
Water quality — Sampling —

Part 23:

**Guidance on passive sampling in surface
waters**

Qualité de l'eau — Échantillonnage —

*Partie 23: Lignes directrices pour l'échantillonnage passif dans les eaux
de surface*



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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.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

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.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

ISO 5667-23 was prepared by Technical Committee ISO/TC 147, *Water quality*, Subcommittee 6, *Sampling (general methods)*.

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

- *Part 1: Guidance on the design of sampling programmes and sampling techniques*
- *Part 3: 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 from treatment works and piped distribution systems*
- *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 the sampling of wet deposition*
- *Part 9: Guidance on sampling from marine waters*
- *Part 10: Guidance on sampling of waste waters*
- *Part 11: Guidance on sampling of groundwaters*
- *Part 12: Guidance on sampling of bottom sediments*
- *Part 13: Guidance on sampling of sludges*
- *Part 14: Guidance on quality assurance of environmental water sampling and handling*
- *Part 15: Guidance on the preservation and handling of sludge and sediment samples*
- *Part 16: Guidance on biotesting of samples*

- *Part 17: Guidance on sampling of bulk suspended solids*
- *Part 19: Guidance on sampling of marine sediments*
- *Part 20: Guidance on the use of sampling data for decision making — Compliance with thresholds and classification systems*
- *Part 21: Guidance on sampling of drinking water distributed by tankers or means other than distribution pipes*
- *Part 22: Guidance on the design and installation of groundwater monitoring points*
- *Part 23: Guidance on passive sampling in surface waters*

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Introduction

Passive sampling devices can be used for monitoring concentrations of a wide range of analytes, including metals, inorganic anions, polar organic compounds (e.g. polar pesticides and pharmaceutical compounds), non-polar organic compounds (e.g. non-polar pesticides), and industrial chemicals (e.g. polyaromatic hydrocarbons and polychlorinated biphenyls) in aquatic environments.

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 period in order to obtain a more representative measure of the chemical quality of a water body. This can 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 device reaches equilibrium with the sampled medium, and provides a measure of the concentration at the time of retrieval from the environment. In the 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. Where uptake into the receiving phase is under membrane control, then passive sampling devices operate as integrative samplers between the time of deployment and an exposure period of up to the time to half maximum accumulation in the receiving phase. Membrane control means that the transport resistance of the membrane is larger than that of the water boundary layer. In stagnant water, uptake is generally controlled by the water boundary layer. Under highly turbulent conditions, uptake is membrane controlled. Where uptake is controlled by the water boundary layer, then the passive samplers behave in a manner similar to those where uptake is under membrane control, but the sampling rate depends on flow conditions. Where flow conditions vary over time, uptake can be under water boundary control when turbulence is low, but change to membrane control when turbulence increases.

Diffusion into the receiving phase is driven by the free dissolved concentration of pollutant, and not that bound to particulate matter and to large molecular mass organic compounds (e.g. humic and fulvic acids). This technique provides a measure of the time-weighted average concentration of the free dissolved fraction of pollutant to which the passive sampling device has been 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 small molecular mass inorganic and organic compounds that can diffuse into and dissociate in the permeation layer. Pollutant bound to large molecular mass compounds diffuses only very slowly into the diffusion layer. The concentration measured by a passive sampling device can be different from that measured 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, polymer or sorbent) that has a high affinity for pollutants of interest and so collects them. This receiving phase can 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, fibre or bulk sorbent which acts as a receiving phase. In such passive sampling devices, the polymer acts as both receiving phase and permeation membrane. The polymers used in these passive sampling devices usually have a high permeation, and uptake is controlled by the water boundary layer. Uptake comes under membrane control only at very high flow rates. Different combinations of permeation layer and receiving phase are used for the different classes of pollutant (non-polar organic, polar organic, and inorganic). 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 the detection of sources of pollution, for example. When appropriate calibration data are available, passive sampling devices can also be used quantitatively for measuring the concentration of the free dissolved species of a pollutant.

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Water quality — Sampling —

Part 23:

Guidance on passive sampling in surface waters

1 Scope

This part of ISO 5667 specifies procedures for the determination of time-weighted average concentrations and equilibrium concentrations of the free dissolved fraction of organic and organometallic compounds and inorganic substances, including metals, in surface water by passive sampling, followed by analysis.

2 Normative references

The following referenced documents are indispensable for the application 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 5667-1, *Water quality — Sampling — Part 1: Guidance on the design of sampling programmes and sampling techniques*

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

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

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 waters*

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

ISO 6107-2, *Water quality — Vocabulary — Part 2*

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

ISO 14644-1, *Cleanrooms and associated controlled environments — Part 1: Classification of air cleanliness by particle concentration*

3 Terms and definitions

For the purposes of this document, 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

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 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

3.4 integrative phase of passive sampling
phase of sampling 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 uptake of the passive sampling device is proportional to the time-weighted average concentration of an analyte in the environment

**3.5 performance reference compound
PRC**
compound that is added to the sampler prior to exposure and has such an affinity to the sampler that it dissipates from the sampler during exposure, and that does not interfere with the sampling and analytical processes

NOTE 1 The offloading (elimination) rates of PRCs are used to provide information about *in situ* uptake kinetics of pollutants.

NOTE 2 Currently PRCs are available neither for passive sampling devices for metals nor for polar organic compounds.

3.6 reagent blank
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 passive sampling device based on the class of pollutant which a passive sampling device is designed to accumulate

NOTE Passive sampling device classes include:

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

3.9**membrane control**

where diffusion through the membrane of the passive sampler dominates the overall mass transfer and resistance to mass transfer of analytes from the bulk water phase into the receiving phase

3.10**water boundary layer**

viscous sub-layer of water adjacent to a surface, caused by complex hydrodynamic interactions of a surface with water, that causes resistance to diffusion from the bulk phase of water to the receiving phase, and that reduces in thickness with increasing turbulence in the bulk phase of water

3.11**sampling rate**
 R_s

apparent volume of water cleared of analyte per time, calculated as the product of the overall mass transfer coefficient and the area of the receiving phase exposed to the external environment

NOTE Sampling rate is expressed in litres per day.

