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Qualité de l'eau

Échantillonnage

Partie 6 : lignes directrices pour l'échantillonnage des rivières et des cours d'eau

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Water quality — Sampling —

Part 6: Guidance on sampling of rivers and streams

Qualité de l'eau — Échantillonnage —

Partie 6: Lignes directrices pour l'échantillonnage des rivières et des cours d'eau

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

This second edition cancels and replaces the first edition (ISO 5667-6:1990), 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* [1)]

— *Part 2: Guidance on sampling techniques* [1)]

— *Part 3: Guidance on the preservation and handling of water samples*

— *Part 4: Guidance on sampling from lakes, natural and man-made*

— *Part 5: Guidance on sampling of drinking water and water used for food and beverage processing*

— *Part 6: Guidance on sampling of rivers and streams*

— *Part 7: Guidance on sampling of water and steam in boiler plants*

— *Part 8: Guidance on 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*

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1) ISO 5667-1 and ISO 5667-2 are currently undergoing joint revision, which will be published as ISO 5667-1.

— *Part 13: Guidance on sampling of sludges from sewage and water-treatment works*

— *Part 14: Guidance on quality assurance of environmental water sampling and handling*

— *Part 15: Guidance on preservation and handling of sludge and sediment samples*

— *Part 16: Guidance on biotesting of samples*

— *Part 17: Guidance on sampling of suspended sediments*

— *Part 18: Guidance on sampling of groundwater at contaminated sites*

— *Part 19: Guidance on sampling of marine sediments*

Introduction

An understanding of the purpose of sampling is an essential prerequisite to identifying the principles to be applied to a particular sampling problem. Examples of the purposes of sampling programmes commonly devised for rivers and streams are as follows:

a) to determine the suitability of the water quality of a river or stream within a river basin for a particular use, such as:

   1) a source of drinking water,

   2) for agricultural use (e.g. all types of irrigation, live-stock watering),

   3) for the maintenance and/or development of fisheries,

   4) for amenity use (e.g. aquatic sports and swimming);

b) to assess the impact of human activities on the quality of water, such as:

   1) to study the effects of waste discharge or accidental spillages on a receiving water,

   2) to assess the impact of land use on river or stream quality,

   3) to assess the effect of the accumulation and release of substances including contaminants from bottom deposits on aquatic biota within the water mass, or on bottom deposits,

   4) to study the effects of abstraction, river regulation and river-to-river water transfers on the chemical quality of rivers and their aquatic biota,

   5) to study the effects of river engineering works on water quality (e.g. addition/removal of weirs, changes to channel/bed structure).

Water quality — Sampling —

Part 6: Guidance on sampling of rivers and streams

1 Scope

This part of ISO 5667 sets out the principles to be applied to the design of sampling programmes, sampling techniques and the handling of water samples from rivers and streams for physical and chemical assessment.

It is not applicable to the sampling of estuarine or coastal waters and has limited applicability to microbiological sampling.

NOTE Procedures for microbiological sampling are given in ISO 19458.

This part of ISO 5667 is not applicable to the examination of sediment, suspended solids or biota.

In cases where naturally occurring or artificially constructed dams result in the retention or storage of water for several days or more, it might be better to consider the stretch of the river or stream as a standing water body for sampling purposes. ISO 5667-4 provides guidance for sampling in these circumstances.

WARNING — The focus of this part of ISO 5667 is the collection and integrity of water samples. The collection of these samples can be hazardous and attention is therefore drawn to the existence in some countries of legislative requirements for the safety of personnel.

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 5667-18:2001, *Water quality — Sampling — Part 18: Guidance on sampling of groundwater at contaminated sites*

ISO 6107-2:1997, *Water quality — Vocabulary — Part 2*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 5667-18 and ISO 6107-2 and the following apply.

3.1
automatic sampling
process whereby samples are taken either discretely or continuously, independently of human intervention, and according to a predetermined programme

[ISO 6107-2:1997]

3.2
incremental sampling
technique in which small samples are taken because of a low flow rate (with the possibility of contamination by bottom deposits) or because of restricted access (e.g. where a sample is obtained through a small aperture), these small samples then being aggregated to form a composite sample

NOTE All the liquid contained in the small samples is used, unlike blending of aliquots used to make a flow-proportional sample (see 8.4).

