

Water quality - Sampling - Part 23: Determination of priority pollutants in surface water using passive sampling

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Qualité de l'eau - Échantillonnage - Partie 23: Détermination des polluants prioritaires dans les eaux de surface en utilisant un échantillonnage passif

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13.060.45 Preiskava vode na splošno Examination of water in general

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Water quality — Sampling —

Part 23:

Determination of priority pollutants in surface water using passive sampling

Qualité de l'eau — Échantillonnage —

Partie 23: Détermination des polluants prioritaires dans les eaux de surface en utilisant un échantillonnage passif

ICS 13.060.45

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ISO/CEN PARALLEL PROCESSING

This draft has been developed within the International Organization for Standardization (ISO), and processed under the **ISO-lead** mode of collaboration as defined in the Vienna Agreement.

This draft is hereby submitted to the ISO member bodies and to the CEN member bodies for a parallel five-month enquiry.

Should this draft be accepted, a final draft, established on the basis of comments received, will be submitted to a parallel two-month approval vote in ISO and formal vote in CEN.

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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.

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The main task of technical committees is to prepare International Standards. Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

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ISO 5667-23 was prepared by Technical Committee ISO/TC 147, *Water quality* - Subcommittee SC 6 *Sampling (general methods)* and by Technical Committee CEN/TC 230, *Water analysis* in collaboration.

ISO 5667 consists of the following parts, under the general title *Water quality — Sampling*:

- Part 1: *Guidance on the design of sampling programmes*
- Part 3: *Guidance on the preservation and handling of water samples*
- Part 4: *Guidance on sampling from lakes natural and man-made*
- Part 5: *Guidance on sampling of drinking water and water used for food and beverage processing*
- Part 6: *Guidance on sampling of rivers and streams*
- Part 7: *Guidance on sampling of water and steam in boiler plants*
- Part 8: *Guidance on sampling of wet deposition*
- Part 9: *Guidance of sampling from marine waters*
- Part 10: *Guidance of sampling of waste waters*
- Part 11: *Guidance of sampling of groundwaters¹⁾*
- Part 12: *Guidance on sampling of bottom sediments*
- Part 13: *Guidance on sampling of sludges from sewage and water-treatment works¹⁾*
- Part 14: *Guidance on quality assurance of environmental water sampling and handling*
- Part 15: *Guidance on preservation and handling of sludge and sediment samples¹⁾*
- Part 16: *Guidance on biotesting of samples*
- Part 17: *Guidance on sampling of suspended sediments¹⁾*

1) Under revision

- Part 18: *Guidance on sampling of groundwater at contaminated sites*
- Part 19: *Guidance on sampling in marine areas*
- Part 20: *Guidance on the use of sampling data for decision making – Compliance with limits and classification systems²⁾*
- Part 21: *Guidance on sampling of drinking water distributed by non-continuous, non-conventional means²⁾*
- Part 22: *Guidance on design and installation of groundwater sample points²⁾*

Introduction

Passive sampling devices can be used for monitoring concentrations of a wide range of analytes (including metals, and inorganic anions, polar organic compounds such as polar pesticides and pharmaceutical compounds, and non-polar organic compounds such as non-polar pesticides, and industrial chemicals such as polyaromatic hydrocarbons and polychlorinated biphenyls) in aquatic environments. For more information on individual type of passive sampling devices, refer to the bibliography section - Reviews of passive sampling devices. Pollutant levels in surface water have traditionally been monitored by spot sampling (also known as bottle or grab sampling). Such sampling gives a snapshot of pollutant levels at a particular time. Pollutant levels in surface water have a tendency to fluctuate over time and so it may be more desirable to monitor pollutants over an extended time period in order to obtain a more representative measure of the chemical quality of a water body. This may be achieved by repeated spot sampling, continuous monitoring, biomonitoring or passive sampling.

Passive sampling involves the deployment of a passive sampling device that uses a diffusion gradient to collect pollutants over a period of days to weeks. This process is followed by extraction and analysis of the pollutants in a laboratory.

Passive sampling devices can be used in kinetic or equilibrium modes. In equilibrium mode the passive sampling devices provide a measure of the concentration at the time of retrieval from the environment. In kinetic mode the passive sampling device samples in an integrative way, and provides a measure of the time weighted average concentration of a pollutant in the water over the exposure period. Passive sampling devices operate as integrative samplers between the time of deployment and an exposure time of up to the time to half maximum accumulation in the receiving phase.

