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Soil quality — Sampling —

Part 8:
Guidance on sampling of stockpiles

Qualité du sol — Échantillonnage —

Partie 8: Lignes directrices pour l'échantillonnage des stocks de réserve

Reference number
ISO 10381-8:2006(E)

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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

The main task of technical committees is to prepare International Standards. Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

ISO 10381-8 was prepared by Technical Committee ISO/TC 190, *Soil quality*, Subcommittee SC 2, *Sampling*.

ISO 10381 consists of the following parts, under the general title *Soil quality — Sampling*:

- *Part 1: Guidance on the design of sampling programmes*
- *Part 2: Guidance on sampling techniques*
- *Part 3: Guidance on safety*
- *Part 4: Guidance on the procedure for investigation of natural, near-natural and cultivated sites*
- *Part 5: Guidance on the procedure for the investigation of urban and industrial sites with regard to soil contamination*
- *Part 6: Guidance on the collection, handling and storage of soil for the assessment of aerobic microbial processes in the laboratory*
- *Part 7: Guidance on sampling of soil gas*
- *Part 8: Guidance on sampling of stockpiles*

This corrected version of ISO 10381-2:2006 incorporates the following corrections.

Clause 3

[ISO 11074-2:1995] was changed to [ISO 11074:2005].

In 3.26, Note 3 was deleted.

Subclause 5.5, **Table 1**

In the third column following “Sampling technique”, “other” was replaced by “different”.

Subclause 6.5.5

In the second sentence of the second paragraph “shaded region” was replaced by “central region”.

Subclause 8.2.3

In the last line of g), “Note 3” was replaced by “item c) 3)”.

Subclause D.4.4

In Equation D.1, the horizontal line of the square-root sign was extended to the right to include “$+ CV$ analysis”.

Subclause H.1.4.4

In the line before Equation (H.5), “(H.4)” was replaced by “(H.5)”.

Subclause H.2.1

In the first line, “less” was replaced by “little”.

In addition, minor editorial changes were made. These changes do not alter the meaning of the text.

Introduction

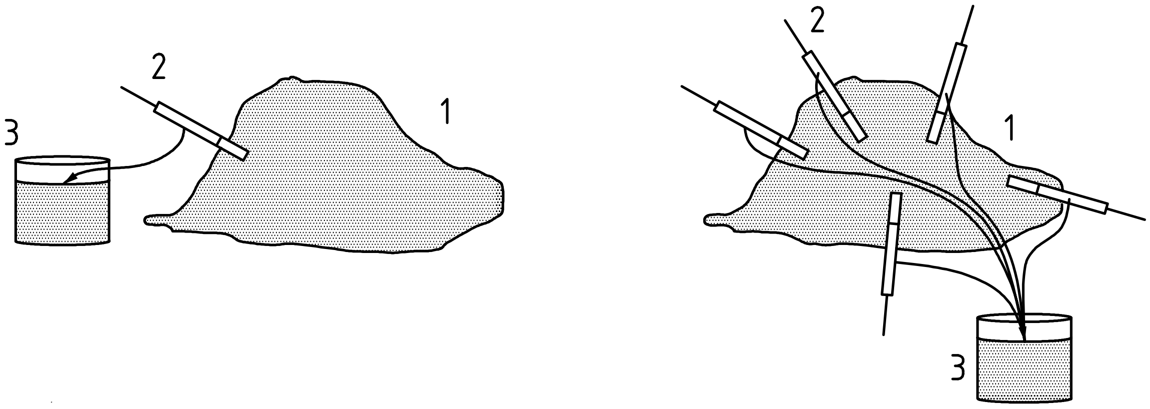
This part of ISO 10381 describes the methods to be applied when sampling soil from stockpiles. The general character of this part of ISO 10381 is a guideline. Nevertheless, many aspects of the sampling of stockpiles are based on well established methods and consequently are described in a prescriptive manner.

This part of ISO 10381 only includes the sampling of the soil material itself, i.e. the solid phase. It defines the different steps in sampling soil from a stockpile and gives instructions on how these steps should be carried out for specific situations.

This part of ISO 10381 is basically a code of practice. It describes what activities, circumstances and requirements should be addressed when sampling soil from stockpiles. As the circumstances can vary enormously, no detailed instructions on how samples should be taken in a specific situation can be given.

For a good understanding of this part of ISO 10381, the distinction between the terms “increment” (3.5), “sample” (3.16) and “composite sample” (3.4) is essential. Figure 1 illustrates this point.

An increment is obtained by a single operation of a sampling device and is per definition put together with other increments in a composite sample. A sample can also be obtained by a single operation of a sampling device, but the obtained material is packed and analysed as an entity.



a) Only material of one sampling action in sample container: sample

b) Two or more sampling actions: gathered material in one sample container: composite sample
Material of each individual action: increment

Key

1	stockpile
2	sampling device
3	sample container

Figure 1 — Sample, composite sample and increment

Soil quality — Sampling —

Part 8:
Guidance on sampling of stockpiles

1 Scope

This part of ISO 10381 defines the methods that should be applied when sampling soil from stockpiles. This part of ISO 10381 only includes the sampling of the soil material itself, i.e. the solid phase. It applies to the sampling of soil material that is present in a stockpile, generally a heap of soil material that is lying above the surface of the location.

The underlying reason for sampling the soil can differ widely as can the subsequent analysis on the obtained samples. This part of ISO 10381 therefore gives guidance on the various aspects that, together, describe the sampling activity:

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

Given the wide differences in circumstances for all of the above-mentioned sampling steps, this part of ISO 10381 provides information on how to obtain clear and simple instructions for the sampling personnel.

2 Normative references

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

ISO 10381-1:2002, *Soil quality — Sampling — Part 1: Guidance on the design of sampling programmes*

ISO 10381-3:2001, *Soil quality — Sampling — Part 3: Guidance on safety*

ISO 10381-5:2005, *Soil quality — Sampling — Part 5: Guidance on the procedure for the investigation of urban and industrial sites with regard to soil contamination*

ISO 11464, *Soil quality — Pretreatment of samples for physico-chemical analysis*

ISO 14507, *Soil quality — Pretreatment of samples for determination of organic contaminants*

3 Terms and definitions

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

3.1
analytical sample
portion of material, resulting from the original sample or composite sample by means of an appropriate method of sample pretreatment, and having the size (volume/mass) necessary for the desired testing or analysis

3.2
actual increment size
amount of material that is present in an increment

NOTE The actual increment size is determined by the minimum increment size, the amount of material needed for the tests or analysis and the number of increments in a composite sample.