3.3
isokinetic sampling
technique in which the sample from a water stream passes into the orifice of a sampling probe with a velocity equal to that of the stream in the immediate vicinity of the probe

[ISO 6107-2:1997]

3.4
light non-aqueous-phase liquid
LNAPL
organic compounds which have low water solubility and a density less than that of water, e.g. petroleum products

[ISO 5667-18:2001]

3.5
random sampling
form of sampling whereby the chances of obtaining different concentration values of a determinand are precisely those defined by the probability distribution of the determinand in question

3.6
river
natural body of water flowing continuously or intermittently along a well-defined course into an ocean, sea, lake, inland depression, marsh or other watercourse

[ISO 6107-2:1997]

3.7
sampling site
general area within a body of water from which samples are taken

[ISO 6107-2:1997]

3.8
sampling point
precise position within a sampling location from which samples are taken

[ISO 6107-2:1997]

3.9
stream
water flowing continuously or intermittently along a well-defined course, as for a river, but generally on a smaller scale

[ISO 6107-2:1997]

3.10
sub-sample
portion removed from a sample and intended to be representative of that sample

3.11
systematic sampling
sampling whereby the samples are taken at predetermined intervals; often equally spaced in time

4 Design of sampling programme

4.1 Sampling point selection

4.1.1 General

The following factors are usually considered in advance of the sampling event. Practical sampling issues, such as accessibility, may make the ideal sampling point impractical. Any change to the designated sampling point on any grounds should be discussed and agreed with the sampling programme originator. The outcome of the deliberations may be recorded in a sampling point file which contains directions to the sampling site, the detailed location of the sampling point, the method of sampling and specific details (e.g. keys required and health and safety issues). It may differentiate between equivalent sampling points that may be used if, for instance, river conditions change. It may also specify the type of sampling to be carried out, e.g. the depth to sample.

4.1.2 Choice of sampling site

In choosing the exact point from which samples are required, two aspects are generally involved:

a) the selection of the sampling site (i.e. the location of the sampling cross-section within the river basin, river or stream);

b) the identification of the precise point at the sampling site.

The purpose of sampling often defines sampling sites (as in the case of the determination of the quality of an effluent discharge), but sometimes the purpose only leads to a general idea of the sampling site, as in the characterization of quality in a river basin.

The choice of sampling sites for single sampling stations is usually relatively easy. For example, a monitoring station for a baseline record of water quality can be chosen to permit the use of a convenient bridge, or to allow an upstream effluent discharge or tributary to be well mixed laterally before the station. Stations for monitoring water supply abstraction points might need to be fixed within narrow limits (i.e. in proximity to the abstractions).

In regions that receive seasonal rainfall only, and that have long periods without rain, river volumes and flows can vary tremendously, and sampling sites for regular use should be chosen so as to ensure that they remain appropriate and practical for sampling during periods of both maximum and minimum flow.

Where it is necessary to carry out sampling through ice in winter, the chosen sampling site should be as close as possible to the sampling site used during other seasons of the year. If sampling is to be carried out near a bridge, the site should be located far enough upstream to avoid contamination from road salt and sand. Deviations from the routine sampling point should be detailed as part of the dataset and recorded with the analytical results.

4.1.3 Importance of mixing

When the effects of a tributary, or an effluent, on the quality in a particular identified stretch of river or the main stream are of interest, at least two sampling sites should be chosen; one should be just upstream of the confluence and the other should be sufficiently far downstream to ensure that mixing is complete.

The physical characteristics of the channels of watercourses largely control distances required for the complete mixing of effluents with stream flow.

Effluents mix in three dimensions in a stream, namely:

a) vertically (from top to bottom);

b) laterally (from one side to the other);

c) longitudinally (levelling out of peaks and troughs in the concentration of effluent constituents as water passes downstream).

The distances over which effluents mix in these three dimensions should be considered in the selection of sampling sites and points and are affected by, amongst other things, the water velocity. Tracer techniques using dyes can be useful in studying mixing processes and conductivity measurements can also be helpful.

NOTE The use of tracer techniques might be subject to licensing by the authority responsible for the watercourse, as there might be concerns over the release of chemicals into the environment. Where this is the case, it might be better to use determinants already present, such as pH, temperature or conductivity, to study mixing processes.

Effluents discharged into most streams mix vertically completely within a kilometre. Normally a stream need not be sampled at more than one depth, although stratification can be induced in slow-moving rivers and streams by thermal and other density effects. In these cases, sampling at several depths might be necessary and preliminary tests should be carried out to assess the degree of stratification (see 4.2 for guidance).

The distance necessary for complete lateral mixing is generally dependent on the occurrence of relatively sharp reverse bends, islets or boulders and is measured in kilometres rather than fractions of a kilometre. Therefore, to obtain representative samples a stream should be sampled at two or more points across its width at sites downstream from an effluent or tributary discharge.

Consideration of longitudinal mixing distances can be important in deciding on the frequency of sampling. To give representative results just below an irregular discharge, more frequent sampling will be required than would be necessary some distance downstream where longitudinal mixing has been completed to a greater extent.

The distance in metres, l, for complete mixing, to within 1 % of complete homogeneity, should be calculated approximately using the following formula (originally published in ISO 555-2):

$$l = \frac{0{,}13b^2c(0{,}7c + 2\sqrt{g})}{gd}$$

where

b is the average width of the reach, in metres;

c is the Chezy coefficient for the reach ($15 < c < 50$);

g is the acceleration due to gravity, in metres per second squared;

d is the mean depth of the reach, in metres.

The following example gives an illustration of the effect of different Chezy coefficients on the longitudinal mixing of a stream.

