
**Kakovost vode - Vzorčenje - 3. del: Konzerviranje in ravnanje z vzorci vode
(ISO/DIS 5667-3:2023)**

Water quality - Sampling - Part 3: Preservation and handling of water samples (ISO/DIS 5667-3:2023)

Wasserbeschaffenheit - Probenahme - Teil 3: Konservierung und Handhabung von Wasserproben (ISO/DIS 5667-3:2023)

Qualité de l'eau - Échantillonnage - Partie 3: Conservation et manipulation des échantillons d'eau (ISO/DIS 5667-3:2023)

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13.060.45	Preiskava vode na splošno	Examination of water in general
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Water quality — Sampling —

Part 3: Preservation and handling of water samples

*Qualité de l'eau — Échantillonnage —**Partie 3: Conservation et manipulation des échantillons d'eau*

ICS: 13.060.45

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ISO copyright office
CP 401 • Ch. de Blandonnet 8
CH-1214 Vernier, Geneva
Phone: +41 22 749 01 11
Email: copyright@iso.org
Website: www.iso.org

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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

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

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

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

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

This sixth edition cancels and replaces the fifth edition (ISO 5667-3:2018), which has been technically revised.

The main changes compared to the previous edition are as follows:

- Addition of a reference to ISO/TS 5667-25 *Water quality — Guideline on the validation of the storage time of water samples*;
- Addition of a flow diagram for preservation and storage of water samples;
- Updated references in [Table A.1](#) and added references in [Tables A.2](#) and [A.3](#);
- [Table A.1](#) has been splitted into an inorganic ([A.1](#)) and an organic ([A.2](#)) part;
- Addition of [Table A.4](#) (Microbiological analysis);
- Addition of types of water to [Tables A.1](#) to [A.5](#);
- Explanation of the added terms used in [Tables A.1](#) to [A.5](#).

A list of all parts in the ISO 5667 series can be found on the ISO website.

Introduction

This document is intended to be used in conjunction with ISO 5667-1, which deals with the design of sampling programmes and sampling techniques.

Where possible this document has been brought into line with current standards. Where new research or validation results have provided new insights, the latest knowledge has been used.

Guidance on validation protocols can be found in ISO/TS 5667-25 and ISO 17034^[86].

ISO 5667-3 provides in [Tables A.1](#) to [A.5](#) where stated validated preservation times or conditions according to ISO/TS 5667-25 or ISO 5667-3:2018, Annex C as well as descriptions of best practice. [Tables A.1](#) to [A.5](#) also refer, for each analyte, to those analytical International Standards available at the date of publication of this version of ISO 5667-3. This is however not an exhaustive list. Other preservation methods may be used when they have been validated. However, it is strongly recommended that where a method validation is not available, the preservation times for the analyte as listed in [Tables A.1](#) to [A.5](#) for ISO test methods be followed. In case more than one storage time is provided in [Tables A.1](#) to [A.5](#) the order of preferred use is:

- Validated;
- Provided by standard;
- Best practice.

The preservation and storage conditions and maximum storage times per analyte as listed in [Tables A.1](#) to [A.5](#) should be regarded as default conditions to be applied in the absence of any other information.

However, if validation of preservation techniques and holding times has been carried out, relative to specific circumstances and matrices, by a laboratory, then, provided that it can produce evidence of this validation where they differ from those set out in [Tables A.1](#) to [A.5](#) of this standard, these validated preservation and storage conditions and maximum storage times are deemed acceptable for use by the validating laboratories. If national standards contain information on preservation, these are to be applied. This means that in these cases the contents of ISO 5667-3 are only to be considered informative.

Attention is drawn to the new part 25 in the ISO 5667 series, which further elaborates on ISO 5667-3:2018, Annex C, and which contains guidelines and the elaboration of the required techniques of how to validate new storage times or preservative methods and details of the techniques described.

Water quality — Sampling —

Part 3: Preservation and handling of water samples

1 Scope

This document specifies general requirements for sampling, preservation, handling, transport and storage of all water samples for physicochemical, chemical, hydrobiological and microbiological analyses and radiochemical analytes and activities.

Guidance on the validation of storage times of water samples is provided in ISO/TS 5667-25.

It is not applicable to water samples intended for ecotoxicological assays, biological assays, passive sampling as specified in the scope of ISO 5667-23 and microplastic particles and fibres.