3.3
actual sample size
amount of material that is present in the sample

NOTE The actual sample size is determined by the minimum sample size, the amount of material needed for the tests or analysis, the size of the sampling equipment and, for composite samples, the number of increments and the actual size of the increments.

3.4
composite sample
two or more increments/subsamples 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 11074:2005]

3.5
increment
sampling unit collected by a single operation of a sampling device and used in a composite sample

[ISO 11074:2005]

NOTE When an individual portion of material is collected in a single operation of a sampling device and this portion is analysed as an individual unit, it is per definition a sample.

3.6
involved parties
individuals involved in the (iterative) process of defining and executing the sampling programme

3.7
judgemental sampling
sampling using methods identified by prior agreement with all involved parties, without sampling in accordance with probabilistic sampling

NOTE Although in general agreement of all parties should be sought, in specific situations some parties are to be considered as more important than others. Whenever there is a hierarchical relation between the different parties, this should be taken into account when no general agreement can be established.

3.8
maximum particle size
D_{95}
particle size that concurs with the mesh width of a sieve on which a maximum of 5 % (mass fraction) of the material remains

3.9

minimum increment size

minimum amount of material in an increment obtained with a sampling device for which the conditions of probabilistic sampling apply

NOTE The fact that every particle in the material to be sampled shall have the same probability of being part of a sample results in requirements for the size of the sampling equipment. These requirements determine the amount of material that is obtained with a single sampling operation.

3.10

minimum sample size

minimum amount of material in a sample for which the variability caused by the individual particles within that material has a negligible effect

NOTE The minimum sample size is calculated based on an equation in which different factors result in an estimation of the minimum sample size. One of these factors is the variability that is accepted to be caused by the differences between individual particles. When a large amount of variability is chosen for this factor, there will no longer be a “negligible effect” as mentioned in the definition. However, in normal circumstances, a low value will be chosen, accepting only a relatively small amount of variability.

3.11

particle size reduction

procedure to reduce the particle size of the whole (sub)sample through grinding or crushing without reducing the sample size (mass)

3.12

population

totality of items under consideration

[ISO 11074:2005]

NOTE In the case of a random variable, the probability distribution is considered to define the population of that variable.

3.13

probabilistic sampling

sampling to ensure that each particle or element in the stockpile (population) has an equal chance of being part of the sample

3.14

project manager

individual responsible for the development of both the sampling plan and the sampling programme

3.15

primary goal

definition of the sampling in short, general statements, giving direction towards the type of sampling, but still lacking the necessary detail to define a sampling plan

3.16

sample

portion of soil material selected from a larger quantity of material

[ISO 11074:2005]

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

3.17

sampler

person or group of persons carrying out the sampling procedures at the sampling locality

[ISO 11074:2005]

3.18
sample division
procedure through which subsamples of smaller size than the original sample are obtained without reducing the particle size of the individual particles

3.19
sample pretreatment
collective noun for all procedures used for conditioning a soil sample to a defined state which allows subsequent examination or analysis or long-term storage

[ISO 11074:2005]

NOTE Sample pretreatment includes, e.g. mixing, splitting, drying, crushing and stabilization.

3.20
sampling goal
technical description of the purpose of sampling

3.21
sampling plan
all information pertinent to a particular sampling activity

NOTE The sampling plan provides the sampler with a predetermined procedure for the selection, withdrawal, on-site pretreatment, preservation and transportation of the portions to be removed from a stockpile (population) as a sample.

3.22
sampling programme
total sampling operation, from the first step in which the purpose of sampling is defined to the last step in which the analytical results are compared with the relevant test level(s)

3.23
sampling technique
correct appliance of appropriate sampling equipment to obtain samples as specified in the sampling plan

NOTE The manner of selection of the sampling technique should be described in the sampling plan.

3.24
secondary goals
detailed definition of the technical aspects necessary for defining the sampling

NOTE The secondary goals address items such as the population to be sampled, the components to be determined, the statistical parameter to be determined, the scale of sampling and the desired precision and confidence.

3.25
stockpile
temporary heap of material

NOTE 1 Within the scope of this part of ISO 10381, the stockpile contains soil material.

NOTE 2 The soil material can be stored in a loosely dumped heap, can be lying in a pre-defined depot, above or below the surface of the location, etc.

3.26
subsample
sample obtained by procedures in which the items of interest are randomly distributed in parts of equal or unequal size

[ISO 11074:2005]

NOTE 1 A subsample may be:

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

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

c) the final unit of multistage sampling.

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

4 Principle

A sampling plan shall be defined and this is carried out mainly as a desk operation. However, the designer of the sampling plan shall have sampling experience and be aware of the specific circumstances of the objectives and location of the sampling. Where knowledge of the site is insufficient, a site visit may be necessary before designing the sampling plan.

The sampling plan design shall include the consideration and formulation of the sampling strategy. This is important as the strategy shall ensure that the samples obtained from the stockpile are representative. Thus, there are two points to be considered in formulating the sampling plan: 1) the sampling strategy; 2) the sampling techniques.

The aim of the sampling strategy is to ensure that the requirements of probabilistic sampling are achieved. This means that all the particles in a stockpile have an equal chance of being present in the sample. This truly representative sample can only be achieved when all the requirements of probabilistic sampling are met. In practice, this may not be possible, in which case sampling should be carried out following the most practicable methods to achieve the sampling objectives.

The sampling plan shall include the sampling equipment chosen, and the sampler should have the necessary experience to ensure correct use of that equipment.

The sampling plan, when completed, should be given to the sampler before sampling commences, though some alterations may be necessary due to situations encountered onsite. Small alterations to the sampling plan may be made in the field without consulting the designer of the sampling plan.

In some cases, the sampling will result in samples which are too large to take to the laboratory and sample pretreatment in the field shall be necessary. There are two basic conditions for pretreatment in the field. Firstly, the sample should not be changed in a way that will affect the subsequent examination, i.e. contamination of the sample and/or involuntary loss of material or components should be avoided. Secondly, there should be no reduction in particle size since that process requires well-defined conditions which can not be achieved in the field and particle size reduction is restricted to being a laboratory operation.

