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

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

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

Material	Validated	Document
Waste	☐	[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.

In the process of defining the Sampling Plan (Key Step 1 in Figure 1 of prEN XXXX), the objective of the testing programme is translated into specific technical instructions for the sampler. Using these instructions the sampler will take the type and number of samples that are adequate to meet the objective of the testing programme, ultimately providing the decision maker with the required information on the material under investigation.

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

In practice, problems arise when translating the objective of the testing programme which is couched at a relatively abstract level (e.g. 'the sludge needs to be assessed to fulfil the demands of land-use regulation') into a technical instruction that corresponds with that same objective (e.g. 'the mean concentration of each truckload should comply with a specified concentration level'). There is a 'gap' between defining the need to evaluate the material and specifying the technical methods that should be applied in order to make an adequate evaluation possible.

This Technical Report provides information and guidance on the process of defining a Sampling Plan (Key Step 1 of the Sampling Plan process map), aiming to 'bridge the gap' between the chosen objective of the testing programme in policy terms, and that same objective defined in technical terms for sampling.

This Technical Report is written for two distinctive groups of users:

- Policy makers involved in sampling. For example, people working for the central, regional or local authority, government or administration, the management of a company involved in the production or disposal of treated sludge and treated biowaste. These people are involved, directly or indirectly, in making policy decisions that are based on the technical information gathered through sampling. Their interest lies in the requirement for testing a material to gain knowledge about the material or to comply with national, regional or local legislation. Typically they have no technical knowledge of sampling, but are responsible for making the right choices. They need help to understand the definition of the testing programme in technical terms, in order to be able to judge if the proposed testing programme is adequate for the purpose.

— Sampling specialists (specifically the project manager). These are the people who have to translate the objective of the testing programme, as defined by the policy maker, into a technically unambiguous Sampling Plan that will instruct the sampler on what to do in the field.

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.

Example: materials to be tested

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 comply with limits set out in legislation. Therefore the treated sewage sludge and treated biowaste must be sampled.

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 provides guidance on 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 1 This Technical Report cannot provide definitive instructions that cover all scenarios. Instead, it discusses the basic statistical approach to be followed, and provides statistical tools that can be applied to determine the amount and type of sampling (e.g. number of samples and sample size) in any given situation to achieve results of adequate reliability (i.e. precision and confidence).

NOTE 2 The document provides considerable detail on current best practice, but is not exhaustive.

2 Normative references

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

EN 3435-1, Statistics – Vocabulary and symbols – Probability and general statistical terms

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

3 Terms and definitions

3.1
background information
information that is essential to understanding the setting of sampling

NOTE Among others, it consists of information on the production process of the material to be tested, the nature of the material, policy aspects and compliance levels set in legislation.

3.2
basic characterisation
sampling that has the goal to describe the character or quality of a population of material

3.3
compliance testing
the process of testing whether sample values meet a pre-defined set of criteria

3.4
composite sample
two or more increments mixed together in appropriate portions, either discretely or continuously (blended composite sample), from which the average value of a discrete characteristic may be obtained

[ISO 11074-2:1998]

3.5
confidence interval
interval within which the value of a particular population parameter may be stated to lie at a specific confidence level

NOTE The bounds of the confidence interval are termed the upper and lower confidence limits.

3.6
confidence level
the value 100(1-α) of the percentage probability associated with a confidence interval

[after ISO 3435-1]

3.7
constituent
property or attribute of a material that is measured, compared or noted

3.8
decision maker
the party that makes a decision based on the results of the testing programme

NOTE In most cases the regulator is the decision maker, but it can also be the producer or manager of the material.

3.9
field sample
the quantity (mass or volume) of material obtained through sampling without any sub-sampling

3.10
increment
individual portion of material collected by a single operation of a sampling device which will not be analysed / investigated as a single entity, but will be mixed with other increments in a composite sample prior to analysis

3.11
involved parties
individuals who have an interest in the results of the sampling and who should therefore be involved in the (iterative) process relating to the exchange of information regarding the testing programme

NOTE Such parties include, for instance, the sampler, the analyst, the client, the regulator and the producer of the material. The person responsible for the overall measurement report is the Project Manager.

3.12
laboratory analyst
the person conducting the analysis of the laboratory sample

3.13
laboratory sample
the sample sent to or received by the laboratory

3.14
legislator
the body responsible for the definition of the rules that should be obeyed

3.15
objective
the underlying motivation for investigating the material

3.16
on site verification
a normally simple test to evaluate if the involved material is indeed the type of material expected

3.17
overall population
the totality of items

3.18
population
the totality of items, or volume of material to be investigated by sampling

NOTE The population will generally be a convenient, well-defined subset of the overall population (e.g. a year's production) that is believed to be typical of the wider population.

3.19
policy maker
person working for the central, regional or local authority, government or administration, the management of a company

3.20
project manager
the person who is responsible for deriving and / or fulfilling the testing programme

3.21
regulator
the body responsible for controlling if the rules of the legislator are met

3.22
reliability
the extent to which a test measures consistently

NOTE1 A collective term for the degree of precision and confidence achieved by a given sampling scheme.

NOTE 2 For scaled scores, a reliability coefficient of 1.00 indicates a test that is perfectly reliable.

3.23
sample
an amount of material taken from a population and intended to provide information on the population

NOTE 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.

3.24
sampler
person carrying out the sampling procedures at the sampling locality

[ISO 11074-2:1998]

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

3.25
sampling plan
all the information pertinent to a particular sampling activity

[IS0 11074-2:1998]

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

3.26
scale
the stated size or volume that is considered appropriate for assessing the material

NOTE A quantity of material (mass or volume), defined in space and / or time.