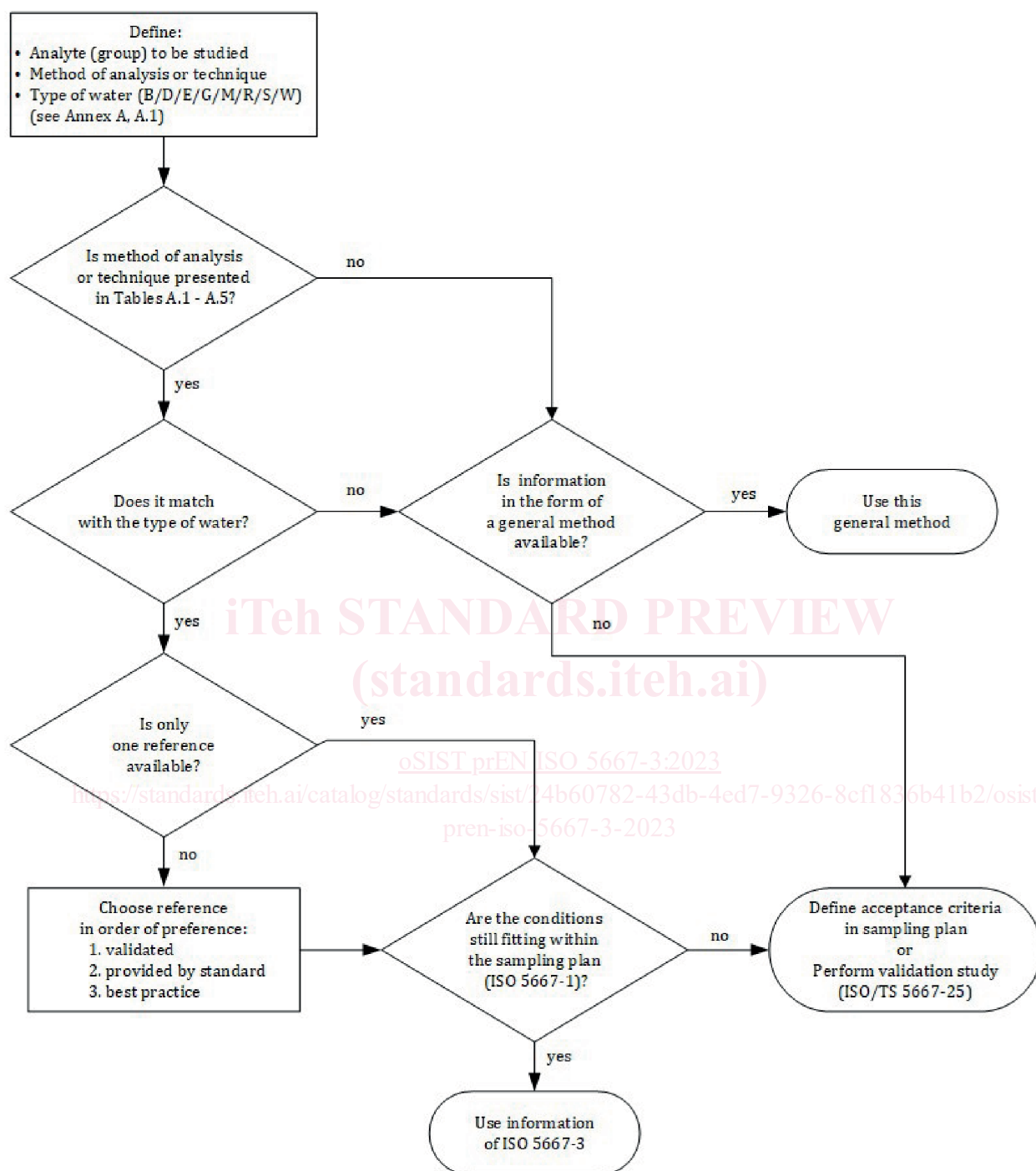
This document is particularly appropriate when samples cannot be analysed on site and have to be transported to a laboratory for analysis.

This document and the analytical International Standards can be used as presented in [Figure 1](#).

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WARNING — ‘Provided by standard’ and ‘validated’ could be based on previous standards and methods and therefore not be in line with ISO/TS 5667-25. This information can be interpreted by a qualified and experienced person.

Figure 1 — Flow diagram for preservation and storage of water samples

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements 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 (all parts), *Water quality — Sampling*

ISO 19458:2007, *Water quality — Sampling for microbiological analysis*

3 Terms and definitions

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

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

- IEC Electropedia: available at <https://www.electropedia.org/>
- ISO Online browsing platform: available at <https://www.iso.org/obp>

3.1

integrity

property that the parameter(s) of interest, information or content of the sample container has not been altered or lost in an unauthorized manner or subject to loss of representativeness

3.2

sample preservation

any procedure used to stabilize a sample in such a way that the properties under examination are maintained stable from the collection step until preparation for analysis

Note 1 to entry: Different analytes may require several samples from the same source that are stabilized by different procedures.

