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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 this 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 soil needs to be assessed to fulfil the demands of sludge-use regulation’) into a technical instruction that corresponds with that same objective (e.g. ‘the mean concentration of each agricultural unit 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:

— <u>Policy makers involved in sampling</u>. 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

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 comply with limits set out in legislation. Therefore the soil must be sampled to establish baseline concentrations.

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 comply with limits set out in legislation. Therefore the soil must 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 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 objective of sampling soils in areas used for agriculture (arable and pasture sites), to either:

— establish the baseline conditions prior to application of treated sewage sludge or biowaste;

— monitor the effect of repeated applications of treated sewage sludge or biowaste;

This Technical Report gives instructions on the various aspects that in total describe the sampling activity:

- definition of a sampling plan;
- choice of an adequate sampling strategy;
- sampling technique to be applied;
- sub-sampling directly after sampling (when necessary and appropriate);
- packing, preservation, storing, transport and delivery of the sample.

This Technical Report provides guidance on how to obtain clear and simple instructions for the sampling personnel.

NOTE The document provides guidance 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

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

3 Terms and definitions

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

3.1
analytical laboratory
The identified laboratory which is to undertake the chemical, biological or physical analysis of samples.

3.2
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.

3.3
basic characterisation
sampling that has the goal to describe the character or quality of a population of material

3.4
characteristic
a property, which helps to identify or differentiate between items of a given population.

[ISO 3534-1:1993, definition 2.2]

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

3.5
compliance testing
the process of testing whether sample values meet a pre-defined set of criteria

3.6
composite sample
two or more increments/sub-samples mixed together in appropriate proportions, either discretely or continuously (blended composite sample), from which the average value of a desired characteristic may be obtained.

[ISO 11074-2:1998, definition 3.10]

3.7
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.

3.8
confidence level
the value 100(1-α) of the percentage probability associated with a confidence interval

[after ISO 3435-1]

3.9
constituent
property or attribute of a material that is measured, compared or noted

3.10
core sample
type of sample, more specifically related to the sampling of solids where augers and other core samplers are used. A vertical or directional sample is taken through the material whereby the integrity of the batch is maintained.

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

3.11
decision maker
the party that makes a decision based on the results of the testing programme

3.12
delivery
Transfer of custody of the sample.

3.13
field sample
The quantity (mass or volume) of material obtained through sampling without any sub-sampling.

3.14
heterogeneity
degree to which a constituent (3.3) is not uniformly distributed throughout a quantity of material.

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

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

3.15
homogeneity
degree to which a constituent (3.5) is uniformly distributed throughout a quantity of material.

3.16
increment
individual portion of material collected by a single operation of a sampling device which will not be analysed/ investigated as a single entity, but will be mixed with other increments in a compose sample

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

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

3.17
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.

3.18
judgemental sampling
samples collected using at best a partially-probabilistic procedure and at worst a non-probabilistic approach

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

3.19
laboratory analyst
the person conducting the analysis of the laboratory sample

3.20
laboratory sample
the sample(s) or sub-sample(s) sent to or received by the laboratory.

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

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

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

3.21
legislator
the body responsible for the definition of the rules that should be obeyed

3.22
mixing
Combining of components, particles or layers into a more homogeneous state.

[ISO 11074-2:1998, definition 6.2]

3.23
objective
the underlying motivation for investigating the material

3.24
on site verification
a normally simple test to evaluate if the involved material is indeed the type of material expected

3.25
packaging
Act of placing a sample into an appropriate sample container for transport and/or storage.

[after ISO 11074-2:1998, definition 4.27]

3.26
policy maker
person working for the central, regional or local authority, government or administration, the management of a company

3.27
population
totality of items under consideration

[ISO 3534-1:1993]

3.28
portion
Each of the discrete, identifiable portions of a material suitable for removal from a population as a sample or as a portion of a sample, and which can be individually considered, examined, tested or combined.

[ISO 11074-2:1998, definition 1.2]

3.29
probabilistic sampling
sampling conducted according to the statistical principles of sampling

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

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

3.30
project manager
the person who is responsible for deriving and / or fulfilling the testing programme

3.31
regulator
the body responsible for controlling if the rules of the legislator are met

3.32
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.

3.33
representative sample
sample in which the characteristic(s) of interest is (are) present with a reliability appropriate for the purposes of the testing programme

3.34
sample
portion of material selected from a larger quantity of material

[ISO 11074-2:1998]

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.

3.35
sample preservation
Any procedure used to stabilise a sample in such a way that the properties under examination are maintained stable from the collection step until preparation for analysis.

[ISO 11074-2:1998, definition 4.29]

3.36
sample storage
Process and the result of keeping a sample available under predefined conditions for a usually specified time interval between collection and further treatment of the sample.

[after ISO 11074-2:1998, definition 4.31]

3.37
sample transportation
Act of transferring a sample from the locality of sampling to the place of subsequent treatment (e.g. laboratory, soil-specimen bank etc.).

[ISO 11074-2:1998, definition 4.30]

3.38
sampler
person carrying out the sampling procedures at the sampling locality.

[ISO 11074-2:1998, definition 1.5]

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

3.39
sampling plan
all the information pertinent to a particular sampling activity

NOTE Predetermined procedure for the selection, withdrawal, preservation, transportation and preparation of the portions to be removed from a population as a sample. [ISO 11074-2:1998]

3.40
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.

3.41
seal (tamper-indicating device)
A device or material designed to provide evidence of unauthorized access.

NOTE 1 A seal is fixed to the container such that the seal must be broken to gain access to the contents.

NOTE 2 A seal does not need to provide resistance to entry; it need only record that it took place.

3.42
stratified sampling
in a population which can be divided into mutually exclusive and exhaustive sub-populations (i.e. strata), sampling carried out in such a way that specified proportions of the sample are drawn from the different strata and each stratum is sampled with at least one sampling unit.

[ISO 3534-1:1993, definition 4.14]

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

3.43
sub-population
a defined part of the population that will be targeted for the purposes of sampling.

3.44
sub-sample
The quantity (mass or volume) of material obtained by procedures in which the characteristics of interest are randomly distributed in parts of equal or unequal size.

NOTE 1 A sub-sample may be:

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

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

c) the final unit of multi-stage sampling.

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

3.45
sub-sampling (sample division)
Process of selecting one or more sub-samples from a sample of population.

[ISO 11074-2:1998, definition 6.3]

3.46
technical goals
objective translated into specific, measurable, action oriented, realistic, timely (SMART) goals

3.47
test sample (analytical sample)
sample, prepared from the laboratory sample, from which the test portions are removed for testing or for analysis.

[ISO 11074-2:1998, definition 3.16]

3.48
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

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.

