CEN BTTF 151

Date: 2007-04-03

CSS99032

prCEN/TR XXXX-3

Secretariat: DS

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

Einführendes Element — Haupt-Element — Ergänzendes Element

Élément introductif — Élément central — Élément complémentaire

ICS:

Descriptors:

Document type: Technical Report
Document subtype:
Document stage: CEN Enquiry
Document language: E

TR3_HZTL_Draft_Revised 03Apr07.doc

Contents

Page

Foreword

This Technical Report (CSS99032) 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:

CSNN99031: 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 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 pre-treatment of samples prior to analysis. This report does not attempt to provide a definitive procedure for each and every situation that might arise from sampling a given material or a specific analytical requirement; rather it aims to expose the factors that influence the selection of these practical field activities to ensure the most appropriate procedure is selected for any given sampling scenario.

The most appropriate approach, tools, and methodology should be chosen on a scenario-specific basis. However, this does not present a barrier to technical innovation, and there is no reason why methodologies other than those detailed in this Technical Report cannot be substituted.

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 Step 2 of the sampling plan development 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 analytical laboratory.

This Technical Report 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 might require additional sub-sampling and/or pre-treatment steps prior to analysis. Field sub-sampling should be carried out in such a way as to obtain, at all stages, a laboratory sample that is representative of the field sample. Specifically this Technical Report supports Clause 4.2.9.2 (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: 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 for sludge and treated biowaste

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 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 might be equally relevant.

This Technical Report is applicable to sub-sampling of sludge, treated biowaste, and soils in the landscape.

2 Normative references

The following documents are indispensable for the application for 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:date, 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 Principles of sub-sampling in the field

Sub-sampling might be necessary when more than one laboratory sample is to be prepared from a single field sample or a composite sample or when it is necessary to reduce the sample mass / volume for transportation to the laboratory.

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

Sub-sampling should be carried out in the field only where the integrity of the sample and sub-samples can be assured, i.e. in an environment that protects against the loss of moisture and volatile components due to evaporation, or the 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 Situations where sub-sampling should be undertaken only under (mobile / field)laboratory conditions or where mixing might be undesirable include:

- samples for microbiological analysis: high risk of cross-contamination;
- samples for moisture, volatile or semi-volatile components: risk of loss by evaporation;
- dry materials: loss of particulates due to air entrainment;
- particulate materials: mixing causes abrasion;
- samples for rheological examination: mixing changes the parameter of interest.

Sub-sampling can be achieved with or without particle size reduction. Particle size reduction consists of crushing or grinding the sample in order to reduce the particle size of the whole or sub-sample without reducing the sample mass / volume. Such reduction procedures are particularly susceptible to the loss of fine particles due to air entrainment in a field environment and such procedures should only be undertaken under laboratory conditions. The procedures in this Technical Report are restricted to methods that exclude particle size reduction by grinding.

If a heterogeneous composite sample cannot be adequately mixed in the field or mobile/field laboratory to produce a homogeneous sample it should be sent to the laboratory for mixing and sub-sampling.

When two or more laboratory samples are required from a composite sample, the sub-sampling process should be specified in such a way that two or more sub-samples of equal mass / volume and composition are generated. In most cases, multiple equal laboratory samples are obtained from the last sub-sampling stage (if the sub-sampling process consists of more than one stage) to ensure that multiple laboratory samples are as identical as possible.

When the required size and number of sub-samples have been obtained they should be transferred to a sample container(s) as specified in the sampling plan.

5 Apparatus

To avoid cross-contamination, the material of the apparatus should not comprise or include any constituent for which the laboratory sample might be analysed.

Suitable apparatus includes:

— clean protective covering to protect the sample from contamination by the mixing surface – heavy duty plastic is suitable;

— scoop;

— spade;

— mechanical shovel;

— balance;

— sheet-metal cross;

— riffle box;

— rotary sample divider.

NOTE 1 Examples of sub-sampling apparatus are given in Annex A.

NOTE 2 In all cases, alternative designs can be used as long as the devices can be used to fulfil the sub-sampling procedures described in clauses 7 to 13.

6 Preparation for mixing and sub-sampling

The following procedure should be followed, where possible, prior to all sample mixing and sub-sampling activities:

— identify an area within a covered area or mobile/field laboratory, or area of hard standing that is sheltered from the effects of the weather;

— choose and area that is flat and large enough to allow ease of access to the sample containers and sufficient space for mixing;

— place a clean protective covering on the mixing surface, if necessary;

— ensure that all apparatus and tools are clean to reduce the risk of cross-contamination.