[SOURCE: ISO 11074:2015, 4.4.20, modified — Note 1 to entry has been added.]

3.3

sample storage

process, and the result of keeping a sample available under predefined conditions, usually for a specified time interval between collection and further treatment of a sample

Note 1 to entry: Specified time is the maximum time interval.

[SOURCE: ISO 11074:2015, 4.4.22, modified — Note 1 to entry has been added; "soil sample" has been changed to "sample".]

3.4

storage time

period of time between filling of the sample container and further treatment of the sample in the laboratory, if stored under predefined conditions

Note 1 to entry: Sampling finishes as soon as the sample container has been filled with the sample. Storage time ends when the sample is taken by the analyst to start sample preparation prior to analysis.

Note 2 to entry: Further treatment is, for most analytes, a solvent extraction or acid destruction. The initial steps of sample preparation can be steps complementary to the storage conditions for the maintenance of analyte concentrations.

4 Sampling and chain of custody

If there is a need to take samples, this is done accordingly to a sampling programme. The first step is to design a sampling programme. Guidance on this topic is given in ISO 5667-1.

Depending on the sample type and matrix, the guidelines found in the relevant part(s) of ISO 5667 and in ISO 19458 shall be consulted.

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The process of preservation and handling of water samples consists of several steps. During this process, the responsibility for the samples might change. To ensure the integrity of the samples, all steps involving the sample shall be documented.

5 Reagents and materials

WARNING — Certain preservatives (e.g. acids, alkalis, formaldehyde) need to be used with caution. Sampling personnel should be warned of potential dangers and appropriate safety procedures should be followed.

The following reagents are used for the sample preservation and shall only be prepared according to individual sampling requirements. All reagents and waters used shall be of at least analytical grade. Acids referred to in this document are commercially available "concentrated" acids.

All reagents shall be labelled with a "shelf-life". The shelf-life represents the period for which the reagent is suitable for use, if stored correctly. This shelf-life shall not be exceeded. Any reagents that are not completely used by the expiry of the shelf-life date shall be discarded.

NOTE 1 Often the shelf-life of reagents is supplied by the receiving laboratory.

NOTE 2 For reagents that in the specific conditions are not likely to change during time, a periodically check of the storage and packaging could be sufficient.

Check reagents periodically, e.g. by field blanks, and discard any reagent found to be unsuitable.

Between on-site visits, reagents shall be stored separately from sample containers and other equipment in a clean, secure cabinet in order to prevent contamination.

Each sample shall be labelled accordingly, after the addition of the preservative. Otherwise, there could be no visible indication as to which samples have been preserved, and which have not.

5.1 Solids

5.1.1 Sodium thiosulfate pentahydrate, $\text{Na}_2\text{S}_2\text{O}_3 \cdot 5\text{H}_2\text{O}$, $w(\text{Na}_2\text{S}_2\text{O}_3 \cdot 5\text{H}_2\text{O}) > 99 \%$.

5.1.2 Sodium hydroxide, NaOH , $w(\text{NaOH}) > 99 \%$.

5.1.3 Sodium tetraborate decahydrate, $\text{Na}_2\text{B}_4\text{O}_7 \cdot 10\text{H}_2\text{O}$, $w(\text{Na}_2\text{B}_4\text{O}_7 \cdot 10\text{H}_2\text{O}) > 99 \%$.

CAUTION — Sodium tetraborate decahydrate is known to be a carcinogen, mutagen and reproductive toxin (CMR).

5.1.4 Hexamethylenetetramine (hexamine, urotropine), $\text{C}_6\text{H}_{12}\text{N}_4$, $w(\text{C}_6\text{H}_{12}\text{N}_4) > 99 \%$.

5.1.5 Potassium iodide, KI , $w(\text{KI}) > 99 \%$.

5.1.6 Iodine, I_2 , $w(\text{I}_2) > 99 \%$.

5.1.7 Sodium acetate, $\text{C}_2\text{H}_3\text{NaO}_2$, $w(\text{C}_2\text{H}_3\text{NaO}_2) > 99 \%$.

5.1.8 Ethylenediamine, $\text{C}_2\text{H}_8\text{N}_2$, $w(\text{C}_2\text{H}_8\text{N}_2) > 99 \%$.

5.2 Solutions

5.2.1 Zinc acetate solution $C_4H_6O_4Zn \cdot 2H_2O$ (100 g/l).

Dissolve 10,0 g of zinc acetate dihydrate in ~90 ml of water. Dilute to 100 ml with water.