When the samples have been taken and, if necessary, pretreated, they should be packaged so that the characteristics are protected. The packaging and any preservation necessary depend on the characteristics which are to be preserved. Preservation of soil samples shall involve two basic methods: 1) cool storage; 2) dark storage.

This part of ISO 10381 gives guidance on the aspects to be considered when storing the samples prior to analysis. This includes storage before and during transport, and storage in the laboratory prior to sample preparation for analysis.

5 Sampling plan

5.1 General

A large number of varying conditions occur where and when soil stockpiles are to be sampled. It is impossible to give detailed instructions on how to sample in all of these possible situations. This part of ISO 10381, therefore, addresses the essential points that should be considered before actual sampling, and does not give detailed instructions for specific sampling situations. Nevertheless, a detailed instruction to the sampler is essential in order to acquire the type and quality of samples which are necessary for the purpose of sampling. This instruction is given by means of a sampling plan.

When taking samples from soil stockpiles in accordance with this part of ISO 10381, a sampling plan should be defined prior to sampling. This part of ISO 10381 gives instructions on the definition of the sampling plan. The elements/aspects that should be part of the sampling plan are given, as well as the (type of) considerations that are relevant when defining the sampling plan.

A simple example of a sampling plan is given in A.1.

The sampling plan is based on the specific purpose of the sampling. It translates the purpose and all the information pertinent to a particular sampling exercise into simple and unambiguous instructions for the sampler. Sampling shall only be carried out when an approved sampling plan is available.

The sampling plan acts as a reference document and provides the means of defining the boundaries and logistics of the sampling. Its formulation requires consideration of a number of key elements/aspects (see Figure 2).

The elements/aspects of the sampling plan may be divided into two groups:

- those which relate to identifying and agreeing the sampling design in consultation with the involved parties;
- those which specify the mechanics of how, when, where and by whom the samples will be collected and precautions that will be taken to protect the sampler and the sample.

5.2 Sampling design

5.2.1 Involved parties

The sampling plan should be prepared under the direction of a project manager in consultation with all appropriate involved parties. Such consultation can involve the owner of the stockpile, the landowner on whose property the stockpile is situated, the final decision-maker, the sampler, the analyst, the customer, the regulator and the material provider.

In cases where the level of complexity is low, a number or all of these roles may be the responsibility of one individual. In certain circumstances, the number of involved parties can be influenced, for example, by legislation. In some cases, the landowner has special (safety) regulations for personnel present on the site. When relevant, the sampler shall be instructed before entering the site.

No further action shall be taken unless or until the purposes and restraints are established.

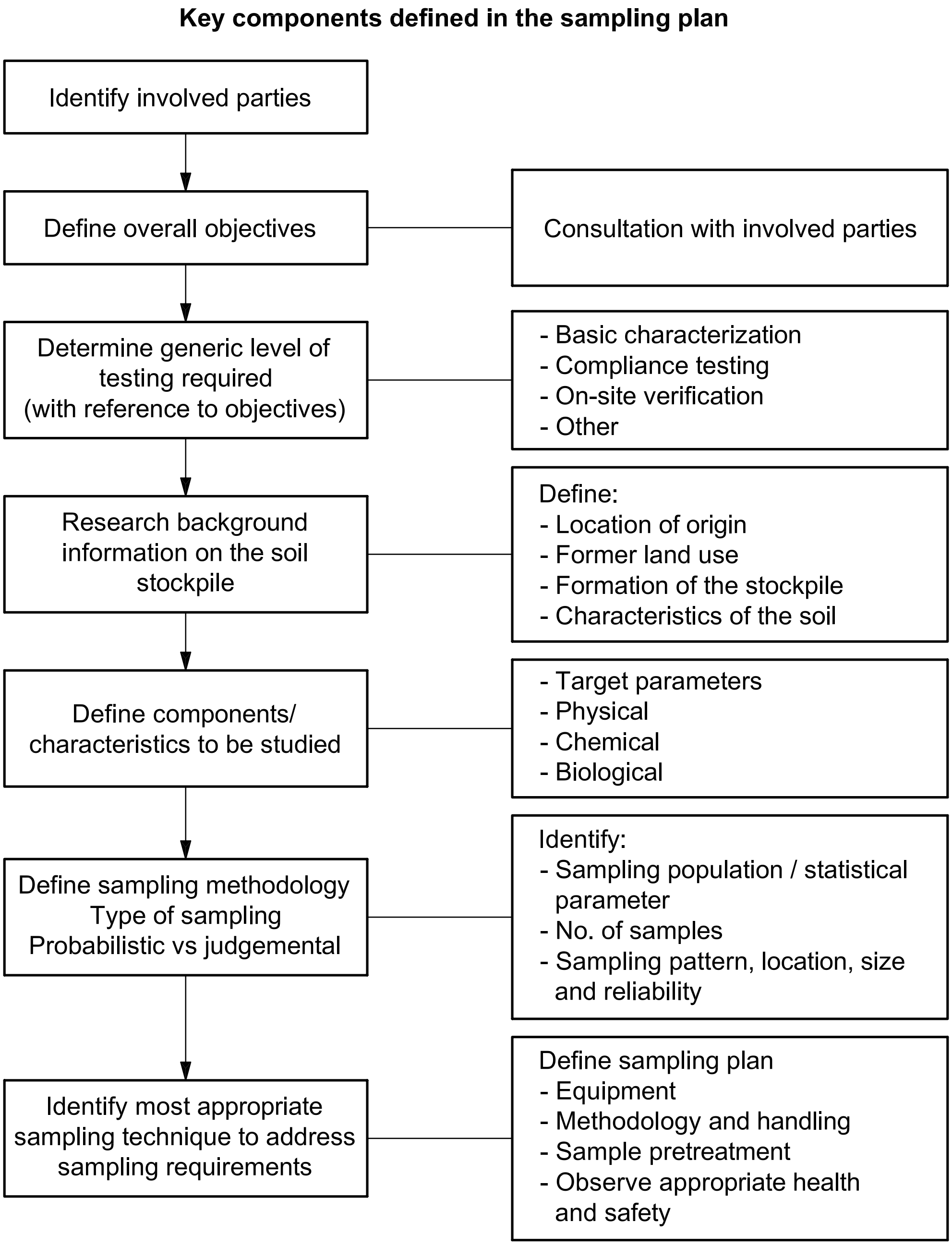


Figure 2 — Key components in the sampling plan

5.2.2 Purpose of sampling

The project manager shall define the purposes of the sampling programme with due consultation of all involved parties. The reasons for sampling can be diverse and can, for example, be to determine the possibilities for re-use, the need for soil clean-up, the assessment of environmental risks.

