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Sludge, treated biowaste, and soils in the landscape – Sampling – Part 4: Guidance on procedures for sample packaging, storage, preservation, transport and delivery

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Foreword

This Technical Report (prCEN/TR xxxx-4) has been prepared by Technical Committee CEN BT TF 151 “Horizontal”, the secretariat of which is held by DS.

This document is currently submitted to the CEN Enquiry.

This document has been prepared under a mandate given to CEN by the European Commission and the European Free Trade Association, and supports essential requirements of EU Directive(s).

The following TCs have been involved in the preparation of this document:

CEN/TC 292 Characterization of waste

This Technical Report is one of a series of five Technical Reports dealing with sampling techniques and procedures, and provides essential information for the application of the European Standard:

prEN xxxxx: Sludge, treated biowaste, and soils in the landscape – Sampling – Framework for the preparation and application of a sampling plan

The subject of the Framework Standard is the preparation of a sampling plan. The Framework Standard can be used to:

— produce standardized sampling plans for use in regular or routine circumstances;

— incorporate specific sampling requirements into national legislation;

— design and develop a sampling plan on a case by case basis.

The Technical Reports display a range of potential approaches and tools to enable the sampling plan to be tailored to a specific testing scenario. This approach allows flexibility in the selection of the sampling approach, sampling point, method of sampling and equipment used.

This Technical Report describes the boundary conditions and procedures, appropriate for application in the field, for the packaging, preservation, storage and transport of samples to assist in maintaining their integrity prior to delivery at the laboratory. The laboratory facility should be consulted on the selection of the most appropriate procedure to ensure compatibility with the chosen analytical methodology and parameters to be tested.

Introduction

Sludge and treated biowaste can be applied to land for the purpose of beneficial land use. The testing of sludge, treated biowaste and soil enables informed decisions to be made on whether land application is appropriate (or not). To undertake valid tests a (number of) representative sample(s) of the sludge, treated biowaste or land will be needed.

The subject of the Framework Standard prEN xxxxx is the preparation of a sampling plan, within the framework of an overall testing programme as illustrated in Figure 1 of prEN xxxxx:date.

The development of a sampling plan within this framework involves the progression through three steps or activities.

1) define the sampling plan;

2) take a field sample in accordance with the sampling plan;

3) transport the laboratory sample to the laboratory.

This Technical Report provides information to support Key Steps 2 and 3 of the sampling plan development and elaborates on methods and boundary conditions for preserving, packaging and storing samples to preserve their integrity, in addition to the transport and delivery of a sample to the designated analytical facility.

Sample integrity might be compromised if insufficient attention is paid to correct packaging, preservation, storage and transport techniques. This might result in a sample which is not representative of the sample population. The selection of the most appropriate procedure needs to be undertaken in collaboration with the analytical laboratory that is to undertake testing to ensure compatibility with the chosen analytical methodology and parameters to be tested. Specifically this Technical Report supports Clause 4.2.9.3 (Procedures for packaging, preservation, storage, transport and delivery) of the Framework Standard.

This Technical Report should be read in conjunction with the Framework Standard for the preparation and application of a sampling plan as well as the other Technical Reports that contain essential information to support the Framework Standard. The full series comprises:

prEN xxxxx: Sampling of sludge, treated biowaste, and soils in the landscape – Sampling – Framework for the preparation and application of a sampling plan

prEN ZZZZ: Sludge, treated biowaste, and soils in the landscape – Sampling – Vocabulary

prCEN/TR XXXX-1: Sludge, treated biowaste, and soils in the landscape – Sampling – Part 1: Guidance on selection and application of criteria for sampling under various conditions.

prCEN/TR XXXX-2: Sludge, treated biowaste, and soils in the landscape – Sampling – Part 2: Guidance on sampling techniques

prCEN/TR XXXX-3: Sludge, treated biowaste, and soils in the landscape – Sampling – Part 3: Guidance on sub-sampling in the field

prCEN/TR XXXX-4: Sludge, treated biowaste, and soils in the landscape – Sampling – Part 4: Guidance on procedures for sample packaging, storage, preservation, transport and delivery

prCEN/TR XXXX-5: Sludge, treated biowaste, and soils in the landscape – Sampling – Part 5: Guidance on the process of defining the sampling plan

The Technical Reports contain procedural options (as detailed in Figure 2 of prEN xxxx:date) that can be selected to match the sampling requirements of any testing programme.