3.12**deployment device**

structure to which passive sampling devices can be attached, or in which they can be contained during deployment, and that is suitable for ensuring that the passive sampling devices are retained in position at the deployment site throughout the deployment period

EXAMPLES A metal mesh, a pole or a cage, with mooring lines, buoys and anchors where necessary.

4 Principle

The general features of a passive sampling device are illustrated in Figure 1. 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 summarized in Table A.1. The procedures commonly used to calibrate the various designs of passive sampling device are summarized in Table A.2.

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). The mass accumulated after an exposure time, t , m_t , is given by Equation (1):

$$m_t = m_{\max} [1 - \exp(-k_e t)] \quad (1)$$

where

m_{\max} is the maximum mass accumulated;

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 (see Note).

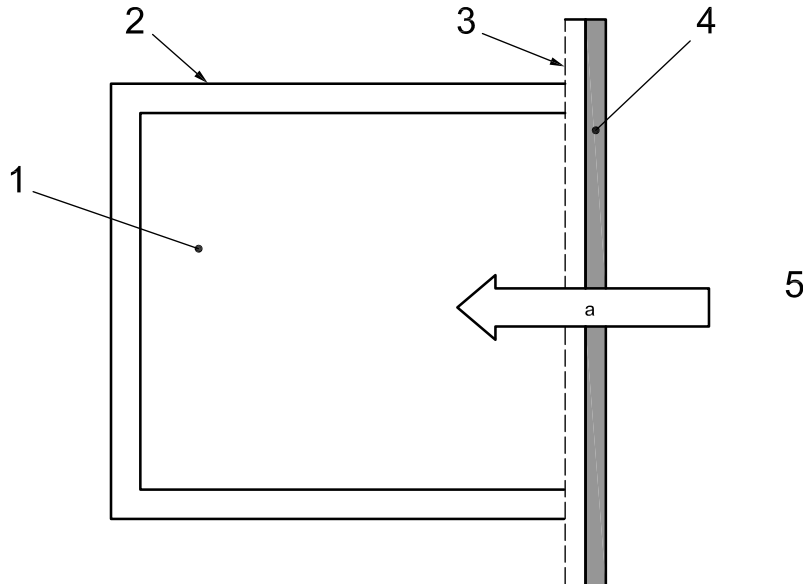
NOTE The parameters that make up the macro rate constant, k_e , are discussed in Clause 13.

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 is linearly related to the 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.

The time to half maximum accumulation in the receiving phase, $t_{0,5}$, is calculated using Equation (2):

$$t_{0,5} = \frac{\ln 2}{k_e} \tag{2}$$

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.



- Key**
- | | |
|-----------------------|---------------------------|
| 1 receiving phase | 4 water boundary layer |
| 2 housing | 5 water |
| 3 permeation membrane | a 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 (e.g. polyethylene strips or silicone rubber sheet) the receiving phase is not held in a housing but is deployed naked on a holding frame. 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, see References [1] to [8].

Figure 1 — Schematic representation of a passive sampling device

5 Handling passive sampling devices

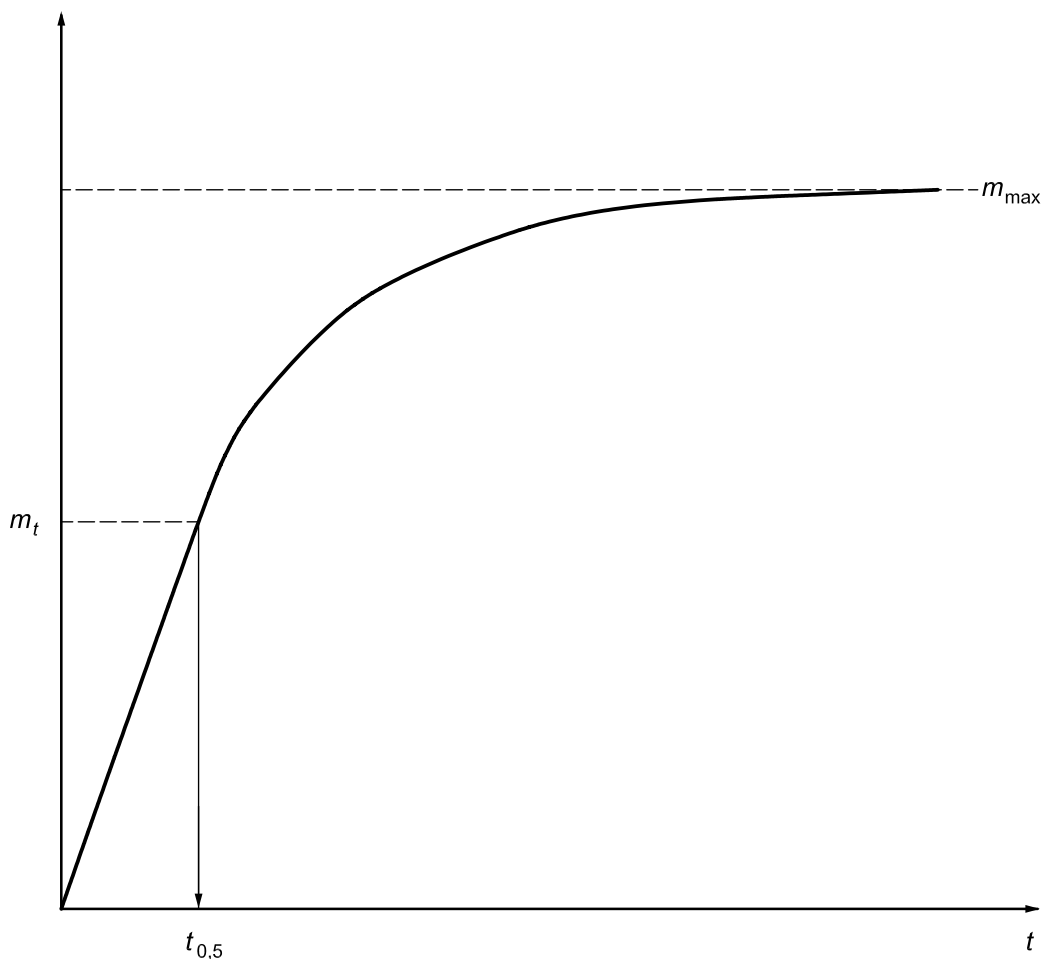
5.1 General

5.1.1 Ensure safety precautions are in place and adhered to for handling all chemicals.

5.1.2 It is essential that passive sampling devices be kept isolated from potential sources of contamination at all times except when being exposed at the sampling site. Ensure that the passive sampling devices are stored and transported in gas-tight containers, of inert materials relevant to the pollutants of interest.