This diversity of purposes affects the location, number, volume and minimum testing requirements for the sampling exercise. It is therefore important that the ultimate purpose of the sampling programme and any specific goals are clearly identified to ensure that the samples collected meet these purposes. The sampling plan shall identify these primary goals of the sampling programme.

The primary goal(s) shall then be translated within the sampling plan into practical and achievable sampling goals (the secondary goals, see 6.3.3) that take into account the characteristics of the soil stockpile to be determined. Any restrictions or limitations on the reliability of the achieved results shall be detailed in addition to the agreement of interested parties.

5.2.3 Primary sampling goal

The project manager, with regard to the purposes of the sampling programme, shall select the level of testing required. Three generically defined levels of testing are described, but other levels are possible as well as other definitions of the three mentioned levels.

- Level 1: Basic (comprehensive) characterization, consisting of a thorough determination of the behaviour and properties of interest of the material.
- Level 2: Compliance testing, consisting of (periodic) testing to determine compliance with specific conditions or reference conditions (e.g. legislation or contract).
- Level 3: On-site verification, consisting of “quick check” methods to establish consistency with either Level 2 results or other formulated documentation.

For each of these levels, but especially Level 1, different types of investigation shall be specified depending on the specific aims of the sampling. The project manager shall take this into consideration when preparing the sampling plan.

5.2.4 Determination of target components

The project manager shall, within the requirements of the appropriate test level, identify the characteristics or components to be investigated, based on information:

- specified in regulations;
- relating to the intended end-use;
- specified in contract;
- ascertained from knowledge, for instance of the process responsible for contamination of the soil;
- agreed between the involved parties.

The parameter(s) to be determined by analysing the samples shall be listed in the sampling plan. Appropriate methods of sample collection and preservation shall be selected to maintain the integrity of the sample with appropriate reference to the analytical method to be used.

It is recommended that the project manager contacts the laboratory which will carry out the analyses to get adequate advice.

If several target parameters are identified, the sampling operation shall be designed in such manner that the parameters of major importance are determining the sampling. If this is not possible (e.g. the required precision for each parameter can not be achieved), separate sampling programmes shall be set up for each group of parameters.

NOTE This can be relevant for the total sampling programme, as well as for a specific part of the sampling programme (e.g. the conditions for preservation, storage and packaging). Keeping the total sampling programme simple — and thus trying to avoid too many deviations — will avoid in itself the occurrence of human errors.

5.2.5 Background information on the soil stockpile

5.2.5.1 General

Background information on the stockpile will often be essential in order to get (general) information on the material to be sampled. Background information shall be obtained in accordance with 5.4 of this part of ISO 10381, Clause 6 of ISO 10381-1:2002 and Clause 6 of ISO 10381-5:2005.

The effort that should be put into obtaining prior information depends on the purpose for sampling in combination with the sampling strategy that is used to fulfil this purpose.

NOTE When for instance the purpose for sampling asks for high quality information, this need for information can potentially be fulfilled in two ways:

- by obtaining a lot of qualitative and/or quantitative information as prior information and using in addition to this prior information a relatively simple sampling strategy;
- by obtaining only little prior information and using an intensive sampling strategy (many increments and/or samples).

Prior information can also be essential for assessing the safety aspects of sampling the particular stockpile (see 5.4).

5.2.5.2 Site details

The project manager shall establish details of the site location and access, including any perceived hazards relating, for example, to high stockpiles, non-consolidated stockpiles or difficult access.

In some situations, there might be a difference between the owner of the site where the stockpile is situated and the client for whom the stockpile is sampled. If so, the project manager should contact the site owner in order to get access to the stockpile and inform if there are site specific health and safety (see 5.4) regulations to comply with.

5.2.5.3 History of the soil

The project manager shall establish a description of the history of the soil stockpile in order to determine the potential environmental risks involved. The history includes the period before the soil was stored in the stockpile. The history of the soil shall be based on the location(s) where the soil originates from and the processes that occurred on that site. It should also include the process in which the stockpile was formed as this can give prior information on the spatial differentiation of soil quality within the stockpile.

5.2.5.4 Material type and dimensions

The project manager shall establish the soil characteristics (e.g. soil type, water content, particle size distribution) and dimensions of the stockpile to be sampled.

As part of the particle size distribution, an estimate of the maximum particle size D_{95} (see 3.8 and Annex B) in the stockpile is necessary for the definition of the sample and increment size.

5.2.5.5 Preliminary investigation

Where the information established under 5.2.5.2, 5.2.5.3 and 5.2.5.4 is deemed by the project manager to be insufficient, a preliminary investigation should be instigated. This preliminary investigation should either be aimed at gaining the lacking information, or should result in a first identification of the soil, by means of which an appropriate sampling plan can be established.

5.2.6 Consideration of statistical methods

5.2.6.1 General

The selection of an appropriate method of sampling defines how, when and where samples shall be taken to obtain the desired quantity of soil, both to be representative as well as being sufficient to meet the testing requirements.

Probabilistic sampling shall be seen as the preferred option. However, in cases where the project manager has identified that the heterogeneity and the inherent variability of the soil demands sample sizes or numbers of increments that exceed the resources available, the project manager shall outline a process of sample collection on a judgemental basis. The available resources can also be exceeded due to a large particle size of the soil material. In both cases, a thorough evaluation of costs versus representativity shall be made before choosing for a form of judgemental sampling that will or can lead to biased testing results.

Based on the variability within the batch, the degree of precision and reliability required from the results, and the resources available, the project manager should select the appropriate sampling strategy. Guidance on the consideration of statistical issues is given in Clause 6.