1 Scope

This Technical Report describes procedures for the packaging, preservation, storage and transport of samples of sludge, treated biowaste, and soils in the landscape. This document gives guidance on how to store and preserve samples for routine testing, e.g. by environmental laboratories, where samples typically are stored for a short time after sampling in order to carry out some additional tests or in order to confirm results found.

Where available and appropriate for field application, requirements for specific storage conditions and/or preservation methods should be selected from the chosen analytical standard and in collaboration with the testing laboratory.

2 Normative references

The following 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.

prEN ZZZZ: Sludge, treated biowaste, and soils in the landscape – Sampling – Vocabulary

3 Terms and definitions

For the purposes of this document, the terms and definitions given in prEN ZZZZ apply.

4 Packing and labelling the laboratory sample

4.1 Selecting a sample container

4.1.1 General

Sample containers can be made from many different types of materials, some of which might react with or contaminate a specific type of sample. To avoid any accidental contamination by the sample container or changes in the sample, advice should be sought, usually from the receiving laboratory, regarding the type and size of sample(s) and container(s), appropriate preservation method(s) if applicable, maximum storage time prior to analysis, and the labelling system. The maximum storage time prior to analysis will indicate the period of time available before the sample needs to arrive at the laboratory. In general, this period has to be as short as possible. Once the details have been agreed and recorded in the sampling plan, the methods detailed in the sampling plan should be followed.

The purpose of the sample container is to protect the sample during transport and storage until it is further treated or analyzed. A container should be compatible with the nature of the material sampled and the constituents to be analyzed.

Where specialized containers and preservatives are advised containers that conform to the characteristics of the analytical procedure to be used.

Table A.1 details types of containers, preservation and storage conditions for different types of parameters associated with a particular analysis or test.

4.1.2 Type of container

Sample containers should be made from materials appropriate for maintaining the natural properties of both the sample and the expected spectrum of constituents. Consideration should be given to the sample preservation method, e.g. if samples are to be frozen, glass is not suitable, and to the suitability of the container for cleaning/decontamination or disposal. The following factors can influence the choice of sample container and container preparation:

— possible loss of the constituents to be measured caused by adsorption onto the walls of the container;

— contamination of the container prior to sampling by improper cleaning;

— contamination of the sample by the material of which the container is made;

— reaction between constituents of the sample and the container;

— resistance to temperature extremes;

— resistance to breakage;

— water and gas tightness;

— ease of reopening;

— size, shape and mass volume;

— ease of filling and removing sample;

— availability;

— differences in the cost if more than one type of container is suitable.

4.1.3 Shape and size of container

The analytical laboratory should be consulted regarding type and size of sample containers. The sample size is a function of the volume of sample needed to complete the analytical suite and the volume of sample needed to be stored for investigation at a later date. Ease of packing for transport to the laboratory or available space in the laboratory if long-term storage is needed might influence the choice of container shape.

For some samples, the amount of headspace above the sample is important. Instruction should be obtained from the analytical laboratory on appropriate headspace, if any, and a container selected that can accommodate the sample volume and sufficient headspace. Either completely fill the container or allow headspace as appropriate for the material being sampled and the planned analyses (see 4.2.2, Filling containers).

4.1.4 Preparation of sample containers

Commonly, instruction on the use of appropriate cleaning protocols is obtained from the analytical laboratory.

NOTE In most cases, containers supplied by the analytical laboratory will have been suitably cleaned.

The choice of cleaning method will depend on the constituents to be analysed. Containers can be cleaned with mixtures of acids followed by rinsing with de-ionized water. Samples that are to be analysed for organic constituents can be stored in containers that are solvent-rinsed and/or heated in a calcination oven. Cleaning procedures might differ depending on the type of material to be packed and the constituents to be analysed. Advice should be to establish the most suitable procedure in each case. In general, the reuse of sample containers is not advised.

Containers should be sterilized after cleaning if sterilized or disinfected sewage samples are to be collected and analysed for microbiological constituents. New, food quality containers can be regarded as suitably sterile.