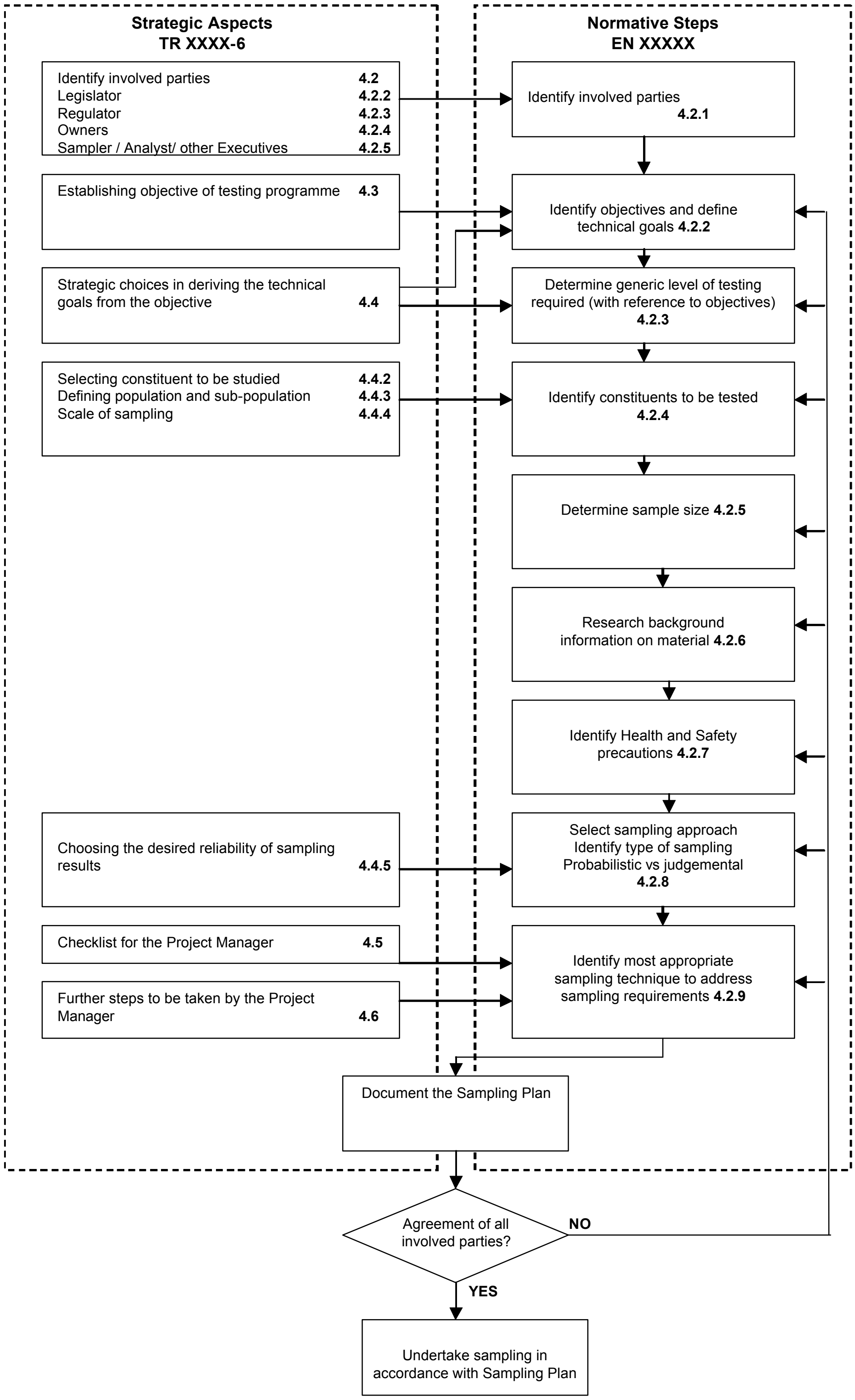

Figure 1 — The process of defining the Sampling Plan, providing information on the elements that are specified in the European Standard and this Technical Report

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 prescribes that the soil should be analysed prior to application of sewage sludge to land, and at intervals thereafter, to ensure that the concentrations of a number of key constituents in the soil do not exceed 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 land owner or user, 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 when desired that the soil complies with the regulatory limits. 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 sewage sludge is to be applied to an area of land for the first time.

Example: multiple parties involved as regulator

Testing of the soil at specified intervals is a requirement governed by legislation and regulated by the formal regulator. Testing is done by the treated sludge or treated biowaste producer and the results of that testing are delivered to the landowner and the regulator.

In addition to the tests that are required by legislation, the producer may choose to test the soil at more frequent intervals to check that it complies with the required limit values. 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

The owner of the land may not 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 may change one or more times after permission is given for treated sludge to be applied.

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 soil 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 soil.

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 establish baseline conditions prior to an activity which might affect the composition or quality of soil;
- to monitor the affect 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 metal content of the soil (to establish if sludge can be applied);
- to compare the quality of the soil with quality levels defined in national and international legislation (does the soil continue to 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 produced at a waste water treatment works has been tested and confirmed to meet the required quality criteria for land application. In order to allow land application, the concentrations in the soil 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 soil fulfils the acceptance criteria for sludge application, 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 soil with quality levels as defined in legislation' has to be translated into technical goals like 'measure the pH and cadmium content of the soil'. 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 soil 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 soil (e.g. 1 kg), the number and size of increment samples and sub-sampling method, 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 soil fulfils the acceptance criteria, which are aimed at protecting the environment, e.g. the concentration of certain metals in the soil. Probabilistic sampling may be more appropriate for this test.

A second objective of the testing programme is to define the nutrient status of the soil, which is aimed at assessing the potential benefit to agriculture (or ecological improvement) and the application rate, based on 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.