7 Sub-sampling granular materials

7.1 General considerations

Materials that might be considered granular include:

- treated biowaste;
- thermally dried sludge granules;
- thermally dried sludge pellets;
- granular lime-treated sludge;
- composted sludge;
- composted air-dried / solar-dried sludge;
- air-dried and solar-dried sludge;
- aged sludge from reedbeds;
- soil.

NOTE Mixing and sub-sampling of very dry materials, e.g. thermally dried sludge, can result in particle abrasion leading to changes in the physical structure and the risk of loss of fine particles. Such materials should only be sub-sampled in a protected environment, i.e. a (mobile/field)laboratory.

7.2 Mixing granular materials

7.2.1 General

Manual mixing of small volumes of granular material to produce a homogeneous sample might be possible in the field, but will depend on the particle size distribution and extent of the cohesive properties of the material.

The following procedure should be carried out:

- prepare a composite sample by combining equivalent quantities (m/m or v/v) of the individual increments;
- determine the quantities (on a dry weight basis) of the increments to be mixed together.

NOTE In most cases where increments are taken from the same stratum, the moisture content of individual increments will be approximately the same and increments can be mixed in the field on an equal volume or weight basis. However, it is possible for the moisture content of multiple increments from a single volume or mass of material to vary considerably. In this situation mixing of the increments by equal weight or volume would result in a composite sample that is biased due to under- or over-sampling of the individual increments. Mixing of increments with variable moisture content should therefore, be carried out in the (mobile / field) laboratory following determination of the dry matter content of each increment.

7.2.2 Methodology

Prepare for mixing and sub-sampling as described in Clause 6, then mix the composite sample by forming a conical heap as follows:

- place all the increments on a clean surface to form a composite sample;
- form a new conical heap on the clean surface by taking a spade or scoopful of the composite sample, putting each subsequent spade or spoonful on the top of the preceding one until all the material is

transferred. To achieve good mixing the size of the spade or scoop should be such that this action needs to be repeated at least 20 times to transfer all the material.

7.3 Sub-sampling granular materials

A sample can be divided into sub-samples by manual or mechanical means. When the material is dry and free-flowing, mechanical techniques should be used 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 (11.3);

— coning and quartering (11.4);

— riffling (11.5).

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

8 Sub-sampling liquid sludges

8.1 Mixing liquid sludges

8.1.1 General

A composite sample can be produced by combining equivalent quantities (v/v or w/w) of the individual increments.

Stratified samples, where differences in the strata are of interest, should be processed under (mobile/ field)laboratory conditions.

Take care to minimize the loss of volatile components.

8.1.2 Methodology

After preparing for mixing and sub-sampling as described in Clause 6, the following procedure should be carried out:

— determine the quantities by volume of the increments to be mixed;

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

— 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.

8.2 Sub-sampling liquid sludges

Mix the composite sample thoroughly (see 8.1.2) then:

— pour the required quantity into a sample container as specified in the sampling plan;

— if multiple sub-samples are required, carry out the mixing procedure between sub-samples.

NOTE This ensures that each sub-sample is representative and is very important for liquid sludges, which are not true liquids, but two-phase mixtures (suspensions).

9 Sub-sampling paste-like sludges

9.1 General considerations

Materials that can be considered paste-like include:

— dewatered sludge cake;

— extruded lime-treated sludge.

9.2 Mixing paste-like sludges

9.2.1 General

A composite sample can be produced by combining equivalent quantities (m/m or v/v) of the individual increments.

NOTE In most cases where increments are taken from the same stratum, the moisture content of individual increments will be approximately the same and increments can be mixed in the field on an equal volume or weight basis. However, it is possible for the moisture content of multiple increments from a single volume or mass of material to vary considerably. In this situation mixing of the increments by equal weight or volume would result in a composite sample that is biased due to under- or over-sampling of the individual increments. Mixing of increments with variable moisture content should therefore, be carried out in the (mobile / field) laboratory following determination of the dry matter content of each increment.

The most suitable method for mixing depends on the water content of the material. Dewatered sludges with high water content might be better considered as gelatinous sludges (see 10). Due to the stickiness of the material, it might not be possible or practicable to mix some sludges using manual methods, especially if the volume to be mixed is large. In this case a mechanical mixer should be used.