4.2 Packaging

4.2.1 General

Using the guidance given in 4.,1select a container that is compatible with the analytical determinants. Wide-neck containers aid sample handling, e.g. for dewatered sludges or coarse-grained treated biowaste.

4.2.2 Filling containers

Either completely fill the container or allow headspace as appropriate for the material being sampled, determinants to be analysed and the method of preservation.

Problems can arise when the volume of the container is much larger than the volume of material it contains:

- enhanced agitation during transport can lead to breakdown of aggregated particles;
- interaction with the gas phase can lead to stripping.

Problems related to complete filling can be:

- oxygen depletion, with possible decomposition, leading to anaerobic conditions and the formation of toxic metabolites (e.g. nitrite, sulphide);
- difficulty homogenizing the sample by shaking or stirring the total volume.

Air space should be minimized, to prevent significant oxidation and/or carbonation reactions, both at the top of the container and, for granular materials, between the particles. When freezing is used as a preservation method, some additional space might be needed within the container to allow for expansion; particularly for samples with high water content, such as liquid sludges.

To avoid loss of volatile species, samples should completely fill the container; overfill the container before capping or sealing.

For samples which are biologically reactive, or have the potential to generate gas or to significantly change in volume with relatively small changes of temperature, some headspace volume is required above the sample and the sample container should be able to withstand an increase in inner pressure. In some cases the internal pressure can be sufficient to cause glass bottles to explode.

For microbiological examination, the sample container should not be filled to the brim so that airspace is left. This aids mixing before examination and minimizes the risk of accidental contamination.

4.2.3 Sealing containers

Laboratory samples for dispatch or transport by third parties and reserved laboratory samples should be sealed in such a manner that the integrity of the sample is protected. Sealing of the samples with a tamper-evident seal might be required when samples are taken for (potential) regulatory investigations.

NOTE The use of certain seals might result in problems reading the label, particularly digital labels such as bar codes.

The container should be secured and sealed in such a manner that it can be opened only by breaking the seal.

Alternatively, the container can be placed in a robust package and sealed in such a manner that no part of the sample can be removed without breaking the seal of the package.

4.2.4 Labelling

Apply a clearly legible, unique, unambiguous code to each sample container, for example by:

a) using adhesive labels pre-printed with the sample identity and bar codes;

b) writing directly on to the container using a permanent marker pen;

c) writing on an adhesive label and sticking it to the sample container.

Attach the label to the main body of the container. When labels are attached to the lid, top or cap of the container either ensure that the lid, top, or cap does not become separated from the container, or that both parts are separately identified as being part of a single unit.

Select a label of a quality capable of remaining firmly attached to the sample container whatever the storage period and prevailing conditions. If there is a risk that the label might become detached, for example as a result of condensation caused by cooling, place the container in a plastic bag and seal closed.

Mark the label with all the information necessary for unequivocal identification of the sample.

NOTE **I**nk, when used, should be indelible and sample codes should be kept short and simple to avoid mistakes when transcribing numbers.

5 Preserving the laboratory sample

5.1 General

Ideally, samples should be analysed as soon as possible after collection, but preservation techniques might be required in the field, during transport to the laboratory and during storage before analysis. The constituents to be determined and the length of time between collection and analysis influence the choice of preservation method. The chosen method of preservation is often the method for the whole period before analysis even if sample pre-treatment prior to laboratory analysis is performed. A change of preservation method on arrival at the laboratory often results in repackaging the sample, which can damage the sample by changing the constituents to be analysed.

The method of preservation relates to the stability of samples. Samples can be regarded as stable is they are unlikely to change physically, chemically or biologically after sampling and before analysis. Preservation methods are considered under the following broad headings:

— samples that are stable;

— samples that are unstable but where stability can be achieved by a preservation method;

— samples that are unstable and cannot be readily stabilized.