3.27
sewage sludge
sludge from urban waste water treatment plants

3.28
sub-population
a defined part of a population that will be targeted for the purpose of sampling

3.29
technical goals
objective translated into specific, measurable, action oriented, realistic, timely (SMART) goals

3.30
testing programme
total sampling operation, from the first step in which the objectives of sampling are defined to the last step in which data are analysed against the objectives

3.31
treated biowaste
biological waste that has been subjected to biological treatment by composting or anaerobic digestion

3.32
treated sludge
sludge which has undergone a treatment process or a combination of treatment processes, so as to significantly reduce its biodegradability and its potential to cause nuisance as well as the health and environmental hazards when it is used on land

4 The process of defining the Sampling Plan

4.1 General description of the process

The project manager is responsible for managing the process which defines the Sampling Plan. A draft of the sampling plan should be discussed with all involved parties. The discussion process should identify the practical implications of the choices made when defining the objectives and translating them into practical instructions. Unrealistic objectives may need to be changed for reasons of practicality. Defining the Sampling Plan may be an iterative process that is repeated several times before it results in an accepted final version. The project manager should actively manage this process.

The first step is to identify the parties that have an interest in the results of the sampling and to ensure their full participation. Supported by the project manager, they must 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 the sampler. These instructions are then recorded in the Sampling Plan by the project manager.

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

Technical goals include statistical terms like the characteristic to be determined, the population, the scale, the confidence level and confidence interval to be reached and technical terms like the constituents that are to be determined, the moment when, or location where, the material will be sampled. Some of these technical goals provide direct input for the Sampling Plan, while others (e.g. the scale, the confidence level) still have to be translated into practical terms, for example the sampling pattern and location, the number of increments and samples and the sizes of increments and samples.

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 required for a given reliability depends on the constituent to be determined and the heterogeneity of the material to be sampled. Balancing the reliability and costs is an important decision to be made by the involved parties when defining a Sampling Plan.

The process of defining the Sampling Plan is provided in Figure 1.

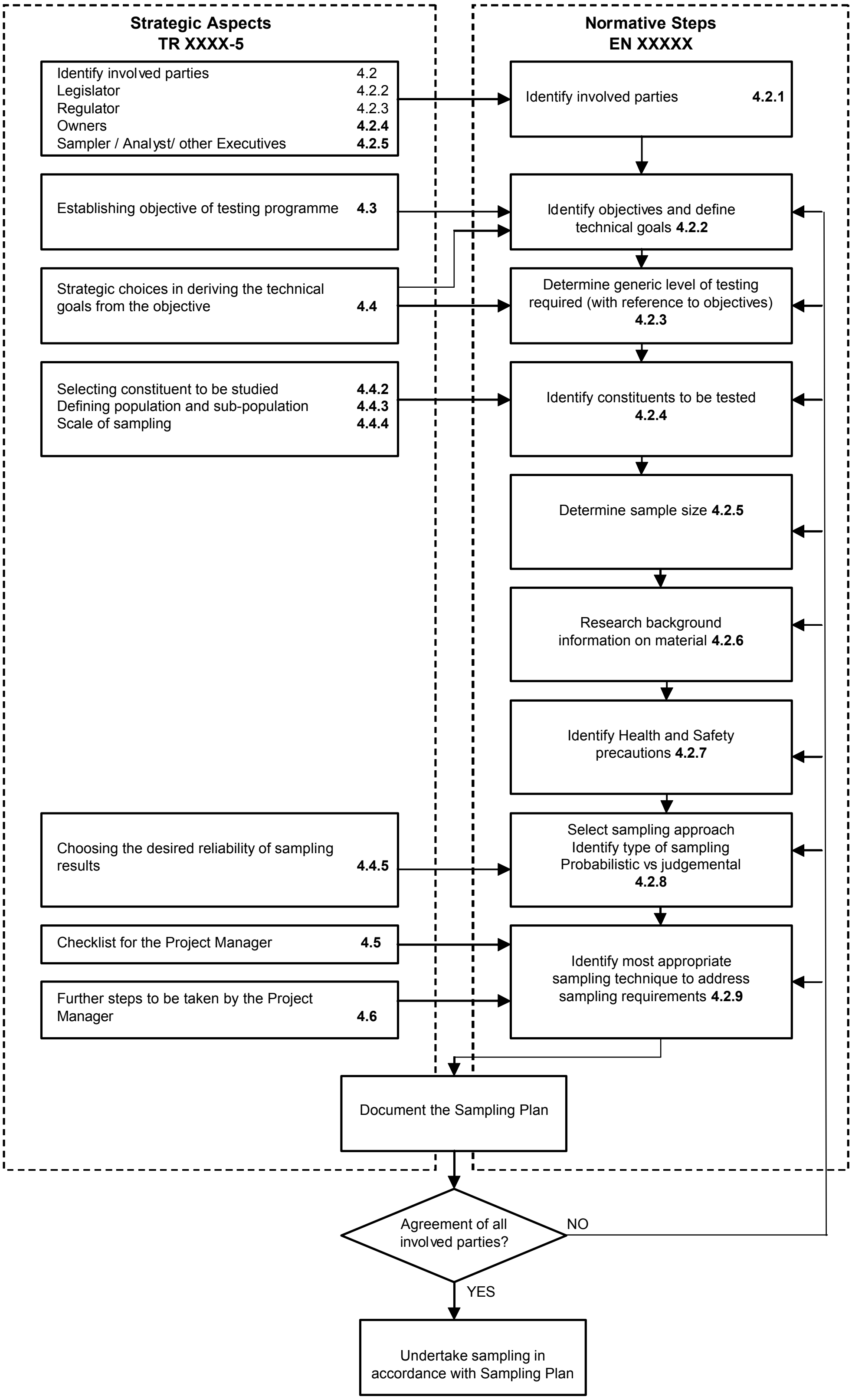

Figure 1 — The process of defining the Sampling Plan, providing information on the elements that are specified in the European Standard and this Technical Report

