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Water quality — Sampling — Part 2 : Guidance on sampling techniques

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Foreword

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Draft International Standards adopted by the technical committees are circulated to the member bodies for approval before their acceptance as International Standards by the ISO Council.

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It has been approved by the member bodies of the following countries :

Australia	Greece	Poland
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Belgium	India	Spain
Brazil	Ireland	Sweden
Czechoslovakia	Japan	Switzerland
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The member body of the following country expressed disapproval of the document on technical grounds :

Canada

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Water quality — Sampling — Part 2 : Guidance on sampling techniques

0 Introduction

This International Standard comprises three parts which are intended to be used in conjunction with each other.

The important factors which need to be considered when designing a sampling programme in relation to water are given in ISO 5667/1, *Water quality — Sampling — Part 1 : Guidance on the design of sampling programmes. ISO 5667/3 deals with the preservation and handling of samples.*

1 Scope and field of application

This part of ISO 5667 gives 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 water, including bottom deposits and sludges.

Detailed instructions for specific sampling situations are not included, but will be given in subsequent International Standards.

Detailed sampling procedures are, similarly, not included.

2 Types of sample

2.1 General

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

Certain parameters, such as the concentration of dissolved gases, should be measured *in situ*, if possible, to obtain accurate results.

It is recommended that separate samples be used for chemical 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 3.

2.2 Spot samples

Spot samples are discrete samples generally collected manually, but which can 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 taken. Automatic sampling is equivalent to a series of such samples taken on a pre-selected time- or flow-interval basis.

Spot samples are useful 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 may be also required 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 that pollutants are present. They may also be taken prior to the establishment of a more extensive sampling programme.

The taking of spot samples may be specified for the determination of certain parameters, such as the concentration of dissolved gases, residual chlorine, soluble sulphides.

2.3 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 during a fixed period into one or more containers, a set volume being delivered to each container.

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

These samples are utilized when variations in water quality criteria and the effluent flow rate are not interrelated. They are also categorized as flow-proportioned samples. An example would be that for each unit volume (for example 10 000 litres) of liquid flow, a constant sample size is removed irrespective of time.

2.5 Continuous samples taken at fixed flow rates (time dependent or time average)

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

2.6 Continuous samples taken at variable flow rates (flow dependent or proportional)

The flow-proportional samples collected are representative of the bulk water quality. If both the flow and composition vary, flow-proportional samples can reveal such variations which may not be observed by the use of spot samples. Accordingly, this is the most precise method of sampling flowing water if both the flow rate and the concentration of pollutants of interest vary significantly.

2.7 Composite samples

Using one of the preceding techniques, samples may be obtained manually or automatically on either of two bases, i.e. individual samples or composite samples, where, on either a flow, time, volume dependent or on flow basis, it is desired to mix several individual samples and reduce the cost and time for their analysis.

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

3 Types of sampling

3.1 Open stream sampling

Sampling in open streams requires that attention be paid to the conditions where the sample is taken to ensure that it is representative. Streams are often poorly mixed for long distances after outfalls and near banks or edges of the water.

Point samples at various depths and at various locations across the stream should be taken to determine the best sampling location.

Turbulent locations, where the stream is well-mixed (if any can be found), are likely to be good sampling sites.

3.2 Closed pipe sampling

Sampling in closed pipes can present similar problems to that of open streams. Inlet probes or sampling tubes should be placed well downstream of inlet flows and the inlet to the sample line should be away from the pipe walls.

Turbulent locations, such as after "Tee" entries, bends, valves, etc., will generally be the best sampling locations due to internal mixing in the pipe. This is not the case, however, for isokinetic sampling.

3.3 Open body of water sampling

Samples may have to be taken at many locations and depths to obtain a representative picture of open bodies of water. Stratification by temperature can cause large differences in the qualities of the water.

3.4 Bottom deposits

Sediments may be sampled by clam-shell buckets, dredges or by coring devices. The process of sedimentation typically results in layers or strata of widely different composition. Moreover, unevenness in the level of the bed and localized stream movements can produce extreme variations in the thickness of the layers.

Composite samples may be obtained using dredges or clam-shell buckets. Coring devices are used when stratification is of interest.

Accordingly, the nature of the sample, whether it is a core or a composite representative of a given depth of material, must be known in order to interpret the analysis and/or examination properly. Furthermore, because of the expected variability, and also because the nature of the bottom may be difficult or impossible to know, taking a large number of samples is recommended.

It is also preferable to obtain analytical values on the individual samples rather than data on composites. The former will be more informative in that it will permit identification of the variability as well as provide the basis for plotting a composition profile.

The guidelines given in clause 5 for water samples will apply to the storage of sediment samples. Ordinarily, large wide-mouthed containers are used. Samples will usually contain excess water, so care may need to be taken to ensure leak-proof closure of the containers.