For those samples that are unstable, it is important to minimize loss or change (physical, chemical or biological) of the constituent. Changes are the result of various environmental factors, including:

— microbiological activity in the sample;

— oxidation of compounds by atmospheric oxygen;

— loss of dissolved volatile constituents due to pressure and/or temperature changes during the sampling process;

— photochemical reactions;

— changes in the chemical nature of certain substances due to changes of temperature, pressure and loss of the vapour phase;

— modification of the pH, conductivity, solubility and carbon dioxide by absorption of CO_2 from the air;

— reaction with carbon dioxide or water;

— irreversible adsorption on the surface of containers of metals in solution or in colloidal state and certain organic compounds.

The choice of preservation method depends on the material to be preserved and the material properties and/or constituent concentrations to be determined. Often several constituents in a sample will be analysed, each needing different methods of preservation. In this case it will be necessary to prepare a number of sub-samples; each preserved using a different method.

The time between sampling and analysis is particularly important for samples in which biological degradation is likely to occur or in which (semi-) volatile organic constituents are to be determined. For these types of samples extended delays between sampling and analysis can result in significant loss of biodegradable, volatile and (semi-)volatile constituents such that the sample is no longer representative of the bulk material that was sampled. It is advisable that the time between sampling and analysis is kept to a minimum.

Common methods of preservation include:

— airtight storage (see 5.2.1);

— dark storage (see 5.2.2);

— cooled storage (2-5 °C) (but might result in precipitation) (see 5.2.3);

— nitrogen atmosphere (see 5.2.4);

— freezing (might change properties by segregation) (see 5.2.5);

— chemical preservation (see 5.2.6);

— drying (might change properties by loss of volatiles (see 5.2.7).

Recommended minimum preservation measures are listed in Table A.1. These methods are further explained in 5.2.

The time given in Table A.1 is the maximum recommended storage time between sampling and the start of analysis and includes the time to transport the sample to the laboratory. To ensure that samples are analysed as soon as possible and not stored beyond the recommended maximum time, samples should be transported to the laboratory as soon as is practical.

5.2 Preservation methods

5.2.1 Airtight storage

When airtight storage is necessary, liquid, paste-like and fine-grained materials should be stored in glass bottles sealed with PTFE (polytetrafluoroethylene) cap liners.

NOTE 1 Plastics are not considered airtight.

NOTE 2 Airtight storage will prevent volatilization of constituents and will reduce biological degradation. However, for granular materials the air volume within the sample is always large; irrespective of tight filling of the sample container. Airtight storage for these types of samples is therefore never a guarantee for keeping the characteristics of the sample constant during the storage period.

NOTE 3 In some cases, inert gas flushing can be used to limit chemical reactions (oxidation, carbonation) or biological degradation. Inert gas flushing might result in losses of volatile/semi-volatile compounds and advice on the necessity and potential detrimental effects on the sample of undertaking such procedures should be sought from the analytical laboratory.

5.2.2 Dark storage

Samples should be kept in the dark to prevent growth of algae and the stimulation of other biological activity.

Dark storage can be achieved by using dark coloured sample containers, wrapping the containers in materials that exclude light or by storing the containers in a dark place.

NOTE 1 In the field a dark environment can be provided by a cool box.

NOTE 2 Bacteria, cysts and oocyst, and virus survival has been shown to be sensitive to light in the UVB and UVC range. When samples are collected for analysis of these organisms, care should be taken to ensure that the container material excludes light in these wavelengths. This is particularly important in low latitude regions.

5.2.3 Cooled storage

When ambient temperatures are above 10°C, samples should be cooled immediately after collection to minimize loss of volatiles and biologically induced change. Cooling necessitates the use of refrigerated vans, refrigerators or cool boxes (containing melting ice or frozen cool packs) at the sampling location.

NOTE 1 Cooling is particularly important for samples collected during warmer periods such as the summer months.

NOTE 2 When cool boxes are used, the volume of the samples, ambient temperature and transport time should be taken into account when deciding the quantity of melting ice or frozen cool packs required.

NOTE 3 Direct contact with frozen cool packs can cause localized freezing of samples with high water content. If this could affect the properties of the sample, preventative action should be taken to ensure that the samples and cool packs are not in direct contact.

5.2.4 Nitrogen atmosphere

When the reducing character of the sample is to be preserved, exposure to oxygen after sampling should be limited. It is common practice to flush the sample container and sample with nitrogen gas. Airtight packaging is then sufficient for short-term storage.