4.2 Identification of involved parties

4.2.1 General

It is the responsibility of the project manager to identify all parties with an interest in the results of sampling and actively involve them in the decision process that is required to define the Sampling Plan. Failure to get input from all involved parties may mean that some parties are not be aware that sampling is about to commence or may not understand the effects that the sampling results could have on their situation. This may lead to resistance and loss of time in later phases of the testing programme.

The involved parties come from differing backgrounds and may have conflicting interests. 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 involved parties are at a minimum the owners, users or buyers of the treated sludge or biowaste and, directly or indirectly, the legislator defining the rules to which the testing programme is related. Additionally, the project manager himself is an involved party.

The following roles can be distinguished in almost every testing programme:

- The legislator;
- The regulator;
- The company or organisation that produces the treated sludge or treated biowaste;
- The company or organisation that accepts the treated sludge or treated biowaste;
- The project manager and related personnel and organisations (like the sampler and the laboratory analyst).

A single person may have more than one role and represent more than one involved party (for example, in some sampling programmes the client and sampler may be the same person).

Example: Identification of the involved parties

Legislation laid out in European Directives prescribe that land application of sewage sludge is permitted only if it can be demonstrated to meet specified treatment requirements and only if the concentrations of a number of key constituents comply with certain limits. The European Commission is a legislator, but is not a directly involved party.

The European Directives are translated into national / regional legislation that defines the compliance levels. The national / regional government can be seen as the legislator. An agency of the national / regional government has responsibility for ensuring that the legislation is complied with. Therefore the government agency can be seen as the regulator.

Other involved parties are the company or organisation that owns the waste water treatment works, the manager of the works, i.e. the treated sludge producer, the project manager and the team that is responsible for the sampling. The project manager is an employee of the company or organisation treating the sewage sludge.

Before sampling, the project manager directly consults the manager of the waste water treatment works. A generic Sampling Plan, developed by the company or organisation owning the works, is available. Directions on sampling are provided in the generic Sampling Plan, whilst more specific conditions are defined in both regional policy and in the contract to supply treated sludge. Therefore the legislator is not involved directly.

The regulatory agency is aware that the treated sludge is to be applied to land. The regulator can check the quality of the treated sludge when desired. Therefore the regulator is not directly involved, but decides if and when the quality of the sludge will be checked.

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 considered when defining of the objective of the testing programme.

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

4.2.3 Regulator

The regulator is the body responsible for ensuring that the rules of the legislator are met. In complex situations, there may be several regulators (as many as there are legislators).

Most legislation authorises a regulator to base a decision on the sampling results provided by the testing programme, or to allow independent sampling by the regulator.

The regulator may 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 may become involved if there is risk of a breach of the rules defined by the legislator.

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

Example: multiple parties involved as regulator
Testing of the treated sludge at specified intervals is a requirement governed by legislation and regulated by the formal regulator. Testing is done by the treated sludge producer and the results of that testing are delivered to the landowner together with the treated sludge. In addition to the tests that are required by legislation, the producer tests the treated sludge periodically to check that it complies with the required specifications. This is self-regulation, a non-legislative definition of regulator. Thus, in this example, two involved parties are acting as regulator.

4.2.4 Owners of sludge or treated biowaste

Ownership of the treated sludge or treated biowaste may change one or more times between production of the material and its final use or disposal. Each of the companies or organisations that take ownership of the material will have an interest in the outcome of the testing programme and should be involved in the definition the objective of the testing programme and the translation of this objective into the Sampling Plan.

Often the project manager is employed by one of these parties, typically the treated sludge or treated biowaste producer, but this does not imply that the producer is the final decision maker.

The companies or organisations taking ownership of the treated sludge or treated biowaste may have conflicting interests, but early discussion may resolve these or at least allow a negotiated compromise, 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 may transfer directly to the manager (landowner or tenant) of the land to which it is to be applied. Alternatively, ownership may transfer to an intermediary organisation acting as a buyer / seller or carrier.

Each owner may 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.

4.2.5 Sampler, laboratory analyst and other executives

The legislator can prescribe procedures to safeguard the quality of sampling. The regulator could make demands on the quality, involvement and responsibilities of other parties like the sampler and laboratory. Other involved parties also may stipulate the quality of the personnel or organisations that conduct the sampling and subsequent analysis of the samples.

These types of demand normally result in demands on certification or accreditation of the companies and / or personnel involved in the testing programme, for example, 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 shall be done by individuals who have been trained in the sampling of treated sludge and treated biowaste and examined under a recognised Sampler Certification scheme.

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

Recognised best practice is for the project manager to select the sampler, laboratory and other executives at an early stage of Sampling Plan development. In many cases, these involved parties 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 make sure that the testing programme is adequate, the reason for investigating the material must be clearly defined: what is the objective of the testing programme?