5.2.6.2 Probabilistic sampling

The project manager shall establish the quantity of sample material to be collected for testing and analysis. When necessary, this shall include provision for replicate samples. Sampling design is based on statistical principles for

- increment size,
- sample size, and
- sampling locations.

Instructions on the determination of the increment and sample size are given in 6.6.

5.2.6.3 Judgemental sampling

The project manager shall specify the procedure for judgemental sampling for each specific situation, including provisions for replicate samples. The basis of the judgemental sampling procedure is that it will resemble, as much as possible, the probabilistic sampling procedure. Depending on the differences that are necessary to enable (judgemental) sampling, the representativity of the samples can be biased or only representative for a specific part of the soil stockpile. The project manager should be aware of this and consider the consequences of the judgemental approach.

5.2.7 Sampling technique

The technique employed to collect samples will be influenced by the characteristics of the soil to be sampled (e.g. soil type, water content, degree of consolidation, accessibility and particle size). Guidance on the selection of appropriate sampling techniques is given in Clause 7.

5.2.8 Sample division in the field

The project manager should select appropriate pretreatment methods to reduce sample size for presentation to the laboratory. Particle size reduction – often necessary to obtain representative analytical samples of the appropriate size – is only allowed when laboratory conditions are met. Guidance on sample pretreatment in the laboratory is given in ISO 11464 and ISO 14507. A selection of pretreatment techniques suitable for sample division in the field is given in Clause 8.

5.2.9 Packing, preservation, storage, transport and delivery

The project manager should select appropriate methods for packing, preservation and storage, while transport and delivery should comply with conditions and quality considerations. More information is given in Clause 9.

5.3 Specifying information in the sampling plan

5.3.1 General information

In preparing the sampling plan, the project manager shall specify the sampling techniques, the pretreatment (in the field), the quantity of increments and samples, the sampling equipment, the number of samples, the sample containers and methods of storage, preservation and transport between sampling and testing and record that information in the sampling plan. An example of a sampling plan is given in Annex A.

The project manager should prepare a sampling plan incorporating, as a minimum, the following:

- company (body) commissioning the sampling (client);
- name of client representative;
- date of placement of commission;
- company performing the sampling;
- name of the project manager;
- name of personnel performing the sampling (the sampler);
- purpose of sampling and there from derived primary and secondary sampling goals;
- details of sampling location.

5.3.2 Stockpile data

- Identification of the stockpile (e.g. location, boundaries, spatial description);
- description of the soil to be sampled (e.g. soil type, estimated water content, particle size distribution);
- size of stockpile to be sampled;
- way in which the stockpile is available for sampling;
- maximum particle size (D_{95}).

NOTE The commissioning company (client) can supply general information on the soil stockpile.

5.3.3 Sampling

- (Applications of) sampling technique (see Clause 7);
- number of increments or samples to be taken (see Clause 6);
- increment size and/or sample size (see Clause 6);
- instructions on safety precautions;
- sampling date.

5.3.4 Sample pretreatment

- Reduction of the size of the sample in the field directly after sampling (see Clause 8).

5.3.5 Packaging, preservation, storage, transport and delivery

- Packaging and preservation of the increment or sample;
- sample coding to be used (see Clause 9);
- storage and transport of the increment or sample;
- name and location of organisation to receive samples;
- date of delivery of samples;
- name of personnel who accepted the samples at delivery.

5.3.6 Actual sampling

Taking the sample(s) in accordance with all the instructions as provided by the sampling plan.

5.3.7 Sampling record

For recording all procedures and results during sampling, a sampling record is produced. Basically, the sampling record corresponds to the sampling plan, but gives sufficient space to write down observations during sampling or (small) alterations in the original sampling plan.

As a first step of sampling, the sampling plan should be checked in the field (see 6.7). In 5.5, guidelines are given on which types of alterations can be made by the sampler without consulting the person who has made the sampling plan (the project manager).

5.4 Health and safety

For health and safety aspects of sampling, see ISO 10381-3.

In addition to the safety instructions in ISO 10381-3, the following sources can provide relevant safety instructions and regulations:

- (inter)national legislation;
- site specific safety instructions.

At most industrial sites, special safety instructions/regulations are in effect. When relevant, the sampler should be informed about these regulations before entering the site, and shall comply with these regulations during the presence on the site. Where (large) stockpiles are part of the (industrial) activities, heavy mechanical equipment can pose an additional threat to the sampler. Operational personnel should be informed about the presence of the sampler on the site.

When sampling non-consolidated stockpiles, the sampling plan should contain additional safety instructions on how the stockpile shall be sampled safely.

5.5 In-field alterations

It can be necessary to make (small) changes to the sampling plan in order to perform the sampling. Some of these changes will not influence the overall purpose of the sampling; the necessary samples are obtained and representativity of the samples is maintained at the desired level. Some changes will however have consequences on the resulting quality of the testing.

In general, minor changes that have no effect on the results of sampling may be made in field by the sampler, while major changes should be made or at least be approved by the project manager prior to sampling.

It is not possible to give detailed instructions on the type of changes that are to be considered minor or major. This depends too much on the specific sampling situation and the desired test level. In general terms, the changes listed in Table 1 are to be considered as major changes. However, any change to the sampling plan – with or without consultation of the project manager – shall be noted and motivated in the sampling record.

Table 1 — List of major changes in the sampling plan for which consultation of the project manager is obligatory

Subject	Major change	Examples of possible reasons for the occurrence of a major change
General information	Sample location	The stockpile is no longer at the location where it was supposed to be. The accessibility of the location has changed (negatively).
Stockpile data	Identity of stockpile	The material looks different from what was expected (e.g. colour, particle size). The stockpile is no longer at the location where it was supposed to be. Two or more stockpiles partly overlap. Size is significantly smaller or larger then expected.
Sampling	Sampling technique	The necessity to use a significantly different sampling technique, for example, due to a different estimate of D_{95}.
	Size of increments and/or samples	Different estimate of D_{95}.
	Number of increments and/or samples	No time available to obtain all the necessary increments and/or samples.
	Sampling strategy	Probabilistic sampling is not possible, for example, due to the size of the stockpile.
Pretreatment	Necessity of pretreatment	Pretreatment was not planned, but appears to be necessary due to, for example, a larger estimate of D_{95}. No pretreatment is necessary due to, for example, a smaller estimate of D_{95}.
	Possibility of pretreatment	There is no clean and unused location at the sampling site. The weather conditions do not allow good quality pretreatment.
Packaging, preservation, storage, transport and delivery	Identity of the samples	Breakage of sample containers. Insufficient number of the appropriate sample containers.
	Quality of the samples	Potential contamination of samples due to, for example, the inability to use proper packaging material or preservation/storage conditions.