When nitrogen gas flushing is not feasible at the sampling site, the time between sampling and flushing with inert gas after arrival at the laboratory should be kept to a minimum.

Place the sample in a gas-tight container; pass nitrogen gas through the container, applying a volume at least 10 times that of the container.

NOTE 1 A suitable container is one that easily allows flushing to take place, for example, a container with gas inlet/outlet connectors on it.

NOTE 2 Storage using nitrogen gas is possible only in containers with a hermetic gas-tight seal such as a glass sample container welded shut. Other containers, particularly plastic containers, are insufficiently gas tight and would expose the sample to oxygen on storage.

Nitrogen gas flushing might result in losses of volatile/semi-volatile compounds.

5.2.5 Freezing

Fast freezing of samples is not a method that is routinely used in the field. If the samples are to be preserved by freezing, this is usually done at the laboratory as soon as possible after receipt of the samples. Liquid nitrogen or dry ice can be used for immediate deep-freezing of samples. Alternatively, place the samples in a freezer at -20ºC.

In most cases, freezing should be avoided as it modifies sludge structure, thus affecting certain measurements.

Glass containers should not be used when freezing samples with high water content.

Fast freezing of sludges might not be appropriate for samples collected for physical examination, e.g. structure, texture or layer formation. Sample integrity is important and agitation- and vibration-free conditions should be maintained during transport.

5.2.6 Chemical preservation

Chemical preservation is appropriate only in a limited number of situations.

Advice should be sought on the type of preservation methods required.

NOTE 1 Care should be taken to avoid unquantifiable reactions between the sample and any chemical preservatives that might occur during sample preparation (e.g. drying or milling).

NOTE 2 When samples are needed for the determination of extractable levels (e.g. using leaching tests), the addition of chemical stabilizing agents can affect the leachability of other constituents and, if not chosen advisedly, can make it difficult to quantify the true concentrations of those parameters within the material.

NOTE 3 It is very difficult to keep solvents of analytical quality free from contamination, therefore the use of solvents in the field for sample preservation may be limited.

5.2.7 Drying

Preserving samples by drying is not a method that is routinely used in the field, and is usually confined to the laboratory.

6 Transporting the sample

Samples should be transported to the laboratory without delay.

The containers holding samples need to be protected and sealed in such a way that they do not deteriorate and do not lose any part of their contents during transport. Packaging material should protect the containers from possible external contamination and breakage, particularly near the opening of the container, and should not be a source of contamination.

It is recommended that glass containers are not used for fermentable samples (nearly all biologically derived sludges). If they are used, they should be wrapped with waterproof adhesive tape or other equivalent measures taken, such as wrapping in plastic mesh. These methods will minimize the dispersion of fragments of the container if an explosion occurs due to gas generation.

NOTE 1 Certain statutory authorities or other organization(s) might have additional requirements for packaging and transporting samples.

During transport, the samples should be kept as cool as practicable and protected from light.

If the time from sample collection to arrival at the laboratory exceeds the maximum time specified in the sampling plan, the integrity of the samples could be compromised. Before a decision is made to proceed with the analysis, it will need to be demonstrated that conditions during transport comply with the recommended storage conditions and maximum storage duration.

NOTE 2 For regulatory samples, a record of sample environment temperature during transport might be required.

7 Chain of custody forms and sample despatch

If specified in the sampling plan, samples should be accompanied by a chain of custody form.

At each transfer of the samples the chain of custody form should be checked, appropriate additional details recorded, the date and time of transfer recorded, and the form signed.

8 Incorporation in the sampling plan

The storage, preservation and transport procedure(s) and necessary equipment, should be specified in the sampling plan prior to commencing sampling.

