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

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Foreword

This Technical Report (CSS99060) 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 wastes

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

CSS99031: 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 attempts to clarify the ‘grey area’ between the description of the overall testing objectives, which are often relatively abstract, and development of the practical sampling plan as unambiguous technical instructions that will provide data to meet those objectives. It specifically provides guidance on the policy aspects that might be relevant for defining the objective of the testing programme and how this will determine the technical methods that can be used to prepare the sampling plan.

Annex A is informative.

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.

This Technical Report complements prEN xxxx, providing guidance on the process of defining a sampling plan within the framework of an overall testing programme.

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.

In the process of defining the sampling plan (Key Step 1 in Figure 1 of prEN XXXX:date), the objective of the testing programme can be translated into specific technical instructions. Using these instructions the type and number of samples that are needed to meet the objective of the testing programme can be taken, ultimately providing the information needed on the material under investigation.

The process of defining the sampling plan, which takes into account both policy and technical requirements to produce technical instructions, is therefore a fundamental step in sampling.

In practice, problems might arise when translating the objective of the testing programme which can be relatively abstract into technical instructions that correspond with that same objective. There is a disparity between defining the need to evaluate the material and specifying the technical methods that should be applied to make an adequate evaluation possible. This Technical Report aims to provide the connection between the objective of the testing programme in policy terms, and that same objective translated into technical terms for sampling.

Annex A provides worked examples of sampling plans for a number of materials and situations in which these materials might be sampled. A number of assumptions have been made to produce each individual example, and therefore – although the examples might represent daily practice – they are case specific and are not necessarily directly applicable to other similar generic situations.

This Technical Report is written for those involved, directly or indirectly, in making policy decisions that are based on the technical information gathered through sampling and for sampling specialists.

In this Technical Report each step of the process of defining the sampling plan is illustrated with examples to clarify the text in more practical terms.

Examples of materials to be tested

Example 1: Sludge and treated biowaste

A waste water treatment works continuously produces treated sewage sludge cake which is put into stockpiles before being transported to agricultural sites for land application.

A works treats biowaste in batches using in-vessel composting; the treated biowaste is stored in piles before being supplied for land application.

Land application is permitted only if the concentrations of a number of key constituents conform to limits set out in legislation. Therefore it is essential that the treated sewage sludge and treated biowaste are sampled.

Land application of sludge and treated biowaste might also limited by nutrient content, e.g. nitrogen and phosphorus. Therefore the sludge or treated biowaste is sampled and tested to establish the nutrient content.

Example 2: Soils in the landscape

It is proposed that treated sewage sludge be applied to land that has not received sewage sludge before. Sludge application is permitted only if the concentrations in the soil of a number of key constituents conform to limits set out in legislation. Sludge application might also be limited by the nutrient content of the soil and sludge. Therefore the soil needs to be sampled to establish baseline concentrations of the key constituents set out in legislation and nutrients.

An area of agricultural land has received several applications of sewage sludge. Further applications of sewage sludge are only permitted if the concentrations in the soil of a number of key constituents still conform to limits set out in legislation. Therefore the soil needs to be sampled for monitoring purposes.

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

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

1 Scope

This Technical Report provides guidance on the process of defining of a sampling plan based on the objective of the testing programme. It specifically deals with the strategic decisions that are needed, based on the sampling objective.

NOTE The document provides considerable detail on current best practice, but is not exhaustive.

2 Normative references

The following documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

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

3 Terms and definitions

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

3.1
biowaste
waste that is derived from a biological source material

NOTE Such as food and garden waste, and paper and paperboard. (See Annex A.7, Council Directive 1999/31/EC Article 2.m.)

3.2
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.

4 The process of defining the sampling plan

4.1 General description of the process

When defining the sampling plan those parties that have an interest in the results of the sampling should be identified and their participation secured.

These parties may represent various backgrounds and might have different interests. However, they need to reach agreement on the objective of the testing programme, the translation of this objective into realistic technical goals and the translation of these technical goals into unambiguous instructions for sampling. These instructions should be recorded in the sampling plan.

The objective of the testing programme determines the desired level of information (e.g. basic characterization or compliance testing) and the desired reliability of the sampling results.

Technical goals include statistical terms such as the characteristic to be determined, the population, the scale, the confidence level and confidence interval to be reached and technical terms such as the constituents that are to be determined and the moment when, or location where, the material will be sampled. Part of these technical goals provide direct input for the sampling plan, while others still need to be translated into practical terms.

Commonly, the reliability of the results improves when the number of samples is increased. This invariably leads to higher sampling and analysis costs. The number of samples and / or degree of reliability might be a legislative requirement. Where this is not the case, the heterogeneous character of the material to be sampled necessitates balancing the desired reliability with the financial input. Balancing the reliability and costs might be the most important decision the involved parties have to make in the process of defining a sampling plan.

The sampling plan should be discussed with and agreed by all involved parties. By doing so, the practical implications of the choices that need to be made in the process of defining and translating the objectives should become clear. For practical reasons, unrealistic objectives might need to be modified.

The process of defining the sampling plan might need to be repeated several times before final agreement is reached. The process of defining the sampling plan is provided in Figure 1.