5.1.3 Avoid physical contact with the receiving phase or membrane of the passive sampling devices, since this may affect the results. Where handling is unavoidable, use powder-free vinyl or latex gloves. Do not re-use gloves.

5.1.4 For some passive sampling devices, it may be necessary to avoid or at least minimize exposure to airborne contaminants during the handling, manipulation and deployment of passive sampling devices, and the subsequent analysis.

**Key** m_t mass accumulated in sampler receiving phase t exposure time**Figure 2 — Profile of uptake of a pollutant into a passive sampling device**

The use of a clean room classified in accordance with ISO 14644-1 or a laminar flow hood is recommended when preparing some passive sampling devices.

5.1.5 Passive sampling devices and resultant extracts should not be stored in proximity to other chemicals, particularly volatile chemicals.

5.1.6 Use suitable pipette tips that are clean and free from contamination for the addition of reagents to extracts.

5.2 Passive sampling devices for organic compounds

5.2.1 Minimize contact of passive sampling devices for organic compounds with plastic materials.

5.2.2 Wash, using an organic solvent such as acetone, all equipment that comes into contact with passive sampling devices during preparation prior to deployment, storage, transport, and preparation for analysis.

5.3 Passive sampling devices for metals

5.3.1 Acid wash all equipment that comes in contact with the extract obtained from the passive sampling device after deployment, other than the passive sampling devices, in accordance with ISO 5667-3.

5.3.2 Use a grade of acid (containing less than 5 µg/kg of any individual heavy metal) that is appropriate for trace metal analysis for addition to samples or for digestion.

6 Estimation of appropriate field deployment time

Where the aim of passive sampling is to estimate the time-weighted average concentration of a pollutant in surface water, the exposure should not extend beyond the linear uptake phase (see Clause 4). Under these conditions, the mass of pollutant collected in the receiving phase is limited by the sampling rate and exposure time. The mass collected in the receiving phase should be above the level of quantification of the analytical method. The time necessary to achieve this depends on the concentration of the pollutant in the water and the sampling rate of the passive sampling device. Where the concentration in the water is low and the sampling rate is low, it may not be possible to estimate a time-weighted average concentration. A passive sampling device where the sampling rate is commensurate with the expected range of concentration of the pollutant should be used.

When equilibrium is approached, then the mass of pollutant collected in the receiving phase is determined by the sorption capacity (the product of the volume of the sampler and the partition coefficient between the receiving phase and the environmental water) of the receiving phase. Under these conditions, information on time-weighted average concentrations is limited.

Where available, the exposure time advised by the manufacturer should be used. For samplers that are not commercially produced, use calibration data provided in peer-reviewed publications.

7 Passive sampling device preparation and assembly

7.1 Passive sampling device preparation

For samplers, e.g. strips or sheets of polymeric material including low density polyethylene and silicone rubber, that are not supplied as ready to use for passive sampling, it is necessary to clean the passive samplers to remove oligomers and contaminants prior to use. This can be achieved by thermal desorption and solvent extraction, e.g. Soxhlet extraction or repeated washing in a solvent like ethyl acetate, during a period of 1 week). Following this extraction stage, any residual extraction solvent should be removed by at least two washes of methanol over a period of 24 h. After cleaning, such polymeric samplers can be stored in methanol for up to 6 months.

Where it is possible to use performance reference compounds (PRCs), select suitable compounds for this purpose according to the compounds to be sampled (see Note). It is not feasible to use a labelled analogue of each compound to be sampled. Select compounds to cover the range of octanol/water partition coefficients of the analytes to be sampled in order to ensure offloading in the range 20 % to 50 % of the individual PRCs spiked into the receiving phase. It is advisable to use PRCs that cover the desired range of log octanol/water partition coefficient in steps of approximately 0,2. Where a labelled analogue of an analyte is not available, it is advisable to use the overall offloading rate constant of a PRC with a slightly lower log octanol/water partition coefficient than that of the analyte in the calculation of the concentration of the analyte in the water.

Prepare PRC solutions for each class of passive sampling device. Select the amount of PRC to be spiked. This should be sufficient to ensure that the residue falls above the limit of quantification of the analytical method. Avoid using larger quantities than necessary, since the materials offload into the environment. Use the solution of PRCs to spike the receiving phase of selected passive sampling devices prior to assembly. Use pure materials and state the use by date for the spiked passive sampling devices. Ensure that the receiving phase is homogeneously spiked.

In some cases, spiking is carried out during manufacture. For passive samplers where the receiving phase is a sorption phase, spiking can be achieved by addition of a solution of the PRCs in a compatible volatile solvent. For passive samplers, e.g. polymeric strips or sheets, that are not supplied ready for use, spiking is achieved (after the cleaning stage) by soaking in a solution of PRCs in methanol and water mixtures. Detailed advice on individual samplers and applications is available in peer-reviewed publications.

NOTE Some commercially available passive samplers are supplied with PRCs already spiked into the receiving phase.

7.2 Passive sampling device assembly

7.2.1 Passive sampling devices that need to be assembled by the user should be assembled in an environmentally controlled room equipped to remove atmospheric contaminants.

7.2.2 Label each passive sampling device in accordance with ISO 5667-3.

NOTE The addition of a suitable label for each passive sampling device by the manufacturer aids passive sampling device identification during deployment and during recovery, and after recovery.

7.3 Passive sampling device storage

It is essential that passive sampling devices be kept isolated from potential sources of contamination during storage. Store prepared passive sampling devices in vapour-tight containers at controlled temperatures. Avoid storing passive sampling devices in proximity to chemicals.

The storage temperature should be selected in accordance with the manufacturer's instructions, where available. Where such instructions are not available, store samplers at 4 °C, and avoid freezing samplers that contain traces of water.