3.5 Ground water sampling

Ground water should be sampled at various depths and times to obtain representative characteristics of the water body.

Preliminary pumping may be necessary before samples are taken for subsequent analysis.

3.6 Precipitation sampling

Precipitation samples can be particularly difficult to take accurately. The sampling technique has to be able to exclude extraneous collection until such time as precipitation actually occurs. Covered samplers, that open only when precipitation occurs, are necessary if accurate results are required.

4 Sampling equipment

4.1 Equipment for sampling for physical or chemical characteristics

4.1.1 Equipment for spot sampling

4.1.1.1 General

Spot samples are usually taken manually under the conditions described in 2.2. The simplest equipment for taking surface samples is represented by a bucket or wide-mouthed bottle dropped into a body of water and hauled out after filling.

For stratified waters, point sampling at selected depths, as described in 4.1.1.3, is recommended. Depth-integrated sampling, as described in 4.1.1.2, can be used if it is considered sufficient to know only the average quality of a vertical profile.

4.1.1.2 Equipment for depth-integrated sampling

This technique requires a mechanism for holding and submerging the bottle. The weighted bottle is lowered through the water at a uniform rate, simultaneously admitting a sample through an orifice throughout the vertical profile.

If the sample has to be of equal aliquots at all depths, the speed of lowering or raising the bottle has to be varied with depth. There also exist variable orifices that maintain constant flow with varying pressure differential. A simple but secure device is required to fasten the bottle to the holder.

4.1.1.3 Equipment for point sampling at selected depths

In practice, a weighted bottle is corked and lowered into the body of water. At a preselected depth, the stopper is removed after which the bottle is filled and withdrawn. Effects of air or other gas may have to be considered as this may change the parameter being examined (for example, dissolved oxygen). Special sampling bottles that avoid this problem (for example, evacuated bottles) can be obtained.

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

4.1.1.4 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. Accordingly, 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.

4.1.1.5 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.

4.1.1.6 Core samplers

Core samplers are used when information concerning the vertical profile of a sediment is of interest. Unless the sample obtained has mechanical strength, care should be exercised in its removal from the coring device to preserve its longitudinal integrity.

4.1.2 Automatic sampling equipment

Instrumented and, often, highly automated sampling systems have been developed and are available from various commercial sources. While the standardization of these is not within the scope of this International Standard, criteria for the selection of suitable equipment is covered in the annex. Equipment may be required to be protected, flushed, heated, cooled, etc.

4.2 Equipment for sampling for biological characteristics

4.2.1 General

As in the case of sampling for physical and chemical analysis, some determinations can be performed *in situ*, but most samples are returned to the laboratory for examination. In the last decade, several devices have been developed to permit manual (by means of a diver) or automated and remote observation and collection of certain biological species or groups of organisms. However, the scope of the sampling described in this sub-clause will deal essentially with simple equipment which is conventionally employed.

4.2.2 Plankton

4.2.2.1 Phytoplankton

Techniques and equipment employed are similar to those described for the taking of spot and point samples for detecting chemicals in water. For most limnological investigations, a bottle of capacity 1 to 3 litres is used. A device is required to unstopper the bottle at the desired sampling depth and to reseal it subsequently.

Collection using nets is not recommended for quantitative assays.

4.2.2.2 Zooplankton

Large samples (up to 10 litres) are required for this group. In addition to the messenger-operated water bottle (see 4.1.1.3), a metered plankton nylon net is employed. Different net sizes are used depending on the species to be examined.

4.2.3 Benthos

4.2.3.1 Periphyton

For quantitative sampling, a standard glass microscope slide (of diameter 25 mm × 75 mm) is the most suitable device. Two types of base mount for the slide are required for two different aquatic situations.

In small shallow streams or littoral areas of lakes where turbidity is not a problem, the slides should be attached to a rack or stationed in frames anchored to the bottom. In large rivers or lakes, where turbidity is a problem, the slides should be mounted on an acrylic plastic rack which is permitted to float by the attachment of polystyrene foam at the ends of the rack.

Prior to sampling, the slides have to be exposed in the manner described for at least 2 weeks. If direct results are required, the periphyton has to be scraped from natural substratum.

4.2.3.2 Macrophytes

For qualitative sampling, the sampling equipment varies according to the specific situation, depending on the water depth. In shallow waters, a garden rake will suffice; for deeper waters, a dredge can be employed. However, diving exploration for this purpose using self-contained underwater breathing apparatus (scuba) has expanded greatly in the last decade.

For quantitative sampling, similar techniques may be applied, except that the areas to be sampled are delimited and the macrophytes are measured, or otherwise assessed, to determine the extent or rate of growth or mass per unit area.

