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## **Introductory element — Main element — Complementary element**

### **Introductory element – Sampling of sewage sludge and treated biowastes – Guidance on sampling techniques**

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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 xxxxx is the mandatory requirement to prepare a Sampling Plan, within the framework of an overall testing programme as illustrated in Figure 1 of prEN xxxxx and 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 describes a selection of sampling techniques that can be used in the recovery of a sample for a wide variety of sludges and treated biowastes. The sampling technique is the physical procedure employed by the sampler to collect part or parts of a discarded or secondary material for subsequent investigations. Specifically this Technical Report provides information to support Clause 4.2.8.1 (Identify the sampling technique) 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

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

## 1 Scope

This Technical Report describes techniques for sampling sewage sludge (liquids, sludges, paste-like and particulate materials) and treated biowaste (particulate materials) found in a variety of locations (pipes, lagoons, tanks, trucks, piles, in motion or in bags). The Technical Report provides information to allow the selection and preparation of equipment and apparatus to be used in the sampling activity.

NOTE 1 This Technical Report provides a shop shelf of example sampling techniques that can be selected to meet a wide range of sampling situations. For a specific situation one of the presented procedures may be appropriate.

NOTE 2 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 3534-1:1993, Statistics – Vocabulary and symbols – Probability and general statistical terms

ISO 5555:2001, Animal and vegetable fats and oils – Sampling.

ISO 9411 (sampling equipment)

ISO 11074-2:1998, Soil Quality – Part 1: Terminology and classification – Section 1.2: Terms and definitions related to sampling

EN 12832:1999, Characterisation of sludges – Utilisation and disposal of sludges - Vocabulary

## 3 Terms and definitions

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

### 3.1

#### **bottom sediment**

solid layer of material on the bottom of liquid storage tanks.

### 3.2

#### **characteristic**

a property, which helps to identify or differentiate between items of a given population.

[ISO 3534-1:1993, definition 2.2]

NOTE The characteristic may be either quantitative (by variables) or qualitative (by attributes).

### 3.3

#### **column sample**

type of sample, more specifically related to the sampling of liquids where column samplers are used. A column of material is of equal length to the depth of the batch at that sampling point.

NOTE A column of material is of equal length to the depth of the sub-population at that sampling point.

**3.4****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]

**3.5****constituent**

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

**3.6****core sample**

type of sample, more specifically related to the sampling of solids where augers and other core samplers are used. A vertical or directional sample is taken through the material whereby the integrity of the batch is maintained.

NOTE A vertical or direction sample is taken through the material whereby the integrity of the sub-population is maintained.

**3.7****directional sample**

geometric sample, usually in only one dimension, which is related to the single principal axis of variability of material in the sampling unit/lot.

**3.8****geometric sample**

type of sample of specific shape, whose dimensions are related to the axes of variability of material in a sampling unit/lot.

**3.9****heterogeneity**

degree to which a constituent (3.5) is not uniformly distributed throughout a quantity of material.

NOTE 1 A material may be homogeneous with respect to one analyte or property but heterogeneous with respect to another.

NOTE 2 The degree of heterogeneity is the determining factor in sampling uncertainty

**3.10****homogeneity**

degree to which a constituent (3.5) is uniformly distributed throughout a quantity of material.

**3.11****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 compose 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 the 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 European Standard this is however an essential condition in the definition of the term 'increment'.

**3.12****judgemental sampling**

samples collected using at best a partially-probabilistic procedure and at worst a non-probabilistic approach

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NOTE Usually these samples are taken from a sub-population which is substantially more restrictive than the overall population.

### 3.13

#### **laboratory sample**

the sample(s) or sub-sample(s) sent to or received by the laboratory.

NOTE 1 The laboratory sample may be used directly as the test sample, or may require further preparation such as sample size reduction, mixing, grinding, or by combinations of these operations to produce the test sample.

NOTE 2 The laboratory sample is the final sample from the point of view of sample collection but it is the initial sample from the point of view of the laboratory.

NOTE 3 Several laboratory samples may be prepared and sent to different laboratories or to the same laboratory for different purposes.

### 3.14

#### **population**

totality of items under consideration

[ISO 3534-1:1993]

### 3.15

#### **probabilistic sampling**

sampling conducted according to the statistical principles of sampling

NOTE 1 The essential principle of probabilistic sampling is that every individual particle or item in the population has an equal chance of being sampled.

NOTE 2 Probabilistic sampling results in boundary conditions for the type of sampling equipment used, the method of sampling (where, when, how) and the minimum size of increments and (composite) samples.

### 3.16

#### **representative sample**

sample in which the characteristic(s) of interest is (are) present with a reliability appropriate for the purposes of the testing programme

### 3.17

#### **sample**

portion of material selected from a larger quantity of material

[ISO 11074-2:1998]

NOTE 1 The manner of selection of the sample should be described in a sampling plan

NOTE 2 The use of the term 'sample' should be supported with a preface 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.

### 3.18

#### **sampler**

person carrying out the sampling procedures at the sampling locality.

[ISO 11074-2:1998, definition 1.5]

NOTE Tools and other devices to obtain samples are sometimes also designated 'samplers'. In this case write 'sampling devices' or 'sampling equipment'.



**3.19**

**sampling plan**

all the information pertinent to a particular sampling activity

NOTE 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]

**3.20**

**sludge**

mixture of water and solids separated from various types of water as a result of natural or artificial processes.

[EN 12832:1999, definition 3.1]

NOTE Attention is drawn to the fact that the term "sludge" is partially defined in the Directives 86/278/EEC and 91/271/EEC.

**3.21**

**stratified sampling**

in a population which can be divided into mutually exclusive and exhaustive sub-populations (i.e. strata), sampling carried out in such a way that specified proportions of the sample are drawn from the different strata and each stratum is sampled with at least one sampling unit.

[ISO 3534-1:1993, definition 4.14]

NOTE The objective of taking stratified samples is to obtain a more representative sample than that which might otherwise be obtained by random sampling.

**3.22**

**sub-population**

a defined part of the population that will be targeted for the purposes of sampling.

**3.23**

**sub-sample**

a sample taken from a sample of a population.

[ISO 3534-1:1993]

**3.24**

**test sample (analytical sample)**

sample, prepared from the laboratory sample, from which the test portions are removed for testing or for analysis.

[ISO 11074-2:1998, definition 3.16]

**3.25**

**viscous liquid**

liquid with high viscosity, resulting in slow flow and adhering to containers and sampling equipment.

## **4 Health and safety**

Personnel responsible for designing sampling programmes and for carrying out sampling operations should ensure that the requirements of relevant (inter)national regulations and site specific safety instructions are taken into account. Sampling personnel should be informed of the necessary precautions to be taken in sampling operations. This should include safe working practices for specific locations, the use of protective clothing and equipment, decontamination and emergency procedures. It is essential that the sampler complies with any health and safety instructions all times when sampling treated sludges or biowastes.

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When the circumstances in the field deviate from the assumed situation in the Sampling Plan it may be necessary to alter the Sampling Plan for reasons of health and safety. Depending on the type of alterations, the project manager who made the sampling plan should be consulted prior to actual sampling.

These guidelines cannot be substituted for local and/or national rules and regulations. The provisions of national health and safety regulations should always be carefully studied and put into effect before sampling occurs.

Safety issues will be specific to the site where sampling is carried out. Examples of the type of risk that may be present with respect to sludge and treated biowaste sampling are:

- a) Treatment plants are often associated with wet flooring which can be covered in minor spillage; presenting a potential for personnel slipping.
- b) Bacterial and parasitic infection can occur through accidental ingestion or infection via unprotected broken skin and mucus membranes. These risks can be minimised by the adoption of strict personal hygiene codes and the wearing of gloves and goggles.
- c) Sampling from sludge cake stockpiles is not without some risk of personal injury, and care should be taken to avoid the risk of becoming trapped by inducing slippage or sinking.
- d) When sampling unconsolidated stockpiles, additional safety instructions may be required.
- e) Regard should be given to the requirements for safe working practice when working near moving machinery, for example conveyor belts and press plates or site vehicles.
- f) It is suggested that the maximum mass of a sample to be lifted by an individual be 25 kg.
- g) Where sludge is sampled using pressure or vacuum systems, care should be taken to prevent exposure of personnel to sludge aerosols formed during the sampling process. Otherwise infectious agents can enter the body via the lungs.

## 5 Sampling equipment

Choose sampling equipment appropriate to the type of material being sampled, the sampling location and the size of sample to be collected. Guidance on selection and descriptions of commonly used equipment are provided in Annex A.

Prior to use all apparatus and tools should be cleaned to reduce the risk of cross-contamination. If sterilized or disinfected samples are to be collected, sterile sampling equipment should be used.

Where more than one sample is to be collected sampling equipment should be cleaned (and sterilised) between samples.

Where it is not possible to clean any sampling equipment between samples this should be recorded in the sampling record.

**NOTE 1** Due to the nature and composition of the materials being sampled, aseptic techniques are difficult or impractical for sampling purposes. Every effort should be made to ensure cleanliness and good hygiene practices.

**NOTE 2** When samples are to be collected from several locations it is advisable to prioritise sampling so that material that has undergone the most treatment is sampled before material that has undergone limited or no treatment.

**EXAMPLE** Untreated sewage sludge is likely to contain a higher number of micro-organisms than treated sludge. If microbiological parameters are of interest, treated sludge should be sampled before untreated sludge.

## 6 Principle of sampling technique selection

This Technical Report details a wide range of sampling techniques that can be used to take a sample. The procedures identified in this document target two fundamental objectives of sampling, as outlined in the Framework Standard prEN xxxx:

- Probabilistic sampling – the preferred method of sampling or recovering material where a quantifiable level of reliability is required in the results for the population being tested. The basis for probabilistic sampling is that each element within the population being sampled has an equal chance of being sampled. This means that the sampler has access to the whole population and can collect a sample that is representative of that population.
- Judgemental sampling – this is used where representative sampling from the whole population is impractical, given available resources (time or money) or when sampling is required to target a specific item or point within the population.

The sampling techniques identified in this Technical Report form only part of the approach required to achieve probabilistic sampling, reference should be made to other Technical Reports in this series to ensure all requirements have been fulfilled. For example, key advice on the design of an appropriate Sampling Plan and selection of an appropriate sampling pattern, number of samples and sample size needed to meet the requirements of probabilistic sampling can be found in TR xxxx-5 and TR xxxx-1 respectively.

Sampling procedures are provided for a wide range of process streams and common storage conditions. The preferred sampling technique will depend on a combination of different characteristics of the material and circumstances encountered at the sampling location. Relevant determining factors include:

- the type of material / the physical state of the material (e.g. liquid, paste, sludge, granules)
- the situation at the sampling location / the way in which the material occurs (e.g. in a tank, a stockpile, in a bag)
- the (expected) degree of heterogeneity (e.g. homogeneous liquids, layered liquids, segregated sludges)
- the level of testing, which may influence the approach to the selection of composite or individual samples as detailed in TR xxxx-1.

A series of process maps or flow charts provide route maps to the user to relevant clauses in the document for a wide range of potential sampling situations that arise when the range of different materials and sampling locations are considered. This approach allows the tools, and methodology to be chosen on a scenario-specific basis. The procedures listed in this Technical Report reflect current best practice, but these are not exhaustive and other procedures may be equally relevant.

## 7 Route map for the selection of sampling techniques

This Technical Report has been structured to address the selection of sampling techniques and equipment by physical form (e.g. liquid, sludge or granules) and nature of the arising (e.g. road tanker, stockpile or bag). This Technical Report does not present a definitive process, but reflects current practice for commonly occurring scenarios, this, however, does not mean that other solutions are not available. The selection of an appropriate sampling technique should be related to the objectives for sampling and the physical form and chemical or microbiological characteristic to be sampled. The route maps presented in this document supports the guidance provided in prEN xxxx - A Framework for the preparation and application of a Sampling Plan. The following flow diagram guides the reader to the appropriate clauses within the Technical Report.