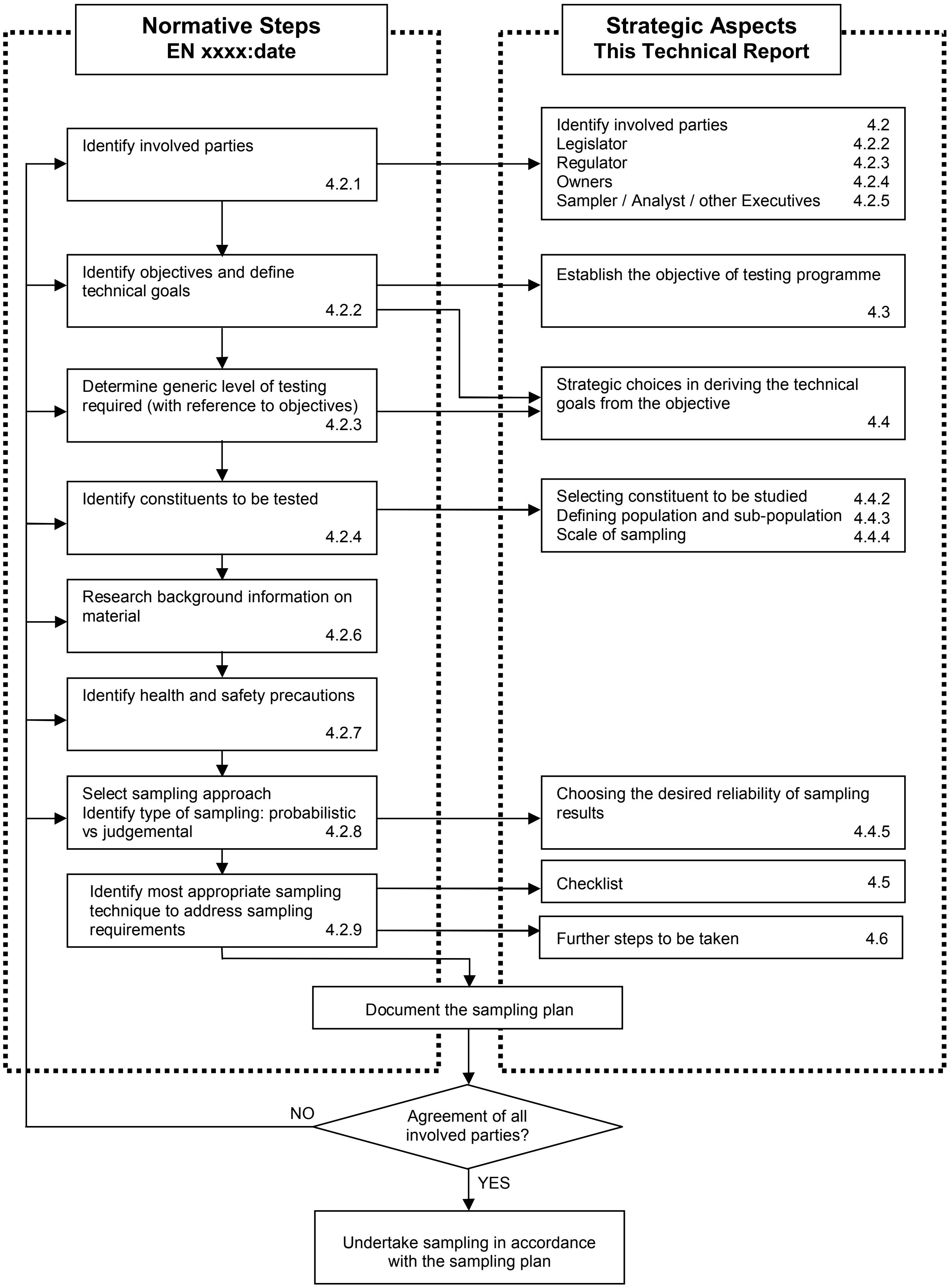

Figure 1 — The process of defining the sampling plan

4.2 Identification of involved parties

4.2.1 General

All parties with an interest in the results of sampling should be identified and actively involved in the decision-making process needed to define the sampling plan. Failure to obtain active participation from all involved parties might mean that certain parties are unaware that sampling is about to commence or might not understand the effects that the sampling results could have on their situation. This can lead to delay in later phases of the testing programme.

Identification of involved parties is not always easy and not all involved parties are easy to access. However, not all parties need to be personally represented.

The following roles can be distinguished in most testing programmes:

— The legislator;

— The regulator;

— The company or organization that produces the treated sludge or treated biowaste;

— The company or organization that accepts the treated sludge or treated biowaste;

— The landowner / manager;

— Related personnel and organizations.

A single person might have more than one role and represent more than one involved party.

4.2.2 Legislator

The legislator is the body responsible for defining the rules for sampling and testing. The requirements of the legislator are usually defined in policy documents, directives and national, regional and local legislation. The legislator will not be involved directly in the process of defining the sampling plan. However, the rules defined by the legislator will have to be taken into account when defining the objective of the testing programme.

In most cases, the legislator will be the European Commission, the national, regional or local government. However company management can also have the role of legislator. Therefore a combination of legislators is possible.

4.2.3 Regulator

Most legislation authorizes a regulator to base a decision on the sampling results provided by the producer and / or buyer of the sludge or biowaste, or allow independent sampling by the regulator.

The regulator might be involved in the process of defining the sampling plan, but the level of involvement will be decided by the regulator on a case by case basis.

EXAMPLE 1 The regulator might become involved if there is risk of a breach of the rules defined by legislation.

EXAMPLE 2 The regulator might be involved when a new sewage treatment works or a biowaste composting plant is being commissioned.

In complex situations, there might be several regulators.

The regulator (but also other parties) could make demands on the quality, involvement and responsibilities of other parties such as the sampler and laboratory. Also, the legislator can prescribe procedures to safeguard the quality of sampling. These types of demand normally result in demands on certification or accreditation of the companies and / or personnel involved in the testing programme; see also 4.2.5.

4.2.4 Owners

4.2.4.1 Owners of sludge or treated biowaste

Ownership of the treated sludge or treated biowaste can change between production of the material and its final use or disposal. Each of the companies or organizations that take ownership of the material will have an interest in the outcome of the testing programme and should be involved in the definition of the objective of the testing programme and the translation of this objective into the sampling plan. Often the sampling plan is provided by the treated sludge or treated biowaste producer, but this does not imply that the producer is the final decision maker.

The companies or organizations taking ownership of the treated sludge or treated biowaste might have different interests, but early discussion can resolve these differences or at least allow a negotiated settlement, well before committing time and resources to the testing programme.

Example: multiple owners

Initially, the owner of the material is the producer, e.g. the operator of the waste water treatment works or biowaste composting plant. Ownership of the material could transfer directly to the manager (the landowner or tenant) of the land to which it is to be applied. Alternatively, ownership could transfer to an intermediary organization acting as a buyer / seller or carrier.

Each owner could be required by the regulator to provide evidence of the properties of the treated sludge, including a description of the sampling and analysis undertaken to quantify the concentrations of substances present in the sludge. Therefore, the owners, directly or indirectly, are involved parties.

4.2.4.2 Owners of land

The owner of the land might not necessarily be the manager of the land.

EXAMPLE In the case of tenancies, the land is managed by the tenant, not by the land-owner from whom the land is rented.

The owner / manager of the land will have an interest in the outcome of the testing programme and should be involved in the definition of the sampling plan, providing background information and records of farming practices on the land.

Ownership / management of the land can change one or more times after permission for treated sludge to be applied has been given.

4.2.5 Sampler, laboratory analyst and other executives

The involved parties could also stipulate the quality of the personnel or organizations that conduct the sampling and subsequent analysis of the samples, e.g. by a system of certification or accreditation of sampler and analyst.

Examples: Certification or accreditation

Member State A has determined on a national level that all sampling should be done by individuals who have been trained in the sampling of sludge, treated biowaste, and soil and examined under a recognized sampler certification scheme.

Member State B has determined on a national level that all sampling should be done by an organization that complies with an accreditation programme for the sampling of sludge, treated biowaste, and soil.

Recognized best practice is for interested parties to be identified and encouraged to participate at an early stage of sampling plan development. In many cases, these involved parties will have practical comments that improve the quality of the testing programme or positively influence the way the sampling should be conducted.

4.3 Establishing the objective of the testing programme

To ensure that the testing programme is fit for purpose, the reason for investigating the material needs to be clearly identified, i.e. what is the objective of the testing programme?

The objective of the testing programme can be partly or fully prescribed by international, national, regional or local legislation or regulation, however the producer of the sludge or treated biowaste should also contribute to the objective setting process. All involved parties should reach agreement about the objective.

Examples of possible objectives of a testing programme are:

— to check the quality of the material because of a change in ownership;

— to determine the (re)usability of the material;

— to assess the human health and / or environmental risks posed by the material;

— to establish baseline conditions prior to an activity which might affect the composition or quality of soil;

— to monitor the effect of direct inputs to the soil.

