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Water quality — Sampling —

Part 15: Preservation and handling of samples of sludge, sediment and suspended matter

Qualité de l'eau — Échantillonnage —

Partie 15: Conservation et traitement des échantillons de boues, de sédiments et de matières en suspension

Reference number
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Foreword

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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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This document was prepared by Technical Committee ISO/TC 147, *Water quality*, Subcommittee SC 6, *Sampling (general methods)*, in collaboration with the European Committee for Standardization (CEN) Technical Committee CEN/TC 308, *Characterization and management of sludge*,in accordance with the Agreement on technical cooperation between ISO and CEN (Vienna Agreement).

This third edition cancels and replaces the second edition (ISO 5667-15:2009), which has been technically revised.

The main changes are as follows:

— 'suspended matter' has been added to the title and 'guidance' has been deleted from the title;

— ISO/TS 5667-25 has been added as an informative reference;

— a flow diagram for preservation and storage of samples of sludge, sediment and suspended matter has been added (in line with ISO 5667-3);

— terms and definitions have been aligned with ISO 5667-3;

— tables from Clause 12 have been moved to Annex A;

— references in the previous Tables 1 to 3 have been added;

— the previous Table 3 has been split into Table A.3 'Hydrobiological analysis' and Table A.4 'Microbiological analysis'.

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

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

Introduction

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

Where possible, this document has been aligned 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[6].

Tables A.1 to A.4 provide the validated preservation times or conditions as well as the descriptions of best practice. Tables A.1 to A.4 also refer, for each parameter, to references available at the time of publication of this document (i.e. ISO 5667-15:2026). 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 listed in Tables A.1 to A.4 for ISO and CEN test methods be followed. In case more than one storage time is provided in Tables A.1 to A.4, the order of preferred use is:

— validated method;

— method provided by reference;

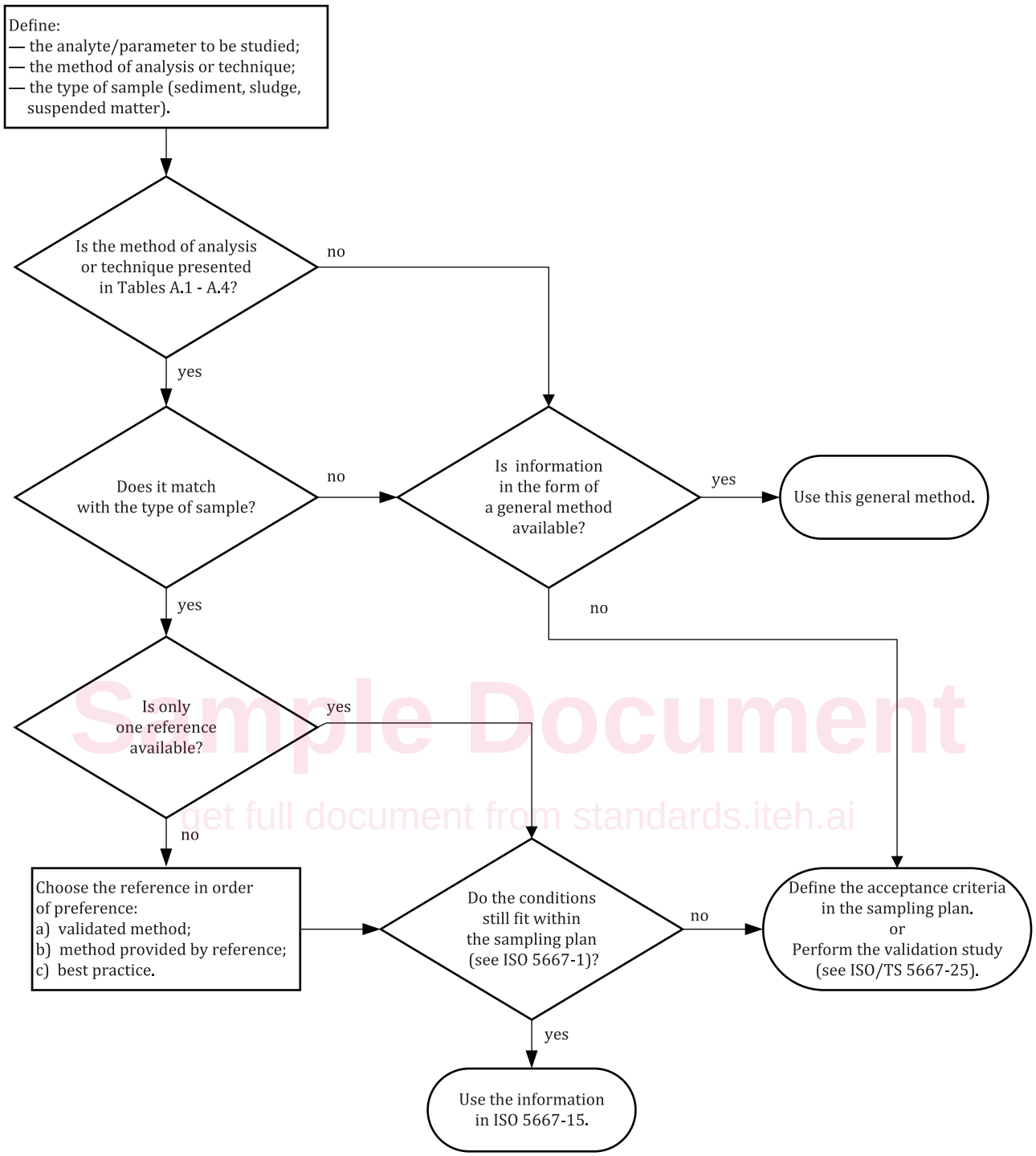
— best practice.