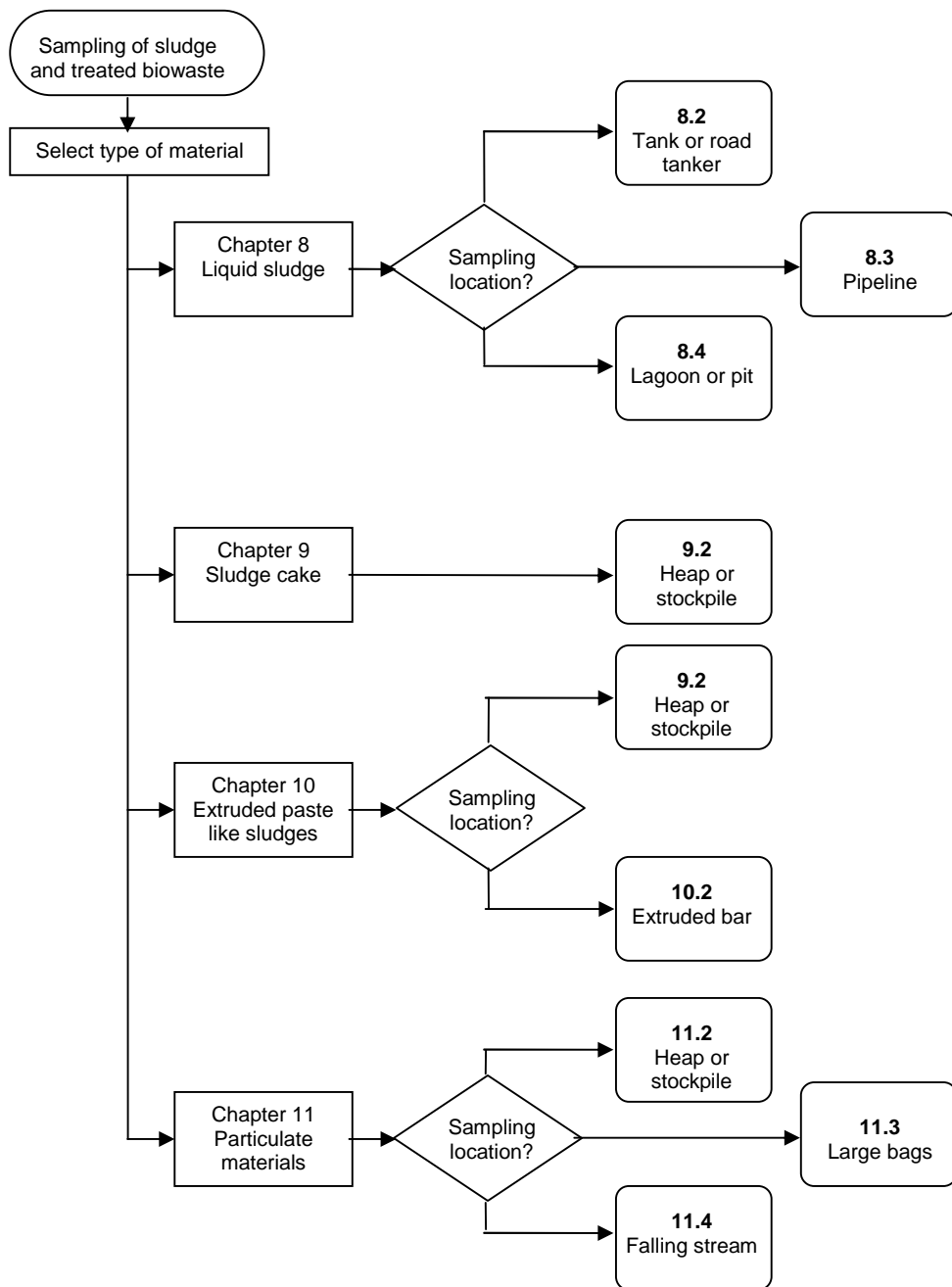


Figure 1 — Generic process map for sampling

## 8 Sampling liquid sludge

### 8.1 General

Often liquid sludges can be readily taken from taps or valves situated on outlet pipework. In other circumstances, it may be necessary to take dipped samples from a road tanker, storage tank or lagoon. Practical and safety considerations may dictate the point at which samples can be obtained.

Where sludges are likely to have settled and stratified the performance of tanks cannot always be gauged from samples taken from inlet and outlet pipelines. The segregation of solids that is likely to occur can be detected by sampling different sections and depths.

### 8.2 Sampling from tanks or road tankers

#### 8.2.1 Probabilistic sampling

##### 8.2.1.1 Preparation for sampling

If the sludge is known to be homogeneous, sample using the procedure described in 8.2.1.2 (Procedure for taking a probabilistic sample where contents are known to be mixed or are homogenous).

NOTE 1 If on-site homogenisation (e.g. circulation) is possible, sampling may be performed as for homogeneous liquids provided the contents have been thoroughly mixed before sampling.

NOTE 2 On-site homogenisation minimises the need for sampling stratified material, since the whole of the sludge volume is treated as a composite sample.

Unless the sludge is known to be homogeneous, assume the sludge is heterogeneous. Either sample using the procedure described in 8.2.1.3 (Procedure for taking probabilistic samples where contents are not mixed or are heterogeneous) or determine the stratification and sample using the procedure described in 8.2.1.4 (Procedure for taking a probabilistic sample from heterogeneous (stratified) liquids).

##### 8.2.1.2 Procedure for taking a probabilistic sample where contents are known to be mixed or are homogenous

If the tank is being emptied, samples may be collected as described in 8.3 (Sampling liquid sludge from a pipeline). Alternatively, use a sampling tube to sample the full depth of the liquid.

Lower the open sampling tube into the tank sufficiently slowly to ensure that the liquid level in the tube does not fall below that of the outside liquid;

Close the tube, withdraw it from the tank then allow any liquid adhering to it to drain from it or wipe the tube dry;

Transfer the contents of the sampling tube to the sample container;

Repeat the procedure until sufficient quantity of sample is collected.

NOTE It is not recommended to use a pumping action to increase the size of the sample taken in any one procedure.

##### 8.2.1.3 Procedure for taking probabilistic samples where contents are not mixed or are heterogeneous

If the tank is being emptied samples may be collected as described in 8.3 (Sampling liquid sludge from a pipeline).

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Access to different strata is often provided by a design feature such as stepped draw-off pipework. Inspection of the tank concerned will usually reveal the presence of these facilities if they have been built in. When this is not the case, vacuum sampling devices could be used (see A.3.2a).

Alternatively, take increments at depth intervals using a sampling tube or a weighted sampler as described in 8.2.2.3 (Procedure for taking a sample at specified depth).

Start sampling from the top layer and take samples at equally spaced intervals, as specified in the Sampling Plan.

If the increments are to be examined individually, label the increments as specified in the Sampling Plan.

If the samples are to be examined combined, mix the increments in the proportions specified in the Sampling Plan to give a representative sample and label as specified in the Sampling Plan.

### 8.2.1.4 Procedure for taking a probabilistic sample from heterogeneous (stratified) liquids

Establish the volume per layer to allow calculation at a later stage of the average concentrations within the tank.

Method 1: Using a transparent sampling tube

- Take a sample of the entire depth;
- Lower the open sampling tube into the tank sufficiently slowly to ensure that the liquid level in the tube does not fall below that of the outside liquid;
- Close the tube, withdraw it from the tank then allow any liquid adhering to it to drain from it or wipe the tube dry;
- If layering is observed, calculate the volume per layer in the tank using the stratification identified in the sampling tube;
- Record the information in the Sampling Record.

Method 2: Mixed surface and bottom sample

- Take a single surface sample as described in 8.2.2.1 (Procedure for taking a surface sample) and transfer the sample to a transparent bottle;
- Take a single bottom sample as described in 8.2.2.2 (Procedure for taking a bottom sample) and transfer the sample to the bottle with the surface sample;

NOTE The surface and bottom samples should be mixed in equal volumes.

- Cap the bottle and invert to mix the samples, then allow to stand for a minimum of 2 minutes;
- If layering is observed, estimate the volume of each layer in the tank;
- Record the information in the Sampling Record.

If layering is not observed, sample using the procedure described in 8.2.1.3 (Procedure for taking probabilistic samples where contents are not mixed or are heterogeneous).

If layering is observed, the liquid is stratified. Collect a separate sample from the centre of each layer using the procedure described in 8.2.1.3 (Procedure for taking probabilistic samples where contents are not mixed or are heterogeneous).

Alternatively, if the tank is being emptied samples may be collected as described in 8.3 (Sampling liquid sludge from a pipeline).

## **8.2.2 Judgemental sampling**

### **8.2.2.1 Procedure for taking a surface sample from a tank**

Lower a bailer or weighted can into the tank to just below the surface of the liquid;

Remove the bailer/can before it fills completely;

Transfer the contents of the can to the sample container, using a funnel if necessary;

Repeat the procedure until sufficient quantity of sample is obtained.

### **8.2.2.2 Procedure for taking a bottom sample from a tank**

Insert a closed sampling tube into the liquid until it touches the bottom of the tank;

NOTE The viscosity of the liquid can affect the choice of the sampling tube (see Sampling Plan).

Open the sampling tube and move quickly allowing the mouth of the tube to traverse the bottom of the tank while the tube is filling;

Close the tube, withdraw it from the tank then allow any liquid adhering to it to drain from it or wipe the tube dry;

Transfer the contents of the sampling tube to the sample container.

### **8.2.2.3 Procedure for taking a sample at specified depth**

Take a sample using a sampling tube or a weighted sampler.

NOTE Suitable weighted sampling apparatus is described in Annex A.

Method 1: Using a sampling tube

- Insert a closed sampling tube to the depth specified in the Sampling Plan;
- Open the sampling tube and allow the tube to fill;
- Close the tube, withdraw it from the tank then allow any liquid adhering to it to drain from it or wipe the tube dry;
- Transfer the contents of the sampling tube to the sample container;

Method 2: Using a weighted sampler

- Close the bottle or can and lower the apparatus to the depth specified in the Sampling Plan;
- Open the bottle or can by pulling sharply on the chain;
- When air bubbles cease to rise, lift the apparatus out of the liquid;
- Carefully pour off the liquid contained in the neck of the bottle or can;

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- Transfer the remaining liquid from the can into the sample container or tightly close the bottle and remove from the cage.

### 8.3 Sampling liquid sludge from a pipeline

#### 8.3.1 Preparation for sampling

Factors such as the nature of the sludge, the flow rate, the diameter of the pipes and the roughness of the pipe can affect the tendency of the dynamic system to allow streaming of the flow. The influence of this potential problem can be minimised by allowing the flow to equilibrate before collecting a sample.

Check the pressure before and during sampling. When taking the sample, visual checks should be made to ensure that the flow rate and consistency remain constant.

**NOTE** Blockages in pipes due to fibrous materials will often influence the nature of the sludge by a filtering action. If this is undetected at the time of sampling, it may be necessary to repeat the sampling to assess the reliability of the results.

Clean all sampling ports prior to sampling. If sampling from a tap or valve allow a small volume to be discharged before sampling. Any side arms or valves utilised in the sampling arrangement should be flushed with at least three times the standing volume to ensure that any stagnant material is removed from the pipework.

#### 8.3.2 Taking a probabilistic sample from the end of a pipeline

If pumping is taking place, correct sampling can be achieved with samples being taken at appropriate intervals at the pump outlet or a similar convenient place.

- Use a funnel to channel the liquid into the sample container. To collect a representative sample put the sample container, with funnel, under the exit stream at regular intervals during the whole of the transfer of the liquid as specified in the Sampling Plan.

**NOTE** Sampling from road tankers can be achieved by using a long-handled ladle.

#### 8.3.3 Taking a judgemental sample from the end of a pipeline

- Use a funnel to channel the liquid into the sample container. Place the sample container, with funnel, under the exit stream for the period specified in the Sampling Plan.

### 8.4 Sampling from a lagoon

#### 8.4.1 Preparation for sampling

If sampling from a tap or valve allow a small volume to be discharged before sampling.

#### 8.4.2 Probabilistic sampling

##### 8.4.2.1 Taking a probabilistic sample when a lagoon is emptied via pumping

As 8.3, Sampling liquid sludge from a pipeline.

#### 8.4.3 Judgemental sampling

##### 8.4.3.1 Taking a perimeter sampling using a weighted bottle

- Lower the weighted bottle sampler or sampling can to the depth specified in the Sampling Plan using the connecting cord or rope to gauge the depth of sampling.



- Give a sharp jerk on the cord connected to the bottle stopper and allow sufficient time for the sampler to fill.
- Withdraw the sampler and remove the bottle.
- Refit the stopper eliminating any air from the neck of the bottle.

#### **8.4.3.2 Taking a perimeter sample using a pond sampler**

- Insert the pond sampler, with a clamped beaker, upside-down into the lagoon;
- Invert the pond sampler at the specified location and depth;
- Withdraw the sampler and transfer the sample to the transparent sample bottle, eliminating any air from the neck of the bottle.

## **9 Sampling sludge cake**

### **9.1 General**

For caked sludge where its storage over a period of time may form part of the treatment process, then appropriate samples should be taken from the stored material. Samples should be taken from below the surface of the stored sludge. The sludge should be free of the bed media, since inclusion of grit or sand will distort measurements.

