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 B. Sampling of sewage sludge and treated biowastes – Technical Report on Sampling – Guidance on selection and application of criteria for sampling under various conditions.

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	
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)	

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 B 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 sub-sampling in the field

Einführendes Element — Haupt-Element — Ergänzendes Element

Élément introductif — Élément central — Élément complémentaire

ICS:

Descriptors:

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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 TCs 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	☐	[reference]
Sludge	☐	
Soil	☐	
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 Step 2 of the Sampling Plan process map and elaborates on the range of potential approaches that can be used to reduce the size of a sample in the field to facilitate the appropriate storage and preservation of the sample and ultimately its transportation to the designated analytical facility.

TR xxxx-3 describes procedures for reducing the overall size of the sample in the field, to aid practical transportation of a sample to the laboratory. It does not deal with sub-sampling in the laboratory to provide a test portion, or the pre-treatment of samples prior to analysis. Samples dispatched to the laboratory may require additional sub-sampling and/or pre-treatment steps prior to analysis. Some samples may be analysed without additional treatment. Field sub-sampling should be carried out in such a way as to obtain, at all stages, a sample that is representative of the field sample. Specifically TR xxxx-3 supports Clause 4.2.8.2 (Procedures for sub-sampling in the field) 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 xxxx-1: Introductory element - Sampling of sewage sludge and treated biowastes: Guidance on selection and application of criteria for sampling under various conditions.

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

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

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

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

TR xxxx-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 suitable for use in the field for reducing the size of field samples, with or without combining increments, to aid practical transportation to the 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).

EN 932-1:1996, Test for general properties of aggregates – Part 1: Methods for sampling

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

3 Terms and definitions

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

3.1
composite sample
Two or more increments/sub-samples mixed together in appropriate proportions, either discretely or continuously (blended composite sample), from which the average value of a desired characteristic may be obtained.

[ISO 11074-2:1998, definition 3.10]

3.2
increment
Individual portion of material collected by a single operation of a sampling device which will not be analysed / investigated as a single entity, but will be mixed with other increments in a composite sample.

NOTE 1 Whenever the portion of material collected by a single operation of a sampling device is analysed individually, the obtained material is called a sample. In such a situation the quantity of material has to fulfil both the criteria for the size of an increment as well as for a sample.

NOTE 2 In some languages the term 'increment' is used without the condition that an increment will never be analysed on its own. For this standard this is however an essential condition in the definition of the term ‘increment’.

3.3
field sample
The quantity (mass or volume) of material obtained through sampling without any sub-sampling.

3.4
laboratory sample
The sample sent to or received by the laboratory.

[IUPAC, definition 2.5.5]

3.5
mixing
Combining of components, particles or layers into a more homogeneous state.

[ISO 11074-2:1998, definition 6.2]

3.6
portion
Each of the discrete, identifiable portions of a material suitable for removal from a population as a sample or as a portion of a sample, and which can be individually considered, examined, tested or combined.

[ISO 11074-2:1998, definition 1.2]

3.7
representative sample
Sample in which the characteristic(s) of interest is (are) present with an uncertainty appropriate for the purposes of the testing programme.

3.8
riffling
Separation of a free-flowing sample into (usually) equal parts by means of a mechanical device composed of diverter chutes.

[ISO 11074-2:1998, definition 6.4]

3.9
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.

3.10
sampling plan
Predetermined procedure for the selection, withdrawal, preservation, transportation and preparation of the portions to be removed from a population as a sample.

[ISO 11074-2:1998, definition 4.3]

NOTE All the information pertinent to a particular sampling activity.

3.11
stratum/ strata
Strata are mutually exclusive and exhaustive parts of a population. They are identified either because they are believed to be different from each other or for the purposes of sampling.

3.12
sub-sample
The quantity (mass or volume) of material obtained by procedures in which the characteristics of interest are randomly distributed in parts of equal or unequal size.

NOTE 1 A sub-sample may be:

a) a portion of the sample obtained by selection or division; or

b) an individual unit of the stratum taken as part of the sample; or

c) the final unit of multi-stage sampling.

NOTE 2 The term ‘sub-sample’ is used either in the sense of a ‘sample of a sample’ or as a synonym for ‘unit’. In practice, the meaning is usually apparent from the context or is defined.

