



SSPI-CT-2003-502411

HORIZONTAL - ORG

**HORIZONTAL STANDARDS ON ORGANIC
MICRO-POLLUTANTS FOR IMPLEMENTATION
OF EU DIRECTIVES ON SLUDGE, SOIL AND
TREATED BIO-WASTE**

Instrument: STREP

**Thematic Priority: PRIORITY 8.1 STREP Topic 1.5
Environmental assessment**

**D 2.2 - part C. Sampling of sewage sludge and treated
biowastes – Technical Report on Sampling – Guidance on
procedures for sample packaging, storage, preservation,
transport and delivery**

Due date of deliverable: 1-1-2006

Actual submission date: 30-1-2006

Start date of project: 1-10-2003

Duration: 3 years

**Organisation name of lead contractor for this
deliverable: University of Reading (partner 4)**

Revision [draft]

Project co-funded by the European Commission within the Sixth Framework Programme (2002-2006)		
Dissemination Level		
PU	Public	PU
PP	Restricted to other programme participants (including the Commission Services)	
RE	Restricted to a group specified by the consortium (including the Commission Services)	
CO	Confidential, only for members of the consortium (including the Commission Services)	

Disclaimer

The European Commission does not accept any liability for the information contained in this document resulting from work carried out under contract with the FP 6 programme of the European Commission. The conclusions herein reflect at this point the opinion of the authors. After consultation, it can be considered to reflect the consensus opinion of the CEN bodies involved in project HORIZONTAL. The opinions are not necessarily consistent with the views of the European Commission.

Acknowledgement

This work has been carried out with financial support from the European Commission, 6th Framework Programme as well as the following EU Member States: UK, Germany, France, Italy, Spain, Nordic countries, Netherlands, Denmark, Austria, EU DG XI and JRC, Ispra.

Document type: European Standard
Document subtype:
Document stage: CEN Enquiry
Document language: E

D:\AdLib eXpress\Special\WebQuality\RestrictedEditing\Input\D2.2 part C Sampling of sludge and treated biowaste.doc STD Version 2.1c2

CEN/TC 151

Date: 2004-12

TC 151 WI 151

CEN/TC 151

Secretariat: DS

Introductory element — Main element — Complementary element

**Introductory element – Sampling of sewage sludge and treated biowastes –
Technical report on sampling – Guidance on procedures for sample
packaging, storage, preservation, transport and delivery**

Einführendes Element — Haupt-Element — Ergänzendes Element

Élément introductif — Élément central — Élément complémentaire

ICS:

Descriptors:

Contents

Page

1	Scope	7
2	Normative references	8
3	Terms and definitions	8
4	Packing and labelling the sample	9
4.1	General	9
4.2	Selecting a sample container	9
4.2.1	General	9
4.2.2	Type of container	10
4.2.3	Size and shape of container	10
4.2.4	Preparation of sample containers	10
4.3	Packaging	11
4.3.1	General	11
4.3.2	Filling containers	11
4.3.3	Sealing containers	12
4.3.4	Labelling	12
5	Sample preservation	12
5.1	General	12
5.2	Preservation methods	13
5.2.1	General	13
5.2.2	Airtight storage	14
5.2.3	Dark storage	14
5.2.4	Cooled storage	14
5.2.5	Freezing	14
5.2.6	Chemical preservation	15
6	Transporting the sample	15
7	Sample record	16
Annex A	(informative) Packaging	17
Annex B	(informative) The modular horizontal system	20
Annex C	(informative) Information on WP 2 Sampling and the project HORIZONTAL	21

Foreword

This document TC 151 WI 151 has been prepared by Technical Committee CEN/TC 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).

For relationship with EU Directive(s), see informative Annex ZA, B, C or D, which is an integral part of this document.

The following TC's have been involved in the preparation of the standard:

This standard is applicable and validated for several types of matrices. The table below indicates which ones.

[table to be filled and amended by the standards writer]

Material	Validated	Document
Waste	<input type="checkbox"/>	[reference]
Sludge	<input type="checkbox"/>	
Soil	<input type="checkbox"/>	
Soil improvers	Not validated yet	

Introduction

Provided certain quality requirements are met, sewage sludge and treated biowaste may be applied to land for the purpose of beneficial land use. The testing of sewage sludge, treated biowastes and soil allows informed decisions to be made on whether land application is appropriate (or not). In order to undertake valid tests a (number of) representative sample(s) of the sewage sludge, treated biowaste or land will be required.