Major variations can be found throughout stockpiles, particularly old ones. When sampling sludge cake from stockpiles it is important to obtain portions of sludge from throughout the mass and not just from the surface layer. Safety requirements may prohibit routine sampling in this manner. In this case, assessing the cross-sections of a heap with a mechanical excavator should be considered if it can be safely undertaken to enable representative sampling.

Depending on the treatment process, lime-treated sludge samples may be similar in texture to caked sludge, in which case sample as for caked sludge. In other cases lime-treated sludge may be extruded as a paste. In this case sample as for paste-like sludges (see clause 10).

### **9.2 Preparation for sampling**

Note and record the thickness and nature of any surface crust. Carefully remove the full depth of the surface crust to a thickness of not less than 100 mm and discard.

**NOTE** Over time the top layers of the sludge desiccate to form crusts which allow anaerobic activity to increase below and aerobic activity to increase in the upper near-surface layers and chemical species may migrate due to leaching in these situations. In this case the surface layers are not representative of the rest of the sludge cake.

#### **9.2.1 Probabilistic sampling**

##### **9.2.1.1 Taking a sample through the depth of a heap or stockpile**

If after assessment of safety requirements and the availability of equipment, core sampling can be considered as a means of obtaining samples, samples should be taken from the depth of the heap/stockpile.

Use a pile sampler if the heap or stockpile has a cross-sectional diameter of 1 m or more.

Insert the sampler into the material being sampled at 0 to 45° from horizontal.

Rotate the pile sampler two or three times in order to cut a core of the sludge cake.

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Slowly withdraw the pile sampler, making sure that the slot is facing upward.

Transfer the sample into the sample container with the aid of a spatula.

Repeat the sampling at different sampling points as specified in the Sampling Plan.

If the increments are to be examined individually, label the increments as specified in the Sampling Plan.

If the samples are to be examined combined, mix the increments in the proportions specified in the Sampling Plan to give a representative sample and label as specified in the Sampling Plan.

### **9.2.1.2 Taking a sample from cross-sections of a heap or stockpile**

Remove and discard the surface crust.

Take a sample from the cleaned surface using a trier or an auger.

Rotate the sampling implement two or three times in order to cut a core of the sludge cake.

Slowly withdraw the sampling implement.

Transfer the sample into the sample container with the aid of a spatula.

Repeat the sampling at different sampling points along the width or circumference of the stored material as specified in the Sampling Plan.

If the increments are to be examined individually, label the increments as specified in the Sampling Plan.

If the samples are to be examined combined, mix the increments in the proportions specified in the Sampling Plan to give a representative sample and label as specified in the Sampling Plan.

### **9.2.2 Judgemental sampling**

Collect individual samples from the part of the stockpile specified in the Sampling Plan following the procedure in 9.2.1.1 or 9.2.1.2.

## **10 Sampling extruded paste-like sludges**

### **10.1 General**

This procedure is suitable for sludges that are extruded under pressure, for example some lime-treated sludges.

**NOTE** For lime-treated sludges, the treatment specification may dictate that sampling or batch release approval should not occur until after the sludge has cooled to below a specified temperature. In this case, sampling should not take place until this temperature has been confirmed.

Typically, the sludge is extruded onto a heap as bars of various lengths. For the purposes of sampling, the sludge may be regarded as a heap or as a number of separate bars.

If the Sampling Plan specifies that samples are to be taken through the depth of the heap or from a cross-section of the heap, follow the procedures for sampling sludge cake (clause 9).

### **10.2 Taking samples from extruded bars**

- Identify the bars to be sampled as specified in the Sampling Plan.

NOTE It is unlikely that the extruder can be stopped prior to sampling. It is advised that the selected bars are removed to a safe place for sampling.

- Take a sample from the bar by taking a cross-section (method 1) or using a scoop (method 2).

#### Method 1: Taking cross-section

- Cut out a piece of the extruded bar of length specified in the Sampling Plan by cutting at right angles through the bar at two places with a knife or cutting wire.
- Remove the sample from between the two cuts and transfer to the sample container.

#### Method 2: Using a scoop

- Scoop out the required amount of material as identified in the Sampling Plan.
- Transfer the sample to the sample container.

## 11 Sampling particulate materials

### 11.1 General

Materials that may be regarded as granular include thermally treated sludges (dried granules and pellets) or treated biowaste (composted materials).

Whenever possible, sampling from the bulk material should be carried out from a moving stream of material, the whole product being sampled.

Thermal drying processes generally produce a product that is of a pellet or granular character and is often bagged or packaged for transport in quantities of 1 tonne. Often it is possible to sample directly into a sample container from an in-line hopper or hatch in the pipework leading to the bagging silo. In this case follow the procedure for sampling from a falling stream (clause 11.4). Otherwise follow the procedure for sampling from large bags (clause 11.3).

All sampling operations should be carried out over a sufficiently short period of time and in such a way as to avoid any alteration in the characteristics of the product delivered or the samples.

NOTE Thermally dried sludges generally possess very low water contents and sampling and storage procedures should take this into account in order that the product does not change significantly after treatment.

It is essential that the sampling system be designed to avoid the following:

- Spillage of the sample;
- Restriction of the bulk material through the system;
- Retention of residual material;
- Contamination of the sample;
- Change in moisture content.

NOTE 1 Some sampling implements may cause degradation of the material and care should be taken to ensure that the properties of the material deemed critical are not affected by the sampling implement.

NOTE 2 Microbiological cross-contamination of samples has been reported when sampling thermally treated sludges from in-line hoppers.

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NOTE 3 Normal handheld shovels should not be used since, unlike sampling scoops, they do not have sides. When a shovel is used sample constancy cannot be guaranteed since sampled material will fall off the shovel after sampling and larger particles will tend to roll off the heap that is formed on a shovel.

### 11.2 Sampling from heaps and stockpiles

#### 11.2.1 General

Samples should be taken from below the surface of the stored material. The media should be free of the bed media, since inclusion of grit or sand will distort measurements.

Sampling during transfer of the material to the stockpile is often to be preferred above sampling from a static stockpile. During transfer probabilistic sampling is often much easier to accomplish than from the stockpile itself. However, in daily practice it is often not possible to sample during transfer and thus sampling will have to be carried out from the static stockpile.

For composted biowaste, where its storage over time may form part of the treatment process, then appropriate samples should be taken from the stored material.

If the stockpile is unconsolidated, it may be difficult or even impossible to reach the selected sampling locations for sampling. If so, the type of sampling equipment should be adapted to this type of sampling situation or the sampling technique should be altered.

#### 11.2.2 Preparation for sampling

The choice of sampling equipment will be determined by the size of the heap or stockpile and the flow characteristics and dryness of the material to be sampled.

The aperture of the sampling equipment should be at least three times as large as the nominal top size of the material being sampled.

#### 11.2.3 Probabilistic sampling

Method 1: Sampling small static volumes

- Push a core sampler through the material at the appointed sampling point and in the identified direction as specified in the Sampling Plan so that the full cross-section is traversed.
- Transfer the sample into the sample container.

Method 2: Sampling large static volumes

- Push a vacuum probe or sampling tube through the material at the appointed sampling point and in the identified direction taking a series of individual samples until the traverse is complete as specified in the Sampling Plan.
- Transfer the directional sample into the sample container.

Method 3: Sampling large static volumes by partial excavation

NOTE To take samples at greater depths, or when the material is unconsolidated, it may be possible to partially excavate a large stockpile. This requires sufficient space for the storage of the excavated material and availability of a suitable excavating machine. The integrity of the sample at the locations defined in the Sampling Plan must be preserved.

- Partially excavate the stockpile using an excavating machine, transferring the excavated material to a suitable clean location.
- Take additional samples from the remainder of the stockpile following the procedure in method 2.

#### 11.2.4 Judgemental sampling

- Push a scoop into the material at the area identified in the Sampling Plan.
- Withdraw the scoop and level off the material so there is none above the sides of the scoop.
- Transfer the sample into a sample container.

### 11.3 Sampling from large bags

#### 11.3.1 Probabilistic sampling

Method 1: Taking a sample through the depth of the bag

- Ignoring material nearer than 50 mm to any surface, push a vacuum probe, core sampler or sampling tube through the material in the direction identified in the Sampling Plan.
- Transfer the directional sample into a sample container.

Method 2: Taking samples when emptying the bag

- Ignoring material nearer than 50 mm to any surface, remove the material from the bag in layers to the depths specified in the Sampling Plan.
- At each depth, take a scoop of material and level off the material so there is none above the sides of the scoop.
- If the samples are to be examined individually, label the increments as specified in the Sampling Plan.
- If the samples are to be examined combined, mix the increments in the proportions specified in the Sampling plan to give a representative sample and label as specified in the Sampling Plan.

#### 11.3.2 Judgemental sampling

- Ignoring material nearer than 50 mm to any surface, push a scoop into the material at the area identified in the Sampling Plan.
- Withdraw the scoop and level off the material so there is none above the sides of the scoop.
- Transfer the sample into a sample container.

### 11.4 Sampling from a falling stream

#### 11.4.1 Preparation for sampling

The following techniques apply only to manual sampling; automatic sampling may exist where the demand is high.

Take samples from as close to point of exit as possible.

#### 11.4.2 Probabilistic sampling

##### 11.4.2.1 Taking a cross-sectional sample with a scoop or sample container

The aperture of the sampling equipment should be at least three times as large as the nominal top size of the material being sampled.

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The sampling action should collect a complete cross-section of the stream; both the leading and trailing edges should clear the stream in the same path.

Method 1: where width and depth of stream is small

- Put a scoop into the stream, at 90 degrees to the falling stream, using a single one directional action.
- Hold the scoop in place for the period specified in the Sampling Plan.
- Remove the scoop in the direction of entry.
- Transfer the sample into a sample container.

Method 2: where width of stream is large and depth is small

- Insert a container at one end of the stream and, at a uniform rate designed to collect the required amount of material, move the container through the width of the stream to the opposite end.

Method 3: where width and depth of stream is large

- Take a first sample following the procedure in method 1.
- Repeat the procedure, taking a second sample at 90 degrees to the first direction of sampling.
- Transfer both samples to the sample container.

### 11.4.2.2 Taking a cross-sectional sample with a receptacle with aperture

The aperture of the receptacle should be larger than the full width of the stream and at least three times as large as the nominal top size of the material being sampled.

- Place the closed receptacle under the falling stream.
- Open the aperture and collect the amount of material as specified in the sample plan.
- Close the aperture and remove the receptacle from the falling stream.
- Transfer the sample to a sample container.

### 11.4.3 Judgemental sampling

The aperture of the container should be at least three times as large as the nominal top size of the bulk material being sampled.

- Put a sample container into the stream to the location identified in the Sampling Plan using a single one directional action.

NOTE The placement of the sampling container may be in the same axis as the falling stream or at 90 degrees to the falling stream. Hold the sample container in place for the period as specified in the Sampling Plan.

- Remove the container in the direction of entry.

## 12 Incorporation in the Sampling Plan

The selected technique(s) for sampling and necessary equipment and preservation techniques should be specified by the Project Manager in the Sampling Plan, see prEN xxxx, prior to commencing sampling.

Check that the outside of all sample containers are wiped clean and labelled as specified in the Sampling Plan.

Follow the sample preservation and handling procedures specified in the Sampling Plan.

Record any deviations from the Sampling Plan.

## Annex A (informative)

### Support on the selection of equipment and apparatus

#### A.1 General

In general, sampling equipment is most practical if it is as simple in design and construction as possible. Tools should be chosen to suit the sampling activity. Permanent equipment may be installed for sampling at fixed points.

All sampling equipment should preserve the characteristics of the material being sampled.

Sampling apparatus and tools should be kept clean and corrosion free.

**NOTE** Old, rusty tools or those with chipped or flaking surface coatings and painted surfaces should not be used, as they may contaminate the samples.

The equipment should be assessed for potential for interference on test results.

**EXAMPLE 1** The use of aluminium extension pipes to a sampling valve would be inappropriate if the samples were being taken for the analysis of an aluminium flocculation assister.

**EXAMPLE 2** High alloy steels should be avoided if trace metals are to be determined.

**EXAMPLE 3** The use of stainless steel tools is routinely adopted, but may be a source of contamination if analyses for elements such as chromium are to be performed.

Equipment to be used to collect samples for microbiological analysis should be suitable for sterilisation.

The size of the sampling tool will vary according to the quantity of sample required and the physical properties of the sample.

**NOTE** For granular samples (e.g. thermally treated sludge or composted biowaste) the size of the sampling tool should be at least three times the maximum particle size.

#### A.2 Equipment selection

Factors to be considered when choosing sampling equipment are:

- suitability for purpose;
- safety in operation;
- ability to take a representative sample;
- capability of preserving sample integrity until it can be transferred to a sample container;
- ability to be cleaned (and sterilised);
- simplicity in use;
- robustness.



