# INTERNATIONAL STANDARD

ISO 5667-4

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# Water quality — Sampling —

Part 4:

# **Guidance on sampling from lakes, natural and man-made**

Qualité de l'eau — Échantillonnage —

Partie 4: Lignes directrices pour l'échantillonnage des eaux des lacs naturels et des lacs artificiels







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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.

The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see <a href="www.iso.org/directives">www.iso.org/directives</a>).

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. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see <a href="https://www.iso.org/patents">www.iso.org/patents</a>).

Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

For an explanation on the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT) see the following URL: <a href="www.iso.org/iso/foreword.html">www.iso.org/iso/foreword.html</a>.

The committee responsible for this document is ISO/TC 147, *Water quality*, Subcommittee SC 6, *Sampling (general methods)*.

This second edition cancels and replaces the first edition (ISO 5667-4:1987), which has been technically revised.

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 and quality control 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
- Part 24: Guidelines for the auditing of water quality sampling

# Water quality — Sampling —

## Part 4:

# Guidance on sampling from lakes, natural and man-made

## 1 Scope

This part of ISO 5667 gives guidelines for the design of sampling programmes, techniques and the handling and preservation of samples of water, from natural and man-made lakes during open-water and ice-covered conditions. This part of ISO 5667 is applicable to lakes with and without aquatic vegetation.

Guidance on sampling for microbiological examination is not included.

#### 2 Normative references

The following documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. 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 5667-14, Water quality — Sampling — Part 14: Guidance on quality assurance and quality control of environmental water sampling and handling

ISO 7027, Water quality — Determination of turbidity

#### 3 Terms and definitions

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

#### 3.1

#### grab sample

single discrete sample collected from a body of water at a specific time, location and depth

#### 3.2

#### depth profile samples

two or more discrete samples collected at two or more depths at a specific time and location on a lake

#### 3.3

#### area profile samples

two or more discrete samples collected from the same depth at two or more locations on a lake

#### 3.4

#### composite sample

two or more *depth profile* (3.2) or *area profile samples* (3.3) that are combined to form a single sample prior to measurement of water quality parameters

#### 3.5

#### integrated sample

single sample collected by a tube or similar sampler that collects a water sample across a range of depths

#### ISO 5667-4:2016(E)

#### 3.6

#### in-situ measurement

measurement of a water quality parameter made within a body of water, not requiring collection of a water sample

Note 1 to entry: The measurement is generally taken by use of an electronic probe.

#### 3.7

#### ex-situ measurement

measurement of a water quality parameter made outside a body of water, and requiring collection and possibly transport of a water sample prior to measurement

#### 3.8

#### open sampling device

open-mouthed vessel, including beakers, buckets, containers or tubes, used for sampling at or near the water surface (<1 m depth)

Note 1 to entry: Open sampling devices are not suitable for sampling for volatile parameters or dissolved gases.

#### 3.9

#### closed sampling device

vertically or horizontally aligned hollow-bodied tube, pipe, box or container fitted with shutters, valves, stoppers or other devices that prevent entry of air into and/or enclosure of air with the water sample and the exchange of water between the collected sample and the surrounding water column

Note 1 to entry: Closed sampling devices are used for collecting water samples from deeper waters or for collection of water samples for the analysis of volatile parameters and dissolved gases.

#### 3.10

#### sampling pole

pole or rod with a terminal apparatus that accepts an open or closed sampling device (3.9) and that is used to extend the reach of samplers

#### 3.11

#### sampling iron

weighted container holder attached to a line and used to extend the reach of samplers or to submerse a sample container to a specific depth

#### 3.12

#### pumping device

hand or motor operated suction or submersible pumps, or pneumatic ejection samplers used for collection of samples from defined depths or a series of depths

#### 3.13

#### filling device

funnel, ladle, churn sample splitter or other device used to transfer sample water from a sampling device to a sampling container

#### 3.14

## negative control

quality control sample that is used to ensure a negative response

## Sampling equipment

#### 4.1 Material selection

Sampling probes, devices and other equipment should be chosen, as far as possible, which do not give rise to any interaction between the water and the material composition. The equipment and devices to be used should be checked at random for the presence of emission, absorption and adsorption of substances or influence of properties that are to be determined in the samples to be taken. Examples of common sampling equipment are summarized in <u>Annex A</u>. Advantages and disadvantages of sampling equipment are provided in <u>Annex B</u>. If a boat, marine vessel or any floatation device is used, care shall be taken so that it does not result in contamination of the samples. Additional details regarding sampling from a vessel are summarized in <u>Annex C</u>. A winch with steel rope and counter is advisable for deeper lakes. This ensures the required sinking and resurfacing velocity of the sampling devices. Any solvents, chemicals or fuels should be stored in sealed non-permeable containers. For example, the use of electric motors on small boats can be of value in eliminating the risk of contamination by combustion exhaust and engine lubricants. ISO 5667-14 provides guidance for checking the uptake and emission of the substances to be measured by equipment and devices that are used for sampling.

## 4.2 Cleaning

A proper quality assurance and quality control (QA/QC) system should be in place to prevent contamination and detect any contamination that could affect analytical results. All equipment and devices should be regularly mechanically and, if appropriate, chemically cleaned, both internally and externally, to prevent contamination of water samples.

#### 4.3 Maintenance

Where equipment uses mechanical or other triggering devices, these mechanisms should be regularly tested. Electronic devices and probes should be tested and calibrated according to the manufacturer's recommendations. A log of testing and calibration dates and results should be maintained.