3.13
sub-sampling (sample division)
Process of selecting one or more sub-samples from a sample of population.

[ISO 11074-2:1998, definition 6.3]

4 Principles of sub-sampling in the field

If several samples have been taken (e.g. from different locations or at different times) they may be combined to produce a composite sample that represents better the population that was sampled. Often combining increment samples produces a composite sample that is much larger than required for a laboratory sample. In this case the combined sample should be thoroughly mixed and sub-sampled to produce one or more laboratory samples.

Samples may require sub-sampling when more than one laboratory sample is to be prepared from a single field sample.

EXAMPLE When several analyses are to be carried out on a sample of sewage sludge or treated biowaste and the analyses require conflicting preservation methods, then the field sample will require sub-sampling to produce more than one laboratory sample.

Sub-sampling should be carried out in the field only if the integrity of the sample and sub-samples can be assured. The sub-sampling environment should protect the sample from loss of moisture and volatile components due to evaporation, or cross-contamination of samples. Sub-sampling activities should ideally be carried out in an appropriately equipped mobile or field laboratory to safeguard sample integrity.

NOTE 1 Where samples are to be analysed for microbiological parameters, the risk of cross-contamination is high and sub-sampling in the field is not recommended.

NOTE 2 Mixing and sub-sampling in the open-air is not appropriate when volatile or semi-volatile components are of interest. It is advised that such samples are returned to the laboratory for sub-sampling.

A method of sub-sampling should be selected that minimises change in the physical, chemical and biological composition of the sample. The ultimate selection of a procedure will depend on the stated objectives of the sampling programme and the tests to be carried out on the sample.

To obtain sub-samples of equal quality, it should be ensured that the bulk sample maintains homogeneity during the sub-sampling process, e.g. by continuous shaking or stirring.

NOTE This holds particularly in the case of two-phase mixtures, e.g. liquid sludges.

If a heterogeneous sample cannot be adequately mixed in the field or field laboratory to produce a homogeneous sub-sample it should be returned to the laboratory for sub-sampling.

When two or more laboratory samples are required from a field or composite sample, all excess material rejected during the first sub-sampling operation should be re-combined, mixed thoroughly and reduced again to provide the second laboratory sample. This should be repeated as necessary to provide the required number of laboratory samples.

It is recommended that when several samples taken at different times are to be combined, the increment samples are kept cool to preserve their integrity.

The instructions for sub-sampling in the field should be stated on the Sampling Plan. Any deviation should be recorded on the Sampling Report (see prEN xxxx).

5 Apparatus

All apparatus and tools should be clean to reduce the risk of cross-contamination.

Suitable apparatus may include:

- Large heavy-duty plastic sheeting
- Scoop
- Spade
- Mechanical shovel

— Sheet metal cross

— Riffle splitter

NOTE In all cases, alternative designs may be used as long as the devices can be used to fulfil the sub-sampling procedures described in clauses 6 to 10.

6 Granular materials

6.1 General considerations

Materials that can be considered granular include:

— Treated biowaste;

— Thermally dried sewage sludge granules;

— Thermally dried sewage sludge pellets;

— Granular lime-treated sewage sludge.

NOTE Mixing and sub-sampling of very dry materials, e.g. thermally dried sewage sludge, can result in particle abrasion leading to changes in the physical structure and the risk of loss of fine particles.

6.2 Mixing granular materials

6.2.1 General

A mixed sample is produced by combining equivalent quantities (m/m or v/v) of the individual increments.

When mixing is carried out in the field, it is not possible to determine the dry matter content of the material. In this case it must be assumed that the dry matter content for all increments is (approximately) the same. In most instances, where increments are taken from the same stratum, this should be the case.

If a significant deviation in dry matter content is expected, the mixing of increments should be carried out in the laboratory after determining the dry matter content of each of the individual increments.

NOTE Mixing increments of the same volume / mass from two or more strata can result in over sampling of one of the strata when the dry matter content of that stratum is significantly higher than the others.

6.2.2 Methodology

Identify a location within a covered area, or an area of hard standing that is sheltered from the effects of wind and rain, that is flat and large enough to allow ease of access around the whole sample when spread on the surface.