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

9.2.2 Methodology

After preparing for mixing and sub-sampling as described in Clause 6, the following procedure should be carried out, depending on the quantity of material:

Mix small quantities of material by stirring, as follows:

— choose a receptacle that is large enough so that, during mixing, there is no loss of sample;

— determine the quantities (by mass or volume) of the increments to be mixed together; place the increments in the mixing container and mix by stirring.

Mix larger quantities of material by turning as follows.

— mix the composite sample by forming a conical heap. Place all the increments on a clean surface to form a composite sample;

— form a new conical heap on the clean surface by taking a spade or scoopful of the composite sample, putting each subsequent spade or spoonful on the top of the preceding one until all the material is transferred. To achieve good mixing the size of the spade or scoop should be such that this action needs to be repeated at least 20 times.

9.3 Sub-sampling paste-like sludges

After mixing the sample thoroughly as described in 9.2.2, the following procedure should be carried out, depending on the quantity of material:

Mix small quantities of material by stirring, then take the required amount using a scoop.

Mix larger quantities of material by turning, using either the long pile-alternate shovel method (see 11.3) or the coning and quartering method (see 11.4)

NOTE 1 The long pile-alternate shovel method is suitable for the reduction of samples in excess of approximately 100 kg.

NOTE 2 The coning and quartering method is suitable for the reduction of samples down to approximately 1 kg.

10 Mixing and sub-sampling gelatinous sludges

10.1 Mixing gelatinous sludges

10.1.1 General

A composite sample can be produced by combining equivalent quantities (m/m or v/v) of the individual increments.

When the moisture content of multiple increments from a single volume or mass of material vary considerably mixing of the increments by equal weight or volume will result in a composite sample that is biased due to under- or over-sampling of the individual increments. Mixing of increments with variable moisture content should therefore, be carried out in the (mobile / field) laboratory following determination of the dry matter content of each increment.

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

10.1.2 Methodology

After preparing for mixing and sub-sampling as described in Clause 6, one of the following two methods of mixing a composite sample should be carried out, where possible.

- The composite sample can be mixed following the procedure described in Clause 9 (Mixing paste-like sludges).
- Alternatively, mix the composite sample by forming a conical heap. Place all the increments on a clean surface to form a composite sample. Form a new conical heap on the clean surface by taking a spade or scoopful of the composite sample, putting each subsequent spade or spoonful on the top of the preceding one until all the material is transferred. To achieve good mixing the size of the spade or scoop should be such that this action needs to be repeated at least 20 times.

The material can be further mixed by employing a technique such as that employed for manual preparation of cement mortar (using a shovel or trowel, create a pile with a hollow centre; working from the outside start to turn the material into the middle. Then mix thoroughly and repeat until the material appears to be homogeneous).

NOTE Due to the stickiness of the material, it might not be possible to mix some sludges following these methods, especially if the volume to be mixed is large. In this case a mechanical mixer should be used.

10.2 Sub-sampling gelatinous sludges

After mixing the composite sample thoroughly, as described in 10.1.2, take the required amount using a scoop.

11 Sub-sampling methods for granular materials

11.1 General

The following sub-sampling methods are based on repeated halving of the sample until the amount of material needed for the laboratory sample remains.

NOTE These methods are not appropriate for liquids.

11.2 Aggregate reduction

11.2.1 General considerations

In some cases aggregate reduction might be required before sub-sampling can be carried out; e.g. soil material or composted biowaste or sludge, where the material has formed clumps.

If the aggregate size is greater than the minimum size of the sub-sample, the size of the aggregates should be reduced during or prior to sub-sampling.

If aggregate reduction is required, it should be stated in the sampling plan.

11.2.2 Aggregate reduction by hand

After preparing for mixing and sub-sampling as described in Clause 6, carry out aggregate reduction by hand as follows:

— place the sample on the protective floor covering and spread evenly to identify all large aggregates within the sample;

— using a spade or trowel gently reduce the size of the aggregates until all oversized material is less than or equal to the required particle size.

NOTE Aggregate reduction by hand can result in the sample being in contact with the air for extended periods and this method should only be applied when sample integrity is not compromised (see Clause 4).

11.2.3 Aggregate reduction using mechanical devices

It is also possible for aggregate reduction to be achieved using mechanical devices.