The principal component of the Standard prEN xxxx is the mandatory requirement to prepare a Sampling Plan, within the framework of an overall testing programme as illustrated in Figure 1 of prEN xxxx. This Standard can be used to:

- produce standardised sampling plans for use in regular or routine circumstances (i.e. the elaboration of daughter/derived standards dedicated to well defined sampling scenarios);
- incorporate specific sampling requirements into national legislation;
- design and develop a Sampling Plan on a case by case basis.

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 process map and elaborates on methods and boundary conditions for preserving, packaging and storing samples to preserve their integrity, in addition to the transportation and delivery of a sample to the designated analytical facility.

Sample integrity may be compromised if insufficient attention is paid to correct packaging, preservation, storage and transport techniques. This may result in a sample which is not representative of the sample population. The selection of the most appropriate procedure must be in collaboration with the laboratory facility designated to undertake testing to ensure compatibility with the chosen analytical methodology and parameters to be tested. Specifically TR xxx-4 supports Clause 4.2.8.3 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 Introductory element - Sampling of sewage sludge, treated biowastes and soils in the landscape – Framework for the preparation and application of a Sampling Plan

TR xxx-1: Introductory element - Sampling of sewage sludge and treated biowastes: Guidance on selection and application of criteria for sampling under various conditions.

TR xxx-2: Introductory element - Sampling of sewage sludge and treated biowastes: Guidance on sampling techniques

TR xxx-3: Introductory element - Sampling of sewage sludge and treated biowastes: Guidance on sub-sampling in the field

TR xxx-4: Introductory element - Sampling of sewage sludge and treated biowastes: Guidance on procedures for sample packaging, storage, preservation, transport and delivery

TR xxx-5: Introductory element - Sampling of sewage sludge and treated biowastes: Guidance on the process of defining the sampling plan

TR xxx-6: Introductory element - Sampling of soils in the landscape: Guidance on the process of defining the sampling plan

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

1 Scope

This Technical Report describes procedures for the packaging, preservation, short-term storage and transport of samples of sewage sludge and treated biowastes. 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 collaboration with the testing laboratory.

NOTE The procedures listed in this Technical Report reflect current best practice, but these are not exhaustive and other procedures may be equally relevant.

2 Normative references

This European Standard incorporates by dated or undated reference, provisions from other publications. These normative references are cited at the appropriate places in the text and the publications are listed hereafter. For dated references, subsequent amendments to or revisions of any of these publications apply to this European Standard only when incorporated in it by amendment or revision. For undated references the latest edition of the publication referred to applies (including amendments).

ISO 5667-3:2003, Water quality – Sampling Part 3: Guidance on the preservation and handling water samples

ISO 5667-16:1998, Water quality – Sampling – Part 16: Guidance on biotesting of samples

ISO 11048:1995 Soil quality — Determination of water-soluble and acid-soluble sulfate

ISO 11074-2:1999, Soil quality – vocabulary – Part 2: Terms and definitions related to sampling

prCEN/TR 15252:2005, Characterization of sludges - Protocol for validating methods for physical properties of sludges

3 Terms and definitions

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

3.1

analytical laboratory

The identified laboratory which is to undertake the chemical, biological or physical analysis of samples.

3.2

constituent

A property or attribute of a material that is measured, compared or noted.

3.3

delivery

Transfer of custody of the sample.

3.4

field sample

The quantity (mass or volume) of material obtained through sampling without any sub-sampling.

3.5

laboratory sample

The sample sent to or received by the laboratory.

[IUPAC, definition 2.5.5]

3.6

packaging

Act of placing a sample into an appropriate sample container for transport and/or storage.

[after ISO 11074-2:1998, definition 4.27]

3.7

sample

Portion of material selected from a larger quantity of material.

[ISO 11074-2:1998, definition 1.3]

NOTE The use of the term 'sample' should be qualified with a prefix as far as possible as it does not indicate to which step of the total sampling procedure it is related when used alone e.g. field sample, laboratory sample.

3.8

sample preservation

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

[ISO 11074-2:1998, definition 4.29]

3.9

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 the sample.