Diffusion into the receiving phase is driven by the free dissolved fraction of pollutant, and not that bound to particulate matter and to large molecular mass organic compounds (e.g. humic and fulvic acids), and so this technique provides a measure of the time-weighted average concentration of the free dissolved fraction of pollutant to which the passive sampling device was exposed. For some passive sampling devices for metals the concentration of analyte measured includes both the free dissolved fraction and that fraction of the analyte bound to large molecular mass organic compounds that can dissociate in the permeation layer. The fraction sampled by a passive sampling device can be different from that sampled in a spot (bottle) sample. In a spot sample the fraction of pollutant measured is determined by a combination of factors such as the proportion of pollutant bound to particulate matter and to large organic compounds, and the treatment (e.g. filtration at 0.45 µm, or ultrafiltration) applied prior to analysis. Passive sampling devices used in surface water typically consist of a receiving phase (typically a solvent or sorbent) that has a high affinity for pollutants of interest and so collects them. This receiving phase may be retained behind or surrounded by a membrane through which the target analytes can permeate. A schematic representation of such a passive sampling device is shown in Figure 1. In its simplest form a passive sampling device is comprised solely of a naked membrane, or fibre, or bulk sorbent which acts as a receiving phase, and in such passive sampling devices where a permeation membrane is absent the boundary layer of water may act as the permeation layer. Different combinations of

2) In preparation

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permeation layer and receiving phase are used for the different classes of pollutant (non-polar organic, polar organic, and inorganic, and passive sampling devices are designed for use with one of these main classes of pollutant.

Passive sampling devices can be used in a number of modes including qualitative or semi-quantitative which can be applied in for instance the detection of sources of pollution. When appropriate calibration data are available, passive sampling devices can also be used quantitatively for measuring the concentration of the free dissolved fraction of a pollutant.

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Water quality — Sampling —

Part 23:

Determination of priority pollutants in surface water using passive sampling

1 Scope

This international standard describes procedures for the determination of time-weighted average concentrations of the free dissolved fraction of pollutants in surface water by passive sampling, followed by analysis.

2 Normative references

The following referenced international standards are indispensable for the application of this international standard. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

EN ISO 14644-1, *Cleanrooms and associated controlled environments – Part 1: Classification of air cleanliness*

EN ISO 13530, *Water quality – Guide to analytical quality control for water analysis*

ISO 6107-2, *Water quality – Glossary – Part 2: Additional terms relating to types of water, and treatment and storage of water and waste water, and terms used in sampling and analysis of water*

ISO 5667-4, *Water quality – Sampling – Part 4: Guidance on sampling from lakes, natural and man made*

ISO 5667-14, *Water quality – Sampling – Part 14: Guidance on quality assurance of environmental water-sampling and handling*

ISO 5667-1, *Water quality – Sampling – Part 1: Guidance on the design of sampling programmes and sampling techniques*

EN ISO 5667-3:2003, *Water quality – Sampling – Part 3: Guidance on the preservation and handling of water samples*

ISO 5667-6, *Water quality – Sampling – Part 6: Guidance on sampling of rivers and streams*

ISO 5667-9, *Water quality – Sampling – Part 9: Guidance on sampling from marine water*

3 Terms and definitions

For the purposes of this international standard, the terms and definitions given in ISO 6107-2 and the following apply.

3.1

analytical recovery standard

compound added to passive sampling device receiving phase prior to analysis and whose recovery levels during analysis are used to provide information about recovery efficiency

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3.2 field control
quality control passive sampling device to record any chemical accumulated in passive sampling devices during manufacture, assembly, storage, transportation, deployment, retrieval and subsequent analysis

3.3 passive sampling
sampling technique based on the free diffusion of an analyte from the sampled medium to a receiving phase in the passive sampling device as a result of a difference between chemical potentials of the analyte in the two media: the net flow of analyte from one medium to the other continues until equilibrium is established in the system, or until the sampling period is terminated without provision of energy from an external source

3.4 integrative phase of passive sampling
phase of sampling in the early period of exposure during which the rate of uptake of an analyte into the receiving phase of the passive sampling device is approximately linear, and during which the passive sampling device can be used to measure the time weighted average concentration of an analyte in the environment

3.5 performance reference compound (PRC)
compound that has moderate to high fugacity from the passive sampling device, which does not interfere with the sampling and analytical processes and which is added to the device receiving phase prior to deployment

NOTE 1 The off-loading (elimination) rates of the PRCs are used to provide information about in situ uptake kinetics of pollutants.

NOTE 2 Currently there are no PRCs available for metals passive sampling devices or for polar organics passive sampling devices.

