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Water quality — Sampling —

Part 2:

Guidance on sampling techniques

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Qualité de l'eau — Échantillonnage —

Partie 2: Guide général sur les techniques d'échantillonnage

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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.

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.

International Standard ISO 5667-2 was prepared by Technical Committee ISO/TC 147, *Water quality*.

This second edition cancels and replaces the first edition (ISO 5667-2:1982), of which clause 3 has been greatly reduced in length, subclause 5.4 has been removed, and the remainder of the text has been brought up to date.

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

- *Part 1: Guidance on the design of sampling programmes*
- *Part 2: Guidance on sampling techniques*
- *Part 3: Guidance on the preservation and handling of 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*

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- *Part 9: Guidance on sampling from marine waters*
 - *Part 10: Guidance on sampling of wastewaters*
 - *Part 11: Guidance on sampling of groundwaters*
 - *Part 12: Guidance on sampling of industrial cooling water*
 - *Part 13: Guidance on sampling of sludges and sediments*
- Annex A forms an integral part of this part of ISO 5667.

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Introduction

ISO 5667 is published in a number of parts. The first three parts are of a general nature and should be read in conjunction with each other. Subsequent parts that have been published are:

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 the sampling of rivers and streams.

Other parts are being developed on the sampling of wastewaters, groundwater, precipitation, marine waters, industrial waters, and sludges and sediments.

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Water quality — Sampling —

Part 2: Guidance on sampling techniques

1 Scope

This part of ISO 5667 provides guidance on sampling techniques used to obtain the data necessary to make analyses for the purposes of quality control, quality characterization and identification of sources of pollution of waters.

Detailed instructions for specific sampling situations and sampling procedures are not included.

2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this part of ISO 5667. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this part of ISO 5667 are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 5667-1:1980, *Water quality — Sampling — Part 1: Guidance on the design of sampling programmes*.

ISO 5667-3:1985, *Water quality — Sampling — Part 3: Guidance on the preservation and handling of samples*.

ISO 6107-2:1989, *Water quality — Vocabulary — Part 2*.

ISO 7828:1985, *Water quality — Methods of biological sampling — Guidance on handnet sampling of aquatic benthic macro-invertebrates*.

ISO 8265:1988, *Water quality — Design and use of quantitative samplers for benthic macro-*

invertebrates on stony substrata in shallow freshwaters.

3 Definitions

For the purposes of this part of ISO 5667, the following definitions taken from ISO 6107-2 apply.

3.1 composite sample: Two or more samples or sub-samples, mixed together in appropriate known proportions (either discretely or continuously), from which the average result of a desired characteristic may be obtained. The proportions are usually based on time or flow measurements.

3.2 snap sample; spot sample; grab sample: A discrete sample taken randomly (with regard to time and/or location) from a body of water.

3.3 sampler: A device used to obtain a sample of water, either discretely or continuously, for the purpose of examination of various defined characteristics.

3.4 sampling: The process of removing a portion, intended to be representative, of a body of water for the purpose of examination of various defined characteristics.

4 Types of sample

4.1 General

Analytical data may be required to indicate the quality of water by determination of parameters such as the concentrations of inorganic material, dissolved minerals or chemicals, dissolved gases, dissolved organic material, and matter suspended in the water or bottom sediments at a specific time

and location or over some specific time interval at a particular location.

Certain parameters, such as the concentration of dissolved gases, should be measured *in situ* if possible, to obtain accurate results. It should be noted that sample preservation procedures should be carried out in appropriate cases (see ISO 5667-3).

It is recommended that separate samples be used for chemical, microbiological and biological analyses, because the procedures and equipment for collection and handling are different.

The sampling techniques will vary according to the specific situation. The different types of sampling are described in clause 5. Reference should be made to ISO 5667-1 for planning of sampling programmes.

It is necessary to differentiate between sampling from standing and flowing waters. Spot samples (4.2) and composite samples (4.6) are applicable to both types of water. Periodic sampling (4.3) and continuous sampling (4.4) are applicable to flowing waters, whereas series sampling (4.5) is more applicable to standing waters.

4.2 Spot samples

Spot samples are discrete samples, usually collected manually but which can also be collected automatically, for waters at the surface, at specific depths and at the bottom.