Place a clean protective covering, preferable heavy-duty plastic sheeting, on the floor of the field laboratory or on the ground to protect the sample from contamination by the floor surface.

Mix the material by forming a conical heap on the clean surface. Take a spade or scoopful of the material and put it on the top of the preceding one. The size of the spade or scoop should be of such size that this action should be repeated on at least 20 occasions in order to transfer the full amount of material.

6.3 Sub-sampling granular materials

A sample can be divided into sub-samples by manual or mechanical means. When the material is dry mechanical techniques usually are preferred as they produce sub-samples that are more representative of the whole sample.

Methods of sub-sampling that are suitable for granular materials include:

— Long pile-alternate shovel method (10.1);

— Coning and quartering (10.2);

— Riffling (10.3).

NOTE A mechanical divider should be avoided when the particles in the sample are wet or behave cohesively as the divider may not function properly and can block.

7 Liquid sludges

7.1 Mixing liquid sludges

7.1.1 General

A mixed sample is produced by combining equivalent quantities (v/v) of the individual increments.

Mixing of stratified samples, where the stratification must be maintained, should be undertaken in the laboratory.

7.1.2 Methodology

Identify a location within a covered area, or an area of hard standing that is sheltered from the effects of wind and rain, that is flat and large enough to allow ease of access around the sample container(s).

The receptacle into which all samples are placed for mixing should be large enough to contain all the increments and space for mixing and ensure that there is no loss of sample during mixing.

Determine the quantities by volume of the increments to be mixed.

Place the increments in the mixing container.

Mix the contents of the container by stirring or, if the container can be sealed, by rolling or shaking.

7.2 Sub-sampling liquid sludges

Mix the combined sample thoroughly.

Transfer the required quantity to a smaller container by pouring, taking care to minimise the loss of volatile compounds.

If multiple sub-samples are required, the mixing procedure should be carried out between sub-samples.

NOTE This ensures that each sub-sample is representative.

Transfer the sub-sample to an appropriate sample container either:

a) in accordance to TR xxxx-4, or

b) as specified in the Sampling Plan.

8 Paste-like sludges

8.1 General considerations

Materials that can be considered paste-like include:

— Dewatered sewage sludge cake;

— Extruded lime-treated sewage sludge.

8.2 Mixing paste-like sludges

8.2.1 General

A mixed sample is produced by combining equivalent quantities (m/m or v/v) of the individual increments.

When mixing is carried out in the field, it is not possible to determine the dry matter content of the material. In this case it must be assumed that the dry matter content for all increments is (approximately) the same. In most instances, where increments are taken from the same stratum, this should be the case.

NOTE The method for mixing depends on the water content of the material. Dewatered sludges with high water content may be better considered as gelatinous sludges.

8.2.2 Methodology

Identify a location within a covered area, or an area of hard standing that is sheltered from the effects of wind and rain, that is flat and large enough to allow ease of access around the whole sample when spread on the surface.

Place a clean protective floor covering, preferable heavy-duty plastic sheeting, on the floor of the field laboratory or on the ground to protect the sample from contamination by the floor surface.

NOTE Extruded sludge should first be cut up into a number of small parts of roughly equal size.

Transfer the sample onto the clean surface to form a conical heap by taking a spade or scoopful of the material and putting it on the top of the preceding one. The size of the spade or scoop should be of such size that this action should be repeated at least 20 times in order to transfer the full amount of material.

The sample is mixed by turning the heap over, for example with a shovel, to form a new cone, the operation being carried out three times. Each conical heap should be formed by depositing each shovelful of material on the apex of the cone, so that the portions which slide down the sides are distributed as evenly as possible, and the centre of the cone is not displaced.

8.2.3 Sub-sampling paste-like sludges

Sub-sampling methods that are suitable for paste-like materials are:

— Long pile-alternate shovel method

NOTE This method is suitable for the reduction of samples in excess of approximately 100 kg.

— Coning and quartering method

NOTE This method is suitable for the reduction of samples down to approximately 1 kg.

9 Gelatinous sludges

9.1 Mixing gelatinous sludges

9.1.1 General

A mixed sample is produced by combining equivalent quantities (m/m or v/v) of the individual increments.