In these examples the objectives are defined in very general terms and provide no specific direction on how to evaluate the material through sampling and analysis. A technical objective might be:

— to determine the nutrient content of the treated sludge or treated biowaste (to calculate the application rate);

— to compare the quality of the sludge or treated biowaste, or soil with quality levels defined in national and international legislation (does the material meet the compliance levels?);

— to determine the metal content of the soil (to establish if sludge can be applied);

— to compare the quality of the soil with quality levels specified in European and national legislation (does the soil continue to conform to the quality levels?)

However, this is not technical description that can be used as an unambiguous sampling plan. The derivation of the sampling plan from the technical objective is discussed in paragraph 4.4

Example situation

Sewage sludge is treated at a waste water treatment works. The treated sludge is put into stockpiles before being applied to land. Land application is not permitted unless the concentrations of a number of key constituents in the sludge and the soil to which it is to be applied conform to quality criteria set out in legislation, which are aimed at protecting the environment, i.e. the soil quality and groundwater quality, and providing benefit to agriculture, e.g. nitrogen and phosphorus nutrients.

The first objective of the testing programme is to test the sludge to determine its suitability for land application.

The second objective of the testing programme is to establish baseline conditions (e.g. metals content, fertilizer requirements) prior to an activity which might affect the composition or quality of the soil.

4.4 Strategic choices in deriving the technical goals from the objective

4.4.1 General

The objective of the testing programme defines what the involved parties want to achieve by investigating the material. To investigate the material, samples should be gathered and analysed to produce analytical results that are fit for purpose to satisfy the objective. Therefore the sampling operation should be planned in detail and be appropriate to produce results that meet the needs of the testing objectives. The detailed plan and technical description of the sampling operation should be formalized in the sampling plan.

In deriving the sampling plan, the original objective needs to be translated into one or more technical goals. The relationship between the testing programme, the objective, the technical goals and the sampling plan are depicted in Figure 2.

Example: Specifying instructions in the sampling plan by defining the technical goals of the objective

The objective ‘compare the quality of the treated sludge with quality levels as defined in legislation’ needs to be translated into technical goals such as ‘measure the pH and cadmium content of the treated sludge’. In the sampling plan, technical goals (e.g. measurement of the pH) should be translated into specific instructions. For instance, specifying the amount of sample to be taken and the necessary sample conservation measures for determining the pH.

In this example, the technical goals are to take an *adequate amount of treated sludge* and conserve its characteristics by using an *adequate sample container*. In the sampling plan the terms ‘adequate amount’ and ‘adequate’ as used in these technical goals is replaced by a statement of the amount of treated sludge needed (e.g. 1 kg) and prescription of the type of sample container (e.g. dark glass and air tight) respectively.

A testing programme can have more than one objective. In principle, each objective might result in a different sampling plan because the technical description of the necessary samples and the quality level to be achieved might vary between the different objectives. As a result it might be necessary to define more than one sampling plan to fulfil all the objectives of the testing programme.

Example: Situation where the testing programme has more than one objective

The first objective of the testing programme is to define whether the composition of the sludge conforms to the acceptance criteria, e.g. the concentration of certain metals in the sludge.

A second objective of the testing programme is to define the nutrient quality of the sludge, which is aimed at assessing the potential benefit to agriculture (or ecological improvement) and the application rate, e.g. the nitrogen and phosphorus concentrations in the sludge. Judgemental sampling might be sufficient for determining the nutrient content.

The two objectives might require different sampling approaches; therefore two different sampling plans might have to be defined.

The fact that there are two different sampling plans does not imply that the sampling cannot take place at the same time.

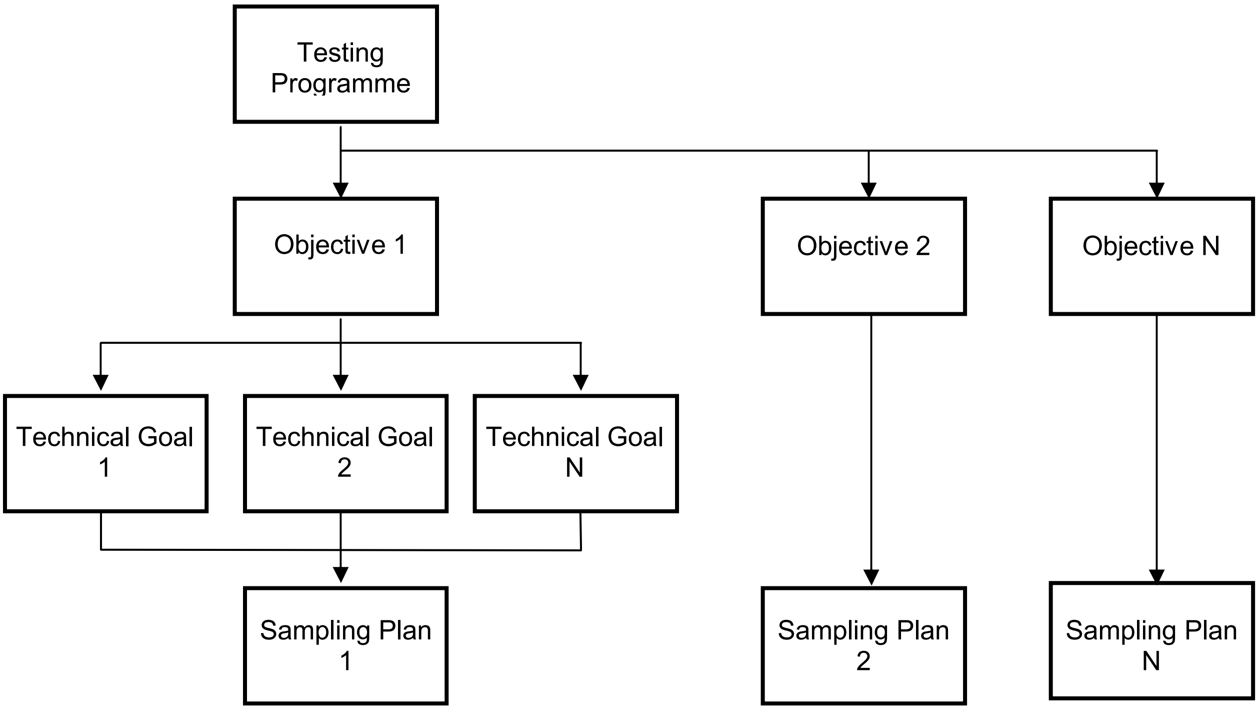

Figure 2 — Translation of objectives into technical goals and instructions in the sampling plan

The technical goals are related to many elements of the sampling plan, including[1]:

— constituents to be studied (4.4.2);

— the population that is represented by the sampling results (4.4.3);

— the scale of sampling (4.4.4);

— the desired reliability of the results (4.4.5);

— statistical parameter to be determined;

— choice of sampling methodology (probabilistic or judgemental);

— adequate sampling technique;

— sample pre-treatment.