[after ISO 11074-2:1998, definition 4.31]

3.10

sample transportation

Act of transferring a sample from the locality of sampling to the place of subsequent treatment (e.g. laboratory, soil-specimen bank etc.).

[ISO 11074-2:1998, definition 4.30]

3.11

seal (tamper-indicating device)

A device or material designed to provide evidence of unauthorized access.

NOTE 1 A seal is fixed to the container such that the seal must be broken to gain access to the contents.

NOTE 2 A seal does not need to provide resistance to entry; it need only record that it took place.

4 Packing and labelling the sample

4.1 General

Sample containers can be made from many different types of materials, some of which may react or contaminate a specific type of sample. To avoid any accidental contamination by the sample container or derogation of the sample the Project Manager should seek scientific advice, 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 has to arrive at the laboratory. In general, this period has to be as short as possible. Once the details have been agreed, the Sampler should follow the methods detailed in the sampling plan.

4.2 Selecting a sample container

4.2.1 General

The Sampling Plan should state the sample container requirements, including container type, size and shape, cleaning, closure, seal and any special filling requirements, e.g. headspace.

Advice on selecting appropriate sample containers should be sought from the analytical laboratory.

The purpose of the sample container is to protect the sample during transport and storage until it is further treated or analysed. A container should be compatible with the nature of the material sampled and the components to be analysed. In general:

TC 151 WI 151:2004 (E) Sampling

- Collect samples for inorganic analysis in plastic containers;
- Collect samples for organic analysis in glass containers;
- Select a sample container having a size relative to the volume of the required sample;
- Select a sample container capable of being sealed.

NOTE Tamperproof seals are commonly used when performing regulatory sampling.

Where specialised containers and preservatives are advised the analytical laboratories should be encouraged to provide 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.

4.2.2 Type of container

The following points should be considered when selecting and preparing sample containers:

- adsorption into 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;
- cost.

4.2.3 Size and shape of container

The sample size is a function of the amount required to complete the analytical suite and by any requirement to store samples for investigation at a later date. The sample container should be of sufficient size to accommodate the sample, taking into account any headspace required.

The container shape may be important when considering storage space.

4.2.4 Preparation of sample containers

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

NOTE 1 The choice of cleaning method will depend on the components to be analysed. Containers may be cleaned with mixtures of acids followed by rinsing with de-ionised water. Samples that are to be analysed for organic components may be stored in solvent-rinsed containers and/or heated in a calcination oven.

NOTE 2 Samples that are to be analysed for microbiological components may require that containers be sterilised after cleaning. Sterilised containers should be used if sterilized or disinfected sewage samples are to be collected.

NOTE 3 Cleaning procedures may differ depending on the type of material to be packed and the components to be analysed. Advice should be sought from the analytical laboratory or other experts in order to establish the most suitable procedure in each case. In general, the reuse of sample containers is not advised.

4.3 Packaging

4.3.1 General

Select a container compatible with the analytical determinants using the guidance given in Table A.1.

NOTE 1 When several analyses are to be carried out, more than one laboratory sample may be required if there is a conflict in the packaging requirements for the different analyses.

NOTE 2 The test method may specify constraints on laboratory sample packaging.

4.3.2 Filling containers

Wide neck rigid containers aid sample handling, e.g. for dewatered sludges or coarse-grained treated biowaste.

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 related to partial filling can be:

- enhanced agitation during transport, leading to breakdown of aggregated particles;
- interaction with the gas phase, leading to stripping;
- oxidation of substances, leading e.g. to precipitation of compounds of heavy metals.

Problems related to complete filling can be:

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

NOTE 1 Air space should be minimised, 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 is needed within the container to allow for expansion.

NOTE 2 To avoid loss of volatile species, samples should be collected in a completely filled container, overfilling it before capping or sealing.

NOTE 3 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 tolerate a slight increase in inner pressure.

NOTE 4 For microbiological examination, the sample container should not be filled to the brim so that airspace is left. This aids mixing before examination and avoidance of accidental contamination.

TC 151 WI 151:2004 (E) Sampling

4.3.3 Sealing containers

Laboratory samples for dispatch or transport by third parties and reserved laboratory samples should be sealed in such manner that the integrity of the sample is protected. Sealing of the samples may be required when samples are taken for (potential) regulatory investigations. The label shall be incorporated in to the seal.