4.2.3.3 Macroinvertebrates

The sampling equipment that is currently available is not considered suitable to provide quantitative data for all types of habitat. Sampling is usually restricted to certain designated areas. In some cases, the excessive number of replicates and the time required to process them may require the analyst to rely mainly on qualitative sampling.

In making comparison surveys of the macrobenthos, care has to be exercised to note the effect of differences in physical habitat among the various sample stations selected. However, because of the large variety of sampling techniques and equipment available, the types of habitat to be studied are relatively unrestricted. The specific type of sampler to be used will be dependent upon many parameters — depth of water, current flow, physical and chemical properties of substrate, etc.

The equipment used for sampling macroinvertebrates falls into the following categories :

a) grabs or dredges;

b) hand nets;

c) cylinders and box samplers;

d) coring devices (for sediments);

e) air lifts;

f) artificial substrates;

g) drift nets.

4.2.4 Fish

Fish are collected either actively or passively. Active sampling methods include the use of seines, trawls, electrofishing, chemicals, and hook and line. Passive sampling methods involve entanglement (gill nets and trammel nets) and entrapment devices (hoop nets, traps, etc.). Limitations imposed on qualitative and quantitative fish assays are the selectivity of the sampling equipment and the mobility and "rapid recruitment" (i.e. rapid rate of population increase) of the fish.

4.3 Equipment for sampling for microbiological characteristics

For the majority of samples, sterilized glass or plastic bottles are suitable. To collect samples considerably below the water surface, as in lakes and reservoirs, various deep sampling devices are available. The point samplers described in 4.1.1.3 are suitable for such purposes.

All apparatus used, including the pumps and pumping equipment, have to be free from contamination (for example by flushing) and should not introduce new micro-organisms themselves.

4.4 Equipment for sampling for radioactivity characteristics

Depending on the objective and national legal regulations, most of the sampling techniques and equipment available for sampling waters and waste waters for chemical constituents are generally applicable for obtaining samples for the measurement of radioactivity.

The samples should be collected in plastic bottles. Several devices are available for the continuous monitoring of radioactivity in streams, effluents and process waters, thus making sample collection unnecessary.

5 Sample containers and associated equipment

The following guidelines are given to assist in the selection of containers for use in general sampling procedures. ISO 5667/3 will give recommendations for specific sampling situations.

5.1 Materials

The chemical constituents (determinants) in water, which are analysed to evaluate the water quality, range in concentration from the submicrogram quantities, to trace quantities to gross quantities. In addition, interaction between constituents, photodecomposition, etc., require reduction of the holding time and restrictions on exposure to light, heat, etc. An additional consideration could be biological activity. The most frequently encountered problem consists of absorption onto the walls of the containers, contamination of the container prior to sampling by improper cleaning, and contamination of the sample by the material constituting the container.

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

Other factors involved in selection of the sample container used to collect and store the sample include resistance to temperature extremes, resistance to breakage, efficiency to tight sealing, reopening, size, shape, mass, availability, cost, potential for cleaning and re-use, etc.

For a great majority of samples containing inorganic chemicals, the current trend is towards the use of plastic containers made of polyethylene, fluoroplastic and polycarbonate polymers. The conventional high density polyethylene is considered highly satisfactory for silica, sodium, total alkalinity, chloride, specific conductance, pH, and hardness analysis of water. For light-sensitive materials, light-absorbent glass is used. Stainless steel has also been suggested for samples of high temperature and/or pressure, or for trace concentrations of organic material.

As a general rule, glass bottles are used for organic chemical compounds and biological species, and plastic containers for radionuclides, and for elements which are major constituents of glass. It is more important to note that the sampling equipment available often utilizes 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 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 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 of 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.

5.2 Sampling lines

Sampling lines are ordinarily 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. Accordingly, the guidelines for the selection of materials for sample containers apply to the selection of materials for sampling lines.

5.3 Types of sample container

5.3.1 General

For conventional sampling for the determination of physical and chemical parameters of natural waters, polyethylene and borosilicate glass bottles are used. Other, more chemically inert materials are preferred, but are often too expensive for routine use. Screw-cap bottles and several types of narrow- and wide-mouthed bottles are available which may be fitted with cork stoppers (wrapped in relatively inert metal foil), rubber stoppers (poor for organic material and in relation to some microbiological considerations) or ground glass stoppers (susceptible to seizing with alkaline solutions). These bottles are readily available and inexpensive. However, if the samples are being 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.

5.3.2 Special sample containers

In addition to the considerations already mentioned, the storage of samples with 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. A specific need exists in the collection and analysis of samples containing dissolved gases or constituents that would be altered by aeration. The narrow-mouthed biochemical oxygen demand (BOD) bottles are fitted which pointed glass stoppers to minimize air occlusion, and thus require special provision for sealing during transportation.