4.4.2 Selecting constituents to be studied

The selection of constituents starts with an inventory of constituents that are raised in relevant legislation or have a direct bearing on the interests of individual parties. The constituents identified by legislation are often a

1 Choosing the constituents to be studied, defining the population and scale of sampling and choosing the desired reliability of the sampling results are most important because these choices influence to a large extent the efficiency and effectiveness of the testing programme.

reflection of their potential to cause human, environmental and economic risks. Background data on the material might also identify further relevant constituents.

Example: Selection of constituents

Specific quality criteria for sludge acceptance for land application are based on strong acid digestion and compliance levels are set for several constituents: Cd, Cu, Cr, Hg, Ni, Pb and Zn. Additional characteristics to be tested include dry matter, organic matter, pH, nitrogen and phosphorus.

Based on regular sampling and analysis, Cu concentration in the sludge is considered 'critical', since the 95-percentile value of earlier analyses show this parameter is most likely to exceed the compliance levels. Thus in the example the statistical definition of 'critical' is that there is more than 5% probability that the mean concentration of a constituent in a stockpile will exceed the compliance level.

Background information about the composition of inputs to the production process that leads to the production of the material can be crucial in selecting the constituents to be studied.

Example: Background information

Different types of background information are available for the treated sludge. There is technical information on the production process and input materials. There is also analytical information obtained from a previous basic characterization as well as previous compliance testing.

An example of non-analytical information is that a waste water treatment works with a capacity to treat 25,000 t sludge actually treats 17,500 t per annum. This results in 12,500 t of thermally dried sludge pellets annually that can be available for land application.

Compositional data from regular testing of the treated sludge provides information that the component that is to be considered as ‘critical’ is Cu. From analysis of data collected over the operational period of the works, the Cu content in the last 4 years ranges from 370 to 900 mg/kg. The mean Cu concentration is 505 mg/kg with a standard deviation of 112 mg/kg. These types of data are required as a basis for determining the requirements of any future sampling programme.

4.4.3 Defining population and sub-populations

4.4.3.1 General

The population is the total amount of material on which information is to be obtained by sampling.

In its most simple form, the population is a container, stockpile or lorry of sludge or treated biowaste or an area of land to which these materials are to be applied. In this case identifying the population in terms of space and time is simple.

Where a production process results in a continuous stream of material, identifying the population is less straightforward. For example, in a continuous production process the population might be the amount of material that is produced in a given period and the time period of production would need to be specified. The population might thus be defined as the amount of material produced at a certain place in a year, month, week, or other period.

Similarly, the area of land to which sludge is to be applied could be large, include different soil types or land forms, and be farmed for multiple purposes. In this situation, identifying the population is less straightforward. For example, the population might be defined as an area of land that is farmed for the same purpose and / or an area of land not exceeding a specified area. For this reason the term ‘overall population’ is used to describe the total area of land to be sampled.

Depending on the objective of the testing programme and the available resources, a choice needs to be made between various options for defining a population. Furthermore, it might be necessary to divide a population into sub-populations. From the perspective of sampling, a sub-population can be seen as the unity that is sampled separately and for which sampling results provide information (see 4.4.4).

The production process of treated sludge and treated biowaste determines to a great extent the definition of the population and the necessity to divide the population in sub-populations. For the purpose of sampling, the following production processes can be identified:

— one off production of material (for example: container, stockpile, lorry);

— continuous production of a homogeneous material;

— continuous production of a heterogeneous material.

The size of the land area to which sludge is to be applied, variations in farming purpose, and variations in soil type and landform determine to a great extent the population and the necessity to divide the populations into sub-populations. For the purpose of sampling, the following set of circumstances can be identified:

— An area of land that is smaller than a specified maximum size and is farmed for the same purpose (the population is the whole area of land);

— An area of land that is larger than the specified maximum size and is farmed for the same purpose (the land is divided into sub-populations based on area);

— An area of land that is farmed for different purposes (the land is divided into sub-populations based on farming purpose).

4.4.3.2 One off production of material

The simplest situation is a one-off production of material stored in a container, stockpile, lorry or other unit, clearly defining the population in terms of space (the amount of material in a certain location). In this case there is no need to use any term other than 'population'.

In a more complex situation, the one-off material is stored in more than one container, stockpile, lorry or other unit, but still the set of 'units' is limited[2] and can be defined in terms of space. Potentially it might be possible and desirable to identify different sub-populations in relation to, for example, the method of storage. Then the population will consist of different sub-populations.

Identification of sub-populations is, from the perspective of sampling, only necessary when these sub-populations are sampled and assessed on an individual basis; for example, when it is expected that sub-populations differ in quality or have different destinations with different acceptance criteria. However, this does not imply that each individual sub-population needs to be sampled.

4.4.3.3 Continuous production of a homogeneous material

In contrast to one-off production, a continuous production process generates a continuous stream of material. The population will now be defined in time rather than in space (the amount of material that is produced by a particular production process in a given time span). Definition of the population in space is however also possible, depending on the location where the samples will be taken.

In some cases, a continuous production process produces a homogeneous stream of material (Figure 3). The quality of this homogeneous material can be established with relative ease by a limited number of samples of appropriate size (4.4.4). It is not necessary to divide the population into sub-populations as these will not be sampled and assessed on an individual basis.

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2 There is a certain overlap between production of a 'limited' number of units and continuous production. This overlap depends on the actual number of units and the time span they are produced in. See 4.4.3.3 and 4.4.3.4.

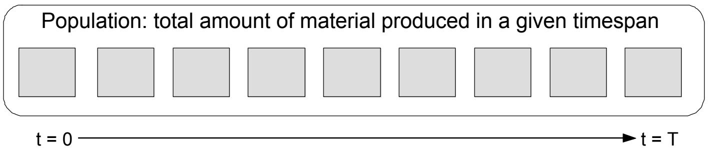

Figure 3 — Continuous production of a homogeneous material

4.4.3.4 Continuous production of a heterogeneous material

A continuous production process can also result in a stream of heterogeneous (of variable quality) material; for example, because the quality of primary inputs can change over time or because of variations in the production process.

The heterogeneity of the resulting material might lead to a situation where it can be reasonably expected that part of the population is not suitable for the planned destination or use. If it might be expected that specific parts of the material stream exceed the relevant specifications (e.g. compliance levels), the sampling should be organized in such a manner that these parts of the material stream can be identified.

Thus the results of the testing programme should give insight into the heterogeneity of the material. To accommodate sampling and to get insight into the heterogeneity within the population, the population has to be divided into several sub-populations. The sub-populations should preferably be physically separated until the results of the testing programme are available, allowing separate actions as a consequence of the potentially variable quality.

Any change in the production process that is expected to have an influence on the quality of the material, such as a new primary input or the introduction of a new machine in the production process, can result in a new sub-population (Figure 4). Identification of sub-populations from a production perspective provides information on the (potential) heterogeneity of the population.