When mixing is carried out in the field, it is not possible to determine the dry matter content of the material. In this case it must be assumed that the dry matter content for all increments is (approximately) the same. In most instances, where increments are taken from the same stratum, this should be the case.

Sludges that have a gelatinous appearance behave more like a jelly than a mineral solid like gravel, and are unlikely to be homogenised by the technique used for sludge cake.

9.1.2 Methodology

Identify a location within a covered area, or an area of hard standing that is sheltered from the effects of wind and rain, that is flat and large enough to allow ease of access around the whole sample when spread on the surface.

Place a clean protective floor covering, preferable heavy-duty plastic sheeting, on the floor of the field laboratory or on the ground to protect the sample from contamination by the floor surface.

Transfer the sample onto the clean surface to form a conical heap by taking a spade or scoopful of the material and putting it on the top of the preceding one. The size of the spade or scoop should be of such size that this action should be repeated at least 20 times in order to transfer the full amount of material.

The sample is mixed by employing a technique such as that employed for the hand or mechanical preparation of cement mortar.

9.1.3 Sub-sampling gelatinous sludges

Sub-sampling methods that are suitable for gelatinous sludges are:

— Coning and quartering method

NOTE This method is suitable for the reduction of samples down to approximately 1 kg.

10 Sub-sampling methods

10.1 Long pile-alternate shovel method

10.1.1 Principle

The sample is laid out in a long pile, the pile is separated into two equal piles by using a shovel and placing alternate shovel loads to either side to form two mounds. Then one mound is randomly selected and the process continued to reduce the sample size.

NOTE This sub-sampling method is suitable for the reduction of samples in excess of approximately 100 kg.

10.1.2 Method

Shovel the sample into a conical pile on the clean surface, placing each shovelful on the top of the preceding one.

NOTE For samples in excess of 500 kilograms, a mechanical shovel should be considered.

When the entire sample is on the floor moving mix it thoroughly by turning it over to form new cone adjacent to the first. Repeat this operation at least three times. When forming the new cones, deposit each shovelful on the peak of the new cone in such a way that the sample runs down all sides of the cone and is evenly distributed so that different particle sizes become well mixed.

Form the final cone into a long pile as follows:

— Take a shovelful from the base of the cone and spread the material into a ribbon having an initial width equal to that of a shovel and a length of 1.5 to 3.0 metres.

— Take the next shovelful from a different point at the base of the cone and spread directly over the previous shovelful, but in the opposite direction.

— Repeat the above step until one long pile is formed

Discard half the sample in the following manner:

— Take a shovelful from the bottom of one end of the pile and set aside.

— Take the next shovelful immediately adjacent to the first by advancing along the side of a pile a distance equal to the width of the shovel and discard.

— Again, advancing in the same direction a distance of one shovel width, take the third shovelful and add to the first.

— Continue along the pile following the above procedure, discarding alternate shovelfuls so that the pile is decreased gradually and uniformly.

Repeat the above procedure (from forming the coning to halving the pile) until the retained amount of material is equal to the desired size of the sub-sample.

Transfer the sub-sample to an appropriate sample container either:

a) in accordance with TR xxxx-4, or

b) as specified in the Sampling Plan.

10.2 Coning and Quartering

10.2.1 Principle

Coning and quartering is the reduction in size of a granular sample by forming a conical heap which is spread out into a circular, flat cake. The cake is divided radially into quarters and two opposite quarters are combined. The other two quarters are discarded. The process is repeated as many times as necessary to obtain the quantity required for the laboratory sample.

NOTE This procedure is suitable for producing sub-samples to approximately 1 kg.

10.2.2 Method

Mix the increment samples to produce a composite sample (see 6.2).

Shovel the sample into a conical pile on the clean surface, placing each shovelful on the top of the preceding one.

NOTE For samples in excess of 500 kg, it is common to use a mechanical shovel.

When the entire sample is on the floor, mix it thoroughly by turning it over to form a new cone adjacent to the first. Repeat this operation at least three times. When forming the new cones, deposit each shovelful on the peak of the new cone in such a way that the sample runs down all sides of the cone and is evenly distributed so that different particle sizes become well mixed.

Flatten the final cone by inserting the shovel repeatedly and vertically onto the peak of the cone to form a flat heap which has a uniform thickness and diameter. The height should be less than or equal to the blade depth of the shovel or spade used.

