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**Water quality — Sampling —**

Part 15:

**Guidance on the preservation and  
handling of sludge and sediment samples**

*Qualité de l'eau — Échantillonnage —*

*Partie 15: Lignes directrices pour la conservation et le traitement des  
échantillons de boues et de sédiments*

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## Foreword

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

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

The main task of technical committees is to prepare International Standards. Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

ISO 5667-15 was prepared by Technical Committee ISO/TC 147, *Water quality*, Subcommittee SC 6, *Sampling (general methods)*.

This second edition cancels and replaces the first edition (ISO 5667-15:1999), which has been technically revised.

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

- *Part 1: Guidance on the design of sampling programmes and sampling techniques*
- *Part 3: Guidance on the preservation and handling of water samples*
- *Part 4: Guidance on sampling from lakes, natural and man-made*
- *Part 5: Guidance on sampling of drinking water from treatment works and piped distribution systems*
- *Part 6: Guidance on sampling of rivers and streams*
- *Part 7: Guidance on sampling of water and steam in boiler plants*
- *Part 8: Guidance on the sampling of wet deposition*
- *Part 9: Guidance on sampling from marine waters*
- *Part 10: Guidance on sampling of waste waters*
- *Part 11: Guidance on sampling of groundwaters*
- *Part 12: Guidance on sampling of bottom sediments*
- *Part 13: Guidance on sampling of sludges from sewage and water-treatment works<sup>1)</sup>*

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1) In preparation. (Revision of ISO 5667-13:1997)

- *Part 14: Guidance on quality assurance of environmental water sampling and handling*
- *Part 15: Guidance on the preservation and handling of sludge and sediment samples*
- *Part 16: Guidance on biotesting of samples*
- *Part 17: Guidance on sampling of bulk suspended solids*
- *Part 19: Guidance on sampling of marine sediments*
- *Part 20: Guidance on the use of sampling data for decision making — Compliance with thresholds and classification systems*
- *Part 21: Guidance on sampling of drinking water distributed by tankers or means other than distribution pipes*
- *Part 22: Guidance on the design and installation of groundwater monitoring points*
- *Part 23: Determination of priority pollutants in surface water using passive sampling*

This part of ISO 5667 may be used in conjunction with the other parts available within the ISO 5667 series.

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# Water quality — Sampling —

Part 15:

## Guidance on the preservation and handling of sludge and sediment samples

**WARNING** — Persons using this International Standard should be familiar with normal laboratory practice. This standard 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 and to ensure compliance with any national regulatory conditions.

### 1 Scope

This part of ISO 5667 provides guidance on procedures for the preservation, handling and storage of samples of sewage and waterworks sludge, suspended matter, saltwater sediments and freshwater sediments, until chemical, physical, radiochemical and/or biological examination can be undertaken in the laboratory.

The procedures in this part of ISO 5667 are only applicable to wet samples of sludge, sediment and suspended matter.

**NOTE** Samples of sludge, sediment and suspended matter that are dried or freeze-dried behave similarly to dried soils. For guidance on long- and short-term storage of (freeze) dried samples, see ISO 18512. For guidance on freeze-drying, see ISO 16720.

### 2 Normative references

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 3696, *Water for analytical laboratory use — Specification and test methods*

### 3 Terms and definitions

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

#### 3.1

##### **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

[ISO 11074:2005, 4.4.20]

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

NOTE Adapted from ISO 11074:2005, 4.4.22.

**3.3 storage duration**  
period of time between sample collection and start of the analysis of the sample in the laboratory, for a sample stored under pre-defined conditions

## 4 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 used should be of at least analytical reagent grade.

**4.1 Deionized water**, Grade 3 quality as specified in ISO 3696.

**4.2 Sodium sulfate**,  $\text{Na}_2\text{SO}_4$ , monohydrate.

Heat the sodium sulfate before use for at least 6 h at  $(500 \pm 10)^\circ\text{C}$ . Store in an desiccator after heating.

**4.3 Zinc acetate**,  $(\text{CH}_3\text{COO})_2\text{Zn} \cdot 2\text{H}_2\text{O}$  (10 % mass fraction)

**4.4 Methanol**,  $\text{CH}_3\text{OH}$ .

**4.5 Ethanol**,  $\text{C}_2\text{H}_5\text{OH}$  (volume fraction of 96 %).

**4.6 Sodium tetraborate** ( $\text{Na}_2\text{B}_4\text{O}_7 \cdot 10\text{H}_2\text{O}$ ), **sodium phosphate** ( $\text{Na}_4\text{P}_2\text{O}_7 \cdot 10\text{H}_2\text{O}$ ) or **hexamethylene-tetramine** [ $(\text{CH}_2)_6\text{N}_4$ ].

**4.7 Formaldehyde solution**,  $\text{CH}_2\text{O}$  (volume fraction of 3,7 %).

Add 37 % formaldehyde neutralized to pH 7 with sodium tetraborate, sodium phosphate or hexamethylenetetramine (100 g/l formalin solution) to give a final solution of 3,7 % formaldehyde (corresponding to a 1 to 10 dilution of formalin solution).

NOTE 37 % formaldehyde is 100 % formalin.