The objective of the testing programme can consist partly or fully of preconditions defined by international, national, regional or local legislation or regulation. It is important that all involved parties reach agreement about the objective.

Examples of possible objectives of a testing programme are:

- to check the quality because of a change in ownership (is it the type of material expected?);
- to determine the (re)usability of the material;
- to assess the human health and / or environmental risks posed by the material.

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 treated sludge or treated biowaste with quality levels defined in national and international legislation (does the material meet the compliance levels?).

However, this is still not a technical specification that will allow the definition of an unambiguous Sampling Plan. The deduction 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. In order to allow land application, the concentrations of a number of key constituents should comply with the acceptance criteria set out in legislation.

The objective of the testing programme is to define whether the composition of the treated sludge fulfils the acceptance criteria, which are aimed at protecting the environment, i.e. the soil quality and groundwater quality.

4.4 Strategic choices in deriving the technical goals from the objective

4.4.1 General

The purpose for investigating the material is defined by the objective of the testing programme. To investigate the material, samples should be gathered and analysed to produce analytical results that are adequate to satisfy the objective. Therefore the sampling operation should be planned in detail and be appropriate to produce adequate results. The detailed plan and technical specification for the sampling operation are formalised in the Sampling Plan.

In deriving the Sampling Plan, the original objective has to be translated into one or more technical goals. The relation 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' has to be translated into technical goals like 'measure the pH and cadmium content of the treated sludge'. In the Sampling Plan, technical goals (e.g. measurement of the pH) are translated into specific instructions to the sampler. 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 term 'adequate' as used in these technical goals are replaced by actually stating the necessary amount of treated sludge (e.g. 1 kg) and prescription of the type of sample container (e.g. dark glass and air tight).

Note that a testing programme may have more than one objective. In principle, each objective will result in a different Sampling Plan because the technical specifications for the necessary samples and the quality level to be achieved will vary between the different objectives. As a result it might be necessary to define more than one Sampling Plan to fulfil all 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 treated sludge fulfils the acceptance criteria, which are aimed at protecting the environment, e.g. the concentration of certain metals in the sludge. Probabilistic sampling may be more appropriate for this test.

A second objective of the testing programme is to define the nutrient quality of the treated 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 may be sufficient for determining the nutrient content.

The two objectives may require different sampling approaches; therefore two different Sampling Plans will 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.

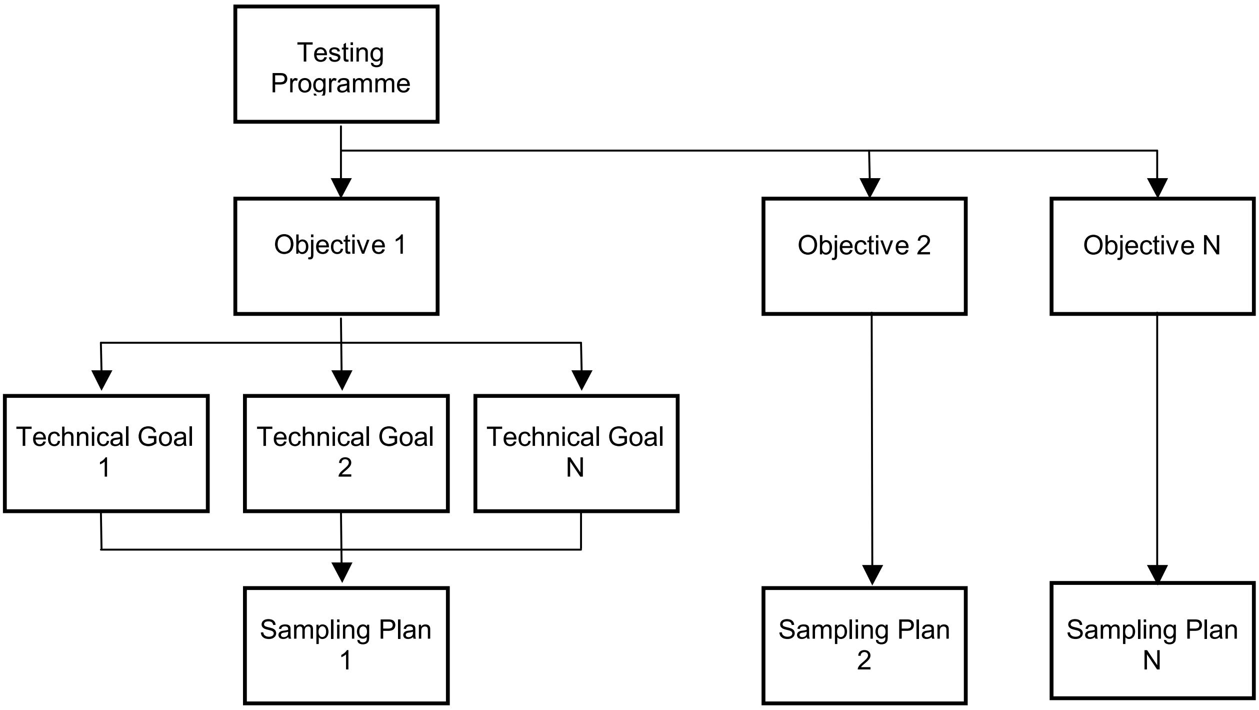

Figure 2 — Translation of objectives into technical goals and instructions in the Sampling Plan

The technical goals are related to the following elements of the Sampling Plan:

— 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.

Not all technical goals need to be discussed with (all) the involved parties. 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.