- Information should be sought on:
  - a) The characteristics of the material to be sampled:
    - the solids content;
    - the consistency;
    - the physical structure;
    - particle size distribution.
  - b) Accessibility and safety:
    - where and how the material is being stored (e.g. heap, bag, lagoon);
    - the accessibility of sampling points;
    - the hazard assessment of the sampling activities;
    - the safety procedures to be implemented during sampling.
  - c) The size and purpose of the sample:
    - the final quantity of sample required;
    - how the sample will be analysed (i.e. physical, chemical or microbiological analyses).

Suggested applications for generic types of sampling equipment are listed in Table A.1. Detailed descriptions of the equipment are presented in Figures A1-A16 and accompanying text (A.3).

**Table A.1 – Suggested applications for generic types of sampling equipment**

<b>Generic sampling apparatus</b>	<b>Liquid sludge</b>	<b>Sludge cake</b>	<b>Paste-like sludges</b>	<b>Particulate materials</b>
Bailer	+	-	-	-
Dipper	+ (1)	-	-	-
Weighted bottle	+ (2)	-	-	-
Depth sampler	+	-	-	-
Pond sampler	+	-	-	-
Column sampler (liquid)	+	-	-	-
Pump/vacuum probe	+	-	-	+
Sampling tube	+	-	-	+
Tap	+	-	-	+ (5)
Auger	-	+	+	-
Corer	-	+	+	+
Pile sampler	-	+	+	-
Spatula	-	+	+	+ (3)
Scoop	-	+	+	+ (3)
Trowel	-	+	+	+ (3)

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Thief/trier	-	-	+	+(4)
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### Key

+ appropriate  
- not appropriate

### Notes

- (1) For liquid depths < 3.5 m
- (2) Not suitable for viscous liquids
- (3) Not suitable for deep containers
- (4) May be difficult to retain the sample with very dry granular material
- (5) If gravity fed

### A.3 Description of sampling equipment

#### A.3.1 Bailers

Weighted depth samplers, pond samplers and column samplers or other sampling tubes are classified under the generic term 'bailer' for the purposes of this document. Their probabilistic application is liquid sludge sampling.

##### a) Weighted bottle samplers

The sampler consists of a glass or plastic bottle, sinker, stopper and line that is used to lower, raise and open the bottle and is used to sample liquids of free-flowing slurries (Figure A.1). The bottle may either be lowered to a specified depth (e.g. to collect upper, middle and lower samples) or all-level samples may be collected, depending on the time of removal of the cork (by a sharp jerk of the chain) and the speed at which the sampler is withdrawn. The successful operation of a weighted bottle sampler to collect a representative sample does require a considerable degree of skill. The use of weighted bottles is not advisable for particularly viscous liquids. An advantage of this method of sampling is that the sampling bottle may also be used as the sample container.

##### b) Simple weighted sample can

The simple weighted sample (see Figure A.2) can be used for sampling at various depths in all sizes of tanks. It consists of a cylindrical container (of capacity about 500 ml) made of stainless steel with a weighted base in a separate compartment and conical neck.

A wire loop is fitted to shoulders at the top of the device, with a ring at the apex through which a cord is passed and is then attached to a cork fitting the neck of the can.

The empty sampler with the cork inserted is lowered into the liquid to the required depth. The cord is jerked to remove the cork and the can is allowed to fill with product.

##### c) Weighted cage for sample bottle

The weighted cage (see Figure A.3) is designed to contain a standard glass sample bottle (of capacity about 500 ml) and can be used for sampling at varying depths in all types of tanks. It consists of a weighted base to which the attached three vertical straps with a retaining clip at their upper end. Two of the straps are angled and to these is fixed a wire loop with a ring at the apex.

Also attached to these straps is a wire hoop which is secured to the third strap to retain the bottle in the cage. A cord passes through the ring of the wire loop and is attached to a cork fitting the neck of the bottle.

The sampler is operated in the same way as the weighted can.

##### d) Valve sampling cylinder (sinker sampler)

The valve sampling cylinder (see Figure A.4) consists of an open headed cylinder with a bottom valve. This valve remains open, owing to the pressure of the liquid in the valve whilst the instrument is being lowered through the liquid. This ensures that an even flow of liquid passes through the cylinder. When lowering ceases, the valve closes and a sample of liquid is drawn from the depth reached by the instrument.

Some samplers of this type and function incorporate a light flap valve at the head, which closes off the cylinder when the filled sampler is raised.

##### e) Bottom sampler

- With spring loaded valve

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This bottom sampler (see Figure A.5a) is constructed of stainless steel and comprises a cylindrical body (capacity about 500 ml) with screw-on base and top. The base incorporates a disc valve to permit entry of the liquid into the bottom of the sampler. The top also incorporates a disc valve to permit release of air from the sampler.

Attached to the screw-on top is a fixed hoop, which serves to suspend the sampler from a cord and provides a guide and spring retainer for the central valve spindle.

The valve spindle projects below the bottom of the sampler and, when this grounds on the tank bottom, the spindle is pushed up into the cylinder against the light spring, opening first the valve in the base, followed after a short delay by that at the top. This is made possible by the small gap in the sleeve at the upper part of the cylinder. The purpose of this short delay between opening of the inlet and the outlet valves is to ensure that the liquid first enters through the base. This causes a slight increase in pressure inside the vessel which prevents liquid entering at the top, when the upper valve opens.

Buoyancy may be overcome by adding weights in the form of stainless steel annular rings which are slipped over the body of the sampler and held in place by the screwed base.

### – With deadweight valve

This bottom sampler (see Figure A.5b) is basically similar to the bottom sampler with the spring-loaded valve in design and operation, except that the lower valve is kept closed by deadweight and the release of air is through a reduced section of the valve spindle at its upper end.

### f) Column sampler

This is one of the most important samplers used for containerised materials (Figure A.6).

The main parts of the equipment consist of a hollow PVC tube and concentric PVC rod attached to a neoprene stopper, which form the sampling tube, closure locking mechanism and closure system. The sampler is lowered into the liquid to cut across a column of liquid. Used correctly, the sampling rate must be fairly slow, with the equipment being lowered sufficiently slowly for the liquid height inside and outside the sampling tube to remain approximately equal. The sampler is pushed against the bottom of the container to close the sampler and locked by turning the T-handle. The sampler is withdrawn, wiped with a disposable cloth and discharged into the sample bottle by turning the T-handle to 'open'. Column samplers are manufactured from either plastic (usually PVC) or glass.

### g) 'Slick stick'<sup>TM</sup>

A similar device to the column sampler is the 'Slick Stick'<sup>TM</sup>. It consists of a 1 m length of clear uPVC tube (through which the liquid may be viewed) with an automatic valve assembly and removable strainer at the base. The 'Slick Stick'<sup>TM</sup> is usually used to collect a composite sample of stratified liquids (e.g. floating oils) from different depths.

### h) Tube sampler

The tube sampling procedure is applicable for sampling liquids in drums and cans. The tube consists generally of glass, PTFE or stainless steel. Different sized tubes (500 and 1000 ml) are used to sample drums of different capacities. The technique is crude, but effective; the tube is lowered into the liquid, sealed at the top with a (gloved) thumb and discharged into a sampling device by removing the thumb. Two rings can be attached to opposite sides of the tubes at the upper end for two fingers to slip through, leaving the thumb free to close the opening (Figure A.7 and A.8).

By leaving the upper end open at various levels in the tank, bottom, middle, upper or all-level samples may be taken.

### i) Pond sampler

The pond or dipper sampler consists of a glass, plastic or other non-reactive beaker clamped to the end of a 2 or 3 piece telescopic aluminium or fibreglass pole (Figure A.9). Liquids and free-flowing slurries can be sampled up to 4 metres from the bank. The sampler is inserted into the liquid upside down and inverted at the required sampling depth. When a discharge stream is sampled, the dipper should be passed through the stream at such a rate that it is filled in one pass, making sufficient passes to cover the entire cross-sectional area of the stream. The use of this type of sampler is not advisable when the total depth of the liquid is greater than around 4 metres.

### A.3.2 Pumps

A range of pumps are available for sampling liquids. Vacuum pumps are less suitable for sampling volatile or highly volatile material than other types as the vapour formed reduces the vacuum so the pump no longer works effectively.

#### a) Vacuum sampling device for sampling thick sludge from open vessels

For thick sludges (such as primary sludges), the vacuum sampler illustrated in Figure A.14 has been used successfully in circumstances such as storage tanks that have not been fitted with pipework for stratified sampling. Sample uptake pipes can be set to sample at predetermined depths from the top of the tank. Construction consists of a pipe of 25 mm bore, electrically earthed to the tank, in 2 m sections joined by screw connections which do not reduce the bore, up to a maximum of five sections. This is connected via a flexible pipe and valve to a 10 l bottle or Buchner flask which is surrounded by a guard to prevent injury should it collapse; it may be evacuated wither by hand or by and electrically operated vacuum pump fitted with an intrinsically spark-free motor.

It is necessary to obtain a good vacuum in the bottle before opening the valve to the sampling line. Before taking a sample, some sludge should be withdrawn into another clean 10 l bottle to flush out the pipe. A purge equivalent to three times the standing volume of the sampling arm is recommended. This method is particularly suitable for sampling from digesters, either through a port in the roof or through a sludge seal in floating head types. It is important to remove encrusted sludge from the sampling point before inserting the aluminium pipe. In order to ensure that the sample of sludge is representative, the location of the uptake tube will need careful consideration.

NOTE 1 In situations in which a very viscous layer develops in a stratified sludge, such equipment may draw up less viscous material and overlying rainwater, thus generating an unrepresentative sample.

NOTE 2 The equipment has been demonstrated to be suitable for sampling sludges with dry solids contents greater than 6% to 8% mass fraction.

### A.3.3 Valves

#### a) Apparatus for sampling from pipes under pressure

The recommended valve arrangement (Figure A.15) should be connected to a system of gauges capable of measuring and equating the pressurized pipe to which it is connected. The device acts as a pressure lock to allow a controlled decompression into the sample vessel. This operation is an aid to the safe handling of the sample and considerably reduces the effects of shear on the sludge. The following steps outline the operating procedure:

- Connect the apparatus to the high-pressure line at point A with all valves closed.
- Open valve D and admit compressed air until the pressure in the apparatus is equal to the operating pressure of the filter press or pipe in question.
- Close valve D and open valve B.
- Slightly open valve E to allow air to escape and sludge to be sampled through the open valve B.

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- When sludge appears at the outlet valve E the sampling compartment is full of sludge. Close valve E.
- Close valve B and open valve E to reduce the pressure to atmospheric.
- Open valve C and withdraw the sludge sample.

In order to compensate for the dead volume of sludge in the pressure sampling line A, the above procedure should be repeated to provide a purge equivalent to three times the volume of the pipe. This ensures that new sludge is drawn off as the sample.

### A.3.4 Augers

#### a) Auger

Soils, sand, packed powders, granular material and pierceable solids can all be sampled with a soil auger. However, the equipment may not be useful for non-cohesive solids (i.e. sandy soils etc.).

An auger consists of a hard metal central shaft with sharpened spiral blades which discharge cuttings upwards as the shaft is rotated down through the material. A disturbed sample is obtained (i.e. it is not possible to distinguish layered material taken between the surface and the base of the profile), removed from the catch-pan and bottled (Figure A.10).

#### b) Drill

This equipment is used for drilling hard and tough materials.

#### c) Sampling tubes

The sampling tube is a stainless steel instrument consisting of two concentric tubes closely fitted into each other throughout their entire length, so that one tube can be rotated with the other. Longitudinal openings are cut in each tube. In one position the tube is open and admits the sample and by turning the inner tube it becomes a sealed container (Figure A.11).

The inner tube is 20 mm to 40 mm in diameter and undivided in its length. The two tubes are provided with holes to be aligned when emptying, so placed that the sample contained in the instrument can be drained through them when the longitudinal openings are closed.

The sampling tube type may be made of glass, although extreme care should be taken to avoid breakages. More usually the equipment is of stainless steel, aluminium or PTFE. It is inserted either closed by a gloved finger at the top or open, as desired. It is then closed by the finger and withdrawn.

The sampling tube may be used for taking samples at various levels from drums by keeping top closed until the required sampling depth is reached.

Volatile material quickly boils under vacuum; it is therefore advisable to use a tube with a bottom seal. The ball valve pump is highly suitable. However, it cannot be used for viscous or aggressive liquids.

### A.3.5 Scoops

Dry granular materials in bins or other shallow containers or on conveyor belts may be sampled using a laboratory scoop or shovel. A polypropylene scoop is preferable, being resistant to corrosion and chemical reaction and is disposable. Figure A.12 shows the design of the scoop, and Table A.1 gives the dimensions of the scoop for particles of different sizes.