EXAMPLE Consider two streams both 5 m wide and 1 m deep but with extreme values of the Chezy coefficient; one of 15 (very rough bottom, i.e. the stream is very fast and turbulent) and the other of 50 (very smooth bottom, i.e. a very tranquil, slow-moving stream). When calculated in accordance with the equation given in this subclause, the former will have reached complete homogeneity after 83 m while the latter will not be homogeneous until it has travelled 683 m.

It should be noted that some tests have shown that the above expression can underestimate the mixing length for small streams of about 5 m in width and overestimate the mixing length for rivers of over about 50 m in width. This is most likely due to the fact that the average width, average depth and Chezy coefficient are

usually estimates. Lateral mixing can take place much more slowly than expected and vertical mixing more quickly. There are many literature sources containing alternative calculations that deal with mixing distances (see Reference [15]).

4.1.4 Consideration of time-of-travel data

Time-of-travel data can often be of relevance to the choice of sampling location. For example, sampling sites might have to be arranged to allow certain constituents or pollutants to be traced through a system, particularly from a discrete source of pollution. This necessitates knowledge of the residence time within the system under investigation (i.e. the time of travel). Knowledge of the time of travel is also important in sampling studies to investigate the rate of change of unstable constituents (e.g. in the self-purification of a water body, the time of travel can provide information on kinetic rate coefficients).

In determining the time of travel, one of the three principal methods should be used, namely the use of surface floats (see ISO 748), the use of tracers (originally published in ISO 555-1, ISO 555-2 and ISO 555-3) or the measurement of flow rate with knowledge of cross-sectional areas (see ISO 748 and ISO 1070).

Measurements should be made at a minimum of five different flow rates and the resulting times of travel plotted against the corresponding flow rates, thereby enabling other travel times to be obtained by extrapolation or interpolation. However, extrapolation outside 10 % of a measured flow rate value can provide inaccurate information on time of travel.

It should also be noted that time of travel can vary greatly between seasons in regions that experience seasonal rainfall only.

ISO 5667-1 should be consulted for general guidance on time of travel and ISO/TR 8363 should be consulted for guidance on the measurement of liquid flow in open channels.

4.1.5 Non-homogeneous sites

Problems arise in selecting suitable sampling sites whenever the determinands are not homogeneously distributed throughout the water body of interest. In general, such sampling sites should be avoided, except when the sites themselves are of direct interest, as they might not yield representative samples of the major part of the water body. If there is any possibility of a non-homogeneous distribution of the determinands of interest at the chosen site, experimental tests on the nature and magnitude of any heterogeneity in all three dimensions should be made. If such tests show that the determinands are distributed homogeneously, any sampling point will suffice. Otherwise another site should be sought where the determinands are homogeneously distributed. If it is impossible to find such a sampling site, samples should be taken from sufficient points at the chosen site to ensure representative results.

These samples can often be combined as sub-samples to form one single composite sample representative of the quality at the sampling location, so that it is not necessary to analyse individual samples taken from each of the sampling points. However, this provides no information on the variability in quality between the sampling points. In addition, the combination of sub-samples in this way cannot be undertaken when sampling for dissolved gases or other volatile constituents.

4.2 Frequency and time of sampling

It is essential that the sampling programme be properly statistically designed in order that the statistical summary information produced from the analytical results provides an estimate of the required information to within the tolerance limits of the programme's objectives. If the objectives do not include a definition of the magnitude of the tolerable error, a statistically based sampling programme is impossible. Guidance and recommendations on the application of statistics to sampling frequency are given in ISO 5667-1.

Where cyclic or other persistent variations are present, better precision should be sought in estimating mean concentrations by systematic rather than by random sampling (for any given number of samples), provided that the sampling interval is short enough for consecutive samples to reveal the variations.

When using systematic sampling, it is essential to ensure that the frequency of sampling does not coincide with any natural cycle present in the system, or with some other time-based effect (e.g. a pump just upstream starting once an hour), a study of the effects of which are not part of the sampling objectives.

In river systems, regular cyclic variations in water quality can occur with, for example, periods of one day, one week and one year. When these occur, sampling times should be carefully chosen to assess the nature of these variations. If these variations are not persistent or if the amplitude is appreciably smaller than random variations, it will usually be adequate to choose the sampling times randomly, or alternatively in a systematic manner with samples evenly distributed throughout the period of interest. It is important when systematic sampling is undertaken over a long period that sampling programme designers take account of possible changes in local time throughout the sampling period. In all other cases, the times should be chosen so that different parts of the cycle are sampled, unless the extreme concentrations are of interest, when samples should be taken at the corresponding times of each cycle. Further guidance on these issues is given in ISO 5667-1.

If the sampling programme is designed to detect trends in water quality, care should be taken during its design to ensure that all variations of interest are detected. These temporal surveys show the changes in the chemical and/or physical conditions of the river or stream due to contamination or natural variation over time. The surveys should be carried out using fixed sampling points and using standardized methodology in accordance with an established programme. This might require samples to be taken at the same time, the same day or same month, depending on the possible duration and flushing rate of the trend under investigation.