NOTE 1 The container should be secured and sealed in such a manner that it can be opened only by breaking the seal.

NOTE 2 Alternatively, the container shall be placed in a sealed, robust package in such a manner that no part of the sample can be removed without breaking the seal of the package.

4.3.4 Labelling

Apply a clearly legible, unique, unambiguous code to each sample container either:

- a) by writing directly on to the container using a permanent marker pen; or
- b) by 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, attach an identical label to the container body.

Select a label of a quality capable of remaining firmly attached to the sample container whatever the storage period and prevailing conditions. Where the possibility exists of the label becoming 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 1 Indelible ink should be used and labels should be kept short and simple to avoid mistakes when transcribing numbers.

NOTE 2 If possible, the use of pre-printed labels and bar-coded labels are an advantage.

5 Sample preservation

5.1 General

The time elapsed between sample collection and analysis depends on the type of analysis to be carried out. In all cases, this time should be as short as possible.

It is seldom possible to analyse samples immediately after collection. For this reason preservation techniques are required in the field, during transport to the laboratory and during storage before analysis. The components 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 attributes to be analysed.

The method of preservation relates to the stability of samples. Preservation methods are considered under the following broad headings:

- materials that are stable;
- materials that are unstable but where stability can be achieved by a preservation method;
- materials that are unstable and cannot be readily stabilised.

For those components which are unstable, it is important to minimise loss or change (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 components 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 CO₂ 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.

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 components 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 components such that the sample is no longer representative of the bulk material that was sampled.

Common methods of preservation include:

- airtight storage;
- dark storage;
- cooled storage ($< 4 \pm 2$ °C);
- freezing;
- drying;
- chemical preservation.

5.2 Preservation methods

5.2.1 General

Consultation should be undertaken with the analytical laboratory, or other experts, to obtain advice on the type of preservation methods required for the identified parameters.

It is common practice that action is taken to:

- avoid the introduction of unacceptable contamination through the preservation method(s);
- incorporate the selected method(s) of preservation into the sampling plan;

TC 151 WI 151:2004 (E) Sampling

- arrange for the samples to be analysed within a timescale agreed with the analytical laboratory, and specify this in the sampling plan.

No recommendation can be given for a universal preservation method. A method that is suitable for one group of analyses may interfere with other analyses. 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 are represented.

5.2.2 Airtight storage

When airtight storage is required, store liquid, paste-like and fine-grained materials in glass bottles sealed with PTFE cap liners.

NOTE 1 Plastics are not considered airtight.

NOTE 2 Airtight storage will prevent volatilisation of components 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 (for example using nitrogen) can be used to limit chemical reactions (oxidation, carbonation). Inert gas flushing may 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.

5.2.3 Dark storage

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

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

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

NOTE Virus survival is sensitive to light in the UVB and UVC range. When samples are collected for viral analysis, care should be taken to ensure that the container material excludes light in this wavelength. This is particularly important in low latitude regions.

5.2.4 Cooled storage

When ambient temperatures are above 10°C, samples should be cooled immediately after collection to minimise 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 Wherever a temperature is given for cooling, the temperature of the sample environment is meant (not the temperature of the sample itself).

NOTE 3 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 4 Direct contact with frozen cool packs can cause localised freezing of samples with high water content. Preventative action should be taken to ensure that the samples and cool packs are not in direct contact.

5.2.5 Freezing

Use liquid nitrogen or dry ice for immediate deep freezing of samples.

NOTE 1 This method of freezing is difficult to conduct in the field. It is common practice to ensure that samples are frozen as soon as possible upon return from fieldwork.

NOTE 2 The use of glass containers is not advised when freezing samples with high water content.

NOTE 3 Fast freezing of sludges may be appropriate for samples collected for physical examination, e.g. structure, texture or layer formation, and sample integrity is important and agitation- and vibration-free conditions should be maintained during transport.

NOTE 4 In most cases, freezing should be avoided as it strongly modifies sludge structure, thus affecting certain measurements.

5.2.6 Chemical preservation

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

Consultation should be undertaken with the analytical laboratory, or other experts, to obtain advice on the type of preservation methods required for the identified parameters. For liquid samples it is now common practice for laboratories to provide sample containers with the appropriate chemical preservative added.