## 5 Design of the sampling programme

Proper sampling is critical to ensure the quality of the investigation and resulting data. Developing a detailed sampling strategy prior to collecting samples will minimize any sampling errors and will provide the most representative sample for analysis. General aspects that should be considered in a sampling programme are summarized in ISO 5667-1. These include, but are not limited to, the following:

- a) purpose of the investigation;
- b) parameters to be analysed for each sampling point;
- c) measurements to be carried out at the sampling point as specified in the analytical method, e.g. temperature, dissolved oxygen, pH, turbidity, conductivity;
- d) frequency and times of sampling and type of sample;
- e) sampling site and the number and locations of sampling points;
- f) sampling equipment;
- g) quality assurance procedures to be followed;
- h) transport preservation and storage of samples;
- i) hydrodynamic and morphologic characteristics of the water sampled;
- j) local circumstances such as water depth, vegetation, accessibility of location and other potential obstacles such as floating layers or sludge layers present;
- k) sampling depth(s);
- l) composition and quantity of the water to be sampled;
- m) safety considerations.

## 6 Sampling procedure

#### 6.1 General

As recommended in ISO 5667-1, an investigation plan should be established before a sampling program is initiated. The plan should include: the purpose of the investigation; parameters to be analysed for each sampling site; frequency and times of sampling; the type of collection gear and containers; the number and locations of sampling points; sample preservation requirements; access and safety concerns; hydrodynamic, morphological and biological characteristics of the sampling site(s); the sampling depth(s); and quantity of water to be collected.

Phytoplankton and/or chlorophyll, as well nutrients for water protection issues, should be sampled in the euphotic zone or mixed layer, respectively. The decision down to which depth an integrated sample has to be taken should be done according to EN 16698. The decision depends on lake type, stratification and phytoplankton turbidity. That means before the sampling procedure, the current probe measurement data and Secchi disk readings have to be available.

## 6.2 Sampling location

#### 6.2.1 General

General guidance is given in ISO 5667-1.

Samples from surface layers containing floating material should be taken with special surface samplers.

The spatial distribution of sampling locations can be properly decided only after detailed preliminary work using a large number of sampling locations to provide information to which statistical techniques may be applied.

#### 6.2.2 Horizontal distribution of sampling positions

## 6.2.2.1 Sampling point for characterization of water quality

Morphologically complex lakes, those either consisting of several basins or having a complicated shoreline can show significant heterogeneities in a horizontal direction. In order to evaluate the extent of such heterogeneities, it is necessary to set up several sampling points to carry out preliminary investigations. The data gathered then enable the necessary number of sampling points to be fixed effectively. One sampling point above the deepest part of the lake is generally sufficient for lakes showing no significant heterogeneities in a horizontal direction. Sampling points should be defined clearly, and if possible, marked with buoys. Use navigation devices to identify the sampling points if the surface area is too large to allow the fixing of buoys. If appropriate to the sampling purpose, samples can be collected from the lake shore, ideally at or near the outflow, or jetty or promontory, using a device such as a sampling iron or sampling pole.

## 6.2.2.2 Sampling point for quality control

Negative control samples should be taken such that they are not influenced by potential sources of contamination. These could include other nearby non-impacted areas or other nearby bodies of water that are representative of the body of water being sampled.

#### 6.2.2.3 Sampling point for special investigations

Samples should be taken as single or replicate samples where unusual phenomena have been observed. The sites should be clearly identified in the report, with a map or sketch where possible.

#### 6.2.3 Vertical distribution of sampling points

The water quality in natural and man-made lakes can show large vertical heterogeneities due to stratification. The reasons for these are influences from the water surface (changes of the water quality by photosynthesis in the euphotic zone and changes in water temperature by heating) and influences arising from the sediment (dissolution or resuspension of substances from the sediment). Furthermore, vertical heterogeneities can arise from sedimentation of suspended matter. Large differences in water quality are also frequently observed at the thermocline. For these reasons, the distance between grab sample depths in heterogeneous zones should be minimized. The exact arrangement of sampling levels depends on the information required and the local circumstances. It is therefore advisable to carry out preliminary investigations using measuring probes (for measurement of temperature, if possible, as well as dissolved oxygen concentration, pH value, conductivity, turbidity and chlorophyll fluorescence), which allow either continuous monitoring or monitoring at short intervals. In such cases, stagger the sampling depth to allow the recording of all vertical heterogeneity. Once a sampling programme has been defined, it should be carried out to completion, since if it is altered during sampling, the data gathered will be incompatible. In large and deep bodies of water where internal movement of water can occur, the use of a series of samplers, which all take samples simultaneously, is recommended.

#### 6.3 Frequency and timing of sampling

Detailed guidance, including statistical considerations, is given in ISO 5667-1.

The water quality of natural and man-made lakes varies seasonally. Consequently, the frequency of sampling will depend on the information required.

In general, for lentic waters, an interval of 1 month or longer between the collection of consecutive samples is acceptable for water quality characterization over a long period of time. For the purpose of quality control measurement, a minimum interval of one week is necessary. If rapid changes in water quality are apparent, daily or even continuous sampling can be necessary.

Lake sampling four times a year will allow an acceptable water quality characterization over a long period of time. For the purpose of quality control measurement, a higher frequency may be required.

In addition, quality can vary significantly during a day. Samples should be taken at about the same time of the day. If daily variation is of special interest, sampling every 2 h or 3 h is recommended.

#### 6.4 Choice of sampling method

The choice of sampling method depends on the objective of the sampling programme. Samples taken for special reasons or for quality control purposes will, in most cases, be grab samples. For monitoring water quality, a series of grab samples is used, but composite samples can be useful. The analysis of a series of grab samples can be costly and these are often combined to reduce analytical costs; however, composite samples will indicate only mean values and will not reveal details of extreme conditions or the extent of quality variation. Both methods may be combined by taking composite samples at short intervals and a series of samples at longer intervals.

## 6.5 Choice of the sampling device

The choice of the sampling device depends on the objective of the sampling program. Samples taken for special reasons or for quality control purposes will, in most cases, be grab samples (see Annex A for examples of sampling devices). For monitoring water quality, a series of grab samples is often used, but composite samples may be useful, especially for the investigation of defined water layers, e.g. the epilimnion or the euphotic zone. However, composite samples will indicate only mean values and will not reveal details of extreme conditions or the extent of quality variation.