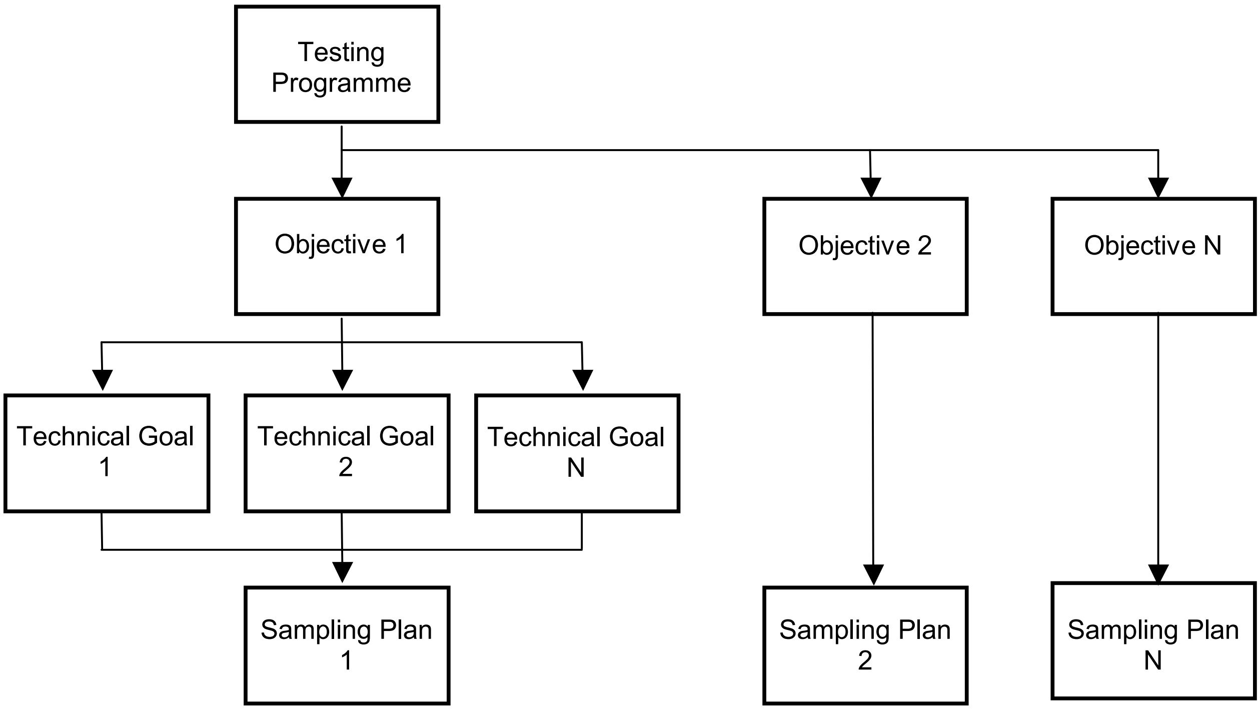

Figure 2 — Translation of objectives into technical goals and instructions in the Sampling Plan

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 sampling site may also identify further relevant constituents.

Example: Selection of constituents

Specific quality criteria for acceptance for land application are based on strong acid digestion of soil samples 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 will exceed the compliance level.

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 an area of land to which treated sludge is to be applied and identifying the population in terms of time (when sludge is to be applied) and space (where it is to be applied) is simple.

In practice, the area of land to which sludge is to be applied may 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 may 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; describing the total area of land to be sampled.

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 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).

Example: Identification of population and sub-populations

Legislation dictates that land that is farmed for a different purpose must 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 applied 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 acres.

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

Because each field is more than 5 hectares, five sub-populations have 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. 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.

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 between individual sample results will be higher for a smaller scale of sampling and will be lower for a larger scale of sampling.

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 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 sample from each 10x10 m area (100 m^2) resulting in 500 samples per 5 hectare area. If each of the 500 samples are analysed, the scale of sampling is 0.01 hectares. If the 500 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 must be taken. One approach could be to take a sample from each 50x50 m area (2500 m^2) resulting in 20 samples per 5 hectare area. In this case, if the each of the 20 samples are analysed, the scale of sampling is 0.25 hectares. However, if the 20 samples 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 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

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 samples 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 divisions (sub-population).

4.4.5 Choosing the desired reliability of sampling results

4.4.5.1 General

Soils are heterogeneous materials for several reasons, including:

- Variability in underlying geology;
- Variability in topography;
- Variability in climate;
- Variability in land use and type of cultivation.

The fact that soil 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[1] 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.

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1 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.

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 3).

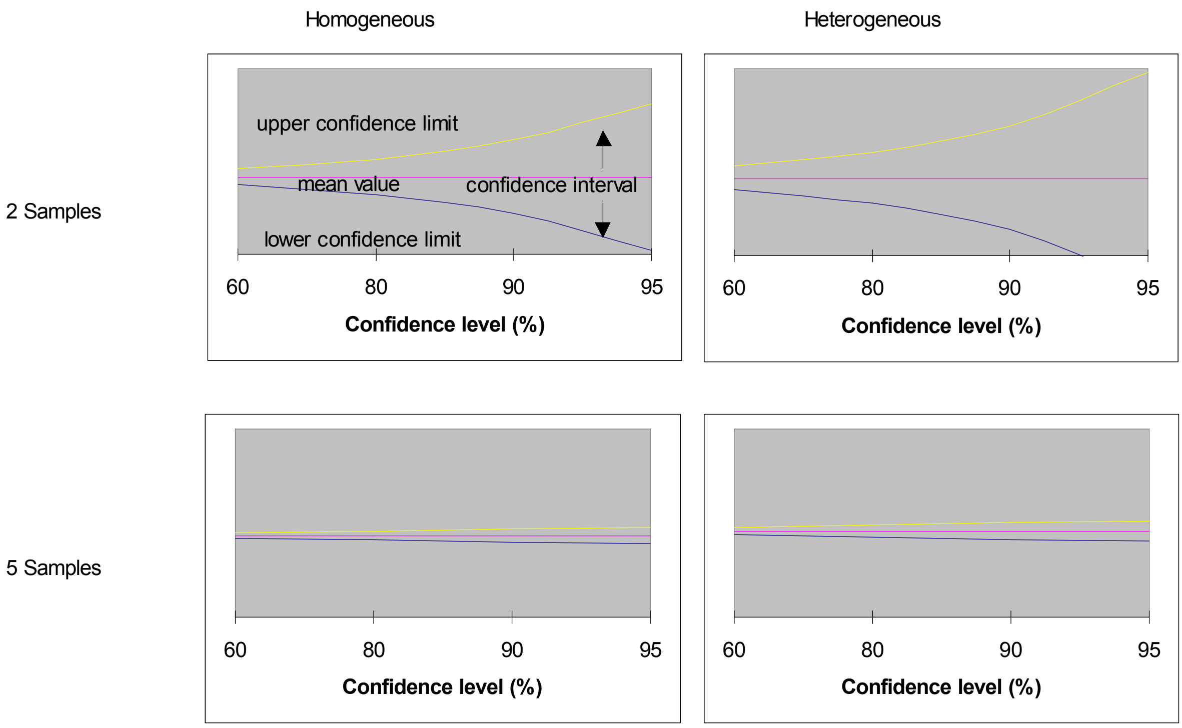

Figure 3 — The confidence interval increases with the desired confidence level and the heterogeneity of the population. The confidence interval decreases when more samples are taken. The narrower the confidence interval, the better the estimate of the mean represents the true mean of the population.

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 3). 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.

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

When taking a large number[2] 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.

[2] 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[3].

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 have been treated differently. For example, where the area to be tested comprises smaller areas that are farmed for different purposes.