Each sample will normally be representative of the water quality only at the time and place at which it is taken. Automatic sampling is equivalent to a series of such samples taken on a preselected time or flow-interval basis.

Spot samples are recommended if the flow of the water to be sampled is not uniform, if the values of the parameters of interest are not constant, and if the use of a composite sample would obscure differences between individual samples due to reaction between them.

Spot samples should also be considered in investigations of the possible existence of pollution, or in surveys to indicate its extent or, in the case of automatic discrete sample collection, to determine the time of day when pollutants are present. They may also be taken prior to the establishment of a more extensive sampling programme. Spot samples are essential when the objective of a sampling programme is to estimate whether a water quality complies with limits not related to average quality.

The taking of spot samples is recommended for the determination of unstable parameters, such as the

concentration of dissolved gases, residual chlorine, soluble sulfides.

4.3 Periodic samples (discontinuous)

4.3.1 Periodic samples taken at fixed time-intervals (time dependent)

These samples are taken using a timing mechanism to initiate and terminate the collection of water during a specific time-interval. A common procedure is to pump the sample into one or more containers for a fixed period, a set volume being delivered to each container.

NOTE 1 The parameter of interest may affect the time interval.

4.3.2 Periodic samples taken at fixed flow-intervals (volume dependent)

These samples are taken when variations in water quality criteria and effluent flow rate are not inter-related. For each unit volume of liquid flow, a controlled sample is taken irrespective of time.

4.3.3 Periodic samples taken at fixed flow-intervals (flow dependent)

These samples are taken when variations in water quality criteria and effluent flow rate are not inter-related. At constant time intervals, samples of different volumes are taken, the volume depending upon the flow.

4.4 Continuous samples

4.4.1 Continuous samples taken at fixed flow rates

Samples taken by this technique contain all constituents present during a sampling period, but in many cases, do not provide information about the variation of concentrations of specific parameters during the sampling period.

4.4.2 Continuous samples taken at variable flow rates

The flow-proportional samples collected are representative of the bulk water quality. If both the flow and composition vary, flow-proportional samples can reveal variations which may not be observed by the use of spot samples, provided that the samples remain discrete and a sufficient number of samples is taken to differentiate between the changes in composition. Consequently, this is the most precise method of sampling flowing water if both the flow rate and the concentration of pollutants of interest vary significantly.

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4.5 Series sampling

4.5.1 Depth profile samples

This is a series of water samples taken from various depths of a body of water at a specific location.

4.5.2 Area profile samples

This is a series of water samples taken from a particular depth of a body of water at various locations.

4.6 Composite samples

Composite samples may be obtained manually or automatically, irrespective of the type of sampling (flow, time, volume or location dependent).

Continuously taken samples may be put together to obtain composite samples.

Composite samples provide average compositional data. Consequently, before combining samples it should be verified that such data are desired, or that the parameter(s) of interest do(es) not vary significantly during the sampling period.

Composite samples are valuable in cases when compliance with a limit is based on the average water quality.

4.7 Large volume samples

Some methods of analysis for certain determinands require the sampling of a large volume, namely from 50 litres to several cubic metres. Such large samples are necessary, for example, when analysing for pesticides or micro-organisms that cannot be cultured. The sample can either be collected in a conventional manner, with great care being taken to ensure cleanliness of the container or tanker holding the sample, or by passing a metered volume through an absorbent cartridge or filter, depending on the determinand. For example, an ion exchange cartridge or an activated carbon cartridge can be used to sample some pesticides whereas a polypropylene cartridge filter of mean pore diameter 1 μm is suitable for cryptosporidium.

The precise details of the latter procedure depend on the type of water sampled and the determinand. A regulator valve to control the flow through the cartridge or filter should be used for supplies under pressure. For most determinands, a pump should be placed after both the filter or cartridge and the meter; if the determinand is volatile, it is necessary to place the pump as close as possible to the sample origin, the meter still being placed after the filter or cartridge. When sampling a turbid water containing suspended solids that could blind the filter or cartridge, or if the amount of determinand required

for analysis exceeds the capacity of the largest filter or cartridge available, a series of filters or cartridges arranged in parallel should be used, using inlet and exit manifolds fitted with stopcocks. Initially the sampling flow should be directed through one filter or cartridge, with the others not receiving the flow, and when the flow rate decreases significantly then the flow should be diverted to a fresh filter or cartridge. If there is a danger of the filter or cartridge being overloaded, then fresh filter or cartridges should be connected on-line sequentially before the original one is exhausted, the flow to which is then stopped. When more than one filter or cartridge is used, they should be treated together and considered as a composite sample. If the waste water from such a sampling regime is returned to the body of water being sampled, then it is essential that it be returned sufficiently distant from the sampling point, so that it cannot influence the water being sampled.

