INTERNATIONAL STANDARD

ISO 5667-15

First edition
1999-08-01

Water quality — Sampling —

Part 15: Guidance on preservation and handling of sludge and sediment samples

Qualité de l'eau — Échantillonnage
Partie 15: Guide général pour la préservation et le traitement des échantillons de boues et de sédiments

Reference number
ISO 5667-15:1999(E)

Contents

1 Scope 1

2 Normative reference 1

3 Sample storage, preservation and handling 1

3.1 General considerations 1

3.2 Chemical examination 1

3.3 Physical examination 2

3.4 Biological examination 2

3.5 Feasible precautions 2

3.6 Sample handling 3

3.7 Sample preservation 3

3.8 Sample storage 3

4 Sample record and quality assurance 4

Bibliography 7

International Organization for Standardization
Case postale 56 • CH-1211 Genève 20 • Switzerland
Internet iso@iso.ch

Printed in Switzerland

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 3.

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

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*

— *Part 5: Guidance on sampling of drinking water*

— *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 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 water, wastewater and related sludges*

— *Part 14: Guidance on quality assurance of environmental water sampling and handling*

— *Part 15: Guidance on preservation and handling of sludge and sediment samples*

— *Part 16: Guidance on biotesting of samples*

— *Part 17: Guidance on sampling of suspended sediments*

— *Part 18: Guidance on sampling of groundwater on potentially contaminated sites*

Water quality — Sampling —

Part 15:
Guidance on preservation and handling of sludge and sediment samples

1 Scope

This part of ISO 5667 provides guidance on the procedures for preservation and handling of sewage and waterworks sludges, suspended matter, and saltwater and freshwater sediments for subsequent analysis.

2 Normative reference

The following normative document contains provisions which, through reference in this text, constitute provisions of this part of ISO 5667. For dated references, subsequent amendments to, or revisions of, any of these publications do not apply. However, parties to agreements based on this part of ISO 5667 are encouraged to investigate the possibility of applying the most recent edition of the normative documents indicated below. For undated references, the latest edition of the normative document referred to applies. Members of ISO and IEC maintain registers of currently valid International Standards.

ISO 5667-3 :1994, *Water quality — Sampling — Part 3: Guidance on the preservation and handling of samples.*

3 Sample preservation and handling

3.1 General considerations

Storage begins when the samples are taken. All storage methods will affect the sample to some extent, and the choice of preservation technique depends mainly on the objective of the sample collection. It is important that the effects that preservation and storage techniques can have on sample quality and the results of analysis are understood.

Sludge and sediment samples are subject to chemical, physical and biological changes as soon as they are collected. If guidance is required on the type of sampling technique to be used, guidance is given in ISO 5667-12 and ISO 5667-13. Sample handling, preservation and storage shall be designed to minimize any changes in composition of the sample by retarding chemical and/or biological activity and by avoiding contamination. Specific preservation techniques are often necessary for a representative evaluation of the sludges and sediments, and a variety of chemical, physical and biological investigations can be performed on the collected samples.

No single preservation method is applicable to all constituents. The objective of the sampling programme and the nature of the analytical method will determine the handling operation or preservation technique.

3.2 Chemical examination

In this type of investigation, the nature and amounts of the substances which have become absorbed or adsorbed by the sludges and sediments can be determined. Partition of chemicals between solid phase and water phase is influenced by several factors, such as particle size, amount of organic matter, pH, redox potential or salinity. The study of such attributes may be a sampling objective and therefore the preservation needs of 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 pollutants. In addition to biodegradation of organics, volatilization is a principal mechanism of loss of volatile compounds during sample handling.

Anoxic samples require appropriate preservation techniques such as oxygen exclusion during sample handling. Where refrigeration is not available at the time of collection of liquid sludge samples, particularly in countries with high ambient temperatures, preservation of the samples for sulfide determination can be achieved by raising the pH to greater than 10,5. Analysis should still be carried out as soon as possible after collection. 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.

3.3 Physical examination

In this type of examination, the structure, texture and, for sediments, the layer formation, are determined. Sediment fabric 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 sample integrity is important, agitation- and vibration-free conditions should be maintained during transport; fast-freezing of the sludges and sediments may be appropriate.

3.4 Biological examination

Biological studies include toxicological, ecotoxicological and ecological examinations. The same factors mentioned in relation to chemical investigations could alter bioavailability and toxicity of compounds. Chemicals might biodegrade, volatilize, oxidize or photolyze during storage. Therefore, careful consideration should be given to these processes and the storage conditions needed to avoid such alterations. However, 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 sludges or sediments. On the other hand, microbial activity may also be of interest to characterize samples and can only be determined without fixation. Microbial activity may be responsible for changes in the nitrate-nitrite-ammonia content, for decreases in biochemical oxygen demand, or for reducing sulfate to sulfide.