All sampling equipment and procedures should be documented and any field observations and measurements recorded on appropriate field sheets or in an appropriate log book in order to facilitate accurate repeat surveys in accordance with the temporal scale on which the surveys are being undertaken.

5 Preparation for sampling

River sampling often involves the sampling operative working in isolation for the majority of the day; therefore both the sampling operative and his/her vehicle should be self-contained. All sampling staff should be properly trained and receive clear sampling instructions which may be in the form of a sampling folder/manual containing details of each sampling site including the above items together with a description of the sampling site, a site plan and information about any special features of the sampling site (e.g. key holders, safety precautions).

The following information should be available as a minimum:

a) a precise description of and documentation on the sampling point;

b) the type of sample required;

c) the applicable sampling techniques;

d) information, if necessary, about any sub-samples, e.g. bottles, filtration, preservation, or any field measurements, etc.;

e) the order of filling bottles to minimize contamination.

Many of the considerations taken into account for storing equipment at the depot apply to the sampling vehicle as well (see 8.6 and 10.2). If necessary, a unit that will keep samples between 1 °C and 5 °C for transportation should be available in the vehicle. The vehicle should be fitted with racks to hold the equipment and prevent any movement that might cause a breakage. This is especially important for glass bottles, bottles with preservatives and hand-held meters.

6 Sampling at specific locations

6.1 Sampling from bridges

When selecting the place on a bridge from which to take a sample, ensure that

a) there is a sufficient depth of water to submerge the sampling container;

b) when submerged, the container will not disturb bottom deposits;

c) there is sufficient clearance on the bridge when suspending the container to avoid dislodging potentially contaminating material from the bridge structure;

d) when sampling on the upstream side of the bridge, the sampling operative does not become un-sighted, i.e. the container is not carried under the bridge by the current.

If the depth of water is insufficient, select the most appropriate alternative sampling approach (see 8.3, 8.4 and 9.4). It might be possible to use a small sampling vessel on an extension pole if there is insufficient depth of water to use a vessel on a rope.

6.2 In-stream sampling

In all cases, and in particular where sampling can be a source of contamination or loss of determinand (e.g. pesticides, oils or trace metals), bottles should preferably be filled directly from the body of water to be sampled. This same technique should also be employed at the discretion of the sampling operative where small numbers of sub-samples are to be taken (see 9.6 and 9.7). Care should be taken to avoid sample contamination by disturbance of either the bed or the bank of the watercourse.

6.3 Sampling from the bank side

Where a sample has to be obtained from the bank side, care should be taken to avoid sample contamination by disturbance of either the bed or the bank of the watercourse. Usually, an extension pole will be required, but often a vessel on a rope is used.

6.4 Sampling from craft

When sampling from a boat, care should be taken to avoid contamination of the sample with disturbed deposits and any discharges from the boat. A properly maintained boat that is appropriate for the work should be used. The staff and crew should be properly trained.

Attention is drawn to the existence in some countries of legislative requirements for the safety of personnel and craft.

6.5 Sampling under ice

The winter sampling location should be as close as possible to the one used during other seasons of the year. If an alternative sampling point is chosen because of ice, this should be mentioned in the sampling report. If any ice safety concerns exist, samples should be collected from an alternative sampling location.

7 Sampling methods

7.1 Single, discrete samples

In cases where sub-surface sampling (e.g. within 25 cm of the water surface) is acceptable, it is often sufficient to immerse a container (e.g. a bucket or can) in the river or stream of interest. The contents are then poured into appropriate sample bottles. Alternatively, the sample bottles or containers can be directly

immersed in the river or stream. However, sampling of surface films should be avoided, unless these are particularly required for analysis.

7.2 Sampling from specific depths

When a sample is required from a specific depth, special sampling equipment, which is lowered into the water to enable a single sealed or continuous sample to be taken from the chosen depth, should be used (see ISO 5667-2). This may be in the form of bottles fitted with an opening mechanism to remove the stopper at the required depth or devices that draw a sample into the bottle via an inlet suspended at the required depth.

Continuous sampling systems for rivers should be carefully selected and installed to avoid blockage of the inlet by debris in the water. Surrounding the inlet with both a coarse and fine mesh should protect it, but frequent inspection and removal of accumulated debris can be required and these factors should be borne in mind when selecting the sampling point. Sampling systems at exposed locations (e.g. on river banks) might need protection from vandalism and effects such as extremes of water level and temperature (freezing).

If the rate of pumping is very slow, the effect of gravity on suspended solids can reduce their concentration in the sample. If suspended material or determinands that may adsorb onto it are being investigated, slow pumping rates are not recommended. This often precludes the use of the low-powered peristaltic pumping systems common to many automatic sampling machines. Ideally, sampling should take place under isokinetic conditions but, where this is impracticable, the linear flow velocity within the intake tube should not fall below 0,5 m/s nor exceed 3,0 m/s.