NOTE From the viewpoint of bias, scoops with sides are preferable to shovels in that large particles tend to roll off the heap which is formed on a shovel.

### **A.3.6 Thief**

The sampling thief or grain sampler consists of two concentric tubes of stainless steel or brass, one fitting closely inside the other (Figure A.13a). The outer tube has a conical pointed tip to facilitate sample penetration; the inner tube is rotated to open/close the sampler. Samples of dry granular and powdered material can be collected where the particle diameter is less than one-third the width of the slots. The closed sampler is inserted into the material from a point near a top edge or corner, through the centre to a point diagonally opposite the point of entry. The inner tube is rotated to open the sampler and shaken to allow material to enter the open hatches. The sampler is then closed and withdrawn, placed in a horizontal position (slot upward), the inner tube removed and its contents transferred to the sample container.

### **A.3.7 Trier**

A trier consists of a tube cut in half lengthways, with a sharpened tip. It is used to collect moist or sticky solids with a particle diameter less than half the trier diameter. They may also be used for soft or loose consistency samples up to a depth of 60 cm. Tiers of lengths varying between 60 - 100 cm and 127 - 254 cm are available at laboratory supply stores (Figure A.13b).

### **A.3.8 Pile sampler**

Where samples are in large heaps with cross-sectional diameters of 1 m or more, a much larger trier (pile sampler) may be used.

It is commercially available but easily fabricated from PVC pipe. A 1.5 m length of pipe (e.g. ID 300 mm, wall thickness 0.3 mm) is sawn lengthways (with a 60/40 split) to the last 100 mm. The narrow piece is removed to leave a slot in the pipe, the slot edges are sharpened to allow the sampler to cut into the material and the uncut end is used as a handle, this sampler can also be used for material in bins or trucks where a normal trier is not long enough. Samples are likely to be suspect where particle diameters are greater than half the trier diameter (Figure A.13c).

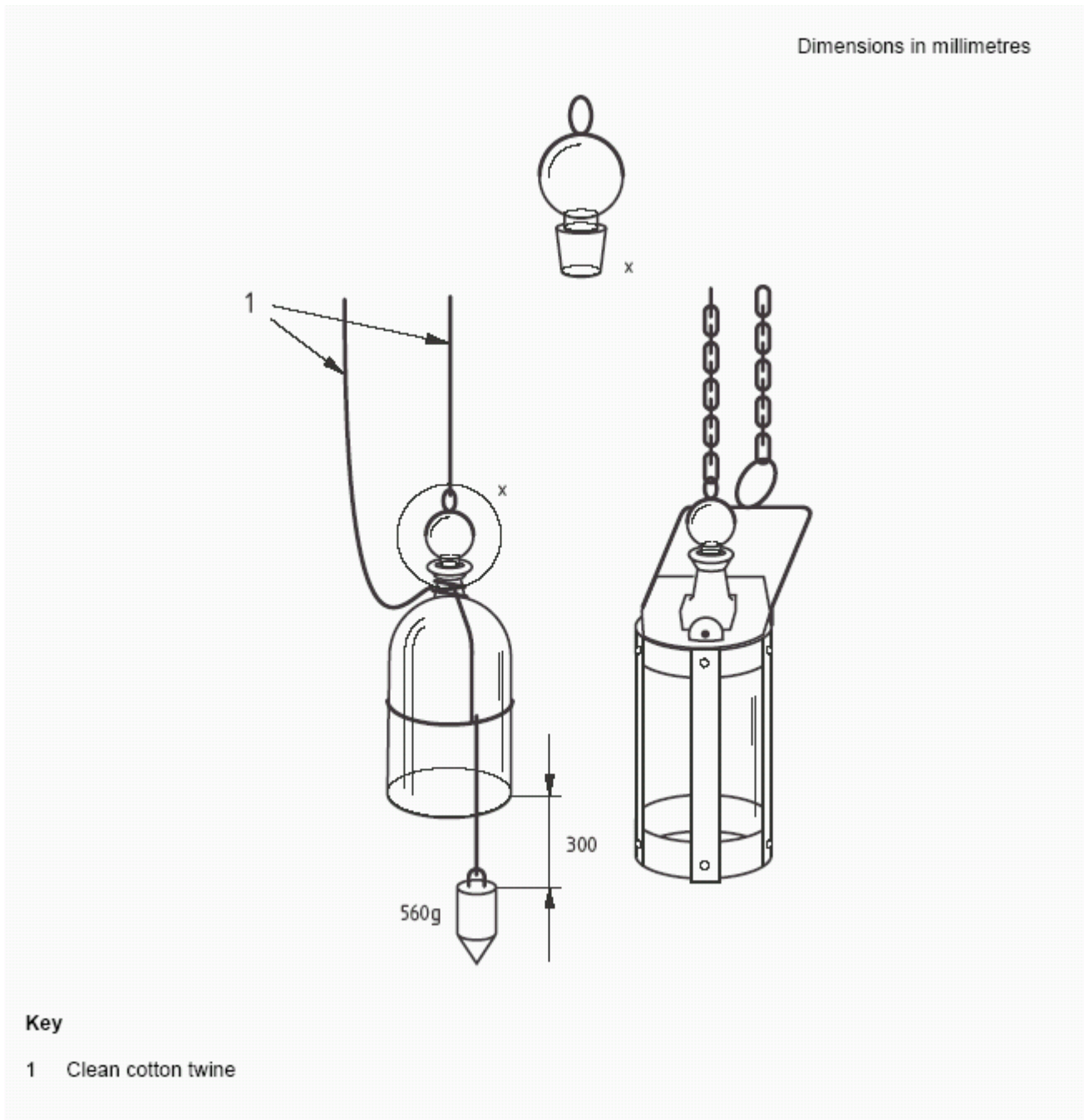


Figure A.1 - Weighted bottle sampler for liquids (after ASTM E300-70, 1973)



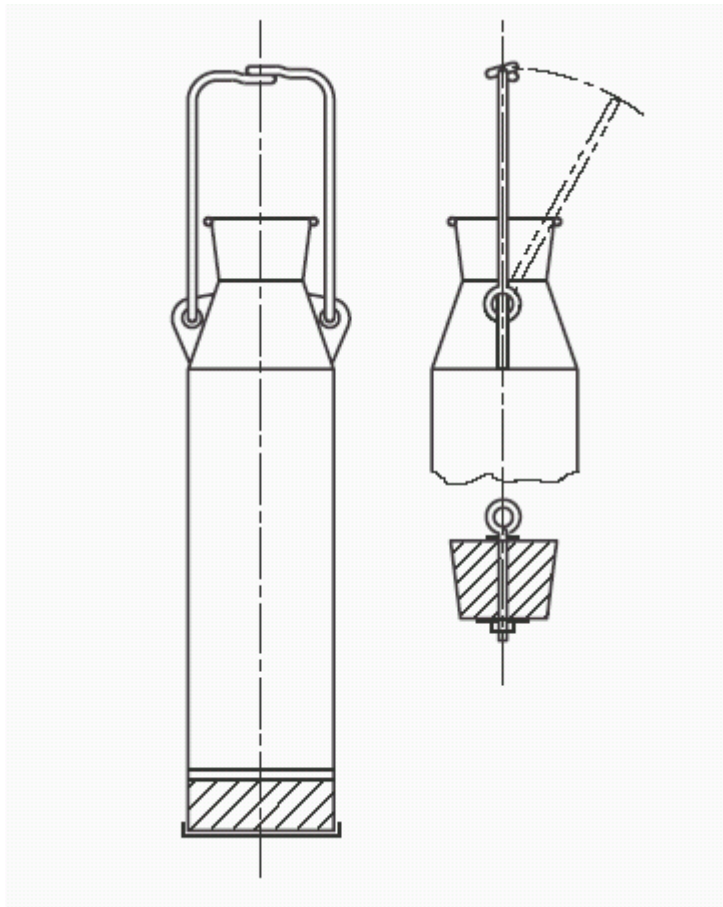


Figure A.2 - Simple weighted sample can [ISO 5555]

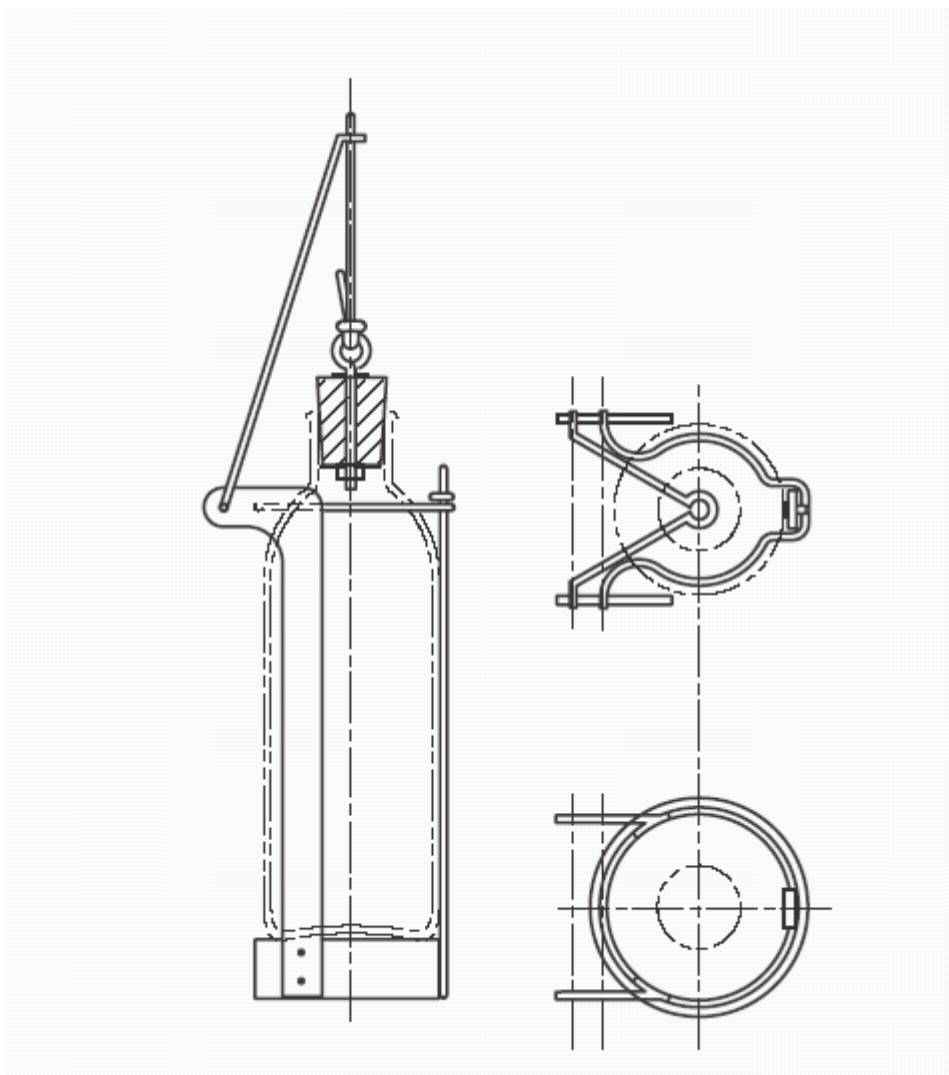


Figure A.3 - Weighted cage for sample bottle [ISO 5555]