8 Quality assurance

8.1 General

Implement quality assurance measures throughout the sampling and handling processes in accordance with ISO 5667-14. Figure 3 illustrates how the quality assurance steps fit into the sequence of processes involved in using passive sampling devices.

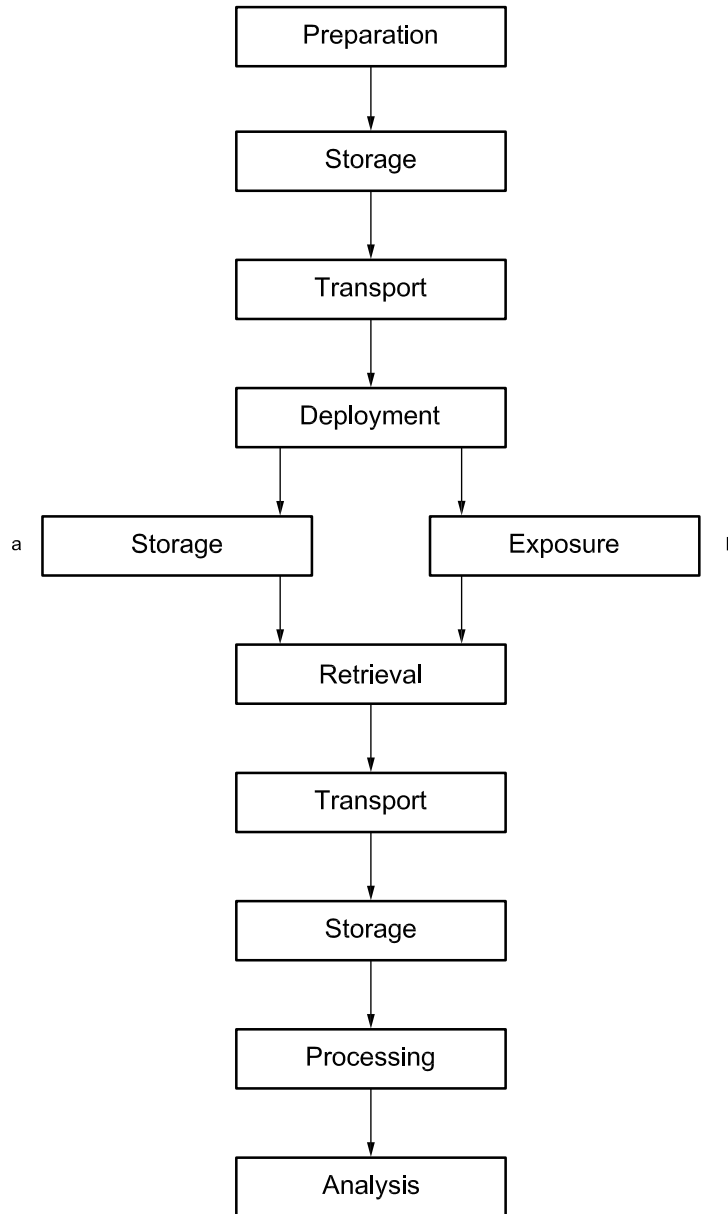
Compare the results of analysis of passive sampling devices deployed together (as specified in 8.2) and of passive sampling devices with passive sampling device controls (as specified in 8.3), in order to calculate uncertainty of the sampling (see Clause 13). Refer to the guidance for analytical quality control in ISO/TS 13530.

8.2 Replicate passive sampling devices in field deployment

The number of replicate passive sampling devices deployed at each site is determined by the design of the sampling campaign, and the precision needed for the purposes of the campaign. If information is needed on temporal changes over a long period, then passive sampling devices can be retrieved at a range of elapsed times after deployment.

8.3 Replicate quality control passive sampling devices

Prepare quality control passive sampling devices at the same time and in the same way as those to be deployed in the field. Use a minimum of one per sampling site for each class (polar organic compounds, non-polar organic compounds and inorganic compounds including metals) of passive sampling device to be used.



NOTE Field controls (a) are stored during the period of field exposure of deployed passive sampling devices (b).

Figure 3 — Scheme showing sequence of processes involved in using passive sampling devices

8.4 Passive sampling device controls

For each passive sampling device set (group of passive sampling devices deployed together), prepare passive sampling device controls in accordance with Table 1. The number and type of controls are dependent upon the required level of confidence, but a minimum of one per sampling site should be used, or two where there is only one sampling site in a campaign.

The average mass of the PRC spike and the associated precision are estimated by using all of the field control passive sampling devices from each batch of passive sampling devices deployed within a field campaign.

For monitoring the time-weighted average concentration of pollutant near the limit of detection, it is possible to combine extracts from a number of passive sampling devices. Under these circumstances, it is necessary to increase the number of control passive sampling devices *pro rata*.

Table 1 — Passive sampling device control requirements

Control type	Number required	Treatment of controls
Field control	At least one per sampling site, or two where there is only one sampling site	<p>Separate the field controls from the passive sampling devices manufactured or delivered together.</p> <p>Transport field controls between the sampling site and laboratory with the set of passive sampling devices.</p> <p>Expose field controls to the air at the sampling site during deployment and retrieval of the passive sampling device set, but only during manipulation. Handle in the same way as the set of passive sampling devices up to the start of deployment, and from the start of recovery from the field.</p> <p>Process and analyse field controls concurrently with and identically to the passive sampling device set.</p>
Recovery spike	At least three per batch of passive sampling devices or for each field campaign if a single batch of samplers is used	<p>Prior to processing of a passive sampling device, fortify recovery spike passive sampling devices with a target compound mixture.</p> <p>Process and analyse recovery spike controls concurrently with and identically to passive sampling devices of the same class in the passive sampling device set.</p>

9 Selection of sampling site and safety precautions

9.1 Selection of sampling site

Select a sampling site in accordance with ISO 5667-1 and ISO 5667-4 for lakes or ISO 5667-6 for rivers and streams or ISO 5667-9 for marine waters.

Before deployment and prior to retrieval of passive sampling devices, carefully inspect sampling site for the following:

- a) sources of vapour-phase contaminants, including engine fumes, oils, tars, gasoline, diesel fuel, paints, solvents, cigarette smoke and asphalt pavement, if passive sampling devices are to be used for organic compounds;
- b) sources of metallic contamination, if passive sampling devices are to be used for metals;
- c) oily films or biofilms on the surface of the water;
- d) it is essential that the passive sampling device be deployed at a point below which water levels do not drop so that there is adequate water depth to ensure that the passive sampling device is kept submerged throughout deployment under all conditions.