3 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.

— 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 (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 (mixing and sub-sampling).

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.

5 Determine the practical instructions

5.1 General

Previous clauses have discussed the steps needed to develop the objective of the Sampling Plan into a number of more detailed goals. The technical goals must now be translated into practical instructions that are given to the sampler prior to sampling. The practical issues that should be considered in identifying these instructions are as follows:

— Choose the sampling pattern;

— Determine the minimum increment and sample size;

— The use of composite versus individual samples;

— Determine the required number of increments and samples.

5.2 Sampling pattern

The sampling pattern defines where, when and how the required samples are selected from the population. There are two fundamental sampling strategies: probabilistic sampling and judgemental sampling. Non-systematic patterns are often used in agricultural / horticultural land investigations. Three probabilistic sampling patterns and two options for judgemental sampling are illustrated in Figure 4.

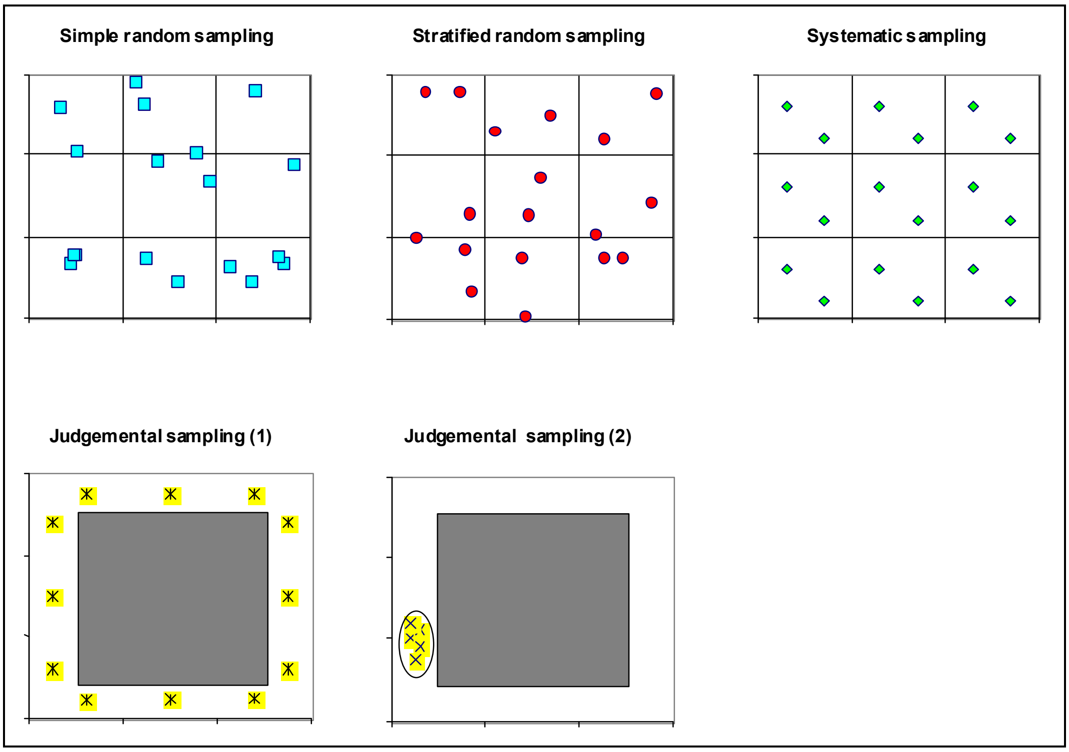

Figure 4 — Possible patterns of sampling

NOTE Figure 4 illustrates the patterns for the context of a two-dimensional spatial area. However, the concepts apply as equally to temporal as they do to spatial components of variability.

5.2.1 Probabilistic sampling

5.2.1.1 Simple random sampling

With simple random sampling, every portion of the population has the same (small) chance of being selected as a sample. However, the resulting samples will not necessarily be very evenly spread across the population. Consequently other more structured forms of sampling are often preferred to simple random sampling.

5.2.1.2 Stratified random sampling

With stratified random sampling, specified numbers of samples are spread randomly over each of a number of strata that are predefined in the population. This preserves the advantages of random sampling (that is, every portion of the population has a known chance of being selected as a sample), whilst ensuring that each stratum is represented by a predetermined number of samples. Where the number of samples in each stratum is proportional to the proportion of the population falling into that stratum, the sampling is termed 'self-weighting'. Often, however, there are advantages in having equal numbers of samples in each stratum, and subsequently weighting the results by the estimated stratum sizes in the population.

5.2.1.3 Systematic sampling

With systematic sampling the samples are evenly spaced across the population, starting from a randomly chosen point (to ensure that each item in the population has an equal chance of being sampled, to fulfil the requirements of probabilistic sampling). This has obvious operational advantages. For the benefits of probabilistic sampling still to apply, however, the approach does rely on the assumption that there are no systematic components of variation within the population that 'run in step with' the chosen sampling frequency.

Systematic sampling should therefore be applied with care when it is used in place of a random or stratified random sampling.

5.2.2 Judgemental sampling

Judgemental sampling can embrace a wide variety of sampling patterns that essentially differ in terms of how far they deviate from a truly probabilistic approach. Judgemental sampling provides no information about the soil except in the immediate vicinity of the samples, and so nothing can reliably be inferred about the quality of the sub-population.

Option (1) in Figure 4 shows a form of judgemental sampling that is based on a probabilistic approach for part of the population. The sampled sub-population is the narrow strip around the shaded region. In contrast, the pattern in Option (2) is fully judgemental.

5.2.3 Non-systematic patterns (irregular sampling)

Non-systematic sampling patterns (see Annex A) are widely used in agricultural / horticultural land investigation. The general premise is that if sub-populations are defined based on topography, soil type and land use, then the distribution of soil constituents is relatively homogeneous. Samples are collected at intervals along the outline of a 'N', 'S', 'W' or 'X' pattern and mixed to provide a laboratory sample.

5.3 Determine the increment and sample size

An <u>increment</u> is the amount of material (mass or volume) that is obtained through one single sampling action. It is not analysed as an individual unit, but is combined with other increments to form a composite sample. Conversely a <u>sample</u> is an increment that does get analysed individually.

The degree to which we have to take account of the increment and sample size will very much depend on the type of material sampled. The <u>minimum</u> increment size is governed by the need for the sampling device to accommodate all particle sizes. Thus it has particular consequences for the sampling of particulate materials. In contrast, there is no practical requirement for a minimum increment size in the sampling of liquids, where the particle size goes down to the molecular scale.

A sample should be sufficiently large in order to minimise or exclude errors caused by the fundamental variability of the material that is determined by the differences between individual particles.