4.4.2 Selecting constituents to be studied

The selection of constituents starts with an inventory of constituents that are raised in relevant legislation. The constituents identified by legislation are often a reflection of their potential to cause human, environmental and economic risks. Background data on the material may also identify further relevant constituents.

Example: Selection of constituents

Specific quality criteria for treated sludge acceptance for land application are based on strong acid digestion and compliance levels are set for several components: 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 testing results, Cu concentration in the treated 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 and 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 characterisation 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 supplied 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 requirements for 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 that we want to obtain information on by sampling.

In its most simple form, the population is a container, stockpile or lorry of material. In this case identifying the population in terms of space and time is simple. But, where a production process results in a continuous stream of material identifying the population is less straightforward. For example, the population might be the amount of material that is produced in a continuous production process. To define the population in this case, the involved parties must specify the time period of production. The population might thus be defined as the amount of material produced at a certain place in a year, month, week or other period. Additionally, this implies that the part of the material produced outside the specified period also needs to be defined. For this purpose the term ‘overall population’ is used; describing the total quantity of material produced.

Depending on the objective of the testing programme and the available resources, the involved parties will have to make a choice between various options for defining a population. Furthermore, it may be necessary sometimes to divide a population in 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 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.

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. The population can easily be defined in terms of space (the amount of material in a certain location). In this case there is no necessity to use any other term 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[1] 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 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 amount 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.

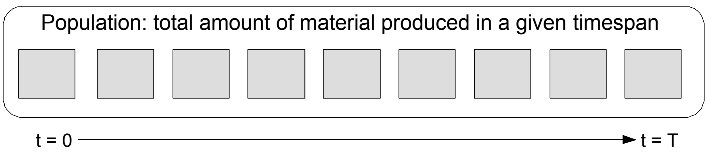

Figure 3 — Continuous production of a homogeneous material

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 may change over time or because of variations in the production process.

[1] 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 paragraphs 4.4.3.3 and 4.4.3.4.

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 organised in such manner that these parts of the material stream can be identified.

Thus the results of the testing programme should give insight in the heterogeneity of the material. To accommodate sampling and to get insight in the heterogeneity within the population, the population has to be divided into several sub-populations. Preferably, the sub-populations are 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, like 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.

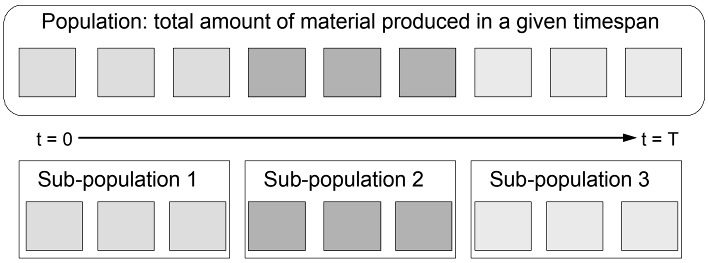

Figure 4— Continuous production of a heterogeneous material: identification of sub-populations from a production perspective

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

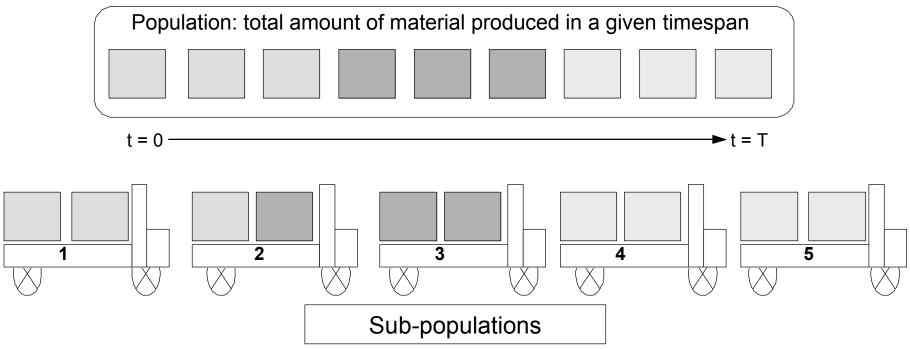

Figure 5— Continuous production of a heterogeneous material: identification of sub-populations from a transport perspective

One may also state 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.

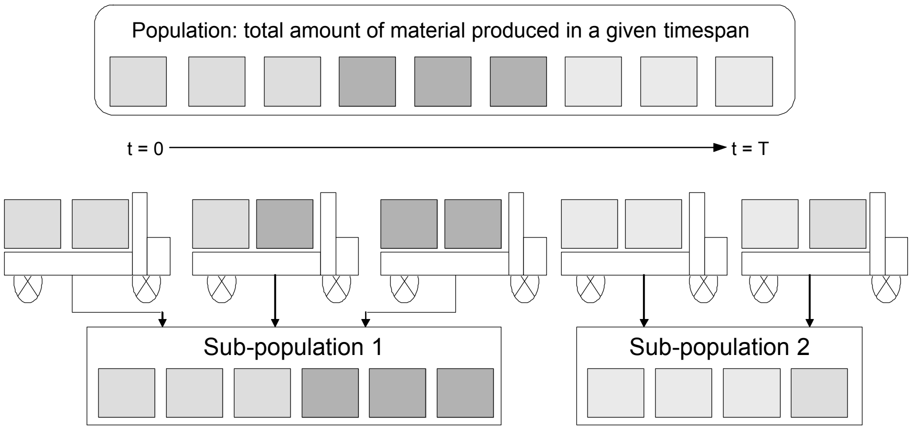

Figure 6 — Continuous production of a heterogeneous material: identification of sub-populations from a destination perspective

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

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

Perspective	**Advantage**	**Disadvantage**
Production perspective	Potentially a clear relation between the sub-population and the production process results in relatively lower costs for the testing programme.	Production process must be known and samples must 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 toxicological perspective.	Variations caused by production, transport and/or mixing of quantities can no longer be identified.