5.2.2 Orthophosphoric acid ($\rho \approx 1,7$ g/ml), H_3PO_4 , $w(H_3PO_4) > 85 \%$, $c(H_3PO_4) = 15$ mol/l.

5.2.3 Hydrochloric acid ($\rho \approx 1,2$ g/ml), HCl , $w(HCl) > 36 \%$, $c(HCl) = 12,0$ mol/l.

5.2.4 Nitric acid ($\rho \approx 1,42$ g/ml), HNO_3 , $w(HNO_3) > 65 \%$, $c(HNO_3) = 15,8$ mol/l.

5.2.5 Sulfuric acid ($\rho \approx 1,43$ g/ml), H_2SO_4 , $w(HNO_3) \approx 49 \%$, $c(H_2SO_4) \approx 9$ mol/l.

Dilute concentrated sulfuric acid (H_2SO_4), $\rho \approx 1,84$ g/ml, $w(H_2SO_4) \approx 98 \%$ 1 + 1 by carefully adding the concentrated acid to an equal volume of water and mix.

WARNING — Adding the concentrated acid to the water can give violent reactions because of an exothermic reaction.

5.2.6 Sodium hydroxide solution ($\rho \approx 0,40$ g/ml), $NaOH$.

5.2.7 Formaldehyde solution (formalin), CH_2O , $\varphi(CH_2O) = 37 \%$ (freshly prepared).

WARNING — Beware of formaldehyde vapours. Do not store large numbers of samples in small working areas.

5.2.8 Disodium salt of ethylenediaminetetraacetic acid (EDTA) ($\rho \approx 0,025$ g/ml), $C_{10}H_{14}N_2Na_2O_8 \cdot 2H_2O$, $w(C_{10}H_{14}N_2Na_2O_8 \cdot 2H_2O) > 99 \%$.

Dissolve 25 g EDTA in 1 000 ml of water.

5.2.9 Ethanol C_2H_5OH , $\varphi(C_2H_5OH) = 96 \%$.

5.2.10 Acidic Lugol's solution, 100 g potassium iodide (5.1.5), 50 g iodine (5.1.6) and 100 ml glacial acetic acid (5.2.16) in 1 000 ml water to pH 2.

5.2.11 Alkaline Lugol's solution, 100 g potassium iodide (5.1.5), 50 g iodine (5.1.6), and 250 g sodium acetate (5.1.7) in 1 000 ml water to pH 10.

5.2.12 Neutralized formaldehyde solution, formaldehyde solution (5.2.7) neutralized with sodium tetraborate (5.1.3) or hexamethylenetetramine (5.1.4). Formalin solution at 100 g/l gives a final solution of $\varphi(CH_2O) = 3,7 \%$ to $4,0 \%$.

WARNING — Beware of formaldehyde vapours. Do not store large numbers of samples in small working areas.

5.2.13 Ethanol preservative solution.

Ethanol (5.2.9), formaldehyde solution (5.2.7) and glycerol (5.2.17) (100 + 2 + 1 parts by volume, respectively).

5.2.14 Sodium hypochlorite $NaOCl$, $w(NaOCl) = 10 \%$. Dissolve 100 g sodium hypochlorite ($NaOCl$) in 1 000 ml of water.

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5.2.15 Potassium iodate KIO_3 , $w(\text{KIO}_3) = 10 \%$. Dissolve 100 g potassium iodate (KIO_3) in 1 000 ml of water.

5.2.16 Glacial acetic acid $\text{C}_2\text{H}_4\text{O}_2$, $w(\text{C}_2\text{H}_4\text{O}_2) > 99 \%$.

5.2.17 Glycerol (glycerin, glycerine) $\text{C}_3\text{H}_5(\text{OH})_3$.

5.2.18 Sodiumhydrogensulphate NaHSO_4 .

5.3 Materials

5.3.1 Container and cap, types as specified in [Tables A.1](#) to [A.5](#).

5.3.2 Filter, membrane, pore size 0,40 μm to 0,45 μm , unless a different filter size is specified in the analytical International Standard.

6 Containers

6.1 Container selection and preparation

The choice of sample container ([5.3.1](#)) is of major importance and ISO 5667-1 provides some guidance on this subject.