Immersion probes may used for a continuous measurement of pH or dissolved oxygen in unstable water samples ( $\underline{Annex E}$ ).

## 6.6 Aids for recovery of lost sampling equipment

When handing sampling devices in small boats, there is an increased risk of losing equipment overboard. It is recommended that the trailing end of the tether line comprises of "floating rope" to aid recovery should the equipment be lost in shallow waters. Floating ropes are designed as safety products that remain near the surface for easy capture and recovery of objects to which they are attached. They are readily available for a range of safety applications and load ratings.

#### 6.7 Blank sample

When collecting and processing water samples, it is necessary to include blank samples such as field blanks, travel blanks and filter blanks to measure the degree of contamination that may have been introduced into the samples as a result of sampling related activities. Detailed guidance on the use of blanks and other quality control samples is given in ISO 5667-14.

#### 6.8 Transport, stabilization and keeping of samples

ISO 5667-3 gives general guidance on sample handling and preservation.

Ensure that sample containers are delivered to the laboratory tightly sealed and protected from the effects of light and excessive heat, because the quality may change rapidly due to gas exchange, chemical reactions and the metabolism of organisms. Ensure that samples which cannot be analysed quickly are filtered (if required by specific analytical method) and stabilized or preserved if required. The method of preservation shall be chosen to avoid interference with the parameters of interest and does not interfere with the subsequent examination or influence the results. Where particles or biological activity in the sample may be expected to influence parameters of interest, the sample may be filtered on site to remove particles or organisms and the filter held separately for analysis. For storage over short periods, cooling to 5 °C  $\pm$  3 °C may be applied; for keeping over longer periods, freezing to –18 °C is advisable. In the latter case, ensure the sample is completely thawed before use as the freezing process can have the effect concentrating some components of the inner part of the sample which freezes last. Samples may be preserved by the addition of chemicals. Record all preservation steps in the report. Measure and record the temperature on site. Ideally, other physical parameters (pH, for example) should be determined on site. If free carbon dioxide is present, measure the pH *in situ*.

## 7 Occupational health and safety

ISO 5667-1 specifies safety precautions including, but not limited to, sampling from boats and ice-covered waters.

NOTE The user's attention is drawn to the existence of national and/or local health and safety regulations.

## 8 Sample containers

Sample containers should be selected on the basis of the parameters to be analysed and be made of materials that do not lead to contamination of samples. ISO 5667-3 provides further guidance on the selection of sample containers. Depending on parameters to be analysed, sample containers can require laboratory pre-treatment, including acid-washing, cleaning or quality assurance/quality control procedures or on-site rinsing to be performed prior to use. Guidance should be sought from the analytical laboratory with respect to the selection and use of sampling containers and the volume of sample required to be collected. This guidance should be documented in formal, written protocols. Where containers are pre-treated, precautions should be taken to avoid exposure to chemicals used in the pre-treatment process.

## 9 Priority of procedure

Prior to taking samples, it is important to take the UTM-coordinates and water depth to ensure samples were collected in the correct location. This is typically the deepest point of the lake.

In order to reduce the risk of contamination of water samples, choose methods that reduce the number of water transfers between sample collection devices, filling devices and sample containers. In order of preference (see <u>Table 1</u>),

- conduct in situ measurement of parameters of interest,
- collect water samples using direct filling of sampling containers for *ex-situ* analysis without the use of sampling devices, funnels or other equipment,
- conduct indirect filling of sampling containers for *ex-situ* analysis where sampling devices are used to collect water samples that are later distributed directly into one or more sample containers, and
- conduct indirect collection of samples for *ex-situ* measurement, whereby sampling devices collect
  water samples that are later distributed into one or more sample containers.

*In situ* water quality measurements including dissolved oxygen, pH, water temperature, conductivity, turbidity and light penetration, by Secchi disk depth or light probe, can guide depth stratified sampling of thermally or chemically stratified waters by permitting pre-sampling assessment of the chemical and physical structure of the water column and therefore should be completed before the collection of physical water samples occurs. Depth measurements should be consistently repeated in stratified waters to ensure depth sensitive parameters such as pH and dissolved oxygen are sampled correctly.

Secchi disks can take a number of different forms including 30 cm diameter white and 20 cm diameter with every quarter of the disk alternating between black and white. Details of the disks and procedure are summarized in ISO 7027. A brief description is listed as follows.

- a) Lower the Secchi disk to the point of disappearance.
- b) Raise the disk until it reappears and then lower it until it disappears a second time.
- c) Note the depth.
- d) Repeat the procedure until a constant result is obtained.

Measurements should be taken such that environmental conditions, e.g. wind and glare from the sun, are minimized. Care shall be taken as to not disturb the sampling area (the use of an underwater viewscope can be desirable). If a motorized vessel with propeller is used, the engine should be shut off at least 10 m from the sampling area. Position and anchor the motorized vessel so that vessel does not contaminate the sampling area.

Table 1 — Priorities in measurement of a water quality parameter including dissolved oxygen, pH, water temperature and turbidity

Measurement of a water quality parameter including dissolved oxygen, pH, water temperature, turbidity							
Priority of procedure	Comments	Records					
Sampling location identified and confirmed Electronic devices and probes tested and calibrat Sampling device cleaned and checked	red		Location of the sampling point Equipment used for measurement and for sampling				
in situ measurement YES	Lower probes slowly in the water Allow time for probes to stabilize	<ul> <li>Values of temperature and oxygen unbiased (no exchange with air and no heating of the sample)</li> </ul>	Method of measurement Values of measurement				
ex situ measurement by immersion YES	Immerse probes in the water sample continuously (see Annex D)	<ul> <li>Risk: Value of temperature biased (heating of the sample)</li> <li>Value of oxygen unbiased (no exchange with air)</li> </ul>	Method of measurement Values of measurement				
ex situ measurement by collection YES	Collection of samples with sampling devices for ex situ measurement	— Risks: Values of temperature and oxygen biased (exchange with air and heating of the sample)	Method of measurement Values of measurement				

## 10 Sample collection, contamination with environmental materials

To avoid contamination of water samples during collection, the following guidelines are provided.