6 Sampling strategy

6.1 General

Sampling strategy is important due to the fact that soil is a particulate material. It is therefore to be considered heterogeneous. As there are different types of soil particles present, the material has a “fundamental” level of heterogeneity. In addition to this “fundamental” heterogeneity, substances of interest can be heterogeneously distributed in the stockpile. As a consequence, the degree of heterogeneity will directly influence the representativity of the samples. Therefore, statistics play an important role in defining the sampling plan. Basic knowledge of the statistical principles are briefly mentioned in 6.2 and discussed in more detail in Annex D.

Before designing the sampling strategy, the purpose of the sampling should be clear because it will determine which type of sampling strategy is adequate and how reliable the sampling should be. The purpose of sampling shall therefore be translated into more technically defined sampling goals. The purpose of sampling and the therefrom derived sampling goals are considered in 6.3.

The practical translation from sampling goals towards the actual sampling activity will also depend on the question of whether the sampling can fulfil the requirements of probabilistic sampling or not. If not, the sampling shall take place on a judgemental basis. The number and character of the diversions from probabilistic sampling will determine the degree of representativity of the judgemental sampling. To understand the consequences of both probabilistic and judgemental sampling, 6.4 considers the principles of both types of sampling.

The sampling strategy deals also with the “where and when” of sampling. In 6.5, this subject is discussed in more detail.

Having determined the sampling locations, the number and type of samples should be determined. This is discussed in 6.6.

Finally, the choices that have been made should be incorporated in the sampling plan, see in general Clause 5, as well as 6.7 for more specific details.

6.2 Statistical principles

The basic statistical principles of sampling are briefly described in Annex D. The text is not intended to be a statistical textbook, and gives only a basic outline of the statistical elements relevant to this part of ISO 10381. The explanation of the statistical principles is grouped under five headings:

- Population (D.2). This is a statistical term for defining the entirety of material – in general the stockpile – about which information is required. Specification of the population should be the starting point of any sampling exercise.
- Types of variability (D.3). A good awareness of the variability in the material being sampled is a vital element in arriving at an effective sampling programme. Linked with this (for sampling granular material) is the need to decide on the “scale” of the sampling, i.e. the maximum volume of material within which variations in quality are of no concern.
- Error (D.4). Apart from the variability characteristic of the material itself, sampling introduces additional uncertainty, known as “sampling error”. Analysis similarly introduces a further degree of uncertainty, termed “analytical error”.
- Statistical parameters (D.5). A variety of summary measures, or “parameters”, may be used to characterize a population (e.g. the mean, the median or the 90-percentile). It is important to consider the choice of parameter, because this can have a big influence on the size of the sampling error.
- Reliability (D.6). The greater the amount of sampling, the more reliable the results are likely to be. The major benefit of a statistical approach is that it enables this link between sampling effort and reliability to be quantified.

6.3 Purpose of sampling

6.3.1 General

The purpose of sampling should be clear prior to selecting a sampling strategy, as this is an essential step towards defining the type and quality of the information that is to be obtained through sampling. The purpose of sampling can be various, for example:

- the necessity to compare the quality of the stockpile with quality levels defined in (inter)national legislation;

- a change in ownership of the stockpile and the necessity for the buyer to know the (environmental) quality of the soil;
- to determine the (re-)usability of the soil;
- to determine the leachability of the soil;
- to assess the human and/or environmental risks;
- other.

These reasons for sampling by themselves give no information on the type and quality of sampling that is necessary for complying with the purpose. Therefore, it is necessary to define the true sampling goal(s) with which the sampling should comply.

A distinction is made between the primary sampling goal (see 6.3.2) and the secondary sampling goal (see 6.3.3). The primary goal defines the sampling in short, general statements, giving direction towards the type of sampling, but still lacking the necessary detail to define a sampling plan. In general, the sampling will have only one primary goal.

NOTE 1 When more than one primary goal is to be achieved with the same sampling programme, the sampling will often be highly complex, if not practically impossible. Therefore, these situations should be omitted.

The secondary sampling goal gives the detailed information that enables the project manager to define the sampling plan. Aspects like the type, size, scale and number of samples to be taken, the way they are selected from the stockpile, and so on, are addressed by the secondary sampling goal. Usually, there are a number of secondary goals coupled with the primary goal.

It is essential that the involved parties agree on the purpose of sampling, and the therefrom derived primary goal and secondary goals, prior to the actual sampling.

When the purpose of sampling or the primary goal does not explicitly define the quality and type of sampling, the project manager has to define these according to his own judgement. Especially in these cases, the acceptance of the sampling plan by the involved parties prior to sampling is essential.

NOTE 2 In some cases, the translation from the purpose of sampling towards a sampling plan is fairly simple, due to the fact that the number of samples, the type of samples and the sampling strategy are already defined in a (inter)national standard or in (inter)national legislation.

EXAMPLE In the Netherlands, a standardized sampling strategy is used for the sampling of soil stockpiles in order to determine if the soil is clean. The primary goal is to determine if the soil is clean in accordance with the Dutch Building Materials Act. In the Dutch Building Materials Act, the sampling of a soil stockpile is described in detail, and the project manager only has to copy this information into the sampling plan.

6.3.2 Primary goals

Three generically defined levels of testing are described in 5.2.3, but, as mentioned there, other levels are also possible, as well as other definitions of the three mentioned levels. These three levels are:

- Level 1: Basic (comprehensive) characterization, consisting of a thorough determination of the behaviour and properties of interest of the material.
- Level 2: Compliance testing, consisting of (periodic) testing to determine compliance with specific conditions or reference conditions (e.g. legislation or contract).
- Level 3: On-site verification, consisting of “quick check” methods to establish consistency with either Level 2 results or other formulated documentation.

These are examples of primary goals. In short, still general terms, the primary goal defines which type of sampling should be carried out.