The terms fundamental variability and heterogeneity due to 'clustering' should not be confused. The latter relates to the preferential presence of a specific type of material to be in a specific part of the population, and can be dealt with by the sampling pattern. Fundamental variability, however, should be overcome by putting a demand on the sample size and hence the number of particles in a sample.

Further detail on determining the increment and sample size is given in TR XXXX-1.

5.4 Health and Safety

Sampling in an agricultural area involves an increased risk to the sampler because the nature of the ground and possible hazards are not necessarily known to the sampler. Physical injury is possible in any sampling situation where machinery is being used; this applies to agricultural sampling as much as to contaminated-site investigations.

Detailed guidance on safety during soil sampling is given in ISO 10381-3.

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

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

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

5.5 Sample collection

5.5.1 Choosing the sampling equipment

Choose sampling equipment appropriate to the type of soil being sampled, the sampling location and the size of sample to be collected. Guidance on selection and descriptions of equipment commonly used for manual sampling are provided in Annex B. Equivalent vehicle-mounted equipment is available.

The type of sample required should be taken into account when choosing sampling equipment, including:

— disturbed samples (mass-proportional sampling, samples obtained without any attempt to preserve the soil structure);

— undisturbed samples (volume proportional sampling, samples obtained using a method designed to preserve the soil structure, samples for VOC analysis).

Prior to use all apparatus and tools should be cleaned to reduce the risk of cross-contamination.

Where more than one site is to be sampled sampling equipment should be cleaned between sites. Unless composite samples are being collected it is advised that equipment is cleaned between samples.

Where it is not possible to clean the sampling equipment as specified in the Sampling Plan this should be recorded in the sampling record.

5.5.2 Taking a sample

Only sampling personnel with appropriate training should be used.

For all topsoil sampling it is important that the whole of the sampling depth is equally represented by each replicate core. Therefore if, on retrieval, part of the core is missing, i.e. the bottom part is left in the ground (this often happens in dry or stony soils) or the top part falls off (because it is dry), discard this core and attempt to take a replacement close by.

Do include the top few centimetres of soil. There is often a different pH and/or concentration of soil nutrients (particularly phosphorus) at the surface in grass land and direct drilled/shallow cultivated fields. It is therefore essential that the top few centimetres are included in the sample.

Take care when taking soil samples. It can be difficult to remove the soil from some push samplers.

Several incremental samples may be combined to produce a composite sample that represents better the population that was sampled. To form a composite sample per area unit, sampling procedures should be selected so that the final sample contains equal parts of the incremental samples, and to ensure that it is representative of all the incremental samples.

Composite samples representative of the media do not require intact undisturbed samples to be collected.

After sampling, large holes should be backfilled to avoid any risk to animals.

5.6 Sub-sampling in the field

5.6.1 General

Combining increment samples may produce a composite sample that is much larger than required for a laboratory sample. In general, it is recommended that sub-sampling, to reduce the mass or volume of field samples to obtain laboratory samples, should be avoided in the field. Instead, the increment samples should be returned to the laboratory for sub-sampling.

If this is impossible, the combined sample should be thoroughly mixed and a representative sub-sample taken to produce one or more laboratory samples.

NOTE 1 When several analyses are to be carried out on a sample and the analyses require conflicting preservation methods, then the field sample will require sub-sampling to produce more than one laboratory sample.

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

Detailed guidance on sub-sampling in the field can be found in TR XXXX-3.

5.7 Sample packaging, storage, preservation, transport and delivery

5.7.1 General

Every effort should be made to ensure that any changes to sample between collection and arrival at the laboratory are minimised. Detailed guidance on sample packaging, storage, preservation, transport and delivery can be found in TR XXXX-4.

5.7.2 Sample containers

The Sampling Plan should state the sample container requirements, including container type, size and shape, cleaning, closure, seal and any special filling requirements, e.g. headspace.

The purpose of the sample container is to protect the sample during transport and storage until it is further treated or analysed. A container should be compatible with the nature of the material sampled and the components to be analysed. In general:

— collect samples for inorganic analysis in plastic containers;

— collect samples for organic analysis in glass containers;

— select a sample container having a size relative to the volume of the required sample;

— select a container capable of being sealed.

Where specialised containers are advised, analytical laboratories should be encouraged to provide containers that conform to the characteristics of the analytical procedure to be used.

5.7.3 Sample preservation

The Sampling Plan should state any preservation and handling procedures.

It is seldom possible to analyses samples immediately after collection. For this reason preservation techniques are required in the field, during transport to the laboratory and during storage before analysis.

As a minimum, it is recommended that samples are transported in containers that minimise loss of water by evaporation, in the dark and are kept cool (4±2 °C).

5.7.4 Packaging and labelling

The Sampling Plan should state the labelling requirements.

Each sample container should be labelled with a clearly legible, unambiguous code either:

a) by writing directly on the container using a permanent marker pen; or

b) by writing on an adhesive label and sticking it to the sample container.

The outside of all sample containers should be wiped clean prior to labelling.

5.7.5 Sample transport

The Sampling Plan should state the transportation method, storage requirements and minimum delivery time.

Samples should be transported without delay. During transportation, the samples should be kept as cool as practicable and protected from light.

5.8 Sampling Record

The sampling report should contain, in addition to information on the sampling location, personnel, observations and sample identification, a proper description of the sampling method and sampling devices used. If the actual sampling procedure differed from that originally planned, this also should be reported, including the reasons for that change.

The sampling report prepared by the field staff should contain the following details:

- sample designation and number (identical to the marking in the sample container);
- date of sampling;
- information on the site (e.g. location, land use, textural class, weather conditions);
- description of the soil profile in special cases;
- information on the procedure (field pattern, sampling equipment, depth of sampling, number of increments or composite samples, etc.);
- information on storage and transport;
- information on the time and place of delivery to the laboratory;
- identification of the sampler;
- counter-signature of customer or programme supervisor;
- confirmation of receipt by the laboratory.

Annex A
(informative)

Non-systematic patterns (irregular sampling)

A.1 Agricultural / horticultural land investigation

Widely used in agricultural/horticultural land investigation are the "N", "S", "W" and "X" patterns of sampling (Figure A.1). The general premise is that the distribution of soil constituents is relatively homogeneous. The patterns used are simplifications of the stratified random sampling method (see TR XXXX-1). Along the outline of such a pattern, a number of samples are taken and then may be bulked and mixed to provide one sample for analysis.