The aim should be that the concentration of determinands in the sample and the main body of water should not be significantly different.

For representative sampling of insoluble materials, the rate of sampling should be adjusted so that the velocity of water in the inlet of the sampling system is the same as that of the water being sampled (i.e. sampling should take place under isokinetic sampling conditions). This also requires that the inlet of the sampling system faces the direction of the river or stream flow.

Where there are significant variations in water level, sampling is facilitated by mounting the sampling system or inlet on a floating platform; however, a floating platform can be vulnerable to damage. Alternatives include the use of submerged inlets suspended from floating buoys (or similar devices) where the floating inlet is connected to the sampling device via flexible tubing anchored to weighted blocks set in the river bed. A more costly but permanent arrangement is to connect the sampling device to a permanent multi-point inlet which enables samples to be taken at the most suitable depth for the particular sampling purpose.

8 Sampling equipment

8.1 Single, discrete samples

Samples are frequently collected directly into laboratory bottles as this method is perceived to be the least contaminating. Where this is not possible, samples should be collected indirectly using open-mouthed vessels.

Before any sampling equipment is used, tests should be performed to show that its use has no effect on the determinand to be analysed. In some cases, for example if samples are required from under ice or when analysis might be compromised by using an indirect method (e.g. for trace organic analysis), various pieces of equipment are available into which the bottles can be fitted and then lowered into the river.

To ease the collection of samples, a range of sampling vessels from 50 ml to 3 l can be employed. In order to achieve the analytical detection limits often required for clean rivers, even larger volumes of sample may be necessary and mechanical handling issues might arise.

Vessels can be lowered by means of a rope or by flexible wire covered in polytetrafluoroethylene (PTFE) or polyethylene. Any material that does not affect the determinand may be used. If the sample is to be taken from a bridge, a small length of stainless-steel chain can be used to connect the wire or rope to the sampling

vessel in order to aid the submersion of the vessel and help prevent contamination. Further information on sampling material is given in ISO 5667-2 and ISO 5667-3.

If the use of a rope may lead to insufficient control of the sampling position, a sampling pole may be used. Poles may be fixed or extending in nature and have either the sampling bottle itself or sampling equipment clamped at the end.

If samples from rivers of varying quality are to be taken or different analytical detection limits are required, it might be necessary to carry different sets of sampling equipment to prevent cross-contamination. In extreme cases, this might require one set of sampling equipment per site.

In cases when the sample should not include the surface layer, two simple alternative processes are available. If it is possible to enter the water safely, a small-mouthed bottle can be lowered to 25 cm below the surface before the stopper is removed. Alternatively, an open bottle can be fixed upside down on a pole, lowered to the required depth, the pole rotated through 180° and the bottle allowed to fill.

8.2 Sampling of surface layers for LNAPL (e.g. oils) or surface films

A wide-mouthed vessel should be used for sampling surface layers. The sampling vessel should be controlled either by hand or using a pole, but not by means of a rope as it is not possible to control the sampling vessel at the surface.

8.3 Devices for sampling from specific depths

In situations where it is essential to sample at specified depths below the surface (or where sampling for dissolved gases), it is essential that specialized sampling devices are used. Guidance and recommendations on the use of such devices are given in 8.4 and ISO 5667-2.

NOTE Bottles or other sampling equipment used for single discrete samples can also be used providing they are fitted with an opening mechanism to remove the stopper at the required depth.

8.4 Automatic sampling devices

Automatic sampling devices can be used in many river and stream sampling situations, since they enable a continuous sample or series of samples to be collected without manual intervention. They are particularly useful in preparing composite samples in situations where samples need to be taken to study variations in river quality with time.

The choice of the most suitable type of machine will depend on the particular sampling situation. For example, sampling in order to estimate the average load of dissolved trace metals in a river or stream might best be carried out using a continuous flow-proportional device, utilizing a peristaltic pumping system.

In all cases, the sampling machine should be tested to ensure satisfactory performance in the situation being investigated.

Simple automatic machines can be programmed to take samples at pre-set time intervals or be operated by an external trigger such as a signal generated by excessive rainfall. More refined flow-proportional machines continuously measure the flow in the river or stream and take samples after a fixed volume of water has passed the sampling point.

It is essential that the automatic sampling machine, or the storage time and conditions of the samples within it, do not result in any significant deterioration. Information on preserving samples is given ISO 5667-3.

Further guidance and recommendations on automatic sampling machines and their use is given in ISO 5667-2 and Reference [14].

8.5 Other sampling equipment

If filtering is required in the field, suitable equipment should be carried and the laboratory can advise on the specification for filtering equipment (see 10.1 and ISO 5667-3).

At some locations, it is necessary to take samples through thick ice during winter. This requires specialized equipment such as an auger or ice drill.

8.6 Provision of storage for sampling equipment and of samples prior to delivery to the analysing laboratory

If necessary, storage will have to be provided for sampling equipment and bottles. Facilities will have to be available so that all sampling equipment can be kept clean. Contamination will have to be prevented at all times.