5.3.3 Trace organic contaminants

The sample bottles generally used are made of glass. Virtually all plastic containers interfere with the highly sensitive analysis. The closure should be glass or PTFE.

5.3.4 Containers for samples for microbiological examination

The primary consideration for containers for samples for microbiological examination is their ability to withstand the high temperatures which occur during sterilization. As in cold sterilization, bottle caps and liner material should follow similar criteria. During sterilization or sample holding, the materials should not produce or release chemicals which would inhibit microbiological viability, release chemicals that are toxic, or encourage growth. The bottles should remain sealed until opened in the laboratory, and should be covered to prevent contamination.

5.4 Cleaning of sample containers

5.4.1 Samples for general chemical analysis

It is recommended that new containers be thoroughly cleaned to minimize contamination of the sample. The type of cleaner used and the container material employed will vary according to the constituents of the sample to be analysed. For general purposes, glass bottles are cleaned with water and detergent to remove dust and packing materials, followed by chromic acid/sulphuric acid cleaning solution, and finally rinsed with distilled water. Detergents containing phosphate cannot be used if phosphates are to be determined, nor can chromic acid/sulphuric acid cleaning solution be used if trace quantities of sulphate and chromium are to be determined.

For polyethylene, it has been recommended that an approximately 1 mol/l solution of hydrochloric acid be used for cleaning, followed by prolonged leaching with dilute nitric acid solution.

To determine the extent of possible contamination of the sample by the material of which the container is made, high purity water should be added to the container under storage conditions simulating those for the field sample, and periodic aliquots should be taken.

5.4.2 Samples for analysis of organic material

The glass bottles used for the determination of traces of organic substances should only be cleaned with inorganic agents. Only if the trace substances are later determined by extraction may the glass bottle be treated with the extraction agent.

In such cases, after conventional cleaning procedures, the bottles should be oven dried, cooled, rinsed with the extraction solvent, for example hexane or petroleum ether, and dried with a stream of carefully cleaned air or nitrogen. Some laboratories have utilized a continuous overnight extraction with acetone, followed by a hexane rinse and drying as described above.

5.4.3 Samples for microbiological examination

For samples for microbiological examination, the glass bottles should be cleaned and rinsed as described in 5.4.1. For enumeration or some other biochemical analysis, bottles should be additionally rinsed with dilute nitric acid solution followed by distilled water to remove any heavy metals or chromate residues. For practical reasons, if the water to be examined contains, or is likely to contain, chlorine or chloramine, sufficient 3 % (m/m) sodium thiosulphate solution should be added to the bottles prior to sterilization. The amount depends on the size of the bottle (for example, 0,1 ml for 170 ml bottles). This inactivates any residual chlorine that may be present in the sample.

5.5 Transport of samples

Considerable attention should be given to the transport of the empty sample containers to the sampling site and to the transport of the filled sample containers back to the laboratory for analysis. The cases may be made of various materials — polyfoams, corrugated cardboard, etc. — to maintain integrity of the sample and to minimize possible damage during transportation. The lid of the packing case is usually lined with insulating material to exert mild pressure on the container stoppers. During the summer, or if biological conversion is feared, the samples should be preserved by refrigeration or by means of ice.

5.6 Quality control

Because of the potential danger of contamination, it is recommended that an active programme of quality control of containers be practised in each laboratory as part of its general quality assurance plan. Water of adequate purity for the determination under consideration should be introduced into randomly selected bottles after the cleaning operation, and subsequently should be analysed to verify the absence of residual impurities. The sampling and storage programme should also be monitored by introduction of test samples for subsequent analysis.

6 Identification and records

6.1 General

The source of the sample and conditions under which it was collected should be recorded and attached to the bottle immediately after filling. A water analysis is of limited value if unaccompanied by detailed information about the sample.

Field notes are extremely valuable in project-type investigations of water quality, but they can be easily misplaced or lost. They should never be relied on to take the place of detailed information accompanying the sample from the point of collection to finished analysed tabulation.

The minimum information required will depend upon the end use of the data.

6.2 Surface waters

At least the following information should be supplied :

Name of water body
Location of site
Point of collection
Date of collection
Time of collection
Gauge height or water discharge
Name of collector
Weather conditions
Nature of pretreatment

6.3 Ground waters

At least the following information should be supplied :

Geographic location
Depth of sample
Size of source
Casing diameter
Nature of pretreatment
Method of collection
Water-bearing formations
Water level
Yield of source
Principal use of water
Name of collector
Date of collection
Appearance at time of collection

6.4 Additional information

The presence or absence of preservatives or stabilizers should be recorded.