The mechanical device should be cleaned to prevent cross-contamination. Where the device cannot be cleaned completely, crush a small volume of the sample in the device and discard the output before reducing the rest of the sample.

11.3 Long pile-alternate shovel method

11.3.1 Principle

The composite 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. One mound is randomly selected and the process is repeated.

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

11.3.2 Method

Shovel the sample into a conical pile on the protective floor covering, placing each subsequent shovelful on the top of the preceding one.

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

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

Form the final conical pile into a long pile as follows:

— take a shovelful from the base of the conical pile and spread the material into a ribbon having an initial width equal to that of a shovel;

— take the next shovelful from a different point at the base of the conical pile 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 conical pile to halving the pile) until the retained amount of material is equal to the desired size of the sub-sample.

11.4 Coning and Quartering

11.4.1 Principle

The composite sample can be formed into 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.

11.4.2 Method

Mix the increments to produce a composite sample (see 7.2).

Shovel the composite sample into a conical pile on the clean surface, placing each subsequent 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 composite sample is on the floor, mix it thoroughly by turning it over to form a new cone adjacent to the first. Carry out this operation at least three times to ensure thorough mixing. 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 amount.

11.5 Riffling

11.5.1 Principle

A riffle box (see Figure A.1) is designed to reduce a sample to half its original size. The sample is placed into a hopper with a control gate that, when opened, allows the sample material to flow through an even number of equally divided compartments or chutes, sending half the sample to the left side and half the sample to the right side into collection pans. The content of one of the pans is randomly selected and the process is repeated.

11.5.2 General

A riffle box can be used when the material is dry enough to allow free flow of the particles through the equipment. Division of samples with a riffle box is generally considered practical only for samples less than approximately 100 kg.

A riffle box should be chosen with slot widths that are at least three times the maximum particle size of the material to be sub-sampled to avoid bridging.

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

If the material is not dry, fine particles can cling to the compartment walls, blocking the divider or producing sub-samples with fewer fines than the original sample and an uneven distribution of the original material over the two collectors; ultimately resulting in biased sub-samples.

The material should not be interlocked, aggregated or fibrous.

11.5.3 Method

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, 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 with the non-discarded material until the mass / volume of material in a sub-sample collector is equal to the desired sub-sample mass / volume.

If the composite sample is too large to go into the riffle box hopper, it can be sub-sampled by:

— dividing the composite sample into sub-samples that are small enough, reducing each by the same number of riffling stages, and then combining the reduced sub-samples, or

— sub-sampling the amount of sample by coning and quartering or the long pile-alternate shovel method, followed by riffling.

12 Incorporation in the sampling plan

The mixing and sub-sampling procedure(s) and necessary equipment should be specified in the sampling plan prior to commencing sampling.

Annex A
(informative)
Examples of equipment for sub-sampling

A.1 Riffle box

The number of slots of a riffle box should be even and not less than eight. The width of the slots should be at least three times the upper aggregate size to avoid bridging.

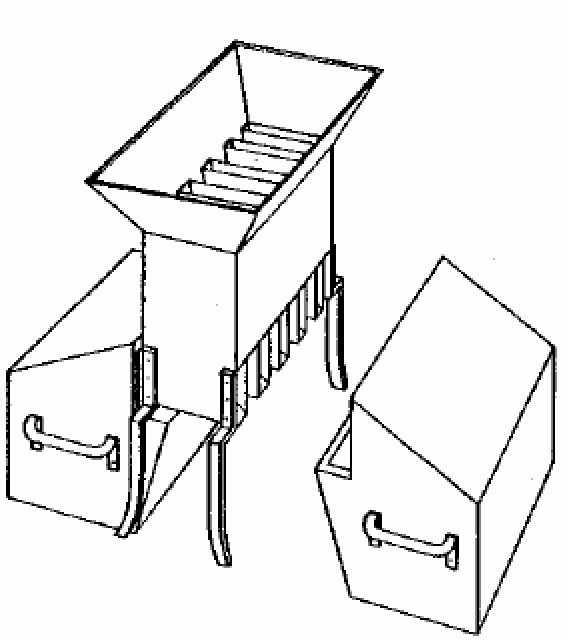

Figure A.1 — Example of a riffle box [Reproduced from EN 932-1]

A.2 Rotary sample divider

When rotating at a constant speed, a rotating sample divider divides the sample into sub-samples which are representative of the composite sample. The rotating dividers consist of a number of prismatic containers, of equal size, mounted round the periphery of a circle which passes under the falling stream of the sample fed from a hopper mounted above the turntable, and off-set from the centre.