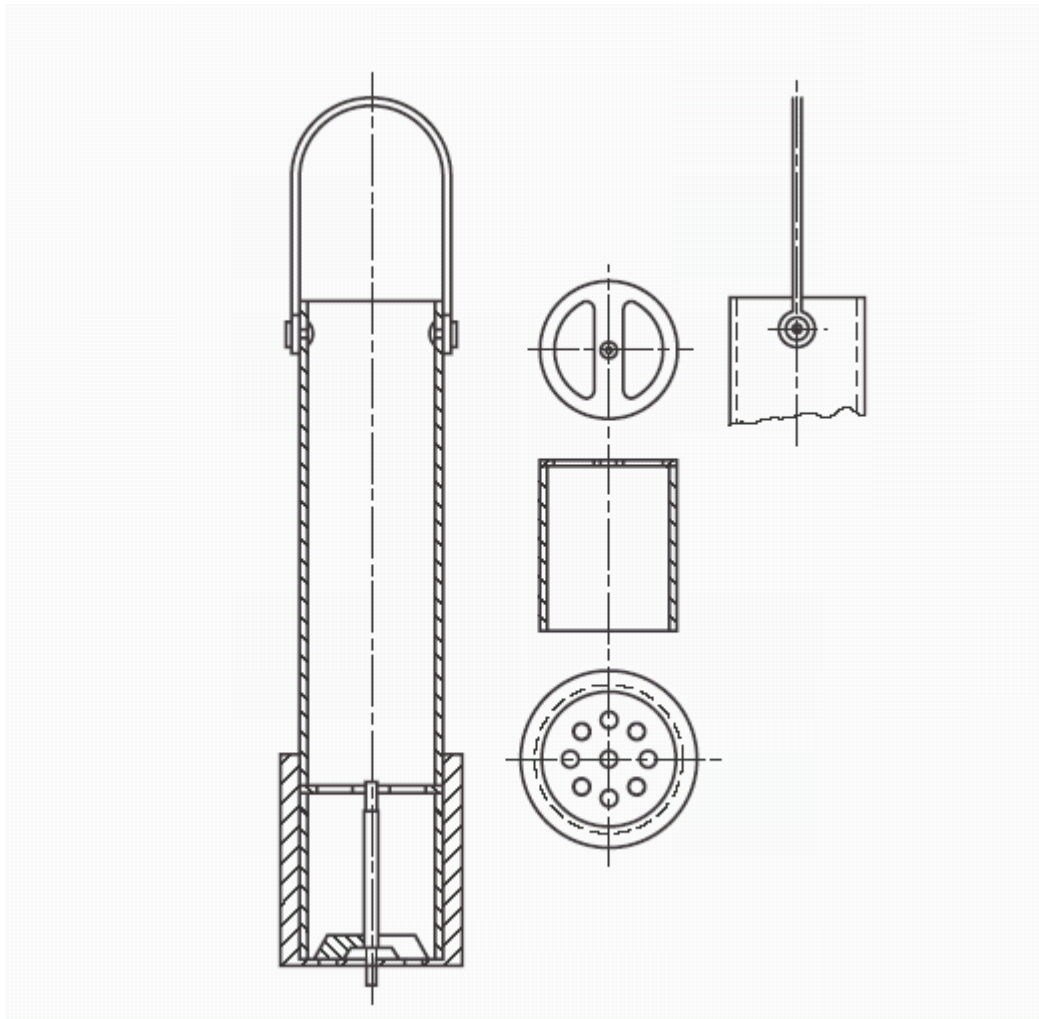


Figure A.4 - Valve opening cylinder (sinker sampler) [ISO 5555]

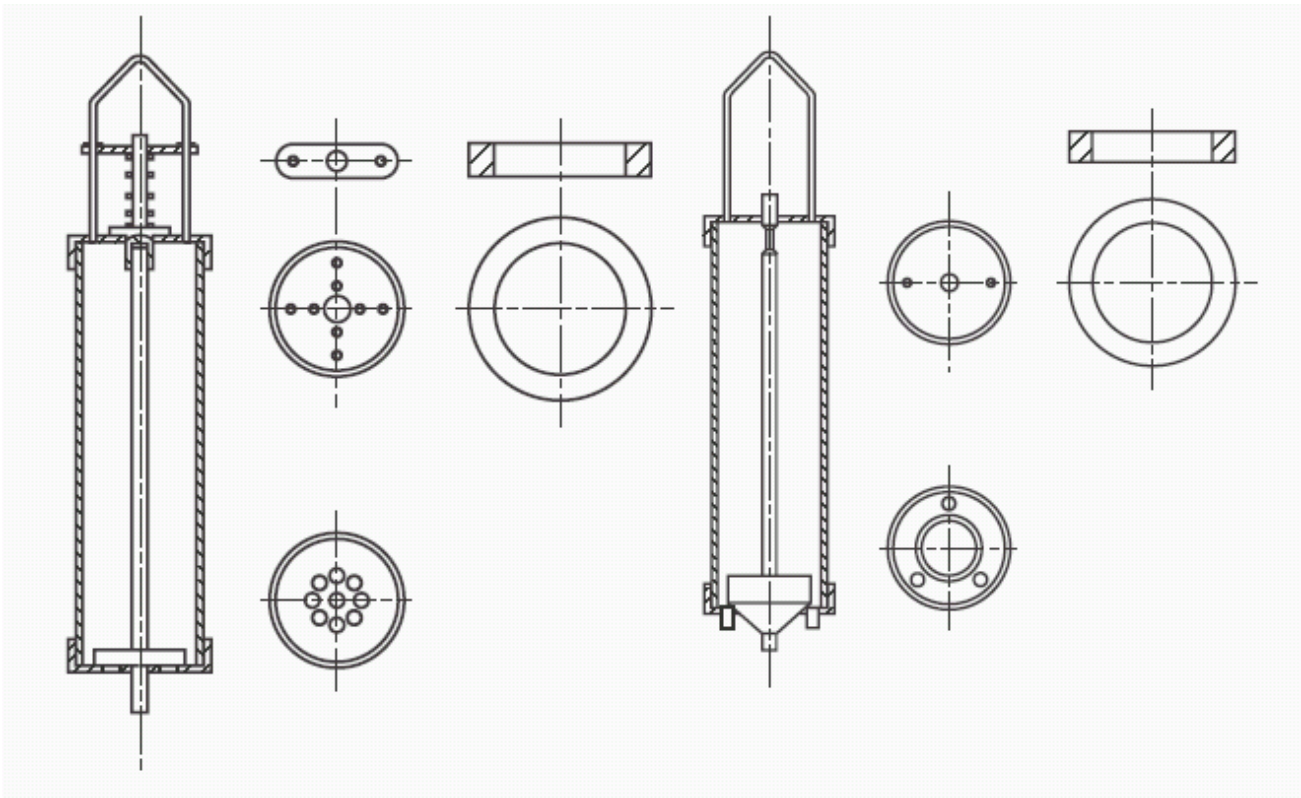


Figure A.5 - Bottom sampler [ISO 5555]

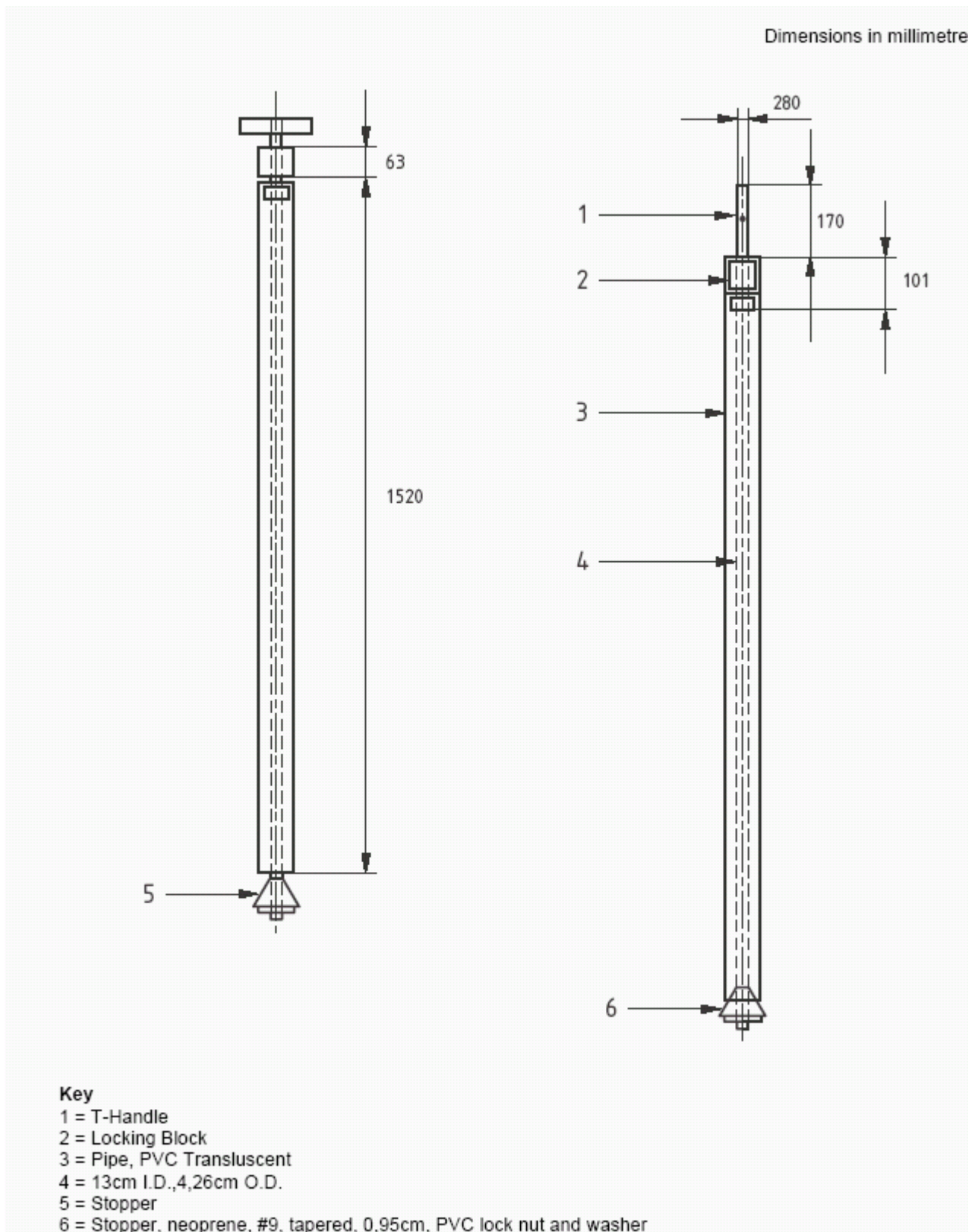


Figure A.6 - Column sampler (after EPA 1994)

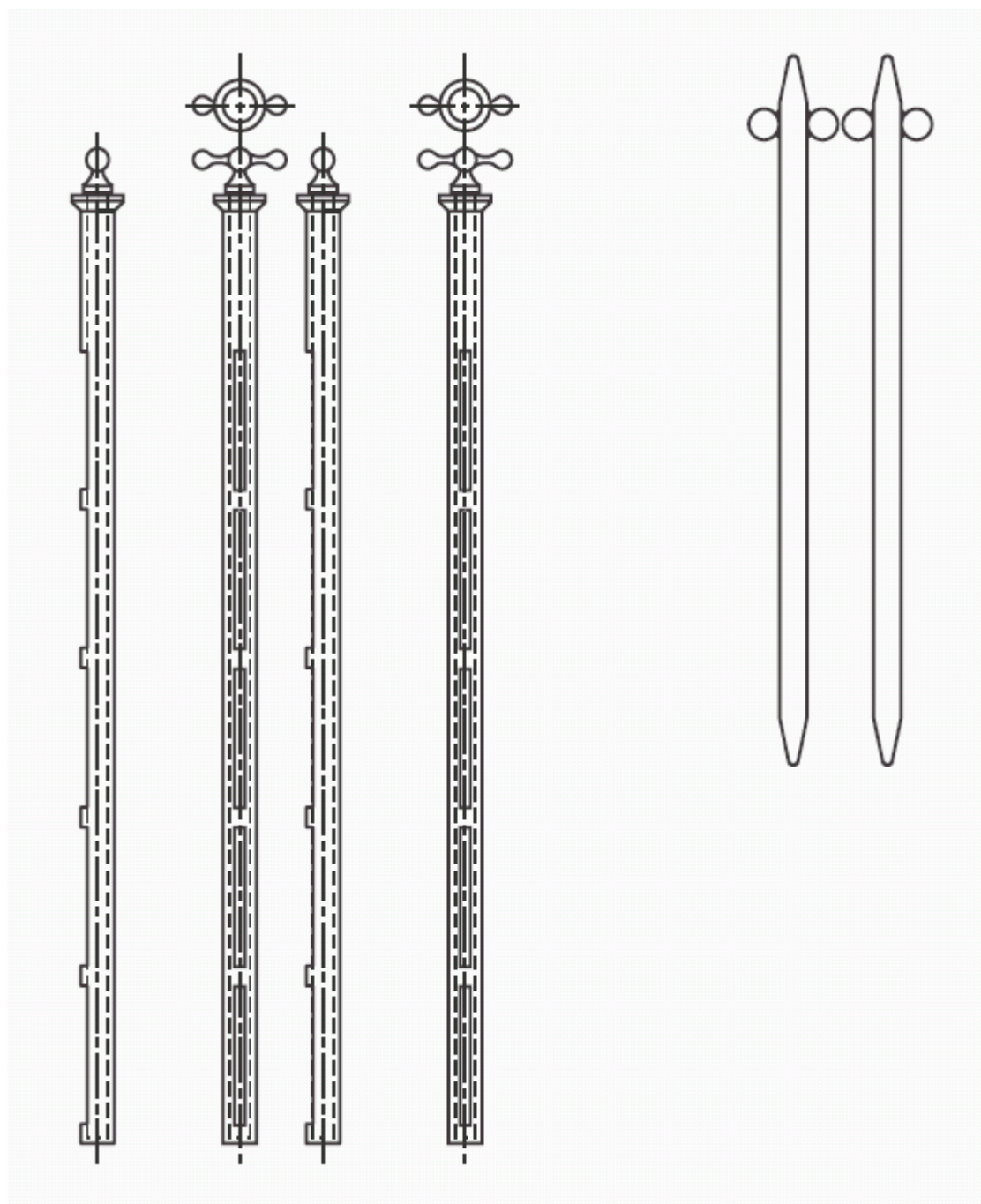
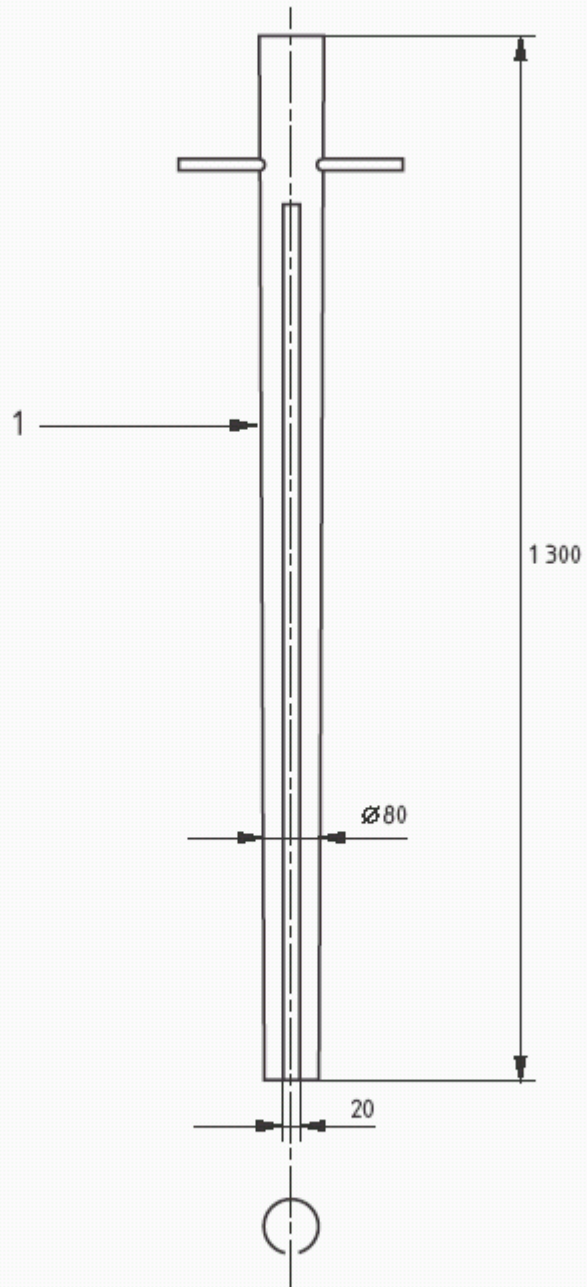