The preservation and storage conditions and maximum storage times per parameter as listed in Tables A.1 to A.4 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.4, these validated preservation and storage conditions and maximum storage times are deemed acceptable for use by the validating laboratories. A national standard can contain information on preservation.

NOTE Samples of sludge, sediment and suspended matter that are dried or freeze-dried behave similarly to dried soils. For guidance on freeze-drying, see ISO 16720[39].

This document and the related analytical references can be used as presented in Figure 1.



WARNING — 'Method provided by reference' and 'validated method' can be based on previous standards and methods and therefore not be in line with ISO/TS 5667-25[6]. This information can be interpreted by a qualified and experienced person.

Figure 1 — Flow diagram for the selection of a method for the preservation and storage of samples of sludge, sediment and suspended matter

Attention is drawn to ISO/TS 5667-25[6], 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 15: Preservation and handling of samples of sludge, sediment and suspended matter

WARNING — Persons using this document should be familiar with normal laboratory practice. This document does not purport to address all of the safety problems, if any, associated with its use. It is the responsibility of the user to establish appropriate safety and health practices.

IMPORTANT — It is absolutely essential that tests conducted in accordance with this document be carried out by suitably qualified staff.

1 Scope

This document specifies the general requirements on procedures for the preservation, handling and storage of samples of sewage and waterworks sludge, suspended matter, marine sediments and freshwater sediments for either chemical, physical, radiochemical, hydrobiological or microbiological examination, or all, in the laboratory.

The procedures in this document are not applicable to dried samples of sludge, sediment and suspended matter.

NOTE The storage conditions given do not necessarily apply for derived samples, e.g. sediment eluates or extracts.

This document is not applicable to samples intended for biotesting with ecotoxicological or biological assays (which is specified in ISO 5667-16[5]) nor intended for microplastics (which is specified in ISO 5667-27[7]).

2 Normative references

There are no normative references in this document.

3 Terms and definitions

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

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

— ISO Online browsing platform: available at https://www.iso.org/obp

— IEC Electropedia: available at https://www.electropedia.org/

3.1
best practice
method based upon consensus or general use and that can be referred to in literature

Note 1 to entry: Given the differences in conditions and circumstances as well as the impossibility to validate all parameters from a *validated method* (3.10) or technique or process, a best practice method based upon the corresponding properties of a validated parameter can be used.

[SOURCE: ISO 5667-3:2024, 3.1[2]]

3.2
integrity
property of the parameter(s) of interest, information or content of a sample stored in a container that has not been altered or lost in an unauthorized manner or that has been subject to loss of representativeness

[SOURCE: ISO 5667-3:2024, 3.2[2]]

3.3
method provided by reference
procedure or technique for the preservation of samples taken from the reference to which it refers

Note 1 to entry: It is not in all cases clear whether the preservation procedure provided by the reference was *validated method* (3.10), a *best practice* (3.1) or which procedure was used for its determination or validation. Where available, the information about the matrices is taken over.

[SOURCE: ISO 5667-3:2024, 3.3[2]]

3.4
sample preservation
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 can require several samples from the same source that are stabilized by different procedures. More subsamples from one location can be needed as some parameters require a different preservation procedure.