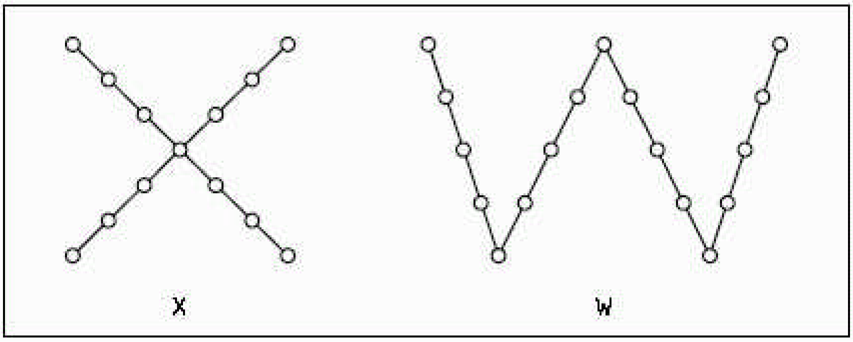

Figure A.1 — Non-systematic patterns [ISO 10381-1:2002]

Wherever there are likely to be differences in soil type or conditions, crop growth, plant species, previous cultivation etc., the site should be subdivided according to these differences and a separate sample taken from each area.

Sampling along a single diagonal of a field or a unit is only recommended in case of strip-like distribution of contaminants on agricultural areas due to application of fertilizers. Applying a diagonal for sampling avoids, by simple and effective means, systematic bias which would arise with strip-parallel sampling. However, a greater number of diagonals is to be preferred. Two diagonals (X-shape) introduce a serious bias to the central area of the field (Figure A.1). This should be considered in the evaluation of the results of the determinations.

Application of diagonal patterns should be based on the following:

— estimation of uniform distribution of substances;

— useful only for uniformly developed areas. Deviating parts of the area should be sampled separately;

— application of more than one diagonal is recommended (e.g. parallel or X-shape);

— distance of sampling points is equal for each diagonal, i.e. shorter diagonals have fewer sampling points;

— selection of sampling point is independent of local characteristics. Points are fixed by pacing preferably.

Traversing the area in a zigzag manner similar to that shown in Figure A.2 is another way of applying a non-systematic pattern.

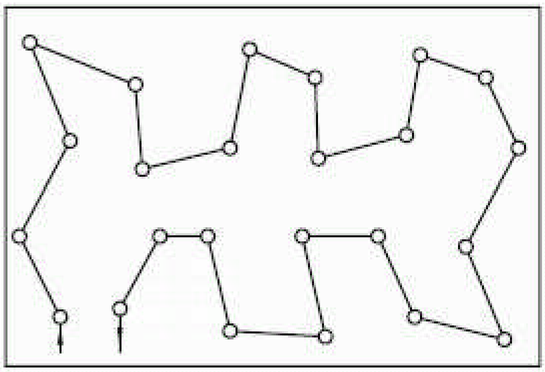

Figure A.2 — Zigzag traverse sampling pattern [ISO 10381-1:2002]

Annex B
(informative)

Support on the selection of sampling equipment

B.1 General

Soil samples may be collected using a variety of equipment, depending on the depth of the desired sample, the type of sample required (disturbed or undisturbed) and the soil type.

Correct sampling requires that an appropriate sampling device be used. Sampling is correct when the probability of collecting all fractions of the soil through the full depth of the sample is equal. It is necessary that the sampling device traverses the entire depth of soil that is to be sampled and extracts an unbiased sample. The optimum device should yield a sample where any biased sample may be easily identified and whose depth can be determined.

In general, sampling equipment is most practical if it is as simple as possible in design and construction. Tools should be chosen to suit the sampling activity. All sampling equipment should preserve the characteristics of the material being sampled. Sampling apparatus and tools should be kept clean and corrosion free.

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

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

EXAMPLE 1 High alloy steels should be avoided if trace metals are to be determined.

EXAMPLE 2 The use of stainless steel tools is routinely adopted, but may be a source of contamination if analyses for elements such as chromium are to be performed.

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

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

B.2 Equipment selection

Factors to be considered when choosing sampling equipment are:

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

Information should be sought on:

a) The characteristics of the material to be sampled:

- the moisture content;
- the consistency;
- the physical structure;
- particle size distribution.

b) Accessibility and safety:

- Sampling location;
- the accessibility of sampling points;
- the hazard assessment of the sampling activities;
- the safety procedures to be implemented during sampling.

c) The size and purpose of the sample:

- the final quantity of sample required;
- how the sample will be analysed (i.e. physical, chemical or microbiological analyses).

Particular attention should be paid to the soil type being investigated and the parameters to be measured (for example, volatile organic compounds, (VOC)).

Suggested applications for sampling devices are listed in Table B.1. Detailed descriptions of the equipment are presented in Figures B1-B3 and accompanying text (B.3).

Table A.1 Hand-driven soil sampling equipment

Equipment	Applicability	Advantages and disadvantages
Screw auger	0 cm to bedrock	Excellent depth range (by adding rod extensions); easy to decontaminate; can be used on all soil samples; results in soil mixing and loss of VOC. Most suitable for moist, cohesive soils; not recommended for stony, dry or non-cohesive soils. Can be operated by one person.
Bucket auger	Soft soil, to 3 m depth	Easy to use; relatively fast sampling method; good depth range; uniform diameter and sample volume; may disrupt and mix soil horizons greater than 15 cm in thickness. Most suitable for cohesive soils; not recommended for stony, dry or non-cohesive soils. Should not be used for collecting samples for VOC analysis, but acetate sleeve may be used to help integrity of VOC samples. Can be operated by one person.
Trier	Soft to firm surface soil	Easy to use and decontaminate; difficult to use in stony soils, dry sandy soils or hard clays.
Tube sampler	Soft soil, 0-3 m	Easy to use; preserves soil core (suitable for VOC and undisturbed sample collection); may be used to help maintain integrity of VOC samples; easy to decontaminate; can be difficult to remove cores from sampler. Most suitable for moist, cohesive soils, but versions are available for non-cohesive soils. Can be used in stony soils. Can be operated by one person.
Split-tube sampler	Soil, 0 cm - bedrock	Excellent depth range; preserves soil core (suitable for VOC and undisturbed sample collection); acetate sleeve may be used to help maintain integrity of VOC samples; useful for hard soils. Ineffective in non-cohesive soils; cannot be used in rocky soils. Is expensive relative to other methods. Some models can require two operators.
Thin-walled (Shelby tube) sampler	Soft soil, 0 cm-bedrock	Excellent depth range. Maintains undisturbed core suitable for VOC. Suitable for cohesive, soft soils. Not durable in rocky soils. Special tips for wet or dry soils are available. Difficult to drive into hard soil. May be difficult to pull form the ground.
Scoop or trowel		Not recommended for collecting representative samples.
Spade or shovel	Medium soil, 0-30 cm	Easy to use and decontaminate; inexpensive; can result in sample mixing and loss of VOC. Not recommended for routine use.