Record any findings for the site.

NOTE Some streams could become dry during periods without rain; deployment of samplers in such cases should be in pools rather than riffles. In tidal waters, passive sampling devices should be deployed at a suitable distance beyond the spring tide low water mark.

9.2 Appropriate precautions against accidents

The enormously wide range of conditions encountered in sampling surface waters can subject sampling personnel to a variety of risks.

Sampling personnel should be informed of the necessary precautions to be taken during sampling operations.

ISO 5667-1 specifies certain safety precautions, including sampling from boats and from ice-covered waters.

ISO 5667-6 specifies safety precautions to be considered when sampling from river banks.

IMPORTANT — Take precautions against accidents, and provide appropriate safety training.

Attention is drawn to the requirements of health and safety regulations.

10 Passive sampling device deployment and retrieval

NOTE A schematic outline of the sequence of processes involved in using passive sampling devices and field controls is provided in Figure 3.

10.1 Materials and apparatus

A check list of materials and apparatus to be taken to the field for use in the deployment of passive sampling devices is provided in Annex B.

10.2 Transport

10.2.1 Follow the storage and handling instructions supplied by the manufacturer or, for samplers that are not commercially produced, use calibration data provided in peer-reviewed publications by competent laboratories.

10.2.2 Use appropriate containers (B.5) to ensure that individual passive sampling devices remain isolated from the environment, from potential sources of contamination, and from each other during storage and transport to the deployment site and back to the laboratory following retrieval.

10.2.3 Ensure that during transport passive samplers are maintained at an appropriate storage temperature, as recommended by the manufacturer. Where such instructions are not available, store samplers at 4 °C, and avoid freezing samplers that contain traces of water. This can be achieved by using an appropriate portable storage facility, e.g. an insulated container with cooling blocks.

10.3 Deployment procedure

10.3.1 Transport the labelled passive sampling devices (B.1) and required control devices (B.2) to the sampling site in sealed containers (B.5) inside a portable storage facility to maintain a low temperature environment (10.2.3), where necessary.

10.3.2 Record any water quality determinands significant to the study, e.g. water temperature, pH, turbidity, water flow rate (B.6). Water temperature and flow rates are necessary in order to be able to select appropriate calibration parameters for passive samplers, e.g. those for metals and polar organic compounds, where PRCs are not available. The pH is necessary to interpret the data when the pollutants being measured are dissociable compounds.

10.3.3 Using gloves (B.4) remove each passive sampling device from its container in accordance with the manufacturer's instructions. Treat field control passive sampling devices in exactly the same way as the field deployed passive sampling devices, but do not deploy them.

For some passive sampling devices, the containers can require opening under water.

10.3.4 Where possible, take care not to touch the membrane of the passive sampling device, unless the type of passive sampling device so requires. Prepare each passive sampling device as necessary and install in the deployment device (B.8) (see Note 1). Where photodegradation of organic chemicals is of concern, protect the passive sampling device from direct light during deployment (see Note 2).

Ensure that exposure of the passive sampling device to the atmosphere during deployment and retrieval is kept to a minimum. This is particularly important when pollutants are present in the vapour phase. Some designs of passive sampling device can rapidly accumulate volatile pollutants from the air.

NOTE 1 Where surface layers of chemicals are visible, precautions can be needed to reduce contamination during the placement of the deployment devices in the water.

NOTE 2 The use of photolysis surrogates, e.g. PRCs, added to the passive sampling device can help determine any potential chemical loss due to photodegradation.

10.3.5 Anchor the deployment device (B.8), to which the passive samplers are attached, to the river, lake or sea bed at the sampling point and suspend it below the surface from the buoy or floats (B.11). Attach weights (B.12) to the deployment device if necessary to keep it submerged at this depth. Record the depth below the water surface at which the passive sampling device is deployed (B.7). Take account of fluctuations in water level so that, if possible, the passive sampling device remains at the same depth below the surface throughout the deployment. In any case, ensure that the passive sampling device remains submerged throughout the exposure period. Passive sampling devices should be deployed in such a way that no air or sediment becomes trapped on the receiving membrane. Air and sediment can reduce uptake rate, or contaminate the passive sampling device.

The deployment device should be positioned so that it is as inconspicuous as possible, in order to guard against tampering or vandalism.

The passive sampling device should be adequately secured and protected against damage and loss during flood events or storms.

10.3.6 When the set of passive sampling devices is being deployed, close the containers that contain the field control(s).

10.3.7 Close the empty passive sampling device containers and place them in the portable storage facility, for transport back to the laboratory along with the field control(s). Where necessary, maintain a low temperature (B.3) during transport back to the laboratory.

10.3.8 Record the date and time of deployment, weather conditions, and the name of the person executing the deployment.

10.3.9 Where necessary, check the integrity of the deployment device and passive sampling devices at recorded intervals. Measure and record relevant water quality determinands at deployment and retrieval (see 10.3.2).

NOTE For some investigations, it can be necessary to recover devices sequentially throughout the deployment period.

10.4 Retrieval procedure

10.4.1 Record any water quality determinands significant to the study, e.g. water temperature, pH, turbidity, water flow rate (B.6).

10.4.2 Retrieve the deployment device. Check whether the deployment device and samplers have been tampered with during deployment. Where samplers have been disturbed during deployment, they should not be used to estimate concentrations of pollutants in the surface water. Using gloves (B.4), retrieve passive sampling devices, taking care not to touch the membrane surface, except where procedures require this.

10.4.3 At the time the passive sampling devices are retrieved, open the container housing the field control(s) (B.2).

10.4.4 Check the integrity of each passive sampling device. Record any damage, e.g. ruptured membrane, and the extent of biofouling. Where the membrane has been damaged, the passive sampling device should not be used to estimate the concentrations of pollutants in water.

10.4.5 Prepare each passive sampling device as necessary. Return each passive sampling device to its original container (B.5). Close the container housing the field control(s).