Details of the type of container used for the collection and storage of samples are given in [Tables A.1](#) to [A.5](#). The same considerations given to this selection of suitable container material shall also be given to the selection of cap liner materials. For microbiological analyses clean sterile bottles shall be used.

Sample containers shall be made of a material appropriate for preserving the natural properties of both the sample and the expected range of contaminants. Suitable types of containers for each analyte to be measured are given in [Tables A.1](#) to [A.5](#).

NOTE For very low concentrations of metals, containers prescribed can be different from those used for higher concentrations. Details can be found in [Table A.1](#) or in the analytical International Standards.

If the samples are to be frozen, suitable containers, such as polyethylene (PE) or polytetrafluoroethylene (PTFE), shall be used to prevent breakage.

The use of disposables is preferred. Some manufacturers supply containers with a certificate of cleanliness. If such a certificate of cleanliness is supplied, it is not necessary to clean or rinse the containers before use.

6.2 On-site filtration

On-site filtration is required in some cases:

- If dissolved metals need to be analysed. Filter on site or as soon as possible after sampling. Acidify to $\text{pH} < 2$ with HNO_3 ([5.2.4](#)).
- If required according to [Tables A.1](#) to [A.5](#), e.g. ammonium, nitrate, nitrite, phosphate, sulfate and silicates.

If immediate filtration on site is impossible where required, then the reason and the time between sampling and filtration shall be added to the test report.

6.3 Filling the container

Containers (5.3.1) should be filled as prescribed in [Tables A.1](#) to [A.5](#) or the analytical International Standard. If there are no instructions regarding the filling of the containers, they should be filled completely unless the samples are to be frozen as part of their preservation, the sample containers shall not be filled completely. This is in order to prevent breakage which may arise from expansion of the water sample during the freezing and thawing process.

If no preservatives are present in the bottle, prerinsing the bottle may be advisable. Guidance on prerinsing can be found in ISO 5667-14.

7 Sample handling and preservation

7.1 Sample handling and preservation for physical and chemical analysis

Waters, particularly surface waters, waste waters and groundwaters, are susceptible to changes as a result of physical, chemical or hydrobiological reactions which may take place between the time of sampling and the commencement of analysis. The nature and rate of these reactions are often such that, if precautions are not taken during sampling, transport and storage (for specific analytes), the concentrations determined are different to those existing at the time of sampling.

The extent of these changes is dependent on the chemical and biological nature of the sample, its temperature, its exposure to light, the type of the container in which it is placed, the time between sampling and analysis, and the conditions to which it is subjected, e.g. agitation during transport. Further specific causes of variation are listed in a) to f).

- a) The presence of bacteria, algae and other organisms can consume certain constituents of the samples. These organisms can also modify the nature of the constituents to produce new constituents. This hydrobiological activity affects, for example, the concentrations of dissolved oxygen, carbon dioxide, compounds of nitrogen, phosphorus and, sometimes, silicon.
- b) Certain compounds can be oxidized either by dissolved oxygen present in the samples, or by atmospheric oxygen [e.g. organic compounds, Fe(II) and sulfides].
- c) Certain substances can precipitate out of solution, e.g. calcium carbonate, metals, and metallic compounds such as $\text{Al}(\text{OH})_3$, or can be lost to the vapour phase (e.g. oxygen, cyanides, and mercury).
- d) Absorption of carbon dioxide from air can modify pH, conductivity, and the concentration of dissolved carbon dioxide. Passage of compounds like ammonia and silicon fluoride through some types of plastics (see [Table A.1](#)) may also affect pH or conductivity.
- e) Dissolved metals or metals in a colloidal state, as well as certain organic compounds, can be irreversibly adsorbed on to the surface of the containers or solid materials in the samples.
- f) Polymerized products can depolymerize, and conversely, simple compounds can polymerize.

Changes to particular constituents vary both in degree and rate, not only as a function of the type of water, but also, for the same water type, as a function of seasonal conditions.

These changes are often sufficiently rapid to modify the sample considerably in a short time. In all cases, it is essential to take precautions to minimize these reactions and, in the case of many analytes, to analyse the sample with a minimum of delay. On-site filtration may be required as a precaution ([6.2](#)).

Details of the sample preservation are given in [Tables A.1](#) and [A.2](#).

7.2 Sample handling and preservation for hydrobiological analysis

The handling of samples for hydrobiological analysis is different from that for samples requiring chemical analysis. The addition of chemicals to the sample for hydrobiological analysis can be used for fixation or preservation of the sample. The term "fixation" is defined as the protection of