B.3 Description of sampling equipment

B.3.1 Augers

Augers can be used when there is no need for an undisturbed sample. Hand-held augers consist of an auger bit, a solid or tubular drill rod and a ‘T’ handle. The auger tip drills into the ground as the handle is rotated, and soil retained on the auger tip is brought to the surface as the soil sample. Power-driven augers are available.

A wide variety of auger bits are available, each designed for use in particular soil types. The following are types commonly used.

a) Screw auger

NOTE Also called spiral auger

Screw augers made especially for examining soils are available, ranging from about 2.5 to 4 cm in diameter and up to 30 cm in length (Figure B.1a). A screw auger cannot be used in very dry or sandy soils since these soil types will not adhere to the bit. In clayey soils, a bit of 2.5 cm may work better than the larger ones. A screw auger is easily carried, but it can be difficult to pull from the bored hole, especially in clay soils. The extracted soil material is disturbed more by a screw auger than by other augers and probes.

The auger is used to bore a hole to the desired depth, and is then withdrawn. The sample is collected directly from the flights. The continuous flight augers are satisfactory if a composite of the complete soil column is required.

Choosing the correct auger bit is essential for effective soil sampling:

— Clay soils are highly cohesive and therefore only require and auger with narrow blades;

— Sandy soils have little cohesion; wider blades are required to help keep the sample inside the auger;

— Coarse sandy soils (and extremely dry sandy soil) have very little or no cohesion; augers for these soils tend to have extra wings to form an almost closed auger that will effectively trap the sample.

Screw the auger vertically into the soil until the auger reaches the required sampling depth; pull the auger out of the ground; inspect the sample obtained. If there are any missing sections of soil, discard the sample and repeat the sampling process, within one metre of the original sampling point, until a sample is obtained without any missing sections.

b) Bucket auger

NOTE Also called barrel, orchard, post-hole and core auger

A bucket auger (Figure B.1b) is made of a cylinder or barrel to hold the soil, which is forced into the barrel by the cutting tips at the lower end. The upper end of the cylinder is attached to a length of pipe with a crosspiece for turning at the top. Although both ends of the cylinder are open, the soil generally packs so that it stays in place while the auger is pulled from the hole. Bucket augers with special closed cutting blades are available for use in sandy soils, very wet soils and very dry soils.

Bucket augers work best in loose or sandy soils and in compact soils. They are not well suited to use in wet or clayey soils, though an open-sided barrel is available that works well. They also work poorly in stony and gravelly soils. Bucket augers are available with different tips designed for specific soil types. For example, the sand auger tips are formed to touch so that very dry and sandy soils are retained. The tips of the mud auger are spaced further apart to allow for easier removal of heavy, wet soil and clays.

Bucket augers can be bulky to carry and bore more slowly than screw augers, but they are easier to remove from the soil and the sample is disturbed less.

B.3.2 Push samplers

a) Trier

A trier (Figure B.2a) consists of a 'T' handle and a tube cut in half lengthways, with a sharpened tip. It is suitable for collecting samples from consolidated soils with a particle diameter less than half the trier diameter. Triers of various length and diameter are available.

Push the trier into the soil and rotate it once or twice to cut a core. Withdraw the trier, making sure the slot is facing upward, and transfer the sample to a labelled sample container. In some cases it is best to insert the trier at an angle to minimise spillage of the sample.

Inspect the sample obtained. If there are any missing sections of soil, discard the sample. Repeat the sampling process within one metre of the original sampling point, until a sample is obtained without any missing sections.

b) Tube sampler

A tube sampler (Figure B.2b) consists of a 'T' handle and a tube with a cutting tip. A variety of cutting tips are available, designed for different soil types. The tube is pushed into the soil then withdrawn, and the sample is collected.

A variety of tube samplers are available, including soil probes, thin-walled tubes and soil recovery probes. Soil recovery probes are used with reusable liners to minimise contact with air, which makes them suitable for collected samples for VOC analysis.

The equipment for the tube sampler is portable and easy to use. Tube samplers are considered better than augers because samples are collected in a continuous core through the entire sampling depth with minimal disturbance of the soil.

Tube samplers are not suitable in all situations. In non-cohesive soils sample retention may be a problem. In overly wet soils soil compression may be a problem, but if the soil is too dry it can be difficult to penetrate.

c) Split-tube sampler

NOTE Also called split-barrel, split-spoon sampler.

A split-tube sampler (Figure B.3) consists of a hollow tube with a circular chisel or cutting shoe threaded onto one end and a driving head or collar threaded onto the other end. The split-tube has a hinge at the top that allows the tube to open and close along its length. This facilitates sample removal and equipment decontamination.

Split-tube sampling can be used to collect undisturbed sample cores up to 60 cm in length. The split-tube is driven in to its sampling depth and the core extracted.

B.3.3 Shovel-like samplers

a) Scoop or trowel

Scoops and trowels are often used to sample soil, but these tools cannot be used to extract a sample from the full depth to produce an unbiased soil sample. They are not recommended for collecting representative soil samples, although they can be used for homogenising and sub-sampling.

b) Spade or shovel

Although it is not recommended, a flat-bladed, square-edged spade or shovel can be used. However, careful sampling is required to extract a sample from the full depth of the soil to produce an unbiased sample. This method is only suitable for cohesive soils.

To take a sample, make a ‘V’ shaped cut into the soil to the required depth (see Figure B.3). Shave a slice, 2-3 cm thick, from one side of the hole to the full sampling depth with the spade. Use the centre portion of the slice, 2-3 cm wide, for the sample carefully taking away unwanted material from either side of a vertical slice on the blade. Transfer the remaining central portion to the sample container.