Example: Identification of population and sub-populations

The population is defined as the amount of treated sludge produced in one year (say, 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 say, 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 (or longer period). 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.

4.4.4 Scale

Scale is one of the essential issues of sampling. 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 may 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 may be one year (the whole population) but also one month, week or day, depending in the objective of the testing programme.

Defining the scale is important, as heterogeneity is a scale-dependent characteristic. Let us assume 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 will 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 (thus really 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 relation 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.

Three examples for which the scale is defined are as follows:

- Situation 1 (Figure 7a) describes a population of 2,000 t from which 50 increments are taken randomly. The resulting composite sample is 10 kg.

  Assuming that the composite sample resulting from these 50 increments represents a good estimate of the mean concentration (but not of the variability) of the whole population, the scale for the composite sample in this example is 2,000 t.

  Note that the variability of the population (on the scale of the increments) is fully incorporated in the composite sample; the sampling method will provide no information on the variability.

- Situation 2 (Figure 7b) describes a population of 2,000 t. Within this population – perhaps only for the purpose of sampling – sub-populations are defined of 50 t each. From each sub-population 50 increments are taken. The resulting composite samples are 10 kg, each representing a sub-population.

  The mass represented by each composite sample is now the mass of the individual sub-populations: 50 t. The scale for each composite sample in this example is 50 t. The mean value of all composite samples yields an estimate of the mean concentration of the whole population of 2,000 t and the estimated variability within the whole population is estimated on a scale of 50 t.

- Situation 3 (Figure 7c) describes a population of 2,000 t. More than one composite sample is taken. However, each composite sample (existing of 50 increments) is obtained by taking random increments throughout the whole population. The mass represented by each composite sample is now equal to the mass of the whole population: 2,000 t.

The scale for each composite sample in this example is 2,000 t. The mean value of all composite samples yields an estimate of the mean concentration and the variability of the whole population of 2,000 t is estimated on a scale of 200 g (the mass of the increments).

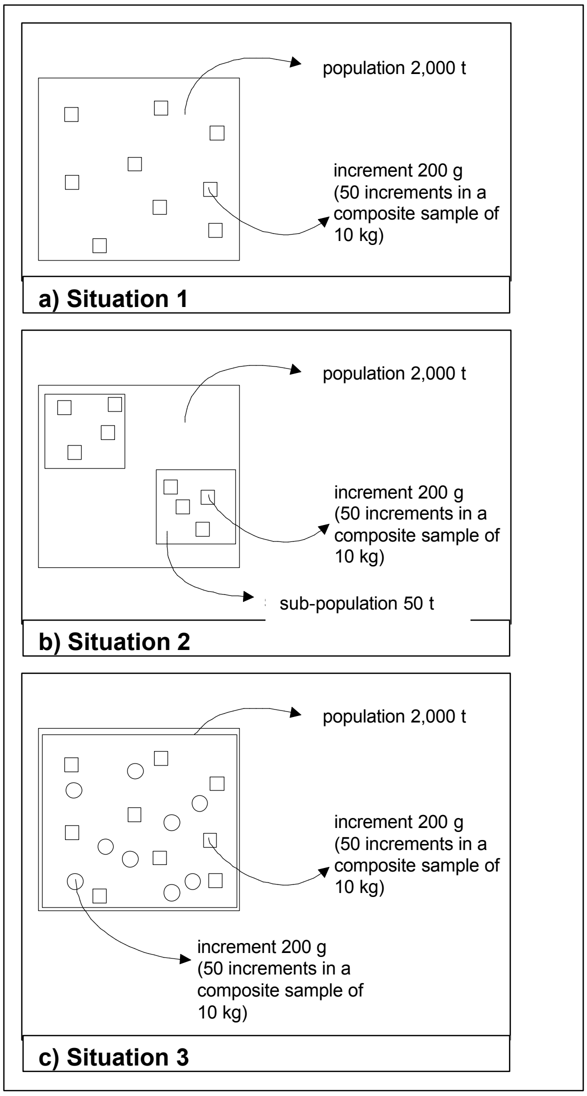

Figure 7 – Examples for which the scale is defined

The following example illustrates the effects of different definitions of the scale of sampling. Depending on the objective of the testing programme, the involved parties must make a choice.

Consider the three sub-populations as shown in Table 2. Each sub-population consists of thirteen individual parts that have a ‘quality’ that is symbolised by a number between 0 and 99. Heterogeneity is quantified by the coefficient of variation: a high coefficient of variation indicates a high heterogeneity.

When the scale of sampling is equal to the size of the sub-population, the sampling result will only be an estimate of the mean concentration for each sub-population. Comparing the sub-populations in Table 2 sub-population 1 and 2 are comparable while sub-population 3 has a higher mean.

When the scale of sampling is equal to the individual parts within each sub-population, we obtain not only an estimate for the mean concentration of the sub-population, but also an estimate for the heterogeneity within that sub-population. Comparing the sub-populations shown in Table 2 now still gives the same result for the mean of the whole sub-population, but additionally we discover that sub-population 2 has a higher degree of variability than sub-populations 1 and 3.