Figure A.7 - Sampling tubes [ISO 5555]

Dimensions in millimetre:



**Key**

- 1 Tube (glass, PTFE or stainless steel)

**Figure A.8 - Probe: slotted tube sampler [NVN 5860]**

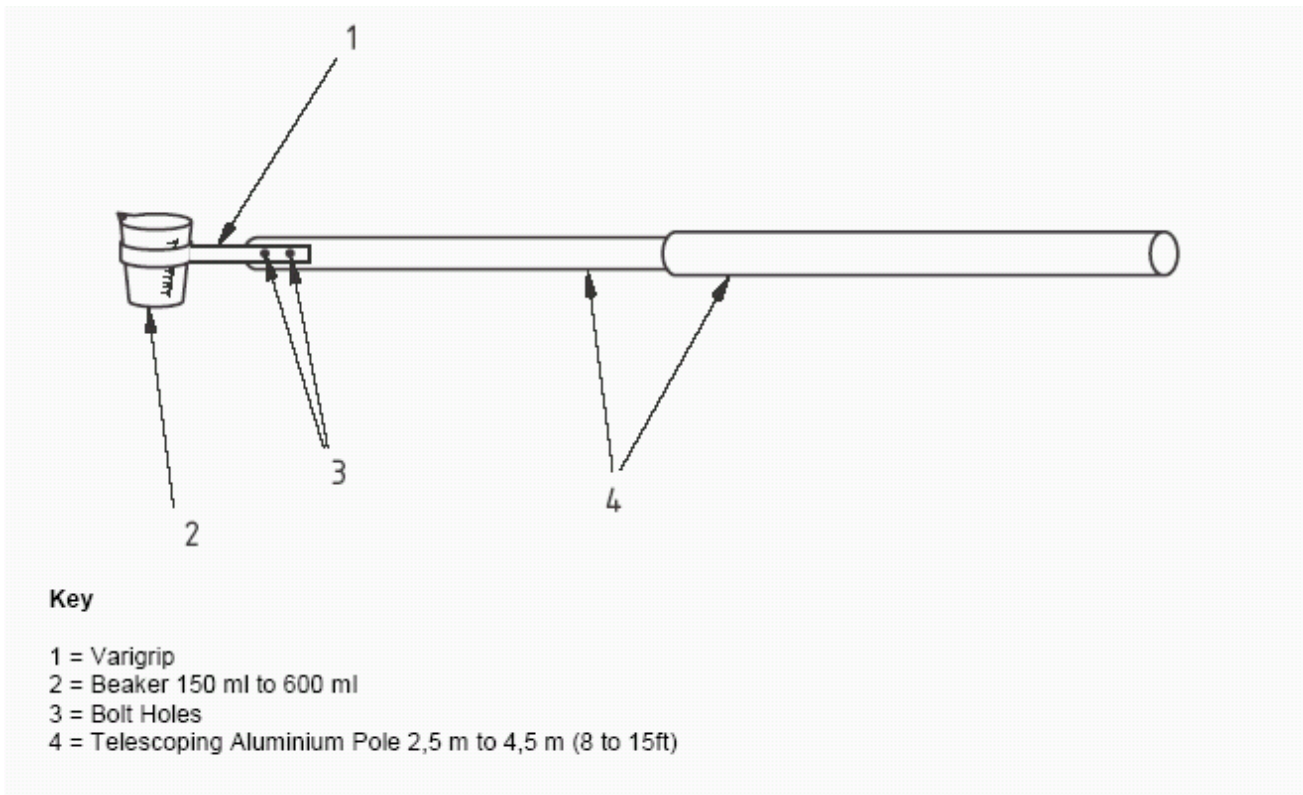


Figure A.9 - Construction of a pond dipper/sampler for liquid sludges (after EPA 1994)



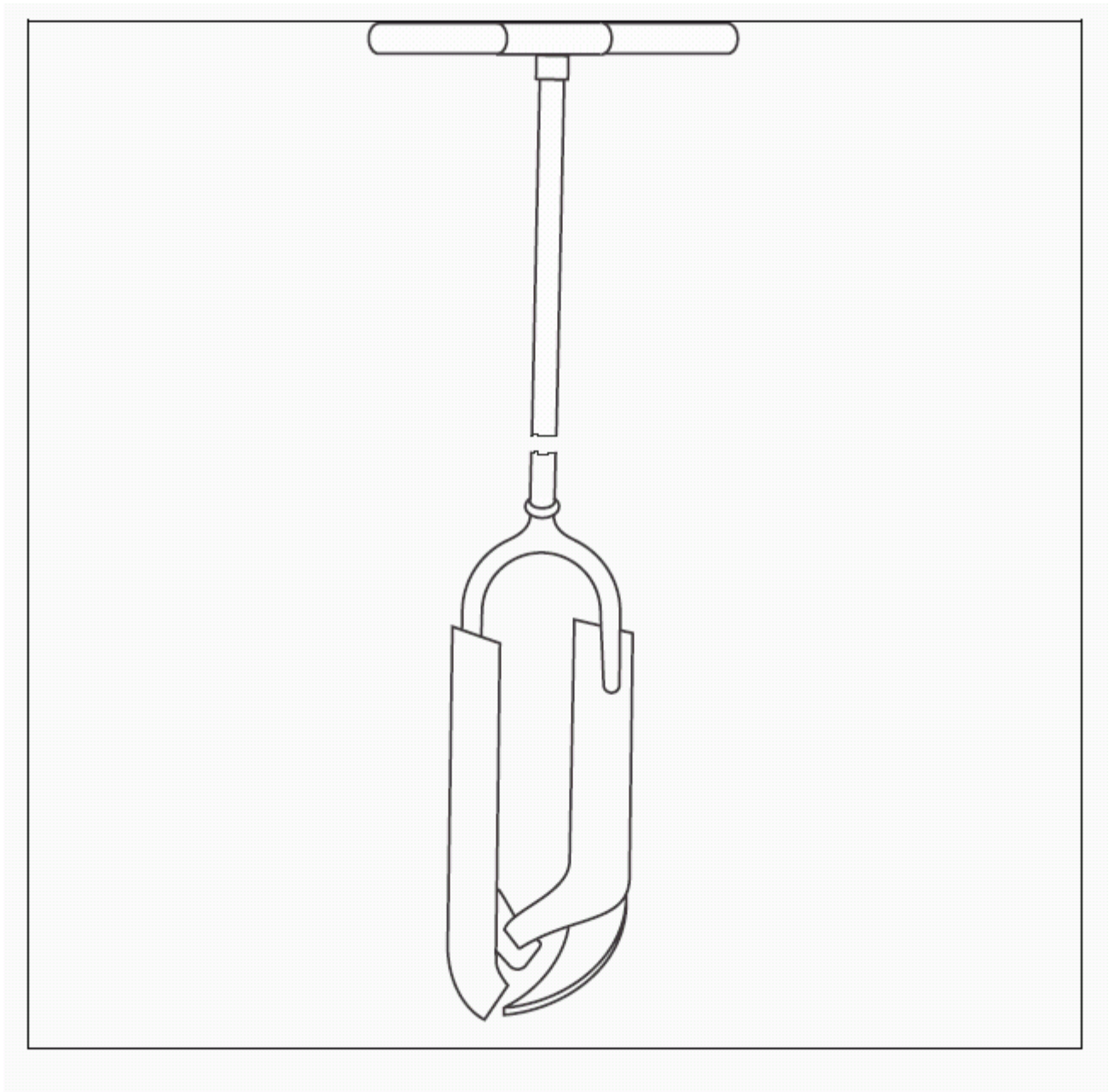


Figure A.10 - Soil auger [NVN 5860]

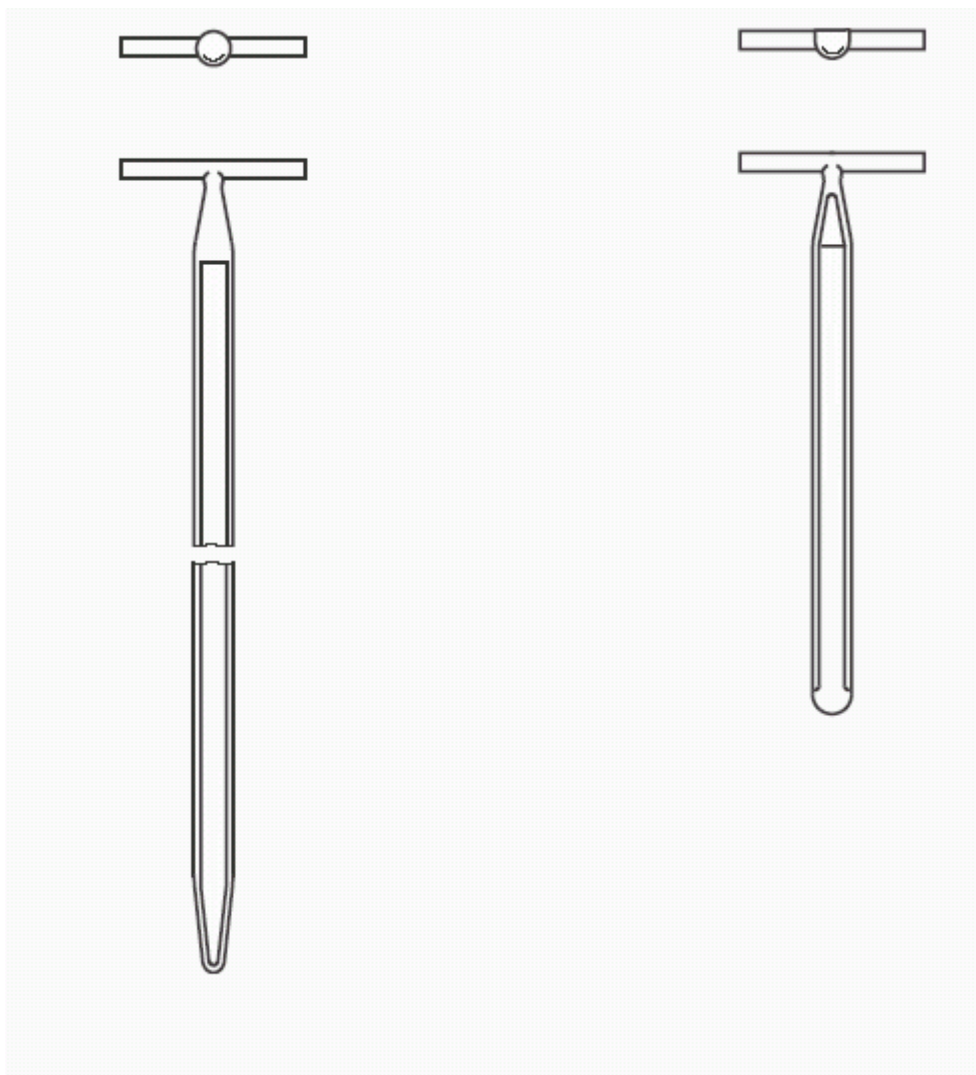


Figure A.11 - Sampling tubes [ISO 5555]