- Samples should be collected from the upwind or upstream side of an anchored boat.
- Samples should be collected from the downwind side of a drifting boat.
- Where depth profile samples are collected, sampling should commence at the surface of the water column and proceed downwards through the water column.
- When samples are collected during the course of wading, samples should be collected forward of the waders path or upwind of the sampler.
- Samples should be collected in a manner that does not permit contamination by motor exhausts, dust, debris and bottom sediments.
- Samples should be collected as to avoid collection devices or containers scraping against docks, poles, preserved lumber and other infrastructure, boat hulls and antifouling coatings.
- Unless specified in the investigation plan, the sample should not include the surface film of the water or floating layers.
- When sampling through ice, ice chips, ice-water slurry, meltwater and snow should not be allowed to enter the sample container.

 When sampling waters covered in vegetation, care should be taken not to disturb the vegetation or allow any of the vegetation to enter the sample container.

## 11 Rinsing the sampling equipment

All the equipment that comes in contact with the water should be rinsed, using water from the body of water to be sampled. Rinse all the equipment used for sampling preferably up to three times.

If the analytical method requires the sample containers to be rinsed, remove the caps prior to taking the rinse water, handling the caps in such a way that the interior surface does not become contaminated, preferably holding them in one hand or keeping them in a polyethylene bag. Follow the instructions in ISO 5667-3 for rinsing sampling containers. It is important that sample containers are not rinsed if they contain preservatives.

If using a rope or chain, pour some of the contents of the sample vessel rinse contents over the final metre of the rope or chain to wash off all traces of previous samples. Remove as much excess liquid as possible by shaking. Do not allow this part of the rope or chain to be re-contaminated by allowing it to come in contact with the ground or any other surface. Similarly, rinse the end of the sampling pole if used.

## 12 Sampling surface or near-surface water with an open sampling device

#### 12.1 General

Extra precautions should be taken for the analysis for volatile parameters including dissolved gases and volatile organic compounds (see <u>Clause 14</u>).

#### 12.2 Sampling

If applicable, attach the sample container to a sampling pole or iron. Displace the floating layer by first making a rotating movement with the container on the water surface. Rinse the sample container, preferably up to three times, with surface water as required by the sampling protocol and as may be directed by the laboratory.

Submerge the sample container to the sampling depth, turn it around and allow the container to fill.

Ensure that bubbles are not collected during the sampling procedure. Hold the sample container at about a 45° angle to allow bubbles to escape the sample container.

If conducting direct sampling, seal the sample container and proceed to post-sampling requirements for the samples.

If indirect water sampling is to be employed, rinse the filling devices and sample container as required by the investigation plan. Fill the filling device and dispense the water sample into the sample container(s). Seal the sample container.

#### 12.3 Filling multiple containers

If filling more than a single sample container from a single sample collection is required, ensure that the sample remains homogeneously mixed during filling of the sample container, by using, for example, a churn sample splitter to dispense sample water among the sample containers.

Where several collections of water samples from a single sampling site are required to fill multiple sample containers, either a) combine the sample collections in a larger container, such as a churn sample splitter, before allocation to sample containers, or b) use sample water from individual collections to completely fill sample containers for independent sets of inter-related parameters. For example, fill all sample containers needed for phosphorus fractions from the same collection, sample containers for anions and cations may be filled from a second collection, sample containers for dissolved and suspended heavy metals from a third collection.

Where depth or area composite samples are to be dispensed into multiple sample containers, mix the constituent samples of the composite sample in a churn sample splitter or similar device prior to dispensing sample water.

Samples should be collected and preserved in regard to specific requirements with respect to the method for which the samples have been taken.

## 13 Sampling with closed samplers

Closed samplers are typically used to sample volatile and non-volatile parameters in deeper waters, generally greater than 1 m depth, and for volatile compounds in shallow waters. In general, horizontal closed samplers are used in shallow or flowing waters while vertical samplers are used for deep, standing waters. Lower the open sampling device slowly into the water column to the required depth, avoiding dynamic pressure, allowing it to rinse as it descends. Seal the device when it has reached the desired depth and filled with sample water. Remove it from the water. Rinse any filling devices and containers with sample water as required. Fill containers as required. When multiple sample containers are to be filled or where multiple collections are to be made, the procedures described in 12.3 should be followed. Samplers with lids that do not uncover the entire cross-section area of the sampling tube while lowered in the water (e.g. Ruttner-sampler) are not appropriate.

## 14 Sampling for volatile parameters

Sampling volatile parameters requires the use of closed sampling or pumping devices. Following collection of the water sample with a closed sampling device, fill the sample containers directly from the device. Extend the outlet of the device with a PTFE tube long enough to reach the bottom of the sample container. Position the PTFE outlet on the bottom of the sample container and fill it with the device. Fill the container until it overflows. Then remove the outlet from the container while the water is still flowing. Seal the sample container immediately after removal of the outlet. The sample container should be completely filled, without any air bubbles.

When using a pumping device to collect the water sample, pump sample water directly into the sample container using an appropriate outlet tube as described above. Suction pumps should not be used for sampling volatile compounds as volatile organics and dissolved gases can be removed from the sample.

Where double sampling containers (see Figure 1) are used to sample volatile parameters in near-surface waters, place the sampling containers at the required depth and wait until both containers have been completely filled. Remove the PTFE stopper with inlet tube from the sampling container and seal this with a ground-glass stopper. The sampling container should be completely filled, without any air bubbles.