New or cleaned bottles should not be stored near those containing preservative.

If the samples have to be stored prior to submission to the laboratory, suitable facilities (which may be co-located with the empty-bottle storage) will have to be provided so that the integrity of the samples is not compromised.

The stability and integrity of the samples is of paramount importance.

A refrigerator should be provided for the storage of samples for up to 24 h. If relatively clean river samples are stored with heavily contaminated ones, either two fridges should be provided to keep such samples separate or the clean and contaminated samples should be kept in separate lidded crates within the same fridge. The refrigerator should be capable of achieving a temperature of 1 °C to 5 °C whatever the ambient temperature. If only one refrigerator is supplied and microbiological samples need to be stored together with chemical ones, it should be capable of maintaining the temperature in the range 2 °C to 5 °C (procedures for microbiological sampling are given in ISO 19458). Maintaining this range is often beyond the ability of domestic fridges.

All preservation steps should be recorded in the report and the storage temperature measured and recorded.

Further guidance and recommendations on the storage of samples are given in ISO 5667-3.

9 Taking the sample

9.1 Arrival on site

If the site procedures so require, the sampling operative(s) should identify themselves to the site management and follow their safety instructions. The sampling site should be confirmed using the information contained in the sampling folder/manual (description, pictures, coordinates, etc.) to make sure that it is the correct location. Global positioning system (GPS) equipment may be helpful as it allows quick and accurate positioning.

9.2 Rinsing the equipment

All the equipment that comes in contact with the water should be rinsed. Take sufficient volume of the body of water to be sampled for a thorough rinsing of all the equipment, using the sampling technique being used at the site. If using a rope, pour some of the contents of the vessel over the final metre of the rope (including the chain, if used) 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 to be re-contaminated by e.g. allowing it to come in contact with the ground. Similarly rinse the end of the sampling pole if used. If, but only if, the laboratory instructions require the sample bottles 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 polythene bag.

Rinse the bottle by filling it with sufficient sample and rotating it to cover all the internal surfaces. Dispose of the rinse water downstream of the sampling site or in such a way that it does not contaminate or disturb the site to be sampled. Disposal of rinse water or excess bulk sample should not itself present a source of pollution. Do not re-stopper the bottle until the sample has been taken unless aerial contamination is likely.

9.3 Direct sampling

Direct sampling provides the minimum risk of contamination while ensuring a representative sample. However, it should not be employed with bottles containing preservatives. Direct sampling should only be used when it is considered to be safe and non-hazardous. Prior to direct sampling, the bottles should be rinsed as described in 9.2.

Enter the body of water to be sampled, face upstream towards the flow of the water, remove the cap of the bottle (if still in place) and retain this in one hand. Plunge the neck of the open bottle under the surface of the water until it is submerged to a depth of about 25 cm. If there is little depth of water, ensure that the sample will not be contaminated by bottom sediment.

Tilt the neck of the bottle such that it points slightly upwards towards the surface and towards the flow. Allow the bottle to fill as needed. In most cases, fill the bottle right to the top to exclude air, as gas exchange might rapidly change the quality of the sample. In some cases, such as when a solvent is added directly to the bottles, as in oil analysis for instance, the bottle should only be filled to the shoulder. Guidance on the filling level of the bottle will be given by the laboratory. When as full as needed, remove the bottle from the water and replace the cap securely. Return to the shore and label the bottle as detailed in 9.9. If sampling has to be directly into a bottle, then it is possible to secure the bottle in a "cage" similar to that used for sampling at specific depths or through ice.

9.4 Indirect sampling using a sampling vessel

Gently lower the sampling vessel to the surface of the water, ensuring the vessel is not contaminated on the descent. Allow the vessel to fill, keeping it in view at all times. Try not to collect a high proportion of liquid from the surface and try to avoid any floating material. Do not let the vessel contact the river bottom. Remove the vessel from the water, again ensuring no contamination occurs.

A pole will give better control, so contamination from the bottom and floating objects can be more easily avoided but, as the volume collected can be much less than with a rope and large vessel, many aliquots can be needed. These aliquots may be used to produce a bulk sample before filling each sample bottle (see 9.7).

Pour the sample carefully into any bottle required, either directly or using a funnel and making sure no sediment has time to settle out. If preservatives are present, ensure that overfilling the bottle does not cause contamination to the watercourse. Stopper the bottles and label as detailed in 9.2 and 9.9.

9.5 Sampling through ice

Clear loose ice and snow from around the sampling location, and drill through the ice with an auger or ice drill. Ensure the area around the hole remains clean and free of potential contamination (gas, dirt from drill and boots, snowmobile exhaust, etc.).

Remove all ice chips and slush from the hole, using a plastic sieve. Wait several minutes for the water to flow freely under the ice, allowing potential contaminants to clear before taking the samples. Take the sample from well below the lower layer of ice.