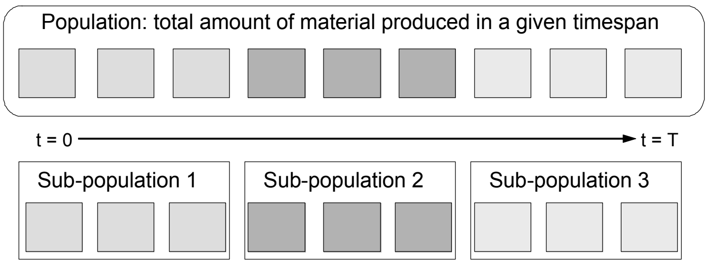

Figure 4— Continuous production of a heterogeneous material: identification of sub-populations from a production perspective

Sub-populations can also be identified from the perspective of transport and destination. For example, if a heterogeneous production stream is collected in trucks to be transported, it might be wise and practical to identify individual truckloads as sub-populations because this allows information to be gathered on the quality of the material that is to be transported (Figure 5).

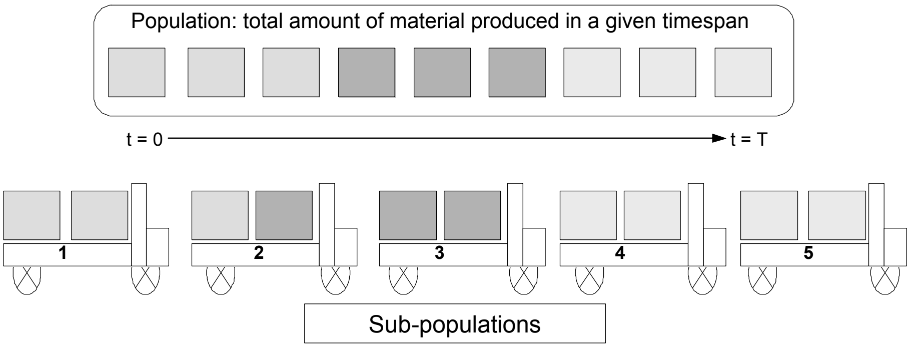

Figure 5— Continuous production of a heterogeneous material: identification of sub-populations from a transport perspective

It can also be stated that the truckload of material is the result of a one-off production and therefore is the population of sampling (4.4.3.2). However, this approach yields no information on the total amount of material that was produced, because only part of the total amount is included in the population.

For efficiency reasons, several truckloads that are transported to the same destination can be joined together in one sub-population (Figure 6). In this case the sub-population is defined from a destination perspective.

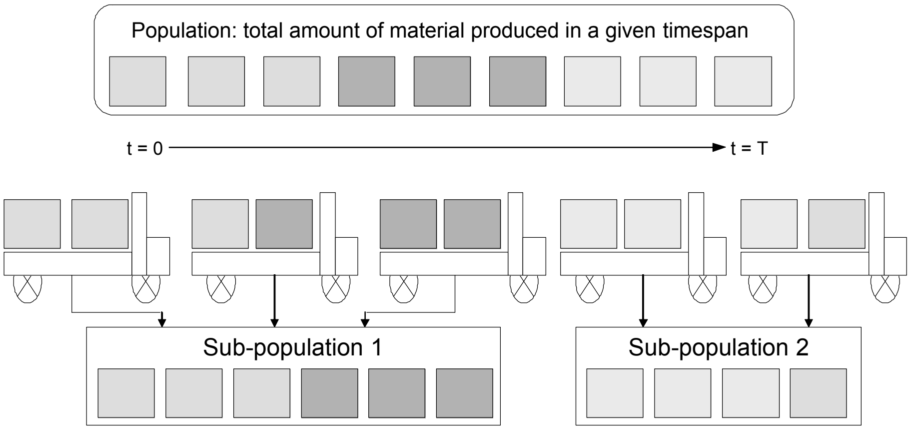

Figure 6 — Continuous production of a heterogeneous material: identification of sub-populations from a destination perspective

Table 1 summarizes the advantages and disadvantages of the various approaches to define sub-populations.

Perspective	**Advantage**	**Disadvantage**
Production perspective	Potentially a clear relationship between the sub-population and the production process results in relatively lower costs for the testing programme.	Production process needs to be known and samples need to be taken during or directly after production.
Transport perspective	Practical from the perspective of sampling.	Might result in high costs when there are a lot of sub-populations.
Destination perspective	Potentially a direct link can be defined between quantities of material that are considered relevant, for example from a	Variations caused by production, transport and / or mixing of quantities can no longer be identified.

	toxicological perspective.	

Table 1 — Advantages and disadvantages of various approaches to define sub-populations in sampling a continuous production of a heterogeneous material

Examples: Identification of population and sub-populations

Example 1: Sludge

The population is defined as the amount of treated sludge produced in one year (for example, 12 500 t thermally dried sludge pellets). However, because it is neither practical nor feasible to assess the production for a whole year, sub-populations have to be identified.

The thermally dried sludge pellets are placed in bags of for example, 1000 kg. So the annual production equals approximately 240 bags each week, based on a 7-day production week.

Sub-populations might be identified based on individual bags. However, that would result in 240 sub-populations to be sampled and assessed each week. To be more cost effective, the sub-population could be defined as the production of a week. In this case, a sub-population consists of 240 bags or 240 t.

In light of this definition of sub-populations, the sludge producer records the location of each week's production. So, when sampling results indicate that a sub-population exceeds the compliance level for a constituent, the sub-population can be easily identified and appropriate measures taken.

Example 2: Soil

Legislation prescribes that land that is farmed for a different purpose has to be sampled as a separate unity. Legislation also defines the maximum area of land that can be sampled as a single unit, say 5 hectares.

The treated sludge is to be considered for application to land of area 20 hectares, comprising two fields. Field 1 is 8 hectares and farmed for purpose 1; Field 2 is 12 hectares and farmed for purpose 2.

The population is defined as the amount of land to which treated sludge is to be applied, i.e. 20 hectares.

Because the land is farmed for more than one purpose, two sub-populations need to be identified, i.e. Field 1 and Field 2.

Because each field is more than 5 hectares, five sub-populations need to be defined, i.e. two for Field 1 and three for Field 2.

In light of this definition of sub-populations, the areas of land to be sampled are mapped. So, when sampling results indicate that a sub-population exceeds the compliance level for a constituent, the sub-population can be easily identified and appropriate measures taken.

4.4.4 Scale

Scale is one of the essential issues of sampling and is described in detail in Annex A of prCEN/TR XXXX-1:date.

The scale defines the volume or mass of material that a sample directly represents. This implies that when the assessment of the material is needed for example on one cubic metre, the sampling results should provide information on a cubic metre scale. Thus the analytical results should be representative for a cubic metre of material.

Depending on the objective of the testing programme, the scale of sampling might be equal to the size of individual particles of the material (for particulate materials), the size of the sub-population or even the whole population.

Scale can also be defined in terms of time: if the population is the total amount of material produced in one year, the scale could be one year (the overall population), but also one month, week or day, depending on the objective of the testing programme.

Defining the scale is important, as heterogeneity is a scale-dependent characteristic. No information is gathered from a volume / mass smaller than the defined scale.

Example: Defining the scale

This example is based on a particulate material that consists of small particles that only vary in colour. The particles in the material are fully mixed.