Quarter the flat heap along two diagonals intersecting at right angles using one of the following methods:

a) using a sheet metal cross

- Place the centre of a sheet metal cross at the centre of the flattened cone and press the lower edges of the metal cross through the sample. The depth of the blades forming the cross should be greater than that of the flattened cone.

- With the metal cross left in position discard opposite diagonal quarters and brush clean the space they occupied.

- Remove the metal cross and mix together the remaining two quarters.

b) using a shovel or spade

- Quarter the flat heap along two diagonals intersecting at right angles using a shovel or spade inserted vertically into the flattened cone.

- Discard one pair of opposite quarters and mix together the remaining two quarters.

Repeat the process of mixing, coning and quartering until the remaining sub-sample is equal to the desired laboratory sample size.

Transfer the sub-sample to an appropriate sample container either:

a) in accordance with TR xxxx-4, or

b) as specified in the Sampling Plan.

10.3 Riffling

10.3.1 Principle

A riffle box (Figure 1) consists of a hopper under which an even number of equal-width chutes is located. Sample material placed in the hopper exits via the base of the chutes, which are alternately directed to the left or the right side of the splitter, into one of two collectors. This process splits the sample in half.

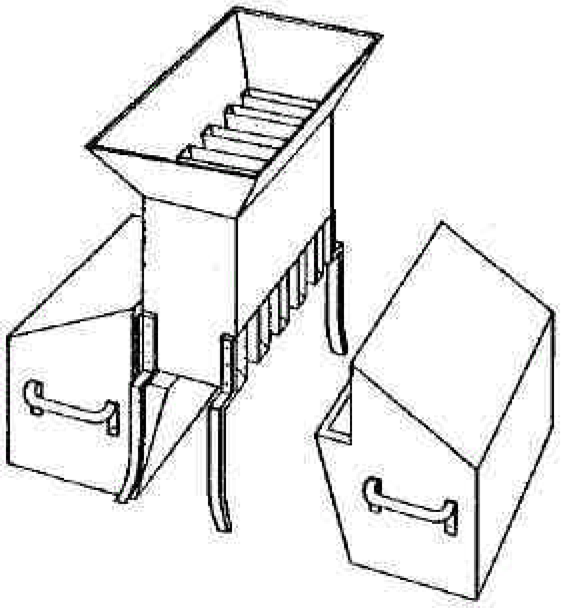

Figure 1 — Example of a riffle box [Reproduced from EN 932-1]

Division of the sample with a riffle splitter is only practical for samples less than approximately 100 kg.

Use the size of riffle splitter best suited for the maximum particle size of the material to be tested.

NOTE Samples of material with particles larger than 45 mm should be split by coning and quartering.

The sample material to be split must be dry enough to allow free flow of the particles through the equipment.

NOTE 1 If the material is not dry, fine particles may cling to the compartment walls, blocking the divider or producing sub-samples with fewer fines than the original sample.

NOTE 2 The material should not be interlocked, aggregated or fibrous.

10.3.2 Method

Check that the slot widths of the riffle splitter are at least three times the maximum particle size of the material to be sub-sampled to avoid bridging.

Place the two sub-sample collectors in position.

Load the sample material into the hopper in a uniform manner. Use a shovel, or transfer directly from a container, to pour the material down the centre of the riffle box hopper. It is essential that the shovel or container is held perpendicular to the axis of the box and that the material is poured evenly across the riffle to prevent biased sub-sampling.

Discard the material that falls into one of the two sub-sample collectors.

Repeat the process of riffling until the volume of material in a sub-sample collector is equal to the desired sub-sample size.

NOTE If the bulk sample is too large to go into the riffle box hopper, either

a) Divide the bulk sample into sub-samples that are small enough, reduce each by the same number of riffling stages, and then combine the reduced sub-samples, or

b) Use coning and quartering or the long pile-alternate shovel method for the first reduction stages, followed by riffling.

Transfer the sub-sample to an appropriate sample container either:

c) in accordance to TR xxxx-4, or

d) as specified in the Sampling Plan.

11 Sub-sampling record

The mixing and sub-sampling 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)

The modular horizontal system

Annex B
(informative)

Information on WP 2 Sampling and the project HORIZONTAL

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