To minimize any changes due to microbial activity, samples should be kept as cool as possible, without freezing, until analysis. For bacteriological examination, sterile glass containers shall be used. Containers should be able to withstand a sterilization temperature of 175 °C for 1 h and should not produce or release at this temperature any chemicals that would influence biological activity. It may be possible to use disposable commercial plastics containers, subject to verification of the absence of interference with the analysis. Manipulation of the samples is often necessary, and the optimal methods depend on the study objectives.

3.5 Feasible precautions

3.5.1 Safety precautions

It is essential for proper health and safety precautions to be observed at all times when sampling potentially hazardous sludges or sediments. Human exposure to pathogenic organisms or pollutants should be avoided by using respirators, safety glasses and appropriate protective gloves. Primary digestion of sludge produces methane, which presents a risk of fire and explosion if a source of ignition is present. Containers should be wrapped with waterproof adhesive tape to minimize the fragmentation of the containers if an explosion occurs. Care should be taken when sampling, transporting and utilizing the sludge, 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.

3.5.2 Other precautions

See ISO 5667-3 for precautions to be taken in the preparation, filling and use of appropriate containers.

Sample containers should be made from materials appropriate for conserving the natural properties of both the sample and the expected spectrum of contaminants. Careful consideration should also be given to their suitability for cleaning/decontamination or disposal.

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

3.6 Sample handling

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, sieving, dilution for concentration-effect determinations, and addition of chemical preservatives all complicate interpretations of *in situ* comparisons. All information regarding sample manipulation, handling and storage should therefore be included in a sampling report.

Generally, samples should completely fill the storage container, leaving no air space. However, note that the final method of analysis may determine or influence the void space in the container. If the sample is to be frozen, enough air space should be allowed for expansion to take place. Sufficient sample volume should be collected to allow:

— separate sub-samples to be preserved for each type of analysis or examination to be undertaken;

— repeat analysis in the event of error checking or the routine quality control requirements of duplicate analysis (see clause 4); and

— the preparation of 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.

3.7 Sample preservation

Because the first few hours after sampling are the most critical for changes to occur in the sample, preservation steps should be taken where possible immediately upon sample collection. No recommendations can be given for a universal preservation or storage technique. A technique for one group of analyses may interfere with other analyses. To overcome this problem, a sufficient sample volume should be taken to allow specific preservation or storage techniques for each specific investigation.

Refrigeration at 2 °C to 5 °C is the recommended basic preservation method. Freezing or addition of chemicals is recommended for determining organic constituents. Samples for particle analyses or biological examinations should be preserved at 2 °C to 5 °C , never frozen or dried. All means of preservation, if practical, should be carried out in the field prior to transportation.

If final preservation methods are not possible in the field, the sample should be transported in coolers filled with ice to retain the integrity of the collected material. To avoid loss of volatile species, samples should be collected in a completely filled container, overfilling it before capping or sealing. Temperature is the most important factor affecting the samples, from the time of sample collection through handling to the final analyses. Refrigeration is easily accomplished with coolers and ice. Samples that are to be frozen may simply be placed in a cooler with dry ice. Any deviation should be recorded in a sampling protocol.

More detailed guidance on specific sample preservation methods is given in Table 1.

3.8 Sample storage

The time elapsed between sample collection and analysis should be as short as possible. Preservation and storage are two linked aspects of sample handling. Samples should be transported and stored wherever possible at 2 °C to 5 °C so as to avoid the possible loss of volatiles and to minimize biologically induced change. Glass containers should be used, with appropriate precautions applied to prevent gas production and gas build-up. If trace organics are not suspected to be significantly volatilized into the gas phase, a regime of regularly opening the container to relieve pressure during storage should be adopted. Fermentable samples (nearly all biologically derived sludges) should, where possible, not be stored in glass containers without rendering them biologically inert to prevent the risk of explosion due to gas generation. Storage in the dark should be maintained to avoid the growth of algae and the stimulation of other biological activity.

The duration of sample storage for chemical evaluations is specific to the chemical analyses to be conducted (see Table 1). For example, for metals (excluding chromium), if samples are not be analysed within one month, they should be frozen or freeze-dried for storage up to 6 months. For ecotoxicological studies, the samples should be tested within two weeks of collection. Bacteriological examinations should be processed within 6 h and microbial activity should be measured immediately. When determination of trace organics is required, the analysis should be

performed on as-received samples. If significant gas-phase volatility is suspected, then analysis should be undertaken as soon as possible after sampling. The storage conditions of samples should allow maintenance of anaerobic or aerobic conditions as appropriate, but a final decision on oxygen exclusion should only be made with a knowledge of the redox potential relative to an aerobic state.