NOTE 1 Care should be taken to avoid unquantifiable reactions that can occur between the sample and additives during sample preparation (e.g. drying or milling). It is common practice to obtain this information from the analytical laboratory.

NOTE 2 When samples are required for the determination of extractable levels (e.g. using leaching tests), the addition of chemical stabilising agents can affect the leachability of other components 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 any contamination. Therefore the use of solvents in the field for sample preservation will be limited.

6 Transporting the sample

Samples should be shipped to the laboratory without delay.

The containers holding samples must 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 all glass containers used for fermentable samples (nearly all biologically derived sludges) be wrapped with waterproof adhesive tape or other equivalent measures taken, such as wrapping in plastic mesh. These methods will minimise the dispersion of fragments of the container if an explosion occurs due to gas generation.

NOTE 1 Packaging should protect the containers from possible external contamination and breakage, particularly near the opening, and should not itself be a source of contamination.

NOTE 2 Each sample should be placed inside an individual waterproof container if possible.

NOTE 3 Certain statutory authorities or other organisation(s) may have additional requirements for packaging and transporting samples.

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

If the time of travel exceeds the maximum recommended preservation time before analysis, whether or not the samples should be analysed should be checked with the client or after consultation with the scientist interpreting the analytical results. If it is decided to proceed with the analysis the time between sampling and analysis should be reported.

TC 151 WI 151:2004 (E) Sampling

NOTE For regulatory samples, a record of temperature during transportation may be required.

7 Sample record

Samples should be accompanied by a chain of custody form (see prEN xxxx).

NOTE 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.

The chain of custody form should be checked and signed at each transfer of the samples.

The storage, preservation and transport procedure(s) and necessary equipment, should be specified by the Project Manager in the Sampling Plan prior to commencing sampling. Any deviation from these procedures should be recorded in the Sampling Report, see prEN xxxx.

Annex A (informative)

Packaging

Table A.1 – Sample containers, preservation and storage conditions for different parameters measured in sediments and sludges and liquids.

Analysis or test	Container	Preservation	Storage Conditions	Storage duration	International Standard
Acidity	Polyethylene/ Glass	Refrigerate	(2 to 5)°C & dark & airtight	14 days	
Alkalinity	Polyethylene/ Glass	Refrigerate	(2 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 to 5)°C & dark & airtight	24 hours	
Conductivity	Polyethylene /Glass	Refrigerate	(2 to 5)°C & dark & airtight	24 hours	
Dry Weight	Glass	Refrigerate	(2 to 5)°C & dark & airtight	1 month	
Anions (e.g. sulphate)	Polyethylene /Glass	Refrigerate	(2 to 5)°C & dark & airtight	28 days	ISO 11048
Nitrate	Polyethylene /Glass	Refrigerate	(2 to 5)°C & dark & airtight	2 days	
Nitrite	Polyethylene /Glass	Refrigerate	(2 to 5)°C & dark & airtight	As short as possible	
Sulfide	Polyethylene /Glass	Refrigerate	(2 to 5)°C & dark & airtight	As short as Possible	
Phosphorus	Glass	Refrigerate	(2 to 5)°C & dark & airtight	1 month	
Orthophosphate	Glass	Refrigerate	(2 to 5)°C & dark & airtight	2 days	
Cyanides	Polyethylene	Freeze	≤ -20°C & dark & airtight	1 month	
Metals	Polyethylene /Glass	Refrigerate	(2-5)°C & dark & airtight	8 days	
		Freeze	≤ -20°C & dark & airtight	6 months	
		Dry (60°C)	ambient temperature dark & airtight	6 months	
Chromium (V1)	Polyethylene /Glass	Refrigerate	(2 to 5)°C & dark & airtight	2 days	
Mercury	Glass/PTFE	Refrigerate	(2 to 5)°C & dark & airtight	8 days	
		Freeze	≤ -20°C & dark & airtight	1 month	