9.6 Sampling of surface layers or films

Sampling can be achieved either by entering the watercourse or by using a sampling pole. If a bottle is to be used directly, remove the stopper and store as described in 9.2. Arrange for the sampling vessel or bottle to face upstream and lay it so that it is horizontal to, and slightly below, the surface of the water, such that half the mouth of the bottle is submerged, and allow the bottle to fill so that it contains a proportion of the surface layer. Remove the vessel from the water as soon as it is full. If it is allowed to overfill, there is a chance that the surface layer might be displaced. Alternatively, or if sampling a thin oil film, the proprietary sampling machines described in ISO 5667-2 can be used.

9.7 Sampling by increments

In conditions of low river flow or where the source of water is difficult to access, a sample can be prepared from small volumes by using small vessels and transferred into a suitable-sized bulk bottle. Care should be taken not to contaminate any of the increments. When there is sufficient volume in the bulk sample, the contents can be transferred homogeneously (using constant swirling) to the individual sample bottles. For the sample to be considered a "single, discrete one", the total time for all the aliquots to be taken should be such that no change in the river composition would be expected. If this is not known, the time for all aliquots to be taken should be less than 5 min.

9.8 Adding preservatives in the field

The preservation of certain types of sub-sample is required in the field. Some sub-sample bottles contain preservatives and others should have the preservative added at the time of sampling, e.g. when sampling for dissolved oxygen. Reference should be made to ISO 5667-3 and specific analytical standards for information on preservation of samples. Follow any specific manufacturer's instructions for adding the preservatives and take care not to contaminate the inside or outside surfaces of any funnel with the preservative. The funnel should be rinsed thoroughly both inside and out with a quantity of the sample before using again.

9.9 Labelling

Samples should be labelled as described in 11.2 at the time of collection and before going on to the next sampling site. For further detail on issues of traceability, chain of custody, quality systems and registration, see 10.3.2.

10 Stabilization, transport and storage of samples

10.1 Stabilization

The stability and integrity of the samples is of paramount importance.

Samples which cannot be delivered to the laboratory within a day should be stabilized or preserved in accordance with the provisions of ISO 5667-3 and appropriate analytical standards. If there is a choice of preservation technique, then the laboratory should be informed which has been used.

The following specific guidance should be noted.

For some applications, sampling will be concerned with an assessment of soluble species (e.g. trace metals in river water). If this is the case, then it is necessary to separate the "dissolved" from the "particulate" material as soon as practicable after sampling (i.e. preferably at the sampling site before transportation to the laboratory). This minimizes changes in composition that can otherwise occur after the sampling operation, but before any subsequent laboratory pre-treatment or analysis. Several techniques are available, but the most convenient for use in the field (i.e. outside the laboratory) is filtration, details of which are presented in ISO 5667-3.

See 8.6 for details of intermediate storage conditions.

All preservation steps should be recorded in the report and the storage temperature measured and recorded.

10.2 Transportation

General guidance on transport, stabilization and storage of samples is provided in ISO 5667-3. However, the following specific guidance should be noted.

Store equipment and sub-samples in the vehicle in a safe and secure manner and in such a way that cross-contamination between heavily contaminated sub-samples and equipment and "clean" sub-samples is prevented, e.g. in separate lidded crates. All samples should be stored in the dark.

When applicable, the samples should be cooled. The vehicle should preferably be fitted with a refrigerator (cool-boxes can be used, but they are not efficient and effectively are only suitable for preventing temperature rise).

Samples which cannot be delivered to the laboratory within a day should be stabilized or preserved in accordance with the provisions of ISO 5667-3 and appropriate analytical standards. If there is a choice of preservation technique, then the laboratory should be informed which has been used.

All preservation steps should be recorded in the report and the storage temperature measured and recorded.

10.3 Security and traceability of samples during storage and delivery

10.3.1 Routine samples

The sampling operative has a responsibility towards the security and traceability of any samples, sub-samples and sample registration documents in their care.

The sampling operative should check that samples, sub-samples, labels, sample registration documents, etc, are undamaged and deposited in the designated place. If any bottles are lost, damaged or broken in transit, this should be recorded by the sampling operative on the sample registration form. Similarly, the courier should make a similar record while the samples are in his/her care. The courier should deliver the samples in accordance with laboratory instructions, especially if delivery takes place when the laboratory is unmanned.

10.3.2 Samples which might be used for legal purposes

Rules which should be followed if samples are to be used for legal purposes can be much more onerous, depending on the legal system operating in a particular jurisdiction.

NOTE Attention is drawn to the existence in some countries of national legislation, with which all persons involved at any stage in the sampling, storage or delivery of samples, or in the associated documentation, should be thoroughly familiar.

11 Quality

11.1 Avoidance of contamination

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

NOTE Some sources of contamination and their 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 the sample and prevent sample contamination.

Examine each sample or sample bottle for large particles such as leaves or detritus. If these are observed, discard the sample and collect a new one.

IMPORTANT — In all cases if contamination is seen, known or suspected to have occurred by any route, the sample should be discarded and sampling repeated. However, if it is not possible to take a sample without contamination from sediment, decant the sample immediately and record the procedure on the sample container.