Table 2— Example of three different sub-populations, characterised on the individual samples, the mean and coefficient of variation (CV).

NOTE A high CV indicates a heterogeneous sample.

	Sub-pop. 1	Sub-pop. 2	Sub-pop. 3	
	20	15	32	
	30	14	36	
	20	22	3	
	30	72	37	
	40	9	38	
	20	23	36	
	30	64	37	
	30	46	30	
	40	5	40	
	20	16	41	
	10	2	17	
	20	17	39	
	30	35	36	
				Population
Mean	26.2	26.2	32.5	28.3
Coefficient of variation	33.3%	84.2%	33.2%	

Finally, when the scale of sampling is equal to the total population we obtain only an estimate of the mean for the whole population.

Different choices can now be made on the scale of sampling:

- The scale of sampling is equal to the scale of the individual parts. It is not possible to define a smaller scale of sampling. The result of this definition of the scale is that information on the heterogeneity within the sub-populations can be obtained by calculating (for example) the coefficient of variation. Additionally, the heterogeneity between the sub-populations and within the population can be calculated. In this approach, the presumptions that led to identification of the sub-population as a relatively homogeneous part of the population can be verified. For example, it may be argued that sub-population 2 in Table 2 is so heterogeneous that at least a part of sub-population 2 will not comply with certain quality standards, although the mean value is within the quality range. Many sub-populations of high heterogeneity may lead to a re-evaluation of the Sampling Plan. An important disadvantage to consider are the costs for measuring the individual parts, in this case thirteen per sub-population[2].

- The scale of sampling is equal to the scale of the sub-populations. Therefore no information on individual parts within a sub-population is gathered. Characterisation of the sub-population is done by means of a composite sample per sub-population in which more than one of the individual items are put together prior to analysis. If this composite sample is taken and analysed correctly, the result of the composite sample

2 Note that it is not necessary (nor practical) to measure each individual item within a sub-population. A sample survey within each sub-population might be sufficient.

will be a good estimate of the true mean of the sub-population. An important advantage of this approach is the low costs for measuring. An important disadvantage is the assumption that a composite sample can be obtained without a considerable sampling error. The analysis of a composite sample might pose problems as the amount of material in the sample will be (much) larger than the amount of material needed for the analysis and thus proper sample pre-treatment is necessary to obtain a representative analytical sample from a – potentially – highly heterogeneous composite sample. Additionally, there will be no information available on the heterogeneity within a sub-population.

- The scale of sampling is equal to the scale of the population. In the example (Table 2) the population is defined as the combination of the three sub-populations. Individual parts are gathered from the involved sub-populations and put together in a composite sample. Now there will be no information available on a smaller scale than the scale of the population. An important advantage is the (very) low costs for measuring, while, as long as it is technically possible to mix a large number of these parts, the result of the composite sample will still be representative for the true mean of the total population. But the population has to be treated as one entity. In case of a heterogeneous population (for example sub-population 2 in Table 2) sampling on the scale of sub-populations or individual parts would have given the involved parties information that may have led to different choices for the destination of sub-populations of different quality.

Given the relation 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 desired, the possible destination, the financial means available and the technical possibilities of working with composite samples.

Example: Scale of sampling

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 impossible to mix this many increments without unacceptable error, an alternative approach must be taken. 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.

In addition to the more technical perspective from which the definition of scale was described in the previous text, 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

When a new biowaste composting facility is built there is validation phase during which there is a requirement that every batch of compost, say, 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, say 6 months, or a given volume, say 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; say 5000 m^3 compost or 6 month's production.

4.4.5 Choosing the desired reliability of sampling results

4.4.5.1 General

Treated sludge and treated biowaste are heterogeneous materials for several reasons:

- 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.

The fact that the materials can be heterogeneous has serious consequences for the Sampling Plan. 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 considered 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. Two types of sampling error will influence the representativeness of the sampling results: the systematic error[3] and the random error.

Due to the fact that one or both of 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 can have serious social, economical or environmental consequences, the reliability of the sampling results often needs to be high; although this does not imply that all sampling in these circumstances should be such. 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 composition 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 us to specify the reliability of the estimate and the chances of an incorrect assessment based on the sampling results. 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 (distance between upper and lower confidence limit) the better the sampling mean estimates the true mean value of the population.

The size of the confidence interval is related to (see TR xxxx-1 clause 7):

- 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. 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 8).

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[3] All activities that are necessary to obtain the analytical results are sources of variability. As long as the measurement in itself is correct, but the results vary due to coincidence, the errors are considered as random error. However, if, for example, the results are per definition too high or too low due to the applied measurement technique, the errors are known as systematic errors. Although limiting the systematic error is essential for a correct assessment of the waste material, the prevention of systematic errors falls outside the scope of this Technical Report. It is a responsibility of the project manager.