10.4.6 Seal each container and place immediately in the portable storage facility to maintain a low temperature (B.3) along with the field control(s) (B.2).

10.4.7 Ensure that passive sampling devices remain isolated from potential sources of contamination during transport to the laboratory and subsequent storage by placing the passive sampling devices in their sealed containers immediately after retrieval. Store passive sampling devices at temperatures recommended by the passive sampling device manufacturer prior to analysis.

10.4.8 Record the date and time of retrieval, the name of the person executing the retrieval, and the weather conditions.

11 Extraction of analytes from passive sampling devices and preparation for analysis

When preparing passive sampling devices and controls for analysis, follow the handling instructions given in Clause 5.

Prepare the receiving phase for analysis so as to avoid contamination from fouling on the passive sampling device surfaces.

NOTE This can be achieved by cleaning the membrane surface or careful disassembly of passive sampling devices.

Use extraction protocols provided by the manufacturer. For samplers, e.g. strips or sheets of polymeric material including low density polyethylene and silicone rubber, that are prepared in-house, use protocols that are reported in peer-reviewed publications.

The extraction uses techniques, e.g. dialysis, solvent extraction, acid digestion or thermal desorption, to extract the analytes of interest, residual PRCs and recovery spike compounds for analysis. The technique used is determined by the type of passive sampling device used, and the class of analyte sampled.

12 Analysis

Using suitable methods, analyse samples from the passive sampling devices and controls to determine the amount of each analyte, PRC and recovery spike compound in the receiving phase, e.g. gas chromatography mass spectrometry (GC-MS), gas chromatography-electron capture detection (GC-ECD), liquid chromatography mass spectrometry (LC-MS), liquid chromatography fluorescence spectrometry (LC-fluor) for organic analytes; atomic absorption spectrometry (AAS) or inductively coupled plasma mass spectrometry (ICP-MS) for metal analytes.

Before commencing analysis, it is essential, using laboratory tests, to establish overall precision, bias and limits of detection of the methods.

Use methods that have been validated by the laboratory that is to perform the analysis.

NOTE Where there is a need to measure very low trace levels of a pollutant, extracts from multiple passive sampling devices can be combined prior to analysis.

13 Calculations

Where the passive sampler has been used in equilibrium mode, calculate the concentration, in nanograms per litre, of dissolved analytes in the water, ρ_a , at the time of recovery of the sampler from the water as follows:

$$\rho_a = \frac{m}{K_{sw} V_s} \quad (3)$$

where

m is the mass, in nanograms, of analyte accumulated in the receiving phase of the passive sampling device at equilibrium;

K_{sw} is the partition coefficient between the receiving phase and water;

V_s is the volume, in cubic decimetres (litres), of the receiving phase.

Partition coefficients and volumes of the receiving phase are provided by the manufacturer for some passive samplers. For samplers, e.g. strips or sheets of polymeric material including low density polyethylene and silicone rubber, that are prepared in-house, use calibration data that are reported in peer-reviewed publications.

Where the concentration of an analyte in the passive sampler has not reached equilibrium with the concentration in the water, but the exposure time is greater than the time to half equilibrium, then it is necessary to use Equation (4) to estimate the concentration of the analyte in the water.

$$\rho_a = \frac{m_t}{V_s K_{sw} \left[1 - \exp\left(-\frac{k_0 t A}{V_s K_{sw}}\right) \right]} \quad (4)$$

where

m_t is the mass, in nanograms, of analyte accumulated in the receiving phase of the passive sampling device after an exposure time, t ;

t is the exposure time, in seconds;

k_0 is the overall mass transfer coefficient, in decimetres per day, of the analyte from water to the passive sampling device;

A is the effective area, in square decimetres, of the passive sampling device.

Where the exposure time for an analyte has not exceeded the time to half equilibrium, the sampler has operated in integrative mode, and the time-weighted average concentration, in nanograms per litre, of dissolved analytes in water, ρ_a , over the deployment time can be calculated as follows:

$$\rho_a = \frac{m}{k_0 A t} \quad (5)$$

This is a very general formulation of the calculation. It assumes unidirectional, linear, integrative uptake of the analyte during the whole deployment period. In the linear phase of uptake (see Figure 2) the sampling rate (the apparent volume, $k_0 A$, in cubic decimetres per day or litres per day, of water cleared of pollutant per time) is independent of the concentration of the pollutant in the water.

Some errors associated with the estimates of ρ_a can be determined from the use of appropriate quality control measures (see Annex C). Examples of calculation and quality control measures for the various types of passive sampling device are provided by the manufacturers of some types of passive sampling device and in the bibliography (see especially References [10], [12], [13], [16], [17], [25], [26], [27], [28]).

The value of the overall mass transfer coefficient for any analyte is in general a device- and site-specific calibration parameter. It is obtained in a variety of ways, depending on the specific passive sampling device used and deployment conditions. It is a substance-specific value dependent on the physicochemical properties of the analytes and environmental variables, e.g. water temperature and hydrodynamic conditions.

The overall mass transfer coefficient can be obtained in a number of ways, including the use of empirical calibration data or from theoretical considerations where the parameters are provided by the manufacturer. For samplers, e.g. strips or sheets of polymeric material including low density polyethylene and silicone rubber, that are prepared in-house, use calibration data that are reported in peer-reviewed publications. Calibration data typically cover a range of temperatures and turbulence conditions. In addition, for some passive sampling devices, PRCs (see Note 1) can be used to correct the overall mass transfer coefficient for fluctuations in environmental conditions (see Figure 4).

The kinetics of offloading of the PRC (see Note 2) and the kinetics of uptake of a pollutant should be related. They should have first order isotropic exchange kinetics (Figure 4). Hence, they are affected in a matching way by environmental variables, e.g. temperature and turbulence. When these conditions are met, then the offloading of the PRC can be used to provide an *in situ* calibration of the passive sampling device. Hence these results can also be used to adjust the overall exchange rate constant for uptake of a pollutant for the effects of temperature and turbulence.

NOTE 1 A PRC is an analyte that is added to the receiving phase of a passive sampling device prior to deployment. A PRC usually has properties similar to those of the pollutants of interest, but a PRC should not be present in the environment in significant concentrations. Typically perdeuterated or ¹³C-labelled analytes are used as PRCs. It is generally possible to measure offloading of the PRC from the receiving phase during exposure of the passive sampling device.