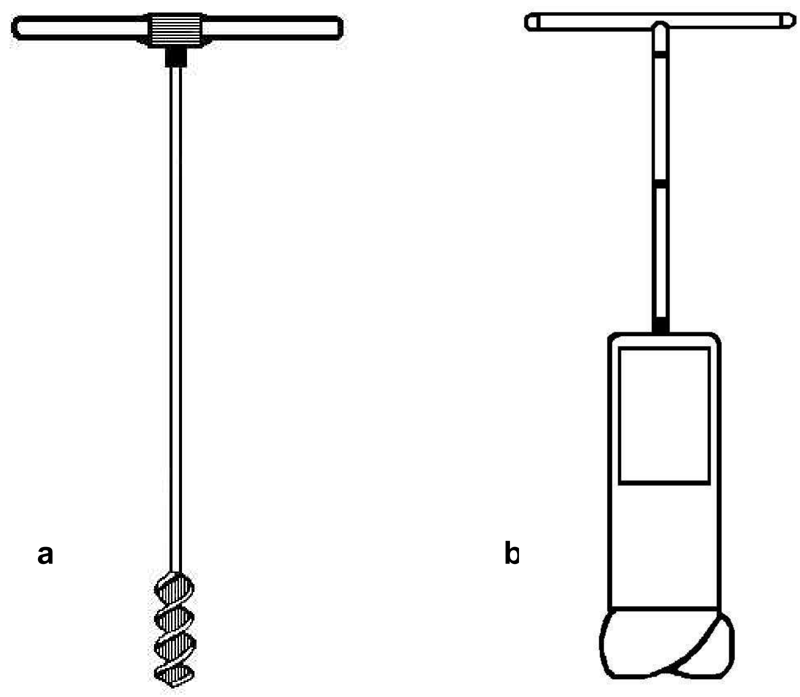

Figure B.1 — Sampling augers (a) Screw auger; (b) Bucket auger

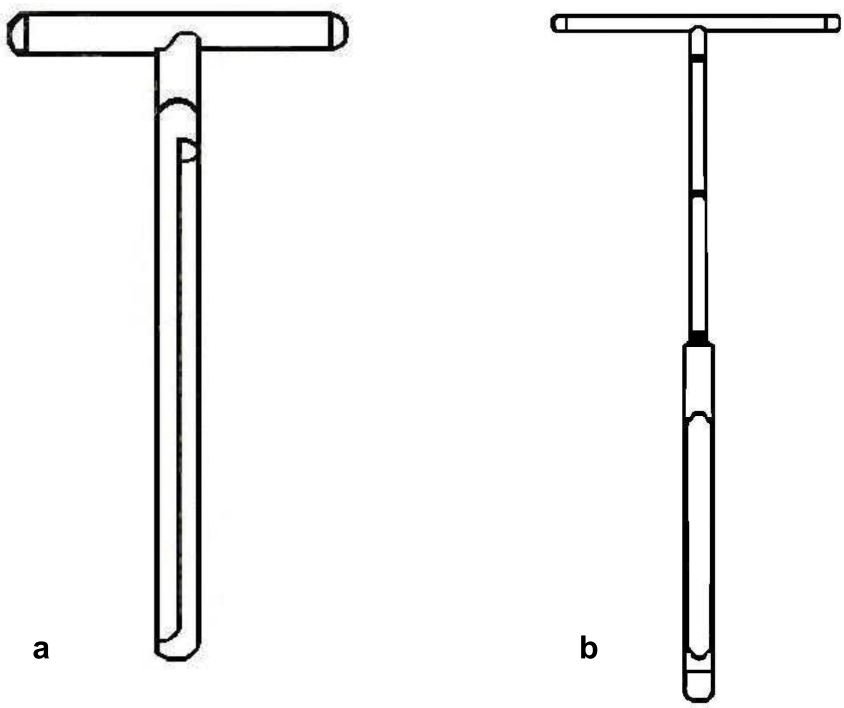

Figure B.2 — Push samplers (a) Trier; (b) Tube sampler

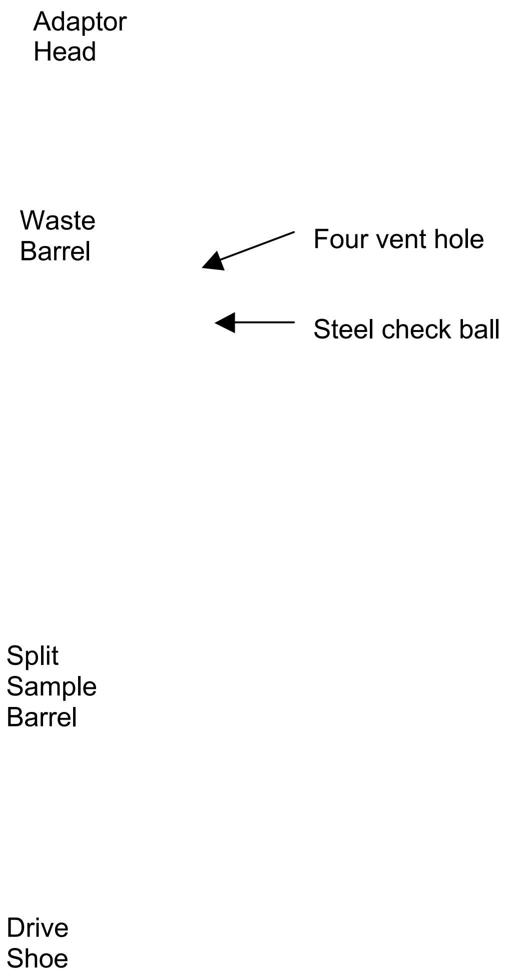

Figure B.3 — Push sampler: Split-tube sampler

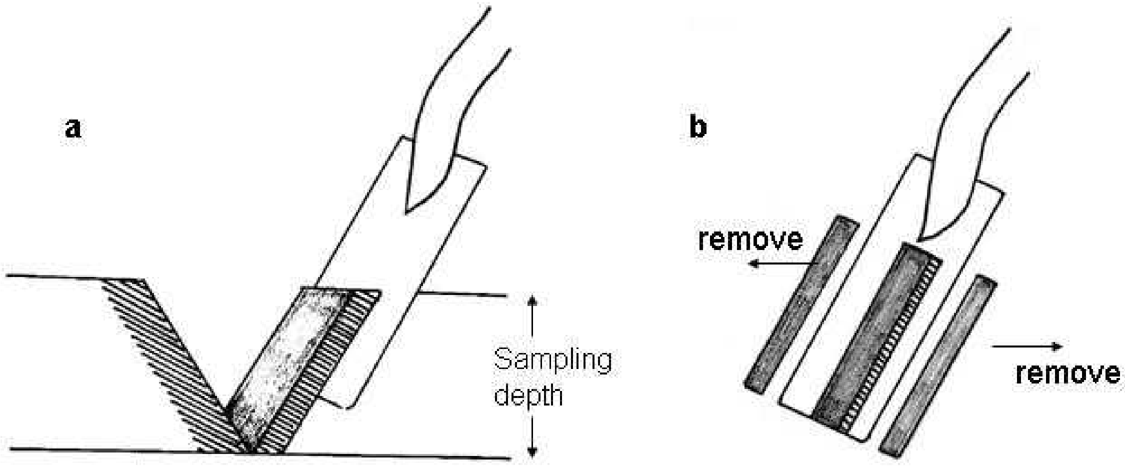

Figure B.3. Sampling with a spade

(a) Shave a slice from the side of a cleanly-dug ‘V’ shaped hole; (b) Use the centre portion for the sample.

Annex C
(informative)

Validation of method

Annex D
(informative)

The modular horizontal system

Annex E
(informative)

Information on WP 2 Sampling and the project HORIZONTAL

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