## 15 Sampling through ice

Sampling through ice requires use of an ice chisel, auger or drill. Use a battery or hand-powered ice auger to drill through the ice or an ice chisel when collecting samples for organics analyses, to reduce the possibility of contamination of samples with gasoline, oil and combustion products. After drilling the hole, remove all ice chips and snow from the hole and surrounding area prior to collection of the sample. Proceed with collection as above but, if applicable, with recognition of the specific challenges of working under severe freezing conditions. Strong gradients of parameters of interest can be present immediately below the ice surface. This should be considered in developing the sampling plan. Wherever possible, when year-round sampling occurs on seasonally ice-covered waters, sampling sites that can be safely accessed and sampled during both open-water and ice-covered periods shall be selected.

#### 16 Severe freezing conditions

Sampling under severe freezing conditions (colder than -15°C) can lead to: failure of battery-operated equipment, rapid damage to electronic sensors due to freezing of internal and external components,

water and/or snow freezing on the interior and exterior surfaces of sampling devices and their drainage tubes or triggering mechanisms, inability of samplers to manipulate delicate or fine mechanisms due to bulkiness of clothing, rapid freezing of samples, damage to glass collection containers due to freezing of samples, and risks to human health.

The following guidelines will assist in maintaining field sampling operability in severe freezing conditions.

- a) Transport and hold temperature-sensitive electronic equipment in a warm location.
- b) Keep a spare set of batteries for each electronic device in a warm location.
- c) Wet or warm sampling devices should not be placed directly onto snow or ice.
- d) Sampling devices, drainage tubes and valves should be immediately and fully drained of sample water.
- e) Frozen sampling devices may be thawed by suspending them below the surface of the water in the ice hole.
- f) Use thermoplastic containers where possible; glass containers are prone to breakage if sample contents freeze.
- g) Warmed freezer packs may be used to maintain sample temperatures above freezing.

## 17 Sample identification and records

Each sample shall be assigned a unique identifier.

The following data should be recorded for each sample (see Annex D): the name of the sampler(s), the location of the sampling point, the description of the sampling point, the depth or depth range (for an integrated sample) sampled, the sampling equipment used, the date and the time at which sampling started and ended, and any preservation steps applied to the sample. In addition, samplers should report any location specific details that can allow for improved interpretation of the data. These include, for example, the presence of any floating layers/oil film, operation of pumping stations, shipping, local application of pesticides, release of effluent from sewage lagoons and other similar types of events, large congregations of wildlife or waterbirds, weather conditions, ice thickness, ice coverage and currents. Immediately record the air and water temperature and other *in situ* parameters of interest (Secchi depth, pH, dissolved oxygen, conductivity and turbidity, for example). If the sampling point is a new site, describe each sampling point in detail. In the case of a long-term program with established site descriptions, note any conditions that differ from the stated description.

## 18 Quality assurance and quality control

#### 18.1 General

Quality assurance and quality control (QA/QC) procedures and practice are important for obtaining good quality analytical results. Quality assurance includes items that ensure the highest likelihood of obtaining quality data. QA procedures include: trained staff, calibrated, maintained and functioning equipment, standard operating procedures (SOPs) and methods, intercalibration studies, as well as tracking and documentation procedures.

Quality control (QC) involves samples and test procedures to measure and assess the quality of the process and results. QC tests include blanks (field blanks, lab blanks, travel blanks, instrument and solvent blanks), spiked samples, reference materials and calibration checks. It is important that QC samples are taken during the sampling procedure to ensure there is no bias or contamination resulting from sampling.

Refer to ISO 5667-14 for full details of such techniques for sampling.

#### 18.2 Avoidance of contamination

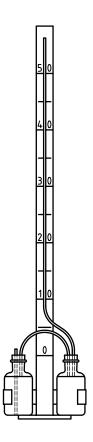
Specific SOPs should be followed to avoid contamination when sampling surface waters. Any deviations should be documented to aid interpretation of results.

Avoiding contamination during sampling is essential. All possible sources of contamination should be taken into account and the appropriate control applied if necessary. Include the following:

- a) environmental factors:
  - exhaust gases (cars/motorboats);
  - painting or use of chemicals in the vicinity of the sampling point;
  - use of plant protection products or fertilizing activities in the vicinity of the sampling site;
- b) method or procedure:
  - disturbance of the lake bottom, such that bottom deposits are also sampled;
  - co-sampling of floating layers;
  - contamination of the sample with algae or "anti-fouling" material as a result of scraping the bucket on the ship's hull or quay;
  - aeration of the sample when filling sampling containers, resulting in a loss of the volatile substances to be sampled. Aeration can also increase the oxygen content if the water is low in oxygen or decrease it if the water is supersaturated;
  - not mixing when filling containers as a result of which the undissolved components present with the impurities bound to them are not evenly spread through the sampling containers;
- c) sampling materials:
  - dirty or insufficiently cleaned sampling containers and/or devices for sampling;
  - incorrect sampling material choice, resulting in adsorption/desorption of the substances to be determined.

Procedures to monitor contamination and its control are presented in ISO 5667-14.

Sampling operatives should wear disposable gloves during the whole of the sampling procedure, both to protect themselves from contact with the sample and to prevent sample contamination. It is important to avoid cross contamination from one sample location to another by handling protective clothing and sampling equipment in such way as to avoid cross contamination.



 $Figure \ 1 - Double \ sampling \ container \ for \ collection \ of \ volatile \ parameters \ from \ shallow \ waters$ 

# **Annex A** (informative)

# **Examples of sampling devices**

## A.1 Vertical sampler

A vertical water sampler system is shown in <u>Figure A.1</u>. This is a tube of 2 l to 5 l content with two closing lids, which uncover the complete cross-section area of the sampling tube while lowered in the water. The closing mechanism is triggered by a messenger.

The shown device is light protected by black foil, which is important for sampling for primary production assays. On top of the activator, the messenger is visible. A winch with steel cable is advisable for that sampling device.