NOTE 2 The kinetics of offloading of the PRC can be described by a first order loss equation:

$$m_{\text{PRC},t} = m_{\text{PRC},0} \exp(-k_p t)$$

where

$m_{\text{PRC},t}$ is the residual mass of PRC in the receiving phase after an exposure time, t ;

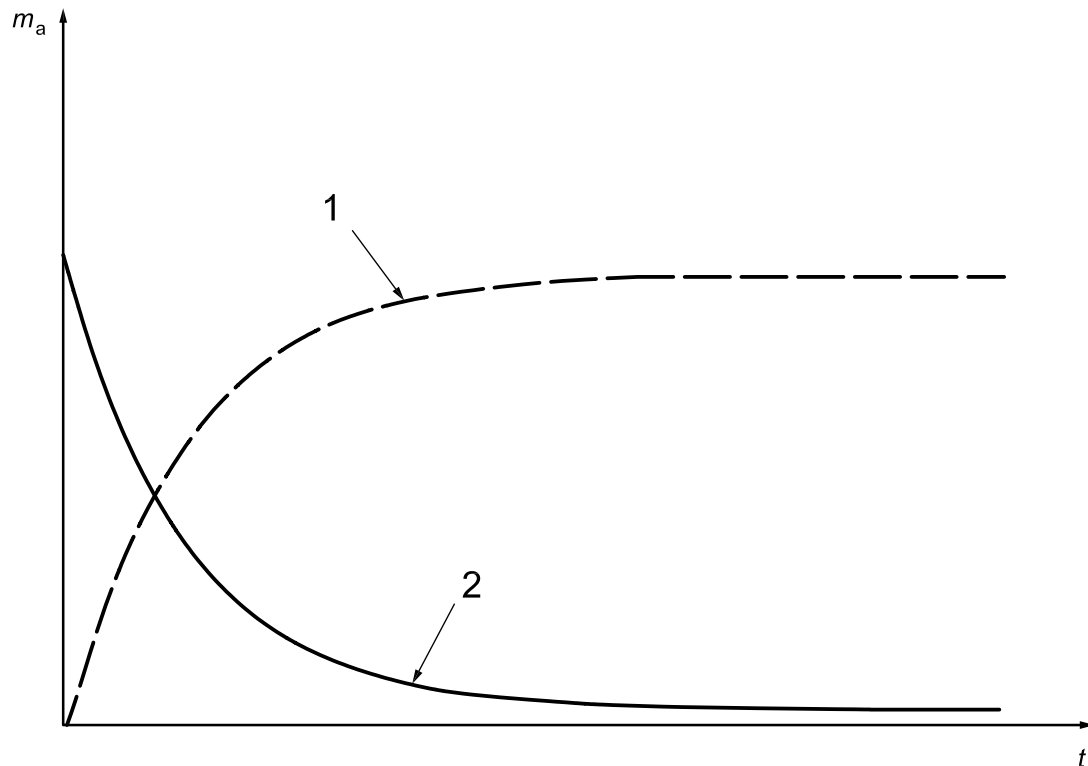
$m_{\text{PRC},0}$ is the mass of PRC spiked into the receiving phase;

k_p is the first order rate constant for offloading of the PRC from the receiving phase.

Since k_p and the overall exchange rate constant for uptake of the pollutant, k_e (Figure 4), are related, the offloading of PRC can be used to adjust uptake rates for the effects of temperature and turbulence. Where the kinetics of uptake and offloading are isotropic, the overall offloading exchange rate constant, k_p , is a function of the overall uptake exchange rate constant, k_e :

$$k_0 A = k_e K_{sw} V_s$$

The overall offloading exchange rate constant, k_p , can be used to calculate, using Equation (2), the time to half maximum accumulation of the pollutant in the receiving phase (Figure 4).

**Key**

m_a	mass of analyte in receiving phase	1	pollutant
t	exposure time	2	performance reference compound

Figure 4 — Time courses of uptake of a pollutant (1) into, and offloading of a performance reference compound (2) from, the receiving phase of a passive sampling device

14 Test report

The test report shall include at least the following information for each analyte in the passive sampling device set:

- a) sampling location;
- b) date and time of deployment and retrieval of passive sampling devices;
- c) site characteristics;
- d) name of person deploying and retrieving the device;
- e) water quality determinands recorded at deployment and retrieval;
- f) levels of target analytes quantified in quality control samples;
- g) amount of the analyte determined in the sampler, with its estimated uncertainty;
- h) time-weighted average concentration of analyte in the surface water;
- i) details of the method used, with reference to this part of ISO 5667 (ISO 5667-23:2011).

At least the following information shall be recorded for each analyte in the passive sampling device set and be available if necessary to aid interpretation of the report:

- 1) identity of set of passive sampling devices;
- 2) passive sampling device type and manufacturer;
- 3) passive sampling device batch number;
- 4) passive sampling device field identification;
- 5) passive sampling device uptake rate and the method used to determine it;
- 6) where applicable, further information on the sample site, e.g. previous levels of any pollutants found;
- 7) any PRCs used and their levels in passive sampling devices before and after exposure;
- 8) recovery spike compounds used;
- 9) sample preparation method;
- 10) analytical method.

Annex A (informative)

Tables providing a summary of the main types of passive sampling devices and a summary of the methods for their calibration

Table A.1 — Structure of main classes of passive sampling devices

Passive sampling device class	Receiving phase	Membrane	Performance reference compound ^a available
Polar organic compounds	Microporous adsorbent materials, e.g. SDB ^b , activated carbon Solid phase microextraction fibres	Polyethersulfone	No
		Polysulfone Regenerated cellulose In the absence of a diffusional membrane the water boundary layer may act as a diffusional layer	No
Non-polar organic compounds	Triolein Octadecyl silica Polydimethylsiloxane Low density polyethylene Organic solvents (e.g. hexane) Polyoxymethylene	Polyethylene Regenerated cellulose Water boundary layer (see Note)	Yes
Inorganic compounds including metals	Chelating resin	Polyacrylamide hydrogel Microporous cellulose acetate	No
NOTE Polymer samplers can act as both permeation membrane and receiving phase at the same time.			
^a See 3.5. ^b Poly(styrene-divinylbenzene) polymer.			