TC 151 WI 151:2004 (E) Sampling

Analysis or test	Container	Preservation	Storage Conditions	Storage duration	International Standard
Particle size	Polyethylene /Glass/Metal	Refrigerate	(2 to 5)°C & dark & airtight		
TOC	Glass with PTFE-lined cap	Refrigerate	(2 to 5)°C & dark & airtight	1 month	
		Freeze	≤ -20°C & dark & airtight	6 months	
Semi- and non-Volatile organic Compounds	Glass with PTFE-lined cap	Refrigerate	(2 to 5)°C & dark & airtight	1 month	
		Freeze	≤ -20°C & dark & airtight	6 months	
(PCBs, PAHs, pesticides, high molecular weight hydrocarbons)	Glass with Aluminium foil cap	Refrigerate	(2 to 5)°C & dark & airtight	1 month	
		Freeze	≤ -20°C & dark & airtight	6 months	
Mineral oil	Glass with PTFE-lined cap	Refrigerate	(2 to 5)°C & dark & airtight	1 month	
Volatile organics as-received	Glass/metal rings with PTFE-lined cap	Refrigerate/ Addition of methanol	(2 to 5)°C & dark & airtight	As short as possible	
		Freeze	≤ -20°C & dark & airtight	1 month	
Ecotoxicological tests	Polyethylene /Glass	Refrigerate	(2 to 5)°C & dark & airtight	14 days ^d	ISO 5667-16
Bacteriological Examination	Sterile Glass	Refrigerate	(2 to 5)°C & dark & airtight	6 hours	
Microbial activity	Sterile Glass	None	None	None	
Ecological Examination	Polyethylene /Glass	70% (v/v) ethanol	(2 to 5)°C & dark & airtight	1 year	ISO 5667-3
		4% (v/v) formalin		1 year	
Bacteria	Polyethylene /Glass	Refrigerate, Do not freeze	(2 to 5)°C & dark & airtight	Complete analysis within 24-48 hours	
Viruses	Polyethylene /Glass	Refrigerate	(2 to 5)°C & dark & airtight	Complete analysis within 24-48 hours	
		Freeze	≤ -20°C & dark & airtight	2 weeks	
Helminth ova	Polyethylene /Glass	Refrigerate, Do not freeze	(2 to 5)°C & dark & airtight	1 month	
Oocysts	Polyethylene /Glass	Refrigerate, Do not freeze	(2 to 5)°C & dark & airtight	1 month	
Settleability/ Thickenability	Polyethylene/ Polypropylene/ Metal bottles	Refrigerate	(2 to 5)°C & airtight	24 hours	prCEN/TR 15252:2005
CST (Capillary)	Polyethylene/	Refrigerate	(2 to 5)°C &	24 hours	prCEN/TR

Analysis or test	Container	Preservation	Storage Conditions	Storage duration	International Standard
suction time)	Polypropylene/ Metal bottles		airtight		15252:2005
Specific resistance to filtration	Polyethylene/ Polypropylene/ Metal bottles	Refrigerate	(2 to 5)°C & airtight	24 hours	prCEN/TR 15252:2005

^a Analysis should be started as soon as possible.

Annex B
(informative)

The modular horizontal system

Annex C
(informative)

Information on WP 2 Sampling and the project HORIZONTAL

Bibliography

- [1] EN 5667-3:1996, Water quality – Sampling – Part 3: Guidance on the preservation and handling of samples.
- [2] EN 5667-13:1998, Water quality – Sampling – Part 13: Guidance on sampling of sludges from sewage and water treatment works.
- [3] EN 5667-15:1999, Water quality – Sampling – Part 15: Guidance on preservation and handling of sludge and sediment samples.
- [4] EN 5667-16:1998, Water quality – Sampling – Part 16: Guidance on biotesting of samples.
- [5] EN 1482:1996, Sampling of solid fertilizers and liming materials.
- [6] EN 12579:2000, Soil improvers and growing media – Sampling.
- [7] EN 932-1:1996, Tests for general properties of aggregate – Part 1: Methods for Sampling.
- [8] EN 932-2:1999, Tests for general properties of aggregates – Part 2: Methods for reducing laboratory samples.
- [9] ISO 10381-6:1993, Soil quality – Sampling – Part 6: Guidance on the collection, handling and storage of soil for the assessment of aerobic microbial processes in soil.
- [10] prCEN/TR 15252:2005, Characterization of sludges – Protocol for validating methods for physical properties of sludges [CEN/TC308]
- [11] IUPAC, 1990, Nomenclature for sampling in analytical chemistry. Pure Appl Chem, **62**: 1193-1208.
- [12] Lambkin, D C, S. Nortcliff and T C White, 2005, HORIZONTAL Desk Study: Sample handling protocols for sludges and treated biowaste for microbiological analysis.