[SOURCE: ISO 11074:2025, 3.401,[10] modified — Note 1 to entry has been added.]

3.5
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: The specified time is the maximum time interval.

[SOURCE: ISO 11074:2025, 3.407,[10] modified — Note 1 to entry has been added; “soil sample” has been changed to “sample”.]

3.6
sediment
matter which settles to the bottom of a liquid, often transported in water before settlement occurs

Note 1 to entry: Sediment samples in this document are a part of water quality analysis. The liquid in this case is therefore water.

[SOURCE: ISO 6107:2021, 3.505,[8] modified — Note 1 to entry has been added.]

3.7
sludge
accumulated settled solids separated from various types of water as a result of natural or artificial processes

[SOURCE: ISO 23880:—,[1)] 3.1.2[48]]

3.8
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.

1) Under preparation. Stage at the time of publication: ISO/DIS 23880:2026.

Note 2 to entry: Further treatment is, for most analytes, a solvent extraction or acid destruction. Initial sample preparation steps may be considered as an extension of the storage conditions for maintaining analyte stability.

[SOURCE: ISO 5667-3:2024, 3.6,[2] modified — Note 2 to entry has been adjusted.]

3.9
suspended matter
solids remaining in suspension in water which can be removed by sedimentation, filtration or centrifugation

[SOURCE: ISO 6107:2021, 3.554[8]]

3.10
validated method
method for which the validity of correctness has been checked by verification or qualification against a number of predefined requirements

Note 1 to entry: A validated method indicates that a preservation method is capable of delivering the intended results with an acceptable degree of uncertainty for the parameter or group of parameters and sample type.

[SOURCE: ISO 5667-3:2024, 3.7[2]]

4 Abbreviated terms

4.1 Plastics

FEP perfluoro(ethylene/propylene)

PE polyethylene

PE-HD high density polyethylene

PET polyethylene terephthalate

PFA perfluoroalkoxy (polymer)

PP polypropylene

PTFE polytetrafluoroethylene

PVC poly(vinyl chloride)

4.2 Analytical chemistry techniques

AAS atomic absorption spectroscopy

AFS atomic fluorescence spectroscopy

AMP amperometric method

CAL calometric method

CLM coulometric method

DIG digestion method

FRZ freeze-drying

GC-ECD gas chromatography - electron capture detector

GC-MS gas chromatography - mass spectrometry

GRA	gravimetric method
IC	ion chromatography
ICP-MS	inductively coupled plasma - mass spectrometry
ICP-OES	inductively coupled plasma - optical emission spectroscopy
LC	liquid chromatography
LC-FD	liquid chromatography - fluorometric detection
LC-MS	liquid chromatography - mass spectrometry
LC-UV	liquid chromatography - ultraviolet detection
MB	microbiological method
MIC	microscopy
POT	potentiometry
SP	spectroscopy
TIT	titrimetric method
VAR	various methods
VM	visual method

5 Sampling and chain of custody

Before samples can be taken, a sampling programme shall be designed. Guidance on this topic is given in ISO 5667-1[1].

Depending on the sample type and matrix, instructions are given in the relevant part(s) of the ISO 5667 series.

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

6 Reagents

WARNING — Sampling personnel should be warned of potential dangers and appropriate safety procedures should be available. Beware of formaldehyde vapours. Do not store large numbers of samples in small working areas.

All reagents and waters used shall be at least of analytical grade.

6.1 Deionized water.

6.2 Sodium sulfate (Na_2SO_4).

Heat the sodium sulfate before use for at least 3 h at 500 °C. Store in a desiccator after heating.

6.3 Zinc acetate solution ($(CH_3COO)_2Zn{\cdot}2H_2O$ (100 g/l).

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

6.4 Methanol (CH_3OH).

6.5 Ethanol (C_2H_5OH), volume fraction of 96 %.

6.6 Formaldehyde solution (formalin; CH_2O), $w(CH_2O)$ = 37 % (mass fraction) (freshly prepared).

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

6.7 Sodium tetraborate ($Na_2B_4O_7{\cdot}10H_2O$).

6.8 Hexamethylenetetramine [$(CH_2)_6N_4$].

6.9 Neutralized formaldehyde solution formaldehyde solution (6.6) neutralized with sodium tetraborate (6.7) or hexamethylenetetramine (6.8).