Annex A
(normative)
Recommendations on containers, preservation and storage conditions

Table A.1 – Sample containers, preservation and storage conditions for different parameters measured in sludges, treated biowastes and soils

Analysis or test	Type of container [a]	Minimum sample size[b] (g)	Preservation technique	Maximum recommended storage time before analysis	International Standard
Acidity	P or G	50	Cool 2-5°C & dark & airtight	14 d	ISO/WD 5667-15
Alkalinity	P or G	50	Cool 2-5°C & dark & airtight	14 d	ISO/WD 5667-15
Ammoniacal nitrogen	P or G	50	Cool 2-5°C & dark & airtight	as short a time as possible (less than 24 h)	ISO/WD 5667-15
Anions (Br, F, Cl, SO_4)	P or G	50	Cool 2-5°C & dark & airtight	1 m	ISO 18512
AOX (adsorbable organic halides)	P or G	50	Cool 2-5°C & dark & airtight	7 d	ISO/WD 5667-15
Bacteria	P or G (Should be sterile if low levels expected)	100	Cool 2-5°C & dark & airtight Do not freeze	6 h (to start of analysis)	Lambkin et. al., 2005
Conductivity	P or G	50	Cool 2-5°C & dark & airtight	24 h	ISO/WD 5667-15
Chromium (VI)	P or G	50	Cool 2-5°C & dark & airtight	2 d	ISO/WD 5667-15
CST (Capillary suction time)	P or M bottles		Cool 2-5°C & dark & airtight	24 h	ISO/WD 5667-15
Cyanides (total)	P	50	Freeze ≤ -20°C & dark & airtight	1 m	ISO/WD 5667-15
Dry Matter	P or G	50	Cool 2-5°C & dark & airtight	1 m	ISO/WD 5667-15
Helminth ova	P or G	Cool, Do not freeze	Cool 2-5°C & dark & airtight	1 m	Lambkin et. al., 2005
Kjeldahl Nitrogen	P or G	50	Cool 2-5°C & dark & airtight	Sludge: 14 d Soil: 1 m	ISO/WD 5667-15 ISO 18512
Mercury	G or PTFE	50	Cool 2-5°C & dark & airtight	Sludge: 7 d Soil: 4 d	ISO/WD 5667-15 ISO 18512
			Freeze ≤ -20°C & dark & airtight	Sludge: 1 m	ISO/WD 5667-15

Analysis or test	Type of container [a]	Minimum sample size[b] (g)	Preservation technique	Maximum recommended storage time before analysis	International Standard
Metals	P or G	50	Cool 2-5°C & dark & airtight	Sludge: 1 m Soil: 6m	ISO/WD 5667-15 ISO 18512
			Freeze ≤ -20°C & dark & airtight	Sludge: 6 m Soil: 10 y	ISO/WD 5667-15 ISO 18512
			Dry (60°C) ambient temperature dark & airtight	Sludge: 6 m Soil: 30 y	ISO/WD 5667-15 ISO 18512
Mineral oil	G with PTFE-lined cap	100	Cool 2-5°C & dark & airtight	Sludge: 1 m	ISO/WD 5667-15
			Freeze -18°C	Sludge: 6 m	ISO/WD 5667-15
			Magnesium sulphate	Sludge: 6 m	ISO/WD 5667-15
Microbial activity	Sterile Glass	100	None	Sludge: 24 h	ISO/WD 5667-15
Nitrate	P or G	50	Cool 2-5°C & dark & airtight	2 d	ISO/WD 5667-15
Nitrite	P or G	50	Cool 2-5°C & dark & airtight	2 d (as short as possible)	ISO/WD 5667-15
Oocysts	P or G		Cool 2-5°C & dark & airtight Do not freeze	1 m	Lambkin et. al., 2005
Organic matter	G with PTFE-lined cap		Cool 2-5°C & dark & airtight	1 m	ISO/WD 5667-15
			Freeze ≤ -20°C & dark & airtight	6 m	ISO/WD 5667-15
Orthophosphate	G	50	Cool 2-5°C & dark & airtight	2 d	ISO/WD 5667-15
Particle size	P or G or M	100	Cool 2-5°C & dark & airtight	1 m	ISO/WD 5667-15
pH	Sampling Device	50	Wet, undisturbed Determine on site	None	ISO/WD 5667-15
pH (with temperature correction)	P or G	50	Cool 2-5°C & dark & airtight	Sludge: 24 h Soil: 7 d	ISO/WD 5667-15 ISO 18512
Phosphorus (total)	G	50	Cool 2-5°C & dark & airtight	Sludge: 1 m Soil: no recommendation	ISO/WD 5667-15 ISO 18512
Phosphorus (available)	G	50	Cool 2-5°C & dark & airtight	Soil: 7 d	ISO 18512