5 Types of sampling

There are many sampling situations, some of which can be satisfied by taking simple spot samples whereas others may require sophisticated instrumental sampling equipment.

The various types of sampling are all examined in some detail in ISO 5667-4 and subsequent parts, and reference should be made to these parts of ISO 5667 whenever possible. The parts published to date, and those still being developed, are given in the foreword.

6 Sampling equipment

6.1 Materials

6.1.1 General

Reference should be made to ISO 5667-3 for specific sampling situations; the guidelines given here are to assist in the selection of materials for general application. The chemical constituents (determinands) in water, which are analysed to evaluate the water quality, range in concentration from submicrogram quantities or trace quantities to gross quantities. The most frequently encountered problems consist of adsorption onto the walls of the sampler or sample container, contamination prior to sampling caused by improper cleaning of the sampler or sample container, and contamination of the sample by the material constituting the sampler or sample container.

The sample container has to preserve the composition of the sample from losses due to adsorption and volatilization, or from contamination by foreign substances.

The sample container used to collect and store the sample should be chosen after considering, for example, resistance to temperature extremes, resistance to breakage, ease of good sealing and reopening, size, shape, mass, availability, cost, potential for cleaning and re-use, etc.

Precautions should be taken to prevent samples freezing, particularly when glass sample containers are used. High density polyethylene is recommended for silica, sodium, total alkalinity, chloride, specific conductance, pH, and hardness determinations in water. For light-sensitive materials, light-absorbent glass should be used. Stainless steel should be considered for samples of high temperature and/or pressure, or when sampling for trace concentrations of organic material.

Glass bottles are suitable for organic chemical compounds and biological species, and plastics containers for radionuclides. It is important to note that the sampling equipment available often has neoprene gaskets and oil-lubricated valves. Such materials are not satisfactory for samples for organic and microbiological analysis.

Thus, apart from the desired physical characteristics described above, the sample containers used to collect and store the samples should be selected by taking into account the following predominant criteria (especially when the constituents to be analysed are present in trace quantities).

- a) Minimization of contamination of the water sample by the material of which the container or its stopper is made, for example leaching of inorganic constituents from glass (especially soft glass) and organic compounds and metals from plastics and elastomers (plasticized vinyl capliners, neoprene jackets).
- b) Ability to clean and treat the walls of the containers, to reduce surface contamination by trace constituents such as heavy metals or radionuclides.
- c) Chemical and biological inertness of the material of which the container is made, in order to prevent or minimize reaction between constituents of the sample and the container.
- d) Sample containers may also cause errors by adsorption of determinands. Trace metals are particularly liable to this effect, but other determinands (e.g. detergents, pesticides, phosphate) may also be subject to error.

NOTE 2 It is recommended that detailed advice be sought from the analyst on the final choice of sample container and sampling equipment.

6.1.2 Sampling lines

Sampling lines are generally used in automatic sampling to supply samples to continuous analysers or monitors. During the residence time, the sample may be considered as stored in a container having the composition of the sampling line. Therefore, the guidelines for the selection of materials for sample containers also apply to sampling lines.

6.2 Types of sample container

6.2.1 General

Polyethylene and borosilicate glass bottles are suitable for conventional sampling for the determination of physical and chemical parameters of natural waters. Other more chemically inert materials, e.g. polytetrafluoroethylene (PTFE), are preferred, but are often too expensive for routine use. Screw-cap, narrow-mouthed and wide-mouthed bottles should be fitted with inert plastics stoppers/caps or ground glass stoppers (susceptible to seizing with alkaline solutions). If the samples are transported in a case to a laboratory for analysis, the lid of the case should be constructed to prevent loosening of the stopper which could result in spilling and/or contamination of the sample.