Formalin solution at 100 g/l gives a final solution of $w(CH_2O)$ = 3,7 % (mass fraction).

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

6.10 Glycerol (glycerin, glycerine), $C_3H_5(OH)_3$.

7 Sample handling and preservation

7.1 General

Sample handling is specific for each determination to be conducted. Manipulation of samples is often required to yield consistent material for analysis. Homogenization, by mixing or sieving, dilution to obtain a suitable concentration and addition of chemical preservatives all complicate interpretations of in situ comparisons.

Any large individuals of macrofauna should be removed from the samples immediately after collecting samples taken for either chemical, physical, radiochemical or biological examination, or all.

The purpose of preservation is to retain the integrity of the collected material as it was on site in relation to the parameters to be analysed. Analytes can be subject to biotransformation, volatilization and chemical transformation (e.g. oxidation, reduction, hydrolysis, photolysis) during storage. Therefore, careful consideration should be given to these processes and the storage conditions needed to avoid such alterations.

The need to preserve sludge, sediment and suspended matter begins immediately after a sample has been taken. The most critical changes to the sample can occur in the first few hours after sampling. Therefore, where possible, preservation steps should be taken immediately upon sample collection.

The choice of preservation technique depends mainly on the objective of the sample collection and the analysis being determined. It is important to understand the effects that preservation and storage can have on the sample quality and the analysis results.

No other general recommendations can be given for a preservation or storage method. A preservation method suitable for one group of parameters can interfere with the analysis of other groups of parameters. To overcome this problem, a number of sub-samples should be collected; each sub-sample should be preserved using a suitable method such that the specific demands of each analytical parameter are met.

Annex A provides techniques for sample preservation accompanied by the information in Tables A.1 to A.4.

7.2 Chemical analysis

Chemical analysis can be performed to determine the nature and amounts of the substances that are contained in the whole sample, dissolved in the aqueous phase and absorbed by sludge, sediment or suspended matter.

Partition of chemical components between the solid phase and the water phase is influenced by several factors, such as particle size, amount of organic matter, pH, redox potential and salinity. The study of such attributes can be a sampling objective. Therefore, the preservation needs for the analytical methods to be employed should be taken into account (see Table A.1). The guidance given in this document is relevant to the determination of components in the sum of the separate phases of sludge, sediment or suspended matter, unless otherwise indicated.

Preservation of samples by freezing can cause mobilization of contaminants by cellular disruption, whereas not stabilizing samples can permit continued microbial transformation of critical parameters of interest. In addition to biodegradation of organics, volatilization is a principal mechanism of loss of volatile compounds during sample handling. Microbial activity can be responsible for changes in the nitrate-nitrite-ammonia content, for decrease in biochemical oxygen demand or for reducing sulfate to sulfide. Anoxic samples require appropriate preservation techniques such as oxygen exclusion during sample handling. Drying, freezing and freeze-drying of anoxic samples alter the binding sites of, for example, heavy metals, making more differentiated investigation of binding forms virtually impossible.

Details of the sample preservation are given in Table A.1.

7.3 Physical analysis

The structure, texture and, for sediments, the layer formation should be determined.

NOTE Sediment matrix changes are obvious if rapid drainage of pore water occurs.

The importance of sludge, sediment or suspended matter integrity to the investigation objectives should be evaluated as it can influence the preservation and handling techniques. In general, any disturbance of the samples should be minimized. Where the physical structure of the material sampled is important for the measurement of parameters (e.g. resistance to filtration), agitation and vibration during transport should be reduced to a minimum. Freezing of the sludge, sediments or suspended matter should be avoided, but can be appropriate. In some cases, thermal techniques can strongly modify sludge structure, thus affecting physical characteristics (e.g. de-waterability, settleability, flowability).

Details of the sample preservation are given in Table A.1.

7.4 Radiochemical analysis

Some sampling sites can have measurable radiochemical activity, e.g. in the soil or air. Some items of domestic equipment within the laboratory can also be a source of radioactive material. Contamination of the sample by its environment should therefore be avoided, especially if the sample activity is likely to be very low.

Details of the sample preservation are given in Table A.2.

7.5 Hydrobiological analysis

Hydrobiological analysis generally involves classifying the species and numbers of either flora or fauna, or both, present on and in fixed sludge or sediments.

Details of the sample preservation are given in Table A.3.

7.6 Microbiological analysis

Microbial activity may also be used to characterize samples and can only be determined without fixation.

Details of the sample preservation are given in Table A.4.