11.2 Sample identification and records

All sampling equipment and procedures should be documented and recorded on an appropriate field sheet/in a logbook in order to facilitate accurate repeat surveys in accordance with the temporal scale on which the surveys are being undertaken.

The statistical power of the sampling for trend data should be robust and suited to the requirements of the study.

Sample containers should be clearly and unambiguously identified, so that subsequent analytical results can be properly interpreted. All details relevant to identification of the sample should be recorded on a label attached to the sample container.

Where the samples are identified through a pre-printed label with the site details and a unique machine-readable code, duplicated on both sample label and laboratory sample registration document, fewer details need to be recorded. Only details that can change, such as date, time and perhaps operative's identification (which may be in the form of a signature), will be required.

No further samples should be taken until all sub-sample bottles have been labelled.

12 Reports

12.1 Analytical reports

The detailed form of the sampling report will depend on the objectives of sampling. All conditions that may influence the analytical results should be noted. Matters that could be considered for inclusion are:

a) the name of the river or stream;

b) the sampling point (i.e. the sampling position in the cross-section at the sampling site);

c) the date and time of sample collection;

d) the name of the sample collector;

e) the weather conditions at the time of sampling (including air temperature) and/or immediately prior to sampling (e.g. amount of rainfall, cloud, sunshine);

f) the appearance, condition and temperature of the water body;

g) the flow condition of the water body (it can also be useful to record any marked variations in flow prior to sampling);

h) the appearance of the sample (e.g. the colour of the water and suspended solids, clarity, nature and amount of suspended solids, odour);

i) the type of sampling device used;

j) information on any sample preservation technique used;

k) information on any sample filtration technique used;

l) information on any sample storage conditions.

12.2 Sampling protocols

A "history" of changes to sampling protocols and procedures should be kept that will allow a person examining data the opportunity to evaluate the impacts of procedural changes in both the field and the laboratory on the series of observations collected. Laboratory changes such as detection limits and precision are usually recorded, but changes in sampling methods, sampling points and personnel should all be part of the record. Sometimes this applies to a specific station, and at other times to an entire network. All too often the understanding of a data record is wrongly attributed (see Reference [16]).

13 Certification/registration/accreditation

In many parts of the world, quality management systems have been developed or adopted and applied to water quality sampling. These systems attempt to control the factors that affect the quality of the final data produced.

NOTE An example is ISO 17025.

The systems themselves do not specify the quality of the data, which is defined by the reason for its production. For instance, water quality data can be used to protect water treatment plant intakes from pollution events. In this case, it is not important to produce highly accurate results but it is important to produce them quickly before the pollution reaches the intake. Conversely, reporting results for regulatory purposes can require the highest accuracy and lowest detection limits possible. These requirements should be specified by the user of the data prior to sampling.

14 Quality control

Quality control measures the quality requirement of a process and uses techniques to correct any deviation from a process. ISO 5667-14 contains full details of such techniques that can be used for river sampling.

15 Safety precautions

The collection of water samples has some elements of danger, particularly when sampling frozen-over rivers or streams, and it is particularly important that relevant safety guidance is followed.

WARNING — If the ice is believed to be unsafe, do not attempt to take a sample.

For general guidance on safety precautions, refer to ISO 5667-1. However, particular attention should be paid to safety aspects when sampling from bridges, bank sides or vessels, at biological treatment works or when standing in water.

Safe access to routine sampling sites in all weathers is particularly important. Failure to satisfy this criterion will normally rule out a given site, even where it is preferred from the point of view of satisfying the technical objectives of the sampling programme.

Sampling operatives should wear disposable gloves during the whole sampling procedure, both to protect themselves from the sample and to protect the sample from contamination.

NOTE Attention is drawn to the existence in some countries of national legislation, with which all persons involved at any stage in the sampling, storage or delivery of samples, or in the associated documentation, should be thoroughly familiar.

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International Standards

[1] ISO 555-1, *Liquid flow measurement in open channels — Dilution methods for measurement of steady flow — Part 1: Constant-rate injection method* [now withdrawn]

[2] ISO 555-2, *Liquid flow measurement in open channels — Dilution methods for the measurement of steady flow — Part 2: Integration method* [now withdrawn]

[3] ISO 555-3, *Liquid flow measurement in open channels — Dilution methods for measurement of steady flow — Part 3: Constant rate injection method and integration method using radioactive tracers* [now withdrawn]

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[5] ISO 1070, *Liquid flow measurement in open channels — Slope-area method*

[6] ISO 5667-1, *Water quality — Sampling — Part 1: Guidance on the design of sampling programmes* [2)]

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[8] ISO 5667-3, *Water quality — Sampling — Part 3: Guidance on the preservation and handling of water samples*

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

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[12] ISO 17025, *General requirements for the competence of testing and calibration laboratories*

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2) ISO 5667-1 and ISO 5667-2 are currently undergoing joint revision, which will be published as ISO 5667-1.

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