Table A.2 — Summary of calibration methods available for passive sampling devices

Calibration method	Passive sampling device types	Conditions	Remarks
Static tank ^a	All	Controlled temperature Fixed stirring rates	Where sampling rates (the apparent volume of water cleared of pollutant per time, in litres per hour) are high, large volumes are needed to avoid depletion of the calibration solution
Static tank with renewal ^a	All	Controlled temperature Controlled stirring rates	
Continuous flow tank ^a	All	Controlled temperature Controlled stirring rates	Flow rate should be higher (at least fivefold) than the total sampling rate of the samplers in the system
Modelling based on diffusion coefficients	Diffusive gradients in thin film samplers, where diffusion limiting layer is thick compared with water boundary layer	Range of temperatures	Calibration data supplied by the manufacturer
Modelling based on partition coefficients	Samplers for non-polar organic compounds		<p>The partition coefficients of extremely hydrophobic compounds between the passive sampling device receiving phase and water are difficult to measure directly because of the low water solubilities of such compounds. These problems are overcome by using the co-solvent method that uses a range of concentrations of methanol to bring into solution the non-polar analytes. The partition coefficients are measured in the range of concentrations of methanol, and then the plot of the log partition coefficient against methanol fraction is extrapolated to zero methanol to yield useful estimates of sampling phase/water partition coefficients.</p> <p>Empirical relationships between sampling phase/water partition coefficients and $\log K_{OW}$ can be used to estimate values for additional compounds</p>
Performance reference compounds (PRCs)	Available for non-polar organic samplers only		<p>PRCs should be used where possible to reduce uncertainties associated with variable temperature and hydrodynamic conditions in the field.</p> <p>Offloading rates for PRCs give a sampling rate over only a limited range ($4,5 < \log K_{OW} < 6$), and for pollutants with $\log K_{OW} > 6$ it is necessary to extrapolate models based on that range</p>

^a Precautions: ensure constant concentration, and measure actual concentration in calibration tank at least daily. Use estimated equilibration rate constants to determine for which analytes the passive sampling devices have operated in kinetic mode, and for which in equilibrium mode.

Annex B (normative)

Materials and apparatus to be taken to the field for use in the deployment of passive sampling devices

- B.1 Passive sampling devices**, labelled in accordance with ISO 5667-1 and ISO 5667-6.
- B.2 Field control passive sampling devices.**
- B.3 Portable storage facility**, e.g. cool box with freezer blocks, to maintain a low temperature recommended for storage and transport.
- B.4 Gloves**, for use in handling the passive samplers in the field. Use powder-free vinyl or latex gloves. Do not re-use gloves.
- B.5 Gas-tight containers**, of inert materials relevant to the pollutants of interest, having low permeability to external contaminants and labelled with the passive sampling device identification.
- B.6 Water quality assessment equipment**, e.g. thermometer, temperature logger, pH meter, flow rate meter, as necessary.
- B.7 Depth measuring device.**
- B.8 Deployment device**, to hold the passive sampling devices in an appropriate orientation during deployment. It is possible to use a variety of deployment devices, e.g. metal stakes, sheets of plastic or metal mesh, or metal cages, depending on the depth of water and water flow at the sampling site. Typically the devices hold the passive samplers so that the permeation membrane is held vertical and perpendicular to the water surface, or horizontal and facing away from the water surface. The former orientation aims to expose the sampling surface to the water flow, and the latter aims to minimize the deposition of sediment on the permeation membrane surface.
- B.9 Anchorage device**, suitable for holding the deployment device at an appropriate depth and in an appropriate orientation with respect to the surface of the water.
- B.10 Ropes.**
- B.11 Buoys or floats.**
- B.12 Weights.**

Annex C (informative)

Quality control measures

C.1 Recovery

Recovery is evaluated on the basis of mean analyte recovery from recovery spikes. The analyte recovery mass fraction, w_a , expressed as a percentage, is determined by:

$$w_a = \frac{\rho_a}{\rho_s} \times 100 \quad (\text{C.1})$$

where

ρ_a is the mean measured analyte concentration;

ρ_s is the recovery spike concentration.

See Reference [22].

C.2 Method detection limits

In order to limit false negative results for samples containing some of the analyte in question, replicate spiked passive sampling devices are analysed to determine the concentration limit of detection, ρ_{LD} . The spiking levels should not be significantly greater than 5 to 10 times the expected s -value. The ρ_{LD} is given by:

$$\rho_{LD} = \left(2 \sqrt{1 + \frac{1}{n_B}} \right) t_{S(n-1, \alpha)} s \quad (\text{C.2})$$

where

t_S is the one-tailed Student t value for a significance level α with $n - 1$ degrees of freedom;

n is the number of replicate spiked samples analysed;

s is the standard deviation of the measured analyte concentration in replicate spikes;

n_B is the number of blank determinations used to correct sample results when using the method for analysing samples from the passive sampling devices — the statistical assumption is that the concentrations of the spike recoveries are normally distributed.

The user needs to decide the level of protection required against false negative results and use the appropriate values of n and t .

See Reference [19].

C.3 Precision

Precision of passive sampling devices is determined by comparison of duplicate, field-exposed passive sampling devices. Coefficients of variation, C_V , are calculated as:

$$C_V = \left[\frac{|\rho_1 - \rho_2|}{0,5(\rho_1 + \rho_2)} \right] \times 100 \quad (\text{C.3})$$

where ρ_1 and ρ_2 are analyte concentrations in duplicate passive sampling devices.

See Reference [10].

Where more than two replicate field-exposed passive sampling devices are used, the precision is measured as the coefficient of variation, C_V , which is calculated as:

$$C_V = \frac{s}{\bar{\rho}} \times 100 \quad (\text{C.4})$$

where

$\bar{\rho}$ is the mean of the analyte concentrations in replicate field-exposed passive sampling devices;

s is the corresponding standard deviation of the concentrations in replicate field-exposed passive sampling devices.

It is assumed that the concentrations of analyte in the replicate field-exposed passive sampling devices are normally distributed.

See Reference [22].

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