Figure A.1 — Vertical sampler (Photo LBH, Germany)

## A.2 Horizontal sampler

Horizontal water samplers (also horizontal van Doorn samplers) have been developed to sample close to the bottom. The cylinder of this sampler is oriented horizontally, while its lids (or sealing balls) are kept open with elastics or springs and are closed by a messenger. Horizontal water samplers for use in flowing water should be equipped with steering fins to stabilize in flow direction. They also should have additional weight and reinforced spacers on the bottom of the cylinder in order to keep a certain distance to the bottom. A horizontal water sampler with these features is shown in Figure A.2.



Figure A.2 — Horizontal sampler (Photo Hydrobios Kiel, Germany)

## A.3 Hose sampler

The components are as follows:

- silicone hose with an inner diameter of at least 1,6 cm (not a rubber hose);
- rope:
- weight (nozzle made of stainless steel at the intake end of the hose).

The hose is equipped with a nozzle made of stainless steel serving as a weight at one end, such that it hangs vertically in the water column. The weight at the end of the hose should be mounted above the hose opening to ensure proper functioning. A rope should be attached to the weighted lower part (see Figures A.3 and A.4).

The weighted and open end of the hose is lowered slowly down into the water to the desired sampling depth such that it starts sampling from the surface. Hoses should only be used if they can be lowered exactly perpendicular to the water surface.

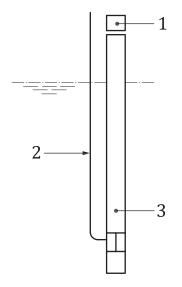
### ISO 5667-4:2016(E)

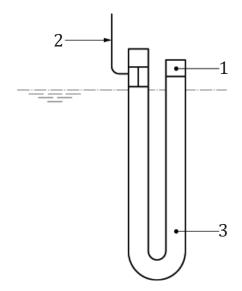
The open, upper end of the hose is tightly closed then with a cork and the lower end of the hose is slowly hauled up using the rope.

The hose should be raised slowly and water in it should be decanted into the mixing container.

After each sampling session, the hose and the mixing container should be thoroughly washed with tap water. All equipment should be thoroughly dried before being stored for future use. The hose should be hung up to dry. It is important to dry the equipment quickly to prevent algal and bacterial growth within the hose.

It should not be used for any other purpose than sampling phytoplankton and accompanying parameters (e.g. nutrients and chlorophyll-a).





- a) lowering of the hose
- b) lifting of the hose to collect the sample

#### Key

- 1 cork
- 2 rope attached to the weighted lower end of the hose
- 3 hose with weighted at the lower end

Figure A.3 — Principle of integrated sampling using a hose





Figure A.4 — Integrating sampling using a hose developed by University Cottbus Germany (Photos Hoehn, LBH, Germany)

## A.4 Tube integrating sampler

The Pauli sampling system (see Figure A.5) was developed as a simple approach to get large sampling volumes. It samples a complete water column in steps corresponding to the length of the tube (1 m or 2 m). The depth intervals are taken from the surface to the bottom of the lake. All aliquots are collected in a vessel (volume depending on the depth to be sampled) on the boat. Two solid rubber balls close the ends of the tube by an elastic band. The rubber balls are drawn from the tube ends and hang to the mounting clamps. In the desired sampling depth, the clamps are released by a messenger. In contrast, the Limnos sampler (see A.4.1) has closing flaps which are released by a messenger.

The content of the tube depends on the length and diameter of the tube.



 $Figure \ A.5 - Tube \ integrating \ sampler \ as \ originally \ developed \ by \ Pauli$ 

## A.4.1 Limnos tube sampler

The lids open vertically. The sampler sinks down while completely open. Closing mechanism is spring free and is triggered by messenger.

The metal parts are made of stainless steel. The container is made of impact-resistant polycarbonate plastic and the lids are made of POM plastic.

The sampler stands on its own base.

There is a wide choice of various volumes. The long models are particularly suitable for plankton sampling. The operation weight, depending on the volume from just under 2,0 kg to 3,5 kg. There is a wide choice of various volumes (Figure A.6, Table A.1).

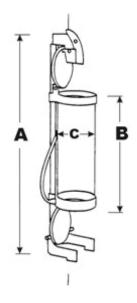




Figure A.6 — Limnos tube sampler

Table A.1 — Sampler dimensions

Sampler volume (l)	2,0	2,6	3,5	4,2	5,0	7,0	
Total height A (cm)	60	69	81	91	102	131	
Cylinder length B (cm)	29	38	50	60	71	100	
Tube diameter C (cm)	Outer/inner diameter 100/95						

### A.5 Mechanical integrating water sampler

This system was developed by UWITEC (Mondsee, Austria). The sampler takes integral samples of vertical water columns with 10~m or 20~m length. It samples 500~ml per metre of a water column of 10~m and 250~ml per metre of a water column of 20~m.

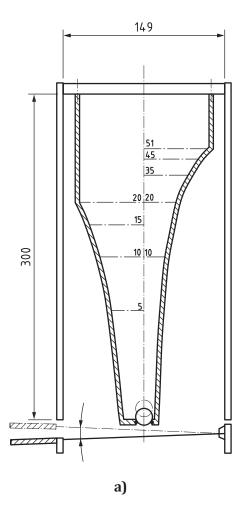
Operating mode: From its own weight, a small chain turns a cogwheel and this raises a piston inside the 5 L Plexiglas tube. The rising of the piston is proportional to the lowering of the sampler, and therefore, water continuously enters the sampler. Sampling can start at the surface or from any required depth operated from a winch and started by a messenger (see Figure A.7).