Depending on the primary goal and the (inter)national legislation, rules, standards, and accepted methods, the primary goal does imply, directly or indirectly, the quality and type of sampling needed. When there is only an indirect relation, the programme manager has to define the quality and type of sampling explicitly.

6.3.3 Secondary goals

The secondary goals coincide with the elements of the sampling plan:

- definition of the stockpile to be sampled (the population);
- definition of the components to be determined and/or tests to be carried out on the samples;
- definition of the statistical parameter to be determined (e.g. mean concentration, degree of heterogeneity, percentile);
- definition of the type of sampling (probabilistic or judgemental);
- definition of the scale of sampling (the use of increments and composite samples or individual samples and the scale on which the soil should be tested);
- definition of the desired precision and confidence.

By defining the secondary goals, part of the information necessary in the sampling plan is generated.

Annex G gives some examples of the definition of types of sampling based on the purpose of sampling and the therefrom derived primary and secondary sampling goals.

6.4 Types of sampling

6.4.1 Probabilistic sampling

The basis of probabilistic sampling is that each soil particle within the population – that is the whole stockpile to be assessed (see D.1) – has an equal chance of being selected by the sampling process. In probabilistic sampling, the sampling design is based on statistical principles for:

- sampling locations (see 6.5);
- increment size (see 6.6);
- sample size (see 6.6).

6.4.2 Judgemental sampling

In contrast, judgemental sampling is where samples are taken in accordance with a non-probabilistic procedure. The most common reason for falling back on judgemental sampling is that representative sampling from the whole population is practically impossible, given the available resources in time and/or money. However, because judgemental sampling may take any form that is convenient it can result in highly biased samples, and so have severe financial and/or environmental consequences. It is therefore preferable for judgemental sampling to depart from probabilistic sampling as little as possible.

Judgemental sampling can be preferred as a sampling methodology over probabilistic sampling when specific parts of a stockpile are to be sampled. This will often be the case when a subpopulation is to be investigated, as the occurrence of that specific subpopulation is often not obvious from “the outside” of the stockpile. Probabilistic sampling of the subpopulation will then be practically impossible.

EXAMPLE 1 In a soil stockpile, specific particles appear to have an unexpected colour. It is decided that these particles should be investigated. It is however impossible to sample these particles according to the conditions of probabilistic sampling. Therefore, only part of these particles – the ones that are present on the outside of the stockpile – will be sampled.

EXAMPLE 2 In a soil stockpile, a small portion of old bricks and building materials is present. There is reason to believe that the concentrations of e.g. heavy metals in the soil are significantly different from the concentrations in the stones and bricks. Therefore, the soil material and the stones and bricks will be sampled individually. The soil will be sampled according to the conditions of probabilistic sampling, whilst the stones and bricks will be sampled as far as they are visible on the outside of the stockpile.

Accordingly, two types of judgemental sampling can usefully be distinguished:

- informative judgemental sampling;
- non-informative judgemental sampling.

6.4.3 Informative judgemental sampling

The use of judgemental sampling will nearly always result in samples being taken from a subpopulation which is substantially more restrictive than the population. Within that subpopulation, however, it may be feasible for the sampling to be probabilistic – in which case, it can be termed "informative" judgemental sampling. This means that the results will still be representative for the part of the population sampled (within which the conditions for probabilistic sampling are met), though it still runs the risk of being biased for the population.

EXAMPLE 1 Samples might be taken at random from the top 50 cm of a stockpile. The advantage of doing this is that it allows statistically sound information to be generated for at least the subpopulation sampled. This makes it easier to assess the possible errors involved in extrapolating to the whole population (i.e. the stockpile), whilst also making explicit the way in which the sampling is unrepresentative.

EXAMPLE 2 Another example is where sampling is restricted to a maximum particle size. For example, samples might be taken using a 3 cm auger for sampling soil with a maximum particle size of 5 cm. The larger particles will not be part of the sample. When the auger size is the only derivation of probabilistic sampling, the samples will be representative for the part of the population. Which part is now more difficult to define as particles of, for example 2,5 cm diameter, will already have only a small probability to enter the auger.

6.4.4 Non-informative judgemental sampling

With non-informative judgemental sampling, in contrast, no attempt is made to achieve even partial representativeness (or perhaps previous attempts have been abandoned). In this situation, there is no way of judging how useful or representative the resulting samples may be.