3.6 reagent control
aliquot of reagent used in treatment of passive sampling devices which is analysed following deployment in order to diagnose any contamination from the reagents used

3.7 recovery spike
quality control passive sampling device, pre-spiked with known mass of analytical recovery standard, used to determine the recovery level of pollutant from passive sampling devices following deployment

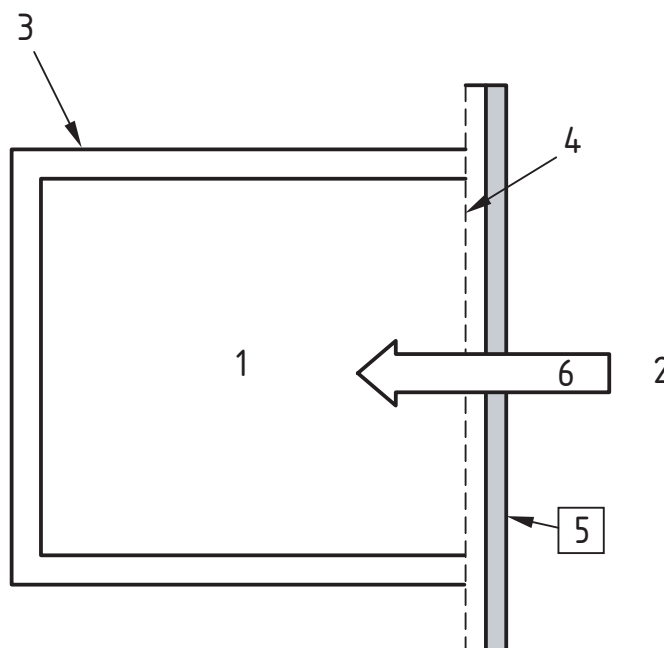
3.8 passive sampling device class
class of pollutant which a passive sampling device is designed to accumulate

NOTE Passive sampling device classes include the following:

- polar organic compounds;
- non-polar organic compounds; and
- inorganic compounds, including metals.

4 Principle

The general features of a passive sampling device are illustrated in Figure 1, and the structures of the types of passive sampling device for the different classes of pollutants (polar organic, non-polar organic, and inorganic (including metals)) are summarised in Table A.1, and the commonly used procedures used to calibrate the various designs of passive sampling device are summarised in Table A.2.



- | | | | |
|---|-----------------|---|------------------------|
| 1 | Receiving phase | 4 | Permeation membrane |
| 2 | Water | 5 | Water boundary layer |
| 3 | Housing | 6 | Diffusion of pollutant |

NOTE 1 The permeation membrane and water boundary layer constitute the permeation layer

NOTE 2 In some passive sampling device designs, the housing is replaced by a membrane that completely encloses the receiving phase. In some passive sampling devices (for instance polyethylene strips or silicone rubber sheet) the receiving phase is not held in a housing but is deployed naked on a holding frame, and in these passive sampling devices there is no permeation membrane, but the water boundary layer acts as a permeation layer. For more information on individual types of passive sampling devices, refer to the Bibliography (Section Passive Samplers).

Figure 1 – Schematic representation of a passive sampling device

Pollutants accumulate in the receiving phase of a passive sampling device over a measured period of time of exposure to surface water. The pollutants are extracted from the passive sampling device in the laboratory and the amount of each pollutant accumulated is determined by chemical analysis.

Uptake of a pollutant into the receiving phase of a sampling device follows a first order approach to a maximum (See Figure 2.).

$$M_{(t)} = M_{max}(1 - e^{-k_e t}) \tag{1}$$

$M_{(t)}$ the mass accumulated after an exposure time (t); M_{max} is the maximum mass accumulated, and k_e is a first order macro rate constant (the overall exchange rate constant) that depends on the properties of the sampler and the pollutant.

Uptake is approximately linear with time throughout the exposure period between time of deployment ($t=0$) and the time to half maximum accumulation in the receiving phase ($t = t_{0.5}$). Under these conditions, and providing that the mass transfer of pollutant varies linearly with concentration in the water, then the passive sampling device operates in integrative mode and can be used to measure the time weighted average concentration of pollutant to which the passive sampling device was exposed.

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The time to half maximum accumulation in the receiving phase ($t_{0.5}$) is calculated:

$$t_{0.5} = \frac{\ln 2}{k_e} \quad (2)$$

At longer exposure times as M_{max} is approached the passive sampling device operates in equilibrium mode, and provides a measure of the concentration only at the time of retrieval of the passive sampling device.

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