In a series of samples, each with the size of an individual particle, each sample might have a different colour. Therefore the observed heterogeneity in colour between these samples will be high.

However, the degree of heterogeneity on a scale of for example, 1 kg, consisting of several thousands of particles, will be low. Each of these samples will have approximately the same mix of colours, and – looking from some distance (on the scale of 1 kg) – the samples will have the same mixed colour. Thus the observed heterogeneity will now be low.

As a consequence of the direct relationship between scale and heterogeneity, sampling results are only valid for the scale that is equal to the scale of sampling or higher scales. In general, the degree of heterogeneity will be higher for a smaller scale of sampling and will be lower for a larger scale of sampling.

Given the relationship between scale and the encountered degree of heterogeneity, the applied scale of sampling might determine if a material is considered homogeneous (i.e. there is little variation between individual sample results) or heterogeneous (i.e. high variation between sample results).

The choice of sampling scale is determined by the type of information that is needed, the financial means available and whether composite samples can provide information that is fit for purpose.

Example: Scale of sampling

Example 1: Sludge

In the example of thermally dried pellet production, the population was defined as the amount of treated sludge produced in one year (12,500 t thermally dried sludge pellets). The sub-population was defined as the production in one week: 240 bags.

In principle, the scale of sampling is equal to this amount of material. It is technically possible to take a sample from each bag and mix them into a composite sample (240 increments) and the scale of sampling is indeed 240 t, or one week's production.

However, when it becomes impracticable to collect this many increments an alternative approach might need to be considered. One approach could be to take a sample from one bag each day and mix the samples at the end of the week, resulting in one composite sample (7 increments) each week. In this case, the scale of sampling would be one week's production.

Example 2: Soil

In the previous example, the population was defined as the area of land to which treated sludge is to be applied. The sub-population was defined as an area of land of not more than 5 hectares that is farmed for the same purpose.

In principle, the scale of sampling is equal to this area of land. It is technically possible to take a composite sample from each 10x10 m area (100 m^2) resulting in 500 laboratory samples per 5 hectare area. If each of the 500 laboratory samples are analysed, the scale of sampling is 0.01 hectares. If the 500 laboratory samples are mixed into a composite sample the scale of sampling is 5 hectares.

However, when it becomes impossible to sample at this intensity, perhaps for reasons of cost, an alternative approach might need to be considered. One approach might be to take one increment from each 50x50 m area (2500 m^2) resulting in 20 increments per 5 hectare area. If the 20 increments are mixed into a composite sample the scale of sampling is 5 hectares.

In addition to the more technical perspective from which the definition of scale can be described, the scale of sampling can also (or even should) be defined by policy considerations. In principle the scale of sampling should be equal to the amount of material which is considered relevant from a policy perspective.

Example: Policy defined scale of sampling

Example 1: Treated biowaste

When a new biowaste composting facility is built there is a validation phase during which there is a requirement that every batch of compost, for example, one week's production, is tested. The scale of sampling is a batch and is achieved by taking a number of increments within the batch. An estimate of the true mean concentration on the scale of one batch is obtained.

After a certain period, for example 6 months, or a given volume, e.g. 30 batches, the validation period is complete and the regulator considers that it is no longer necessary to test every batch. A new scale of sampling is defined; e.g. 5000 m^3 compost or 6 month's production.

Example 2: Soil

Legislation dictates that soil sampling is required to establish baseline conditions prior to application of treated sewage sludge for the first time. The legislation also specifies that soil samples for analysis should be made up by mixing together 25 increments taken over an area not exceeding 5 hectares which is farmed for the same purpose.

If the area to be tested is less than 5 hectares, then the scale of sampling is equal to the area to be sampled. If the area to be tested is greater than 5 hectares, then it is divided into smaller areas (sub-populations) and the scale of sampling is equal to the area of each of the sub-populations.

4.4.5 Choosing the desired reliability of sampling results

4.4.5.1 General

Treated sludge and treated biowaste are heterogeneous materials for numerous reasons, including:

- variability in the sewage sludge or biowaste that are inputs to the treatment process;
- variability in the mix of inputs from multiple sources;
- variability in the treatment process.

Soils are heterogeneous materials for numerous reasons, including:

- variability in underlying geology;
- variability in topography;
- variability in climate;

— variability in land use and type of cultivation.

In principle, it is impossible to know the exact composition of heterogeneous materials. Knowledge of whether the material is consistent or erratic in composition will need to be taken into account in the design of the testing programme. The results of the sampling are always an estimate of the true composition of the material that is studied and the total error or the estimate includes sampling and analytical error. Two types of sampling error will influence the representativeness of the sampling: the systematic error and the random error.

Due to the fact that these errors occur, there is always a chance that the estimated characteristic of the material will lead to an incorrect assessment.

If an incorrect assessment could have serious social, economical or environmental consequences, the reliability of the sampling results often needs to be high. The necessary reliability also depends on how close the measured characteristic is to the relevant quality level (compliance level). Thus, when the (expected) characteristic is much lower than the compliance level, the chance that the compliance level is exceeded is small and poorer measurement reliability might be acceptable. However, when the (expected) characteristic of the material is close to the compliance level, there is a higher chance that the compliance level is exceeded and greater measurement reliability is required to prevent an incorrect assessment.

Statistics enable the reliability of the estimate and the chances of an incorrect assessment based on the sampling results to be estimated. For any random sample, confidence limits can be calculated. Confidence limits specify with a given confidence that the true value of the characteristic will fall within a given range (confidence interval) around the estimate. The narrower this confidence interval the better the sampling mean estimates the true mean value of the population.

The size of the confidence interval (see prCEN/TR xxxx-1) is related to:

— the heterogeneity of the population;

— the number of samples;

— the desired confidence level.

Given a specific sampling effort, a heterogeneous population has a wider confidence interval than a homogenous population. When analytical error is assumed to be negligible, confidence limits decrease when more samples are taken and for a narrow confidence interval more samples are needed than for a wider confidence interval (Figure 7).

The confidence interval increases with the desired confidence level and the heterogeneity of the population. The confidence interval decreases when more samples are taken. The narrower the confidence interval, the better the estimate of the mean represents the true mean of the population.