## 5 Preservation of samples

### 5.1 General considerations

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

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 might biodegrade, volatilize, oxidize, be reduced or photolyse 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 recommendations can be given for a universal preservation or storage method. A preservation method used 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 different method such that the full range of required analyses is represented.

## 5.2 Chemical examination

Chemical analysis can be performed to determine the nature and amounts of the substances that have become absorbed or adsorbed by sludge, sediment and 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 1). The guidance given in this part of ISO 5667 is relevant to the determination of components in the sum of the separate phases of a sludge or sediment, unless otherwise indicated.

Preservation of samples by fast-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.

## 5.3 Physical examination

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 or sediment 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. Fast-freezing of the sludge and sediments may be appropriate. In some cases, thermal techniques should be avoided as they strongly modify sludge structure, thus affecting physical characteristics (e.g. de-waterability, settleability, flowability).

Samples should be stored and preserved in accordance with the conditions given in Table 1.

## 5.4 Radiochemical examination

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

Samples should be stored and preserved in accordance with the conditions given in Table 2.

## 5.5 Biological examination

Biological studies include toxicological, ecotoxicological and ecological examinations. The same factors mentioned in relation to chemical examination (see 5.2) can alter the bioavailability and toxicity of compounds.

The assessment of sludge contamination by laboratory bioassay testing requires different preservation techniques in comparison to ecological or microbial investigation. An ecological investigation generally involves classifying the species and numbers of flora and/or fauna present on and in fixed sludge or sediments.

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

Any large individuals of macrofauna should be removed from the samples immediately after collecting samples taken for the chemical, physical, radiochemical and/or biological examinations.

Samples should be stored and preserved in accordance with the conditions given in Tables 1 and 3.

## 6 Safety precautions

### 6.1 Staff protection

Health and safety precautions should be observed at all times when sampling potentially hazardous sludge, sediments or suspended matter.

Human exposure to pathogenic organisms or pollutants should be avoided by using appropriate protective equipment such as respiratory protective masks, safety glasses and protective gloves. The hazard due to pathogenic organisms can be very high. It is vital that all sampling personnel should receive thorough training and be provided with appropriate medical inoculations.

Degradation of sludge produces methane, which presents a risk of fire and explosion if a source of ignition is present. Containers should be appropriately wrapped to minimize the fragmentation of the containers if an explosion occurs.

If sludge samples are to be taken in locations where there is restricted ventilation, staff should take safety precautions to protect themselves against sulfide, carbon dioxide and methane.

### 6.2 Sample protection

When sampling, transporting and utilizing sludge, care should be taken to prevent a build-up of gas pressure in the sample container. Manual release of pressure during and after transport may be necessary if prolonged storage is required.

Samples collected for the analysis of volatile organic or sulfide compounds should not be homogenized because many of these compounds could be lost while compositing.

## 7 Containers

Sample containers should be made of a material appropriate for preserving the natural properties of both the sample and the expected range of contaminants. Suitable types of container for each analyte to be measured are given in Tables 1, 2 and 3.

If the samples are to be frozen, suitable material such as polyethylene or polytetrafluoroethylene (PTFE) should be used to minimize the risk of breakage.

Careful consideration should also be given to the suitability of the container for cleaning/decontamination or disposal and appropriate action taken. Recommendations for the preparation of containers are given in Annex A.

The choice of sample container is of major importance and ISO 5667-1 provides guidance on this subject.

In 5.3 of ISO 5667-14:1998, guidance is given on measuring the contamination impact of the container. The analyte level in the blank should be negligible compared to the analyte level to be measured in the sample.

NOTE Regular container volumes are 500 g to 1 000 g.

## 8 Sample collection

Samples should be collected in sufficient volumes to allow:

- a) separate sub-samples to be preserved for each type of analysis or examination to be undertaken;
- b) repeat the analysis in the event of error checking or the routine quality control requirements of duplicate analysis;
- c) prepare time-dependent composites; for example, a daily aliquot of sewage works sludge (preserved as appropriate) may be retained to produce a composite for monthly analysis.

For sludge samples, it is recommended that the container is filled to a maximum of 80 % of its capacity, especially if biological activity is expected, in order to reduce the risk of overpressurization and explosion.

If analysis of volatile compounds is required, containers should be completely filled with sample sediment from the first grab, prior to sample homogenization. No headspace should remain in either container.

If the sample is to be frozen, enough headspace should be allowed for expansion to take place.

Where samples are collected for the purpose of microscopic examination, for example of activated sludges, it is recommended to fill the container to no more than 5 % of its capacity to ensure an oxygen supply to the sludge prior to examination.

The temperature of the sample, especially of sludge samples, can influence the properties of the sample. Therefore, the initial temperature of the sludge samples should be measured on site and recorded.

## 9 Identification of samples

Container labels should withstand wetting, drying and freezing without detaching or becoming illegible. The labelling system should be waterproof to allow use in the field.

The exact information given in the sampling report and on the sample labels will depend on the objectives of the particular measurement programme. In all cases, an indelible label should be secured to the sample container.

For each sample, the following information should be provided as a minimum:

- a unique identifier, traceable to the date, time and location of sampling;
- a description and disposition of sample;
- the name of the individual sampler;
- details of preservation used;