Figure A.7 — Mechanic integrating water sampler (Photo UWITEC Mondsee, Austria)

## A.6 Hydrostatic integrating water sampler

The sampler was developed in Reference [2] [see Figure A.8 a)] and produced by Züllig (Rheineck, Switzerland). A continuous water column from the surface to a maximum depth of 21 m can be sampled. The sampler consists of a glass cylinder, which is open on the lower end and closed on the upper end. Inside the cylinder is a glass funnel with a hyperbolic shape. When the sampler is slowly lowered (<1 m/s), water enters the funnel through an open valve. Due to the hyperbolic shape of the funnel, the water enters commensurately to the depth. The air inside the cylinder is compressed depending on the depth. When lifted quickly, the outside flowing water closes the valve and the sampled water remains in the funnel. To compensate for the buoyancy, the cylinder is built, mounted in a metal frame with a weight of 7 kg. The device samples 40 ml per metre [see Figure A.8 b)].





b)

Figure A.8 — Hydrostatic integrated water sampler (Photo Züllig Rheineck, Switzerland)

## A.7 Electronic integrating water sampler

The electronic integrating water sampler (IWS) has been developed by Hydro-Bios (Kiel, Germany). The desired depth range (start and final depth) is entered and stored in the sampler via a hand-held terminal. The recommended speed for lowering (slowly) is shown in the display. A winch with steel cable is advisable for that sampling device to ensure the required sinking velocity. The electronics will regulate if lowering is not constant, e.g. due to moving of the boat or swell. After reaching the final depth, the sampler can be lifted with a total content of 2,5 l or 5 l. Due to the freely selectable depth range, it is possible to get samples from a specific segment of the water column (see Figure A.9).



Figure A.9 — Electronic integrating water sampler (Photo Hydrobios Kiel, Germany)

## A.8 Grab samplers

The standard water sampler according to Ruttner (see Figure A.10), the UWITEC water sampler (see Figure A.11) and the Niskin water sampler (see Figure A.12) are examples for instruments to take water samples from any desired depth. They are lowered (while open) slowly into the water column, avoiding dynamic pressure and allowing them to rinse as they descend. Upon reaching the desired depth, a closing mechanism is activated and closes the lids of the sampling tube.



Figure A.10 — Standard water sampler according to Ruttner



Figure A.11 — UWITEC water sampler



Figure A.12 — Niskin sampler

## A.9 LIMNOS water sampler

The LIMNOS water sampler (see Figure A.13) is an instrument to take water samples up to 30 m depth. It consists of the holder device to hold two exchangeable 1 000 ml glass bottles as sampling containers. This device is lowered into the water in closed condition to prevent sample contamination by surface water. The sampler is activated by a messenger upon reaching the desired water depth. For transport and investigation of the samples, the glass bottles are unscrewed from the holder and closed with a lid. This avoids contamination of the sample by pouring it from one sample container to another.



Figure A.13 — LIMNOS water sampler

## A.10 Integrating water sampler

The integrating water sampler (see Figure A.14) takes integral samples of vertical water columns with a programmable depth interval. A microprocessor controls the sampling procedure according to the preselected scenario. Therefore, it is used for the acquisition of integrated water samples without repeated operations of grab samplers, followed by mixing procedures.



Figure A.14 — Integrating water sampler

# Annex B

(informative)

# Advantages and disadvantages of sampling equipment

Several types of sampling equipment can be used to perform the sampling operation, as described in Table B.1.

Table B.1 — Sampling equipment for sampling operation

Sampling equipment	Advantages	Disadvantages			
Integrated water samplers	<ul> <li>Homogeneous</li> <li>representative sample of the</li> <li>sampling area</li> <li>Inertness of the possible materials: PTFE, PVC coated with TPFE</li> </ul>	<ul> <li>Risk of contamination depending on the nature of the materials used</li> <li>Risk of contamination during the homogenization step on the boat</li> </ul>			
ipe or flexible hose sampling area Lower rick of contamination		<ul> <li>Difficult to use for sampling depths greater than 10 m</li> <li>Equipment not easy to clean</li> <li>Risk of contamination depending on the nature of the pipe used</li> </ul>			
Horizontal water samplers Vertical water samplers	<ul> <li>Sample is representative of the entire sampling area</li> <li>Inertness of the possible materials: glass or PTFE</li> </ul>	<ul> <li>Taking equidistant</li> <li>samples in the sampling area at</li> <li>different depths or position</li> <li>Risk of contamination during</li> <li>the homogenization step on the boa</li> </ul>			
Pump	Allows for variable volumes to be taken depending on analyses requested	<ul> <li>Taking equidistant</li> <li>samples in the sampling area at different depths or position</li> <li>Relatively long sampling time (renewal of water in the apparatus)</li> <li>Risk of contamination depending on the nature of pipes and components of the pump</li> <li>Risk of contamination during the homogenization step on the boat</li> </ul>			

# Annex C (informative)

# Sampling from boats — Maintaining station

#### C.1 General

When sampling in open water from a boat, it can be necessary to maintain a geostationary position in order to ensure that repeated collection activity remains relevant, for example, collecting a conductivity profile, and at the same position, taking samples at discrete depths. Depending on the size and type of vessel, various techniques can be applied to achieve the required positional integrity.

It can also be relevant in terms of repeat sampling from the same location, for example, when determining the impacts of seasonal changes in prevailing wind direction.

## C.2 Large vessels

For the purpose of this guidance, a large vessel is defined as one which can maintain a geostationary position with the use of auxiliary control mechanisms, such as computer controlled bow thrusters.

The ability to achieve the required positional accuracy will, to some extent, depend on prevailing weather conditions and the judgement of the vessel's master. It is important that all the relevant positional information pertinent to the vessel's operation, as well as the weather conditions, are recorded in the sampling documentation in order that the data user can extract the maximum value from the information provided by the sample.

#### C.3 Small vessels

For the purposes of this guidance, a small vessel is defined as one which cannot maintain its geostationary position with the assistance of computer controlled bow thrusters.

It is generally accepted that maintaining a geostationary position in a small vessel presents a greater degree of variability in terms of geo-positional repeatability. Various techniques can be deployed depending on the type of vessel, prevailing weather conditions and the objectives of the sampling plan. It is important that the technique employed is recorded with the sample records together with information relating to the accuracy of the geo-positional record. For example: the make and model of the geo-positional recording equipment; and a record of its claimed accuracy. Typically, this can include the number of satellites the device was able to use to triangulate its recorded position.