Analysis or test	Type of container [a]	Minimum sample size[b] (g)	Preservation technique	Maximum recommended storage time before analysis	International Standard
Semi- and non-Volatile Organic Compounds[c] (PCBs, PAHs, pesticides, high molecular weight hydrocarbons)	G with PTFE-lined cap	50	Cool 2-5°C & dark & airtight	1 m (after extraction)	ISO/WD 5667-15
			Freeze ≤ -20°C & dark & airtight	6 m (after extraction)	ISO/WD 5667-15
	G with aluminium foil-lined cap		Ambient temperature & dark & airtight	6 m (dry)	ISO/WD 5667-15
Settleability/ Thickenability	P or M bottles		Cool 2-5°C & dark & airtight	24 h (as short as possible)	ISO/WD 5667-15
Specific resistance to filtration	P or M bottles		Cool 2-5°C & dark & airtight	24 h (as short as possible)	ISO/WD 5667-15
Sulfide	P or G	50	pH > 10,5; Cool 2-5°C & dark & airtight & anoxic	Sludge: 24 h	ISO/WD 5667-15
			Addition of 5 ml Zn Acetate	Sludge: 7d	ISO/WD 5667-15
TOC / OC	G with PTFE-lined cap	25	Cool 2-5°C & dark & airtight	Sludge: 1 m	ISO/WD 5667-15
			Freeze ≤ -20°C & dark & airtight	Sludge: 6 m	ISO/WD 5667-15
Viruses	P or G		Cool 2-5°C & dark & airtight	Complete analysis within 24-48 h	Lambkin et. al., 2005
			Freeze ≤ -20°C & dark & airtight	14 d	Lambkin et. al., 2005
Volatile organics[c] as-received	G or M rings with PTFE-lined cap	50	Addition of methanol	Sludge: 24 h Soil: 1 m	ISO/WD 5667-15 ISO 18512
			Cool 2-5°C & dark & airtight	Sludge: 1 m (after extraction) Soil: 4 d	ISO/WD 5667-15 ISO 18512
			≤ -20°C & dark & airtight	Sludge: 6 m	ISO/WD 5667-15

NOTE 1 There have been few scientific studies on maximum storage times. Some additional information is provided in EN ISO 5667-3:2003 and ISO/DIS 18512:2006.

NOTE 2 Guidance on preservation techniques should be sought from the chosen analytical laboratory.

NOTE 3 The temperature for cooling refers to the temperature of the sample environment, not the temperature of the sample itself.

a

P = Plastics, e.g. polyethylene, PTFE (polytetrafluoroethylene), PVC (polyvinyl chloride), PET (polyethylene terephthalate)

G = Glass

M = Metal

Analysis or test	Type of container [a]	Minimum sample size[b] (g)	Preservation technique	Maximum recommended storage time before analysis	International Standard
b Minimum field sample mass for one laboratory analysis based on wet material. For sludges, it is necessary to know before sampling how much dry sample is needed for the analysis and the approximate water content due to their high water content.					
c Volatile components are organic and inorganic components with a boiling point less than 300 °C. All organic components with a boiling point above 300 °C are here to be considered as semi-volatile.					

Bibliography

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ISO/WD 5667-15, Water quality – Sampling – Part 15: Guidance on preservation and handling of sludge and sediment samples [ISO/TC 147/SC 6/WG 3]

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prCEN/TR XXXX-1: Sludge, treated biowaste, and soils in the landscape – Sampling – Part 1: Guidance on selection and application of criteria for sampling under various conditions.

prCEN/TR XXXX-2: Sludge, treated biowaste, and soils in the landscape – Sampling – Part 2: Guidance on sampling techniques

prCEN/TR XXXX-3: Sludge, treated biowaste, and soils in the landscape – Sampling – Part 3: Guidance on sub-sampling in the field

prCEN/TR XXXX-5: Sludge, treated biowaste, and soils in the landscape – Sampling – Part 5: Guidance on the process of defining the sampling plan

[1] Lambkin, D C, S Nortcliff and T C White, 2005, HORIZONTAL Desk Study: Sample handling protocols for sludges and treated biowaste for microbiological analysis