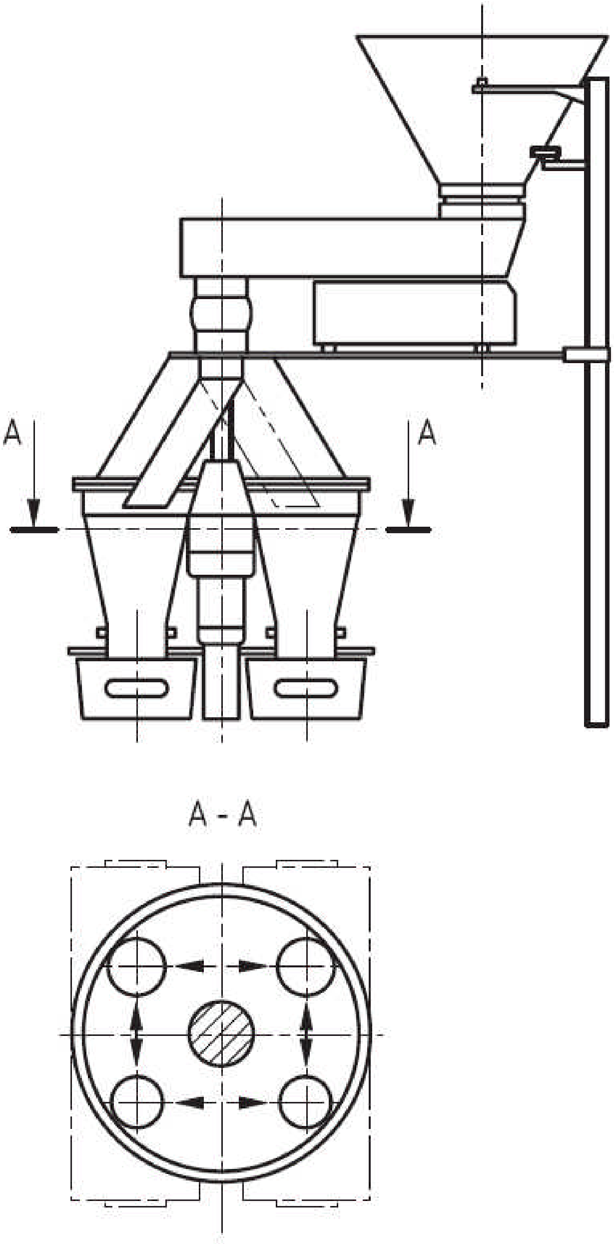

Figure A.2 — Example of a sample divider for large particle sized solid materials [after EN 932-1]

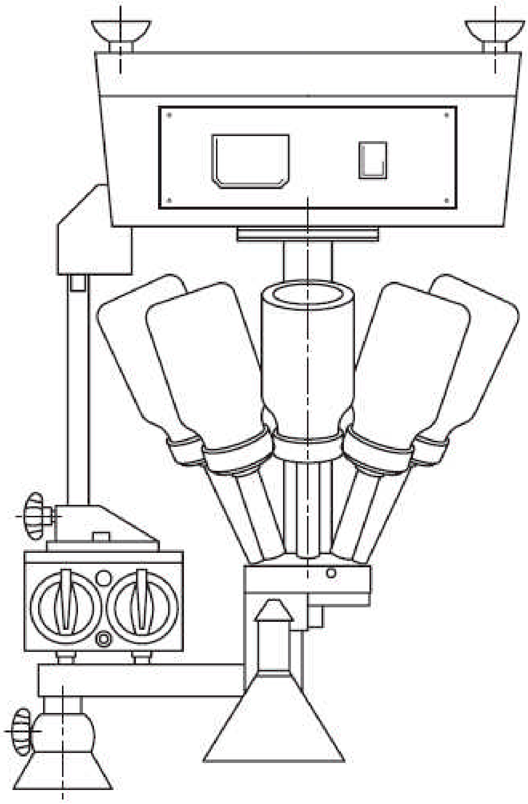

Figure A.3 — Example of a rotary sample divider for small particle sized solid materials [after EN 932-1]

A.3 Sheet-metal cross

A sheet-metal cross is made with four blades joined together at the centre at 90° to each other. (No figure)

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