## C.4 Fixed point sampling

Where repeat sampling from a fixed point is required, and the only collection option is a small vessel, location marker buoys may be used.

NOTE The deployment of such buoys is subject to local regulations concerning navigation of the body of water being studied.

In all cases, if at any time the sample point is no longer required and the marker buoy is to be recovered, the sub-surface equipment should be recovered or rendered safe in terms of negating a risk to navigation or the fouling of motorized vessel propulsion.

## **C.5** Variable location sampling

Where it is not important to return to the same location on repeat occasions, but maintenance of a stationary position is required, various techniques may be deployed depending on the vessel's build and handling characteristics. The choice of technique should be governed by the safety considerations and the depth of the water body assessed by the vessel's master. The techniques, which can be available, include the following:

- a) deployment of a single point anchor system, which allows the vessel to rotate around the anchor point and align with the prevailing wind direction;
- b) deployment of a twin, or multiple, anchor line system, which reduces the ability of the vessel to align with the prevailing wind;
- c) deployment of a drogue to compensate for wind induced drift.

NOTE 1 A drogue is usually constructed to provide substantial resistance when dragged through the water, and is trailed behind the vessel on a long line. It will not hold a vessel on station but it can provide sufficient positional stability for one or two repeat sample recovery operations under some circumstances. It is not considered to be an ideal technique for geo-positional sampling from a vessel. However, it can be a realistic option when conditions are judged appropriate by the vessel's master. A typical drogue design is shown in Figure C.1.

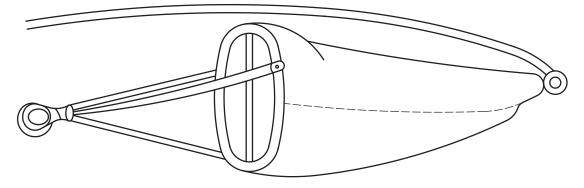


Figure C.1 — Typical drogue design

If an anchoring system is used to maintain a geostationary position, care should be taken with the timing and relative positioning of Secchi disk instrument.

NOTE 2 This is because anchoring a vessel will inevitably increase the risk of disturbing bottom sediments in the vicinity, locally influencing turbidity. The degree of any anchor-related impact on Secchi depth determinations will be largely influenced by the depth of the water body and prevailing wind conditions.

# **Annex D**

(informative)

# Example of a report — Sampling from lakes, natural and man-made

Sampling report for physical-chemical analysis from lakes, natural and man-made										
Project: Sample ID:						Date collected: Sampling time: Start: End:				
Location:				UTM-coordinates: Easting: Northing:			:			
Sample collect	or's name	e:								
Sampling meth	nod:	☐ Grab sample ☐ Depth profile samples ☐ Area profile samples				Depth of the lake: Withdrawal depth:				
		□ Cor	nposite sample egrated sample				Depths for mixed samples:			
Sampling equipment:		Integrated water samplers:  ☐ Pipe: ☐ Horizontal water sampler ☐ Vertical water sampler ☐ Pump			Samples with ship:					
Weather						In situ measurement data				
Day of samplin	ıg	Wind:								
□ Sunny □ Cloudy □ Changeable		Force		Ice cover: Ice thickness:		Turbidity and light by Secchi (cm):  Aquatic vegetation:				
☐ Rainy ☐ Hot ☐ Frosty		Air te	mperature:	Gauge level:			Submers/emers:			
				Rema	arks					
Date/signature	e	_								
			In s	itu measu	rem	ent data				
Sample ID Withdr dept		:h	Water temperature (°C)			Conduc (µS/cm a		Dissolved oxygen/% sat (mg/l/%)	Colour/ turbidity/ odour	
				Rema	arks					
Date/signature	e									

## Annex E

(informative)

## Continuous measurement of water samples with immersion probes

#### E.1 Precautions

For a correct measurement of pH or dissolved oxygen in unstable water samples, the following precautions for handling samples should be taken:

- no temperature variations;
- no degassing of carbon dioxide during the transfer of the sample and during the actual measurement;
- no precipitation of salts during the measurement (carbonate, etc.);
- slow agitation at the electrode (flushing without turbulence or bubbles).

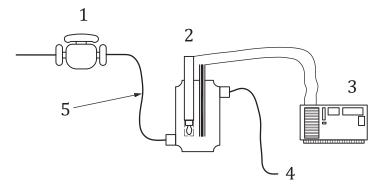
"Pseudo-continuous" satisfies the precautions described above and should be implemented as follows:

- a) continuous supply of the water to be analysed in contact with the electrode;
- b) no disturbances or air bubbles;
- c) use a beaker with bottom sample feed and continuous overflow as shown in <u>Figure E.1</u> and <u>Figure E.2</u>.

#### E.2 Measurement

- Ensure all technical aspects followed for each device (e.g. calibration and verification, temperature stability, maintenance and conditioning of electrodes).
- Make simultaneous temperature measurements.
- Allow the electrode response to stabilize (typically 2 min to 3 min).

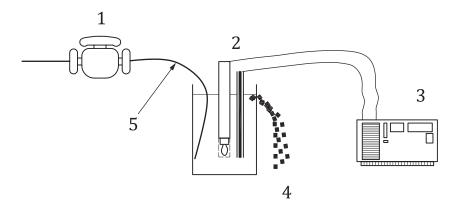
## E.3 Schemes for measuring the pH or dissolved oxygen in lake water



#### Key

- 1 tap sampling
- 2 measurement electrode and temperature sensor
- 3 pH or dissolved oxygen meter
- 4 towards sewer
- 5 flexible hose

Figure E.1 — Measurement with specific container



#### Key

- 1 tap sampling
- 2 measurement electrode and temperature sensor
- 3 pH or dissolved oxygen meter
- 4 towards sewer
- 5 flexible hose

Figure E.2 — Measurement in a beaker with overflow

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