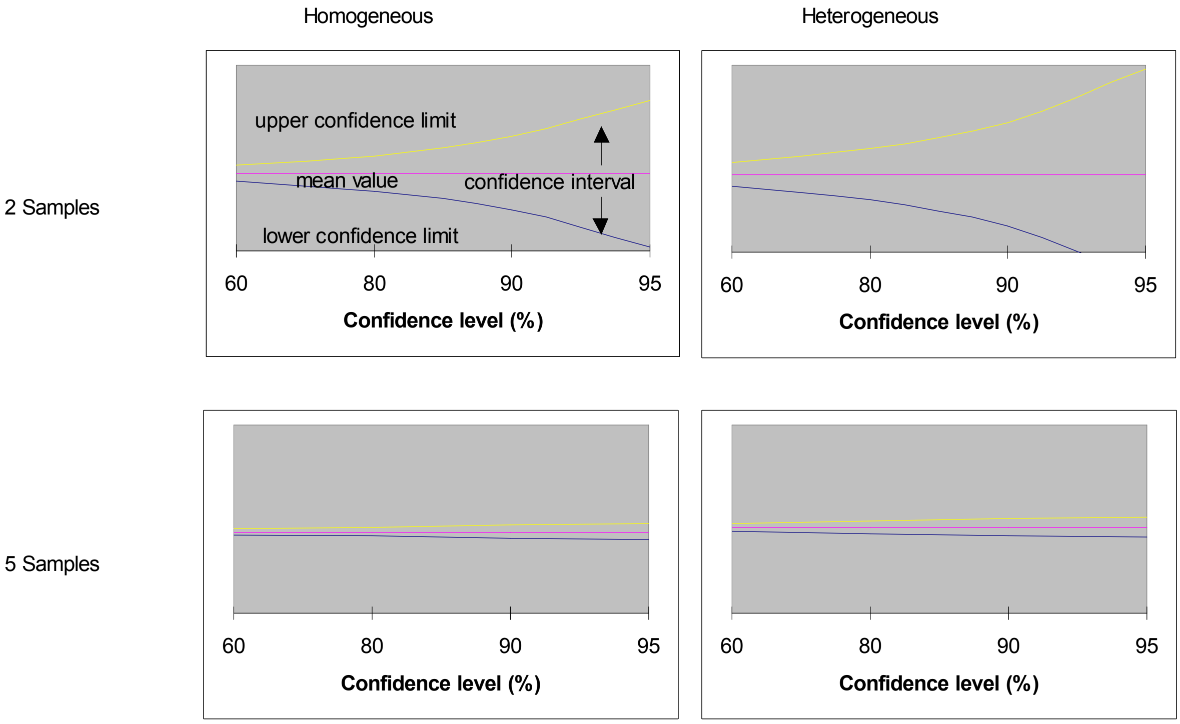

Figure 7 — Confidence levels for homogeneous and heterogeneous samples

The involved parties influence the costs of the testing programme because the number of samples is directly determined by the desired reliability that they specify. The advice of statistical experts and additional research might be necessary to quantify the relationship between heterogeneity of the material, the reliability of the estimate and the number of samples.

<u>It is very important that the involved parties are aware of the impact of their choices on both costs and reliability of the sampling and that they specify the desired reliability of the estimate before a sampling plan is constructed.</u>

In most cases, the reliability of sampling results improves when the number of samples is increased (Figure 7). This leads invariably to higher costs for sampling and analysing. There are two important approaches possible to balance reliability and financial input:

— many field samples versus many increments mixed in a composite sample (4.4.5.3);

— increasing the scale of sampling.

These two approaches can be combined.

4.4.5.2 Probabilistic versus judgemental sampling

It should be noted that whenever the reliability of the sampling is considered important, the type of sampling should conform to that need. Two principally different types of sampling are distinguished: probabilistic sampling and judgemental sampling (see CEN/TR xxxx-1).

4.4.5.3 Many field samples versus many increments mixed in a composite sample

When taking a large number[3] of field samples, the cost of analysis for all these samples might be high in relation to the cost of sampling. On the other hand, when these increments are mixed in a composite sample,

[3] Not only the number, but also the size of samples or increments influences the reliability. The minimum sample size can be calculated (see CEN/TR xxxx-1).

the variability of the sampled material in effect will be summed within the composite sample. In this case, the number of samples and the resulting cost of analysis are relatively low, but more effort (and thus cost) has to be made in sample pre-treatment to accomplish complete mixing of the increments. At the same time information on the range of concentrations that might be expected from the material is lost. Whether that information is important depends on the objective of sampling.

The results of both options are different. Through analysing many field samples, information on the variability of the material is obtained, but analysing a composite sample yields only an estimate of the mean characteristic. Of course, intermediate solutions are also possible where limited numbers of increments are mixed in a limited number of composite samples.

Example: Defining the desired confidence and estimating the resulting number of samples

The involved parties undertook some preliminary research on the number of samples that would be necessary to reach 90% and 95% confidence levels for an estimate with a confidence interval of ± 5% for the constituents (Cd, Pb and Cu). The results of the experiment are shown in the following table.

Confidence level	Number of composite samples (10 increments each)		
	Cd	Pb	Cu
90%	8	8	5
95%	17	18	8

The involved parties agree that the number of samples that is necessary for a 95% confidence for all three elements (18 samples) would lead to unacceptable costs for the testing programme.

However, inspection of the data shows that the critical component is Cu, which frequently reaches 90% of the limit value. The concentrations of Cd and Pb are less critical; Cd and Pb concentrations are typically only 20% of the limit value.

They decide that 8 samples will be sufficient to estimate Cd and Pb with 90% confidence and Cu with 95% confidence.

4.5 Checklist

In the interactive process of deriving technical goals from the objective, the involved parties need to formulate answers to the questions that were raised in 4.4. However, not all sampling plans consist of the same elements, depending on the complexity of the objective. This subclause contains a list of questions that should be addressed in the process of defining the sampling plan[4].

Question: Are all parties involved that should be involved?

See 4.2

Question: Is the objective clear and do all the involved parties agree on the objective?

See 4.3

Question: Are the constituents defined?

See 4.4.2

Question: Is the population defined?

[4] The order in which these questions are addressed might vary. For example the scale of sampling can be specified at various times during the process.

See 4.4.3

Question: Is it necessary to identify sub-populations?

Identifying sub-populations is advisable when:

- parts of the population are going to be treated differently. For example, in case of a continuous production process where part of the annual production is transported every week to a production manager and the quality of this week's production (=sub-population) needs to be known, or where the area to be tested comprises smaller areas that are farmed for different purposes.

- identifiable parts of the population are expected to be significantly different from other parts of the population. For example, where the area to be tested comprises smaller areas that have different soil type.

Question: Is the scale of sampling defined?

In some cases, the scale of sampling is stated explicitly in legislation. However, in most cases scale is not defined explicitly by legislation.

Sometimes the scale can be derived from the objective. For example when information is necessary on the heterogeneity within the population[5], the scale of this information should be known by a specific choice of the size of the samples and the volume / mass these samples represent. In other cases, the scale is not defined a priori but becomes clear after the sampling plan is derived. Also in these situations it is important to identify the scale to check if it is possible to reach the objective by the chosen sampling plan.

Question: Is the desired reliability defined?

See 4.4.5

- confidence interval;

- confidence level;

- probabilistic sampling or judgemental sampling.

Question: Field samples or composite samples?

See 4.4.5.3

- (many) samples: Good estimate of heterogeneity within the population (or sub-population). Mean value of the population can be calculated. Given a specific population, the reliability depends on the number of samples, the sample size (scale) on which the heterogeneity is determined.

- composite sample of (many) increments: Good estimate of the mean of the whole population (or sub-population). Reliability of the estimate depends on the number of increments and the quality of the sample pre-treatment (mixing and sub-sampling).

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5 Or sub-population or lower scales: for example primary particles.