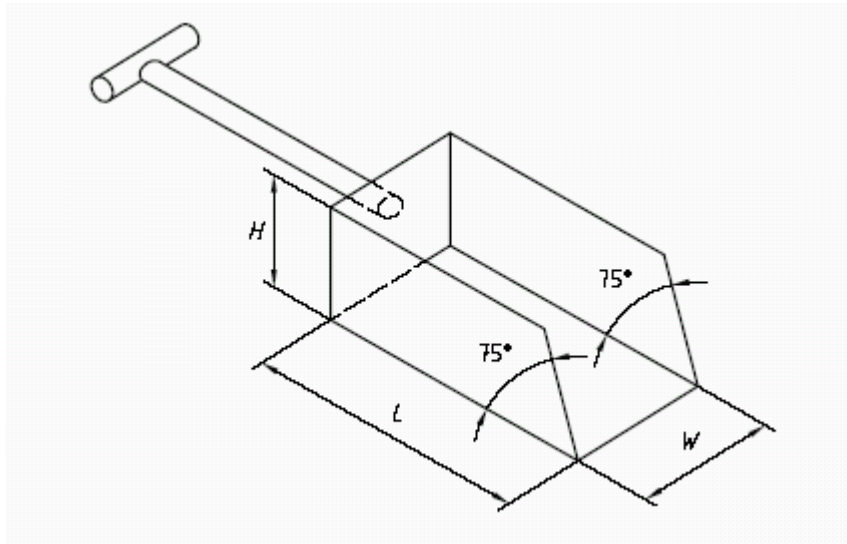


Figure A.12 - Scoops [ISO 11648-2:2001]

Table A.1 — Recommended sizes of scoops [ISO 11648-2:2001]

Nominal top size of material  mm	Recommended dimensions  mm		
	<i>L</i>	<i>W</i>	<i>H</i>
11.2	75	35	30
16.0	110	50	40
22.4	170	70	50
31.5	220	95	80
45.0	300	135	120

NOTE Scoops are not suitable for sampling bulk material with a nominal top size larger than about 45 mm.

Dimensions in millimetres

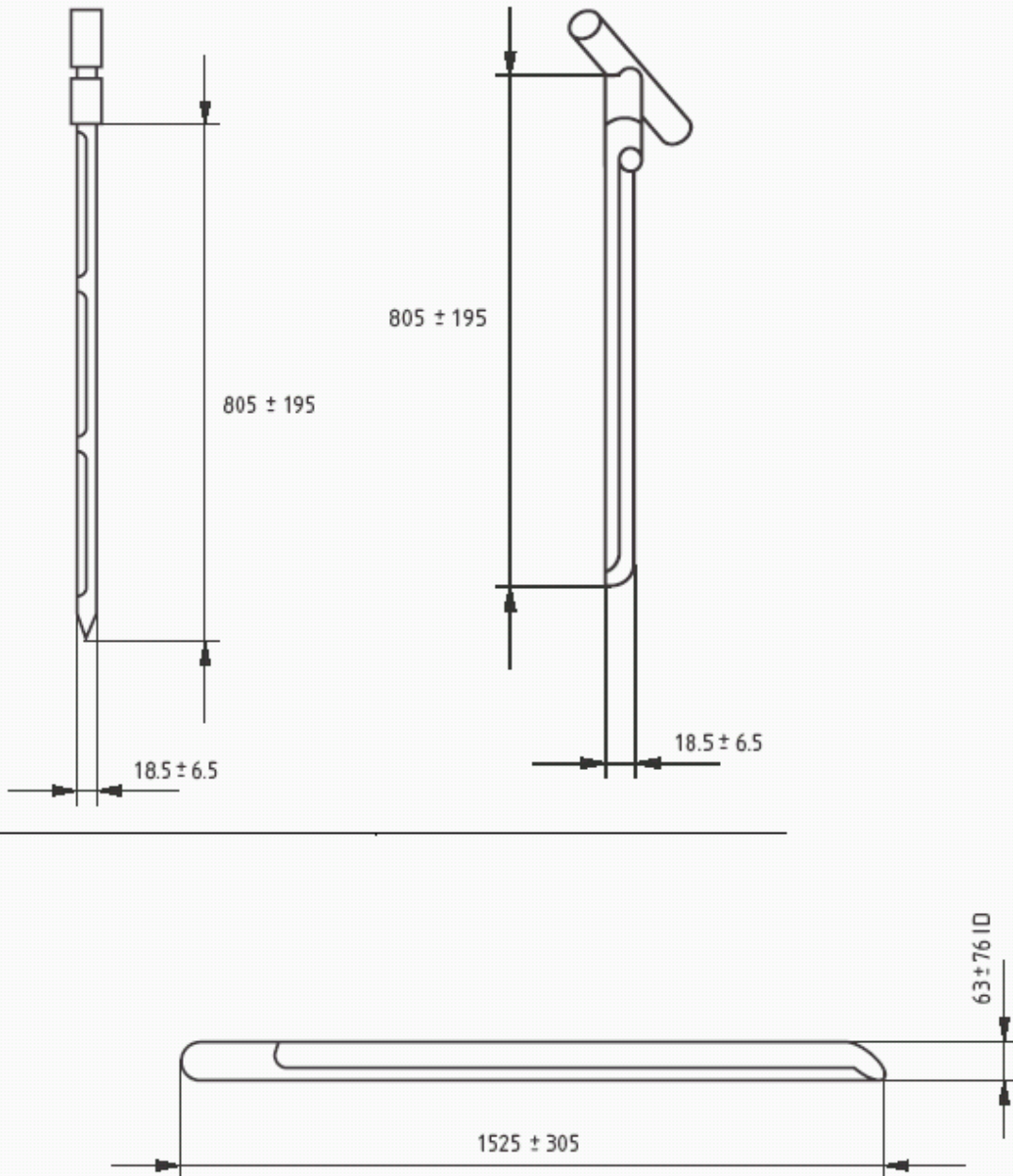


Figure A.13 - Samplers for solid materials a) thief, b) trier and c) pile sampler (after EPA 1980)

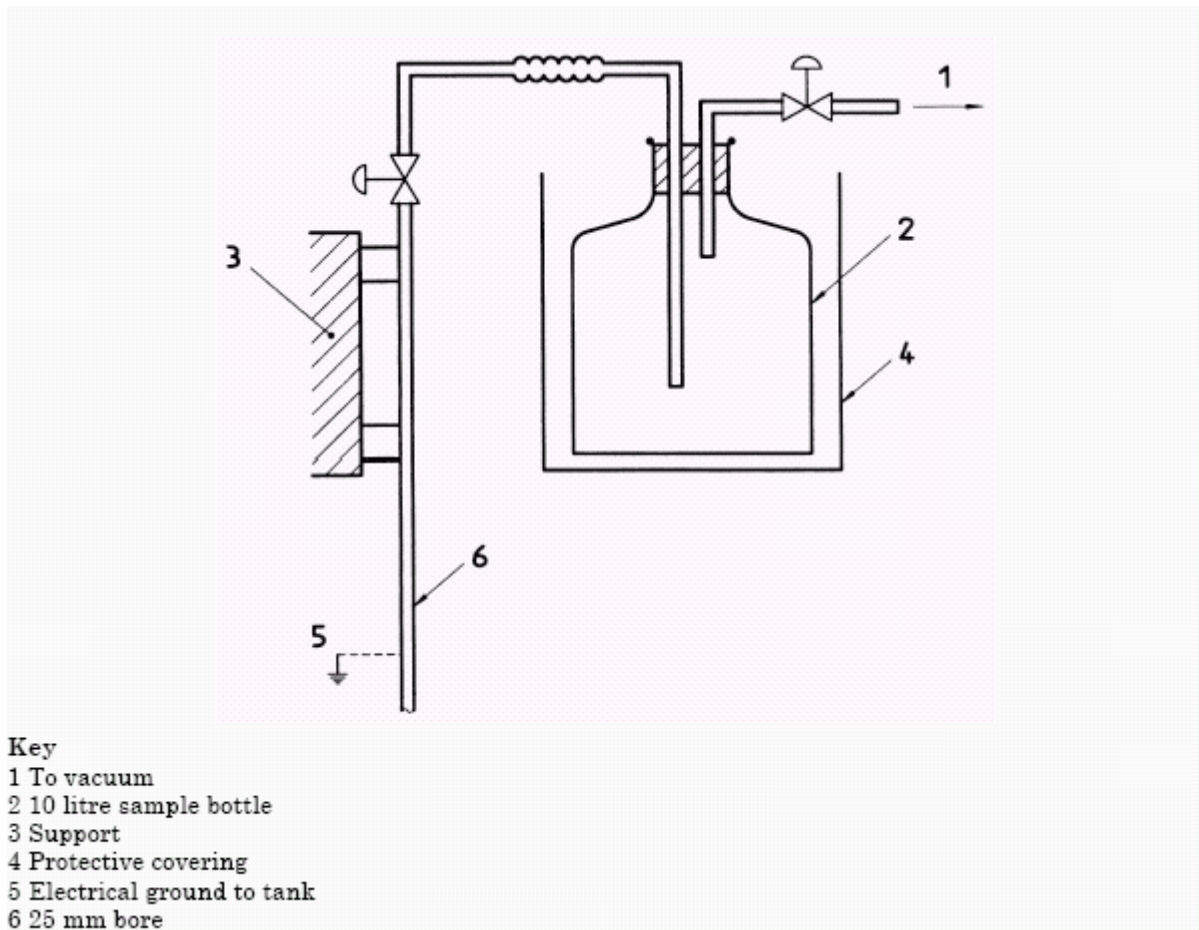
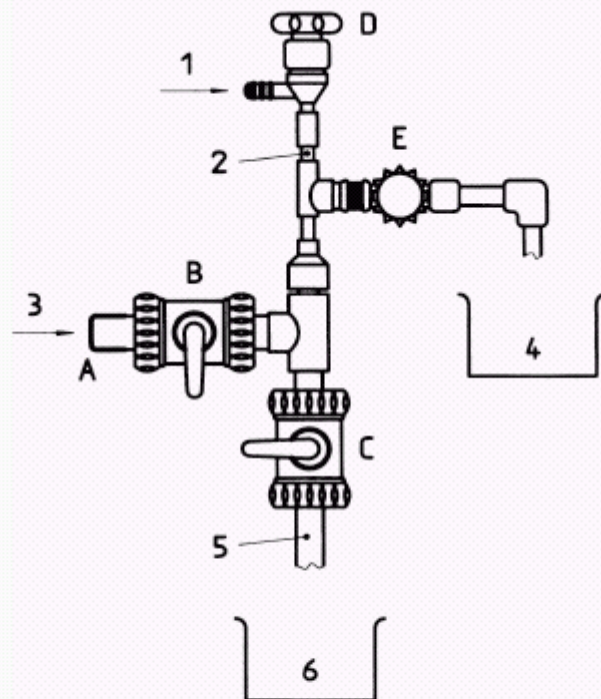


Figure A.14 – Apparatus for the sampling of thick liquid sludge under vacuum [EN ISO 5667-13]



- Key**
- 1 Compressed air
  - 2 PVC pipe 15 mm o.d.
  - 3 High pressure sludge line
  - 4 Container
  - 5 PVC pipe 30 mm o.d.
  - 6 Sample collecting container

Figure A.15 – Recommended valve arrangement for sampling liquid sludge under pressure [EN ISO 5667-13]

**Annex B**  
(informative)

**Validation of method**

**Annex C**  
(informative)

**The modular horizontal system**



**Annex D**  
(informative)

**Information on WP 2 Sampling and the project HORIZONTAL**

## Bibliography

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