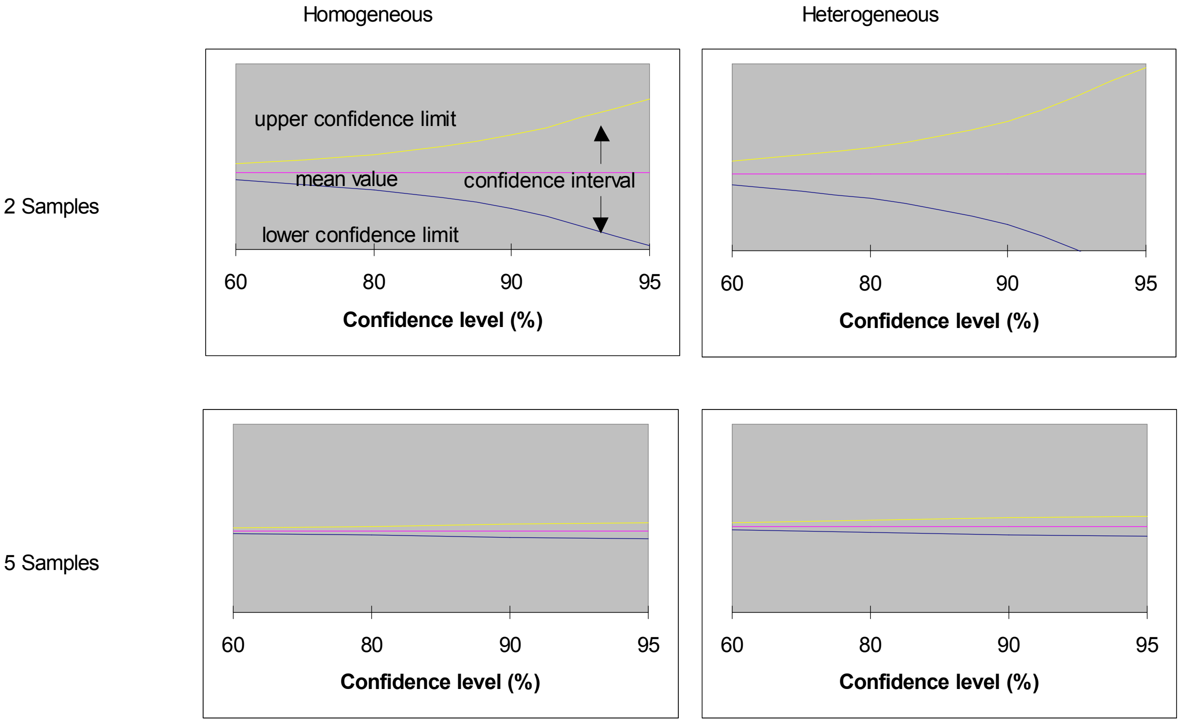

Figure 8 — The confidence interval increases with the desired confidence level and the heterogeneity of the population. The confidence interval decreases when more samples are taken. The narrower the confidence interval, the better the estimate of the mean represents the true mean of the population.

The involved parties influence the costs of the testing programme because the amount of samples is directly determined by the desired reliability that they define. The advice of statistical experts and additional research may be necessary to quantify the relationship between heterogeneity of the material, the reliability of the estimate and the amount 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 8). 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 joined in a composite sample (4.4.5.3);
- increasing the scale of sampling (4.4.4).

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 comply with that need. Two principally different types of sampling are distinguished: probabilistic sampling and judgemental sampling (see TR xxxx-1 clause 5.4).

The essential difference between probabilistic sampling and judgemental sampling is that in probabilistic sampling each individual part of the material to be sampled has an equal chance of being sampled. While in judgemental sampling part of the population will not be considered during sampling. As a consequence the samples obtained by judgemental sampling can never be seen as (fully) representative of the whole population.

When the objective of the testing programme is to determine the type of substance in a material which obviously differs from the rest of the material, spot sampling is often the most appropriate sampling method. This is a specific type of judgemental sampling; only the parts that appear to be different are considered for sampling. In most other cases probabilistic sampling should be considered first and should only be replaced by judgemental sampling when there are good arguments for judgemental sampling. Still, the type of judgmental sampling should be as close to probabilistic sampling as possible in order to assure some degree of representativeness of the samples.

4.4.5.3 Many Field Samples versus many increments joined in a composite sample

When taking a large number[4] of field samples, the cost of analysis for all these samples will be high in relation to the costs of sampling. On the other hand, when these increments are mixed in a composite sample, the variability of the sampled material will effectively be summed within the composite sample. In this case, the amount of samples and the resulting costs of analysis are relatively low, but more effort (and thus costs) 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 a good 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.

[4] Not only the number but also the size of samples or increments influences the reliability. The minimum sample size can be calculated (see TR xxxx-1 clause 6.3).

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

The involved parties did some preliminary research on the amount of samples that is 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 for the project manager

In the interactive process of deriving technical goals from the objective, the involved parties must 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 paragraph contains a list of questions that helps the project manager (and the involved parties) through the process of defining the Sampling Plan[5].

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?

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

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

week to a production manager and the quality of this week's production (=sub-population) must be known.

— Identifiable parts of the population are expected to be significantly different from other parts of the population.

Question: Is the scale of sampling defined?

In some cases, the scale of sampling is stated explicitly in legislation (see example: Policy defined scale of sampling). 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, the scale of this information should be known. 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; see 4.4.4.

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) field samples: Good estimate of heterogeneity within the population (or sub-population). Mean value of the population can be calculated. Reliability depends on the number of field samples.

— 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.

4.6 Further steps to be taken by the project manager

After the identification of the involved parties, the identification of the objective and translation of objective into technical goals, the project manager can make the Sampling Plan. As the policy related decisions are now made, the remaining aspects are purely technical and procedural. They include:

— The statistical parameter to be determined.

— The sample size.

— What sampling technique is adequate?

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

Annex A
(informative)

Validation of method

Annex B
(informative)

The modular horizontal system

Annex C
(informative)

Information on WP 2 Sampling and the project HORIZONTAL

Bibliography

[1] IUPAC (1990) Nomenclature for sampling in analytical chemistry. Pure Appl. Chem, 62: 1193-1208.

[2]