4 Sample record and quality assurance

For general guidance on identification and reception of the samples in the laboratory, proceed in accordance with ISO 5667-3. Documentation of the collection and analysis of environmental samples requires all the information necessary to trace a sample from the field to the final result of analysis. In all steps, systematic or accidental errors can occur. Consequently, a certain number of extra samples should be collected to allow for unexpected problems with sample transportation or preservation.

Quality assurance encompasses a complex integrated system of management activities. It should be utilized if at all possible to achieve optimum confidence in all results. ISO/TR 13530 and ISO 5667-14 should be referred to for details of the procedures to be followed. Personnel should be thoroughly familiar with these procedures before sampling is initiated.

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 (see 3.5.2) and contain at least the following information:

— date, time and location of sampling,

— sample number,

— description and disposition of sample,

— name of sampling personnel,

— type of preservation used,

— type of sample storage used/required, and

— any information regarding integrity and manipulation of the sample.

Table 1 — Sample containers, preservation and storage conditions for different parameters measured in sediments and sludges

Analysis or test	Container	Preservation	Storage conditions	Storage duration	International Standard
Acidity	Polyethylene/ Glass	Refrigerate	2 °C to 5 °C/dark/airtight	14 days	
Alkalinity	Polyethylene/ Glass	Refrigerate	2 °C to 5 °C/dark/airtight	14 days	
pH	Sampling device	Wet undisturbed	Determined in the field	None	
pH (with temperature correction)	Polyethylene/ Glass	Refrigerate	2 °C to 5 °C/dark/airtight	24 h	
Conductivity	Polyethylene/ Glass	Refrigerate	2 °C to 5 °C/dark/airtight	24 h	
Kjeldahl nitrogen	Polyethylene/ Glass	Refrigerate	2 °C to 5 °C/dark/airtight	1 month	

Analysis or test	Container	Preservation	Storage conditions	Storage duration	International Standard
Ammoniacal nitrogen	Polyethylene/ Glass	Refrigerate	2 °C to 5 °C/dark/airtight	As short as possible	
Total residue	Glass	Refrigerate	2 °C to 5 °C/dark/airtight	8 days	
Anions (e.g. sulfate)	Polyethylene/ Glass	Refrigerate	2 °C to 5 °C/dark/airtight	28 days	ISO 11048
Nitrate	Polyethylene/ Glass	Refrigerate	2 °C to 5 °C/dark/airtight	2 days	
Nitrite	Polyethylene/ Glass	Refrigerate	2 °C to 5 °C/dark/airtight	As short as possible	
Sulfide	Polyethylene/ Glass	Refrigerate pH >10,5	2 °C to 5 °C/dark/airtight /anoxic	As short as possible	
Phosphorus	Glass	Refrigerate	2 °C to 5 °C/dark/airtight	1 month	
Orthophosphate	Glass	Refrigerate	2 °C to 5 °C/dark/airtight	2 days	
Cyanides	Polyethylene	Freeze	⩽−20 °C/dark/airtight	1 month	
Metals	Polyethylene Polyethylene Polyethylene/ Glass	Refrigerate Freeze Dry (60°C)	2 °C to 5 °C/dark/airtight ⩽−20 °C/dark/airtight Ambient temperature dark/airtight	1 month 6 months 6 months	
Mercury	Glass/PTFE	Refrigerate Freeze	2 °C to 5 °C/dark/airtight ⩽−20 °C/dark/airtight	8 days 1 month	
Chromium(VI)	Polyethylene	Refrigerate	2 °C to 5 °C/dark/airtight	2 days	
Particle size	Polyethylene/ Glass/Metal	Refrigerate	2 °C to 5 °C/dark/airtight	1 month	
TOC	Glass with PTFE-lined cap	Refrigerate Freeze	2 °C to 5 °C/dark/airtight ⩽−20 °C/dark/airtight	1 month 6 months	
Semi- and non-volatile organic compounds (PCBs, PAHs, pesticides, high molecular weight hydrocarbons)	Glass with PTFE-lined cap Aluminium foil /Glass with aluminium foil	Refrigerate Freeze Dry	2 °C to 5 °C/dark/airtight ⩽−20 °C/dark/airtight Ambient temperature dark/airtight	1 month 6 months 6 months	
Mineral oil	Glass with PTFE-lined cap	Refrigerate Freeze	2 °C to 5 °C/dark/airtight ⩽−20 °C/dark/airtight	24 h 1 month	