4.6 Further steps to be taken

After the identification of the involved parties, the identification of the objective and translation of the objective into technical goals, the sampling plan can be produced. As the policy related decisions are now made, the remaining aspects are technical and procedural. They include:

— the statistical parameter to be determined.

— the sample size.

— what sampling technique is fit for purpose?

— the type of sample pre-treatment necessary in the field to obtain a quantity of material that can be transferred to the laboratory.

Annex A
(informative)
Examples of sampling plans for specific situations

A.1 Basic characterization and compliance testing of sludge from a continuous production process

A.1.1 Introduction

A production process generates a continuous stream of sludge throughout the year. The annual amount of sludge produced is more than 10.000 t per year. Based on preliminary examinations and process information, the quality of the sludge is expected to be constant with variations within certain boundaries during the year.

According to national legislation, the quality of the sludge needs to be characterized before application to land. It is not possible to store the sludge produced within one year prior to sampling. To facilitate acceptance and application to land of the sludge during the one-year period, basic characterization follows specific rules.

A.1.2 Description of the process

Step in process	Results	Description
Identify involved parties Paragraph 4.2	— Legislator — Sludge producer — Sludge owner — Laboratory (also responsible for sampling) — Regulator	The national legislation defines the involved parties
Define objectives Paragraph 4.3	The objectives of the testing program can be divided into objectives for year 1 and objectives for the following years. Year 1: basic characterization to evaluate if: — The mean concentration of the sludge produced within one year of production complies with the limit values. — The sludge has a constant quality so that only compliance testing may be done in year 2. Following years: compliance testing based on a systematic sampling pattern.	In year one, the production period is divided into 4 quarters. The quality of the sludge in the first and the third quarter is characterized by three weekly assessments. If the results of basic characterization show a constant quality below compliance levels in the first year, basic characterization is replaced by compliance testing in the following years. In the first week, composite samples of the sludge produced on three days distributed evenly over the production week (e.g. Monday, Wednesday, Friday) have to be analysed. The sludge can be applied to land until the results of the next weekly assessment become available if the average over the 3 days of every parameter meets the limit value and no single daily result exceeds certain tolerance values. If the mean value over 3 days does not exceed 80 % of the limit value and if the variability between days is not high, in the second weekly

		assessment the three days may be combined to a composite sample representing the weekly production. If this condition is not met the procedure of week 1 is repeated. If the second weekly assessment does not conflict with the criteria, the sludge can be applied to land up to the third weekly assessment. If the values of the third weekly assessment still comply, application to land may continue without sampling until the third quarter. In the third quarter this procedure is repeated. If all the six 'weekly assessments' of the first and the third quarter confirm that the sludge may be applied to land, the sludge is considered to be basically suitable for application to land. This means that in the following years, only compliance tests have to be performed. However, if changes in sludge characteristics are expected (e.g. due to process modifications or changes in input materials) basic characterization has to be done. It is not allowed to mix different types of sludge or sludge with different quality levels caused by changes of the input or the process.
Determine generic level of testing required	Basic characterization in year 1 and compliance testing in the following years.	According to national legislation, the quality of the sludge needs to be characterized before land application. If the results of basic characterization show a constant quality below compliance levels in the first year, basic characterization is replaced by compliance testing in the following years. NOTE One part of each daily sample should be stored for eventual later use. If the measured value of one or more parameter in a composite sample exceeds 90 % of the limit value, the three daily composite samples should be analysed.
Identify constituents be tested <u>Paragraph 4.4.2</u>	Cd, Co, Cr, Cu, Ni, Hg, Pb, Zn, pH, dry matter, organic matter, Nitrogen, Phosphorus	
Research background information on sludge	Production process, causes of variability in the sludge stream, sludge type.	
Identify health and safety precautions	General health and safety precautions.	No particular health and safety precautions are needed. Consequently, the general health and safety precautions prescribed for the site are to be taken.
Select sampling approach <u>Paragraph 4.4</u>	<u>Population</u>: one-year's production of sludge from a continuous production process. <u>Sub-population</u>:	The population is defined by the legislator as the amount of sludge produced within one year because most other regulation concerning sludge also has a year as time period.

	— Year 1: one week's production — Following years: amount of sludge that is produced in a quarter <u>Scale</u>: — Year 1: First week of the first and third quarter: the amount of sludge that is produced in one day, in other weeks the amount of sludge produced in a week. — Following years: amount of sludge that is produced in a quarter. <u>Required level of confidence</u> — = 90 %, <u>Required precision</u> — = 20 %	In the first weekly assessment of year 1, one composite sample is analysed per day. In the following assessments one composite sample per week is analysed. This composite sample consists of composite samples from three days. Therefore the result of testing is a weekly mean value. Daily composite samples have to be stored for eventually later use. In the following years, the three weekly composite are combined into a quarterly composite sample that is analysed. As a consequence, information on the scale of one week's production is not available. The lowest scale information is available for is the quarterly production. If the stored daily composite samples are analysed, information is obtained on the scale of a day.
Comments The sample size and number of samples are calculated following the guidance given in prCEN/TR XXXX-1. For the first weekly assessment approximately 50 increments need to be taken.		

A.2 Testing of agricultural soil

A.2.1 Introduction

No person is permitted use sludge on agricultural land unless the requirements set out in legislation are fulfilled. The legislation stipulates that the soil on the land has to be tested to ensure that the concentration of specified parameters will not exceed the limits specified in the legislation as a consequence of sludge application. Sludge cannot be applied to soil with a pH less than 5. The nutrient needs of the plants grown on the land should be taken into account. According to national legislation, the quality of the soil has to be characterized before application to land and at prescribed periods afterwards.

A.2.2 Description of the process

Step in process	**Results**	**Description**
Identify involved parties Paragraph 4.2	— Legislator — Sludge producer — Sludge owner — Landowner / manager — Laboratory (also responsible for sampling) — Regulator	The national legislation defines the involved parties
Define objectives Paragraph 4.3	Basic characterization prior to sludge application and subsequent periodic compliance testing	According to national legislation, the quality of the soil has to be characterized before land application. If the results of basic characterization show that the quality of the soil conforms to specified criteria, sludge application is permitted. Basic characterization is replaced by periodic compliance testing in the following years.
Identify constituents be tested Paragraph 4.4.2	Cd, Co, Cr, Cu, Ni, Hg, Pb, Zn, pH, Nitrogen, Phosphorus	
Research background information on land	Large-scale maps showing field boundaries Information on previous/planned use Soil depth	
Identify health and safety precautions	General health and safety precautions.	No particular health and safety precautions are needed. Consequently, the general health and safety precautions are to be taken.
Select sampling approach Paragraph 4.4	Population: An area of land comprising one or more agricultural fields Sub-population: — An agricultural unit: An area of land of no more than 5 hectares that is	The sub-population is defined by the legislator The sampling approach is specified in legislation: For each agricultural unit on which sludge is to be used, a representative sample of soil has to be obtained by mixing together 25 separate core samples, each taken to the depth of the soil or 25 centimetres, whichever is the lesser depth.

	farmed for the same purpose Scale: — 5 hectares Required level of confidence — = 90 %, Required precision — = 20 %	

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