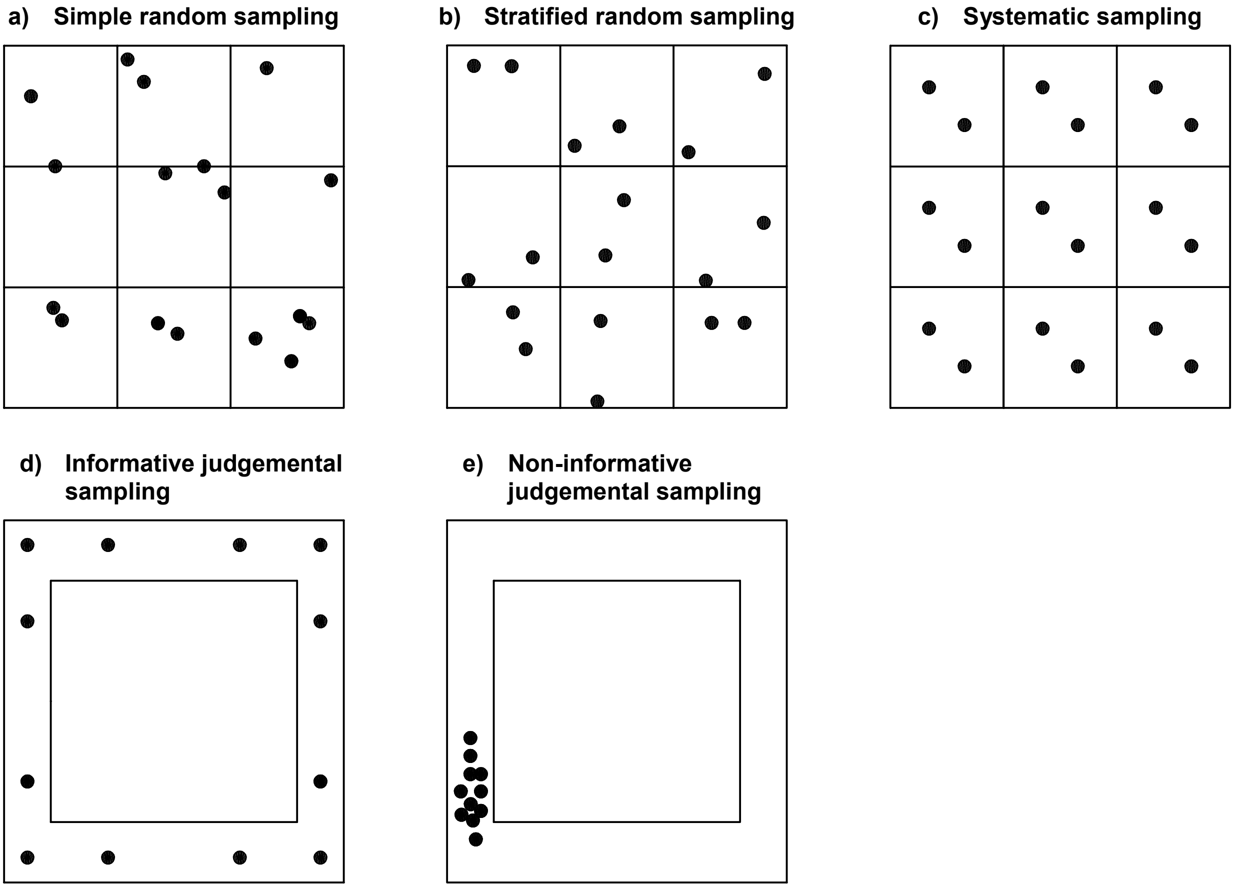
6.5 Sampling locations

6.5.1 General

The location where the samples or increments shall be taken is determined by the sampling pattern. The sampling pattern defines the way in which the samples are selected from the population. Three probabilistic sampling patterns and two options for judgemental sampling are illustrated in Figure 3.

NOTE 1 Figure 3 only provides a conceptual definition of the sampling locations, for simple understanding displayed in a two dimensional sampling situation. A more realistic example of the three-dimensional definition of sampling locations in a soil stockpile is provided in Figure 4. The systematic sampling pattern displayed in Figure 4 is explained in H.1.3.

This Subclause only gives the concepts of the definition of the sampling locations. In Clause 7, methods are given for the sampling of a stockpile according to probabilistic sampling (simple random sampling, stratified random sampling and systematic sampling) as well as judgemental sampling (spot sampling and directional sampling).



a) Simple random sampling

b) Stratified random sampling

c) Systematic sampling

d) Informative judgemental sampling

e) Non-informative judgemental sampling

Figure 3 — Different types of sampling patterns for probabilistic and judgemental sampling

NOTE 2 The figure illustrates the patterns for the context of a two-dimensional spatial area. However, the concepts equally apply to a three-dimensional spatial area, as well as temporal variability.

6.5.2 Simple random sampling

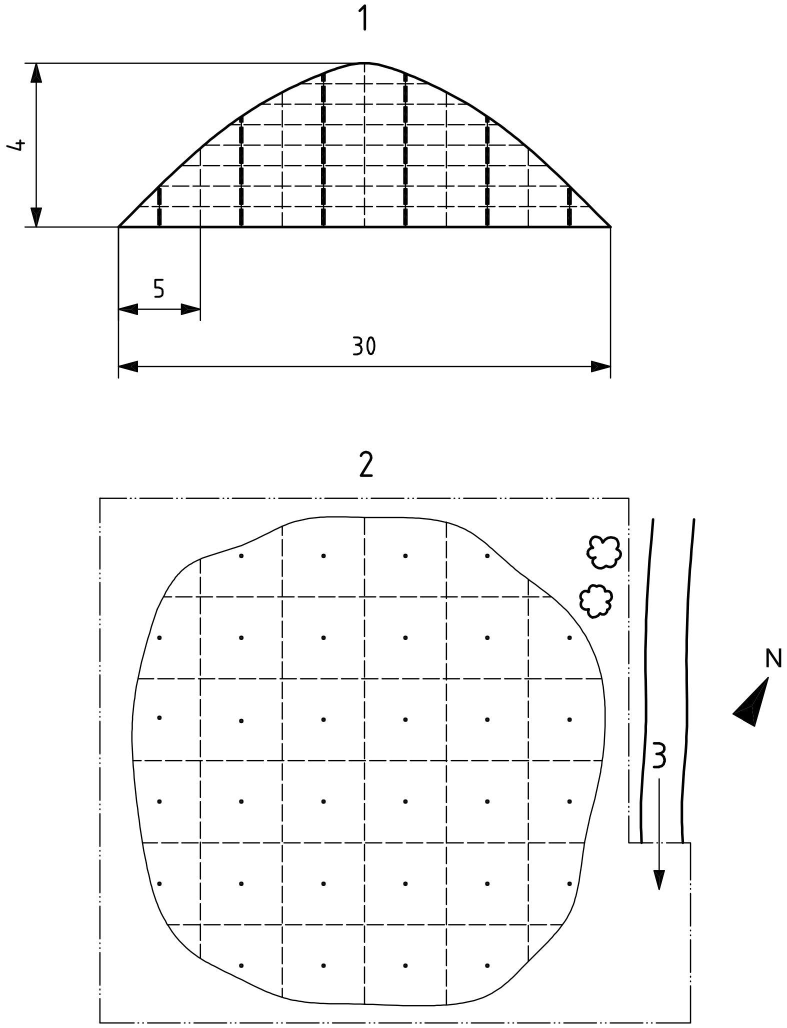
With simple random sampling, every portion of the population has the same (small) chance of being selected as a sample. However, the resulting pattern will not necessarily be very evenly spread across the population. Consequently, other more structured forms of sampling are often preferred to simple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

6.5.3 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, 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. This is the easiest when all strata are of the same size.

NOTE Potentially, it is possible to define the strata based on, for example, the process that resulted in the material to be sampled or knowledge of a spatial differentiation within the material. However, this is not advisable for sampling soil stockpiles, as, in general, the spatial differentiation within the stockpile is unknown or at least uncertain.

Dimensions in metres



Key

1 cross section

2 top view

3 