6.2.2 Special sample containers

In addition to the considerations already mentioned, the storage of samples containing photosensitive materials, including algae, requires their protection from exposure to light. In such cases, containers constructed of opaque materials or non-actinic glass are recommended, and they should be placed in light-proof cases during extended periods of storage. The collection and analysis of samples containing dissolved gases or constituents that would be altered by aeration poses a specific problem. The narrow-mouthed biochemical oxygen demand (BOD) bottles should be fitted with pointed glass stoppers to minimize air occlusion, and thus require special provision for sealing during transportation.

6.2.3 Trace organic contaminants

The sample bottles should be made of glass, as virtually all plastics containers interfere with the highly sensitive analysis. The closure should be of glass or polytetrafluoroethylene.

6.2.4 Sample containers for microbiological examination

Sample containers for microbiological examination should be able to withstand the high temperatures which occur during sterilization. During sterilization or sample storage the materials should not produce or release chemicals which could inhibit microbio-

logical viability, release toxic chemicals, or encourage growth. The samples should remain sealed until opened in the laboratory, and should be covered to prevent contamination.

Sample bottles should be of good quality glass or plastics material and free from toxic substances. A capacity of about 300 ml is sufficient for most routine purposes. The bottles should be fitted with ground-glass stoppers or screw caps fitted, if necessary, with silicone rubber liners that will withstand repeated sterilization at 160 °C.

6.3 Sampling equipment for physical or chemical characteristics

6.3.1 Introduction

The volume of sample collected should be sufficient for the required analyses, and for any repeat analyses. The use of very small sample volumes may cause the samples collected to be unrepresentative. In addition, small samples may also increase problems of adsorption because of the relatively small volume to area ratio.

Effective samplers should

- a) minimize the contact time between the sample and the sampler;
- b) use materials such that no sample contamination occurs;
- c) be simply designed to ensure ease of cleaning, with smooth surfaces and the absence of flow disturbances such as bends and with as few taps and valves as possible (all samplers should be checked to ensure that no bias is being introduced);
- d) be designed after considering the system suitability in relation to the required water sample (i.e. chemical, biological or microbiological).

For sampling of dissolved gases, reference should be made to 6.7.

6.3.2 Equipment for spot sampling

6.3.2.1 General

Spot samples are usually taken manually according to the conditions described in 4.2. The simplest equipment for taking surface samples is a bucket or wide-mouthed bottle dropped into a body of water and hauled out after filling.

6.3.2.2 Equipment for spot sampling at selected depths

In practice a weighted bottle is stoppered and lowered into the body of water. At a preselected depth, the stopper is removed and the bottle is then filled and withdrawn. The effects of air or other gases may have to be considered as this may change the parameter being examined (e.g. dissolved oxygen). Special sampling bottles that avoid this problem (e.g. evacuated bottles) are available.

For stratified water bodies, a graduated glass, plastics or stainless steel cylinder, open at both ends, can be lowered to obtain a vertical profile of the water body. At the sampling point, the cylinder is stoppered at both ends by a mechanism before withdrawal to the surface (messenger-operated water bottle).

6.3.2.3 Grabs or dredges for sampling sediments

Sediments may be sampled by grabs or dredges, designed to penetrate the substrate as a result of their own mass or leverage. Design features vary and include spring-activated, or gravity, modes of jaw closure. They also vary in the shape of the substrate bite, from square to sharp angle, and in the area and size of sample taken. Therefore, the nature of the sample obtained is affected by such factors as

- a) the depth of penetration of the substrate;
- b) the angle of jaw closure;
- c) the efficiency of closure (ability to avoid obstruction by objects);
- d) the creation of a "shock" wave and resultant loss or "wash-out" of constituents or organisms at the mud-water interface;
- e) the stability of samples in rapidly moving streams.

In selecting dredges, the habitat, water movement, area of sample, and boat equipment available need to be considered.

6.3.2.4 Clam-shell buckets

Clam-shell buckets resemble similar equipment used in land excavation. Usually operated from a boom, they are lowered at a selected sampling site to obtain a relatively massive composite sample. The resulting sample is more precisely defined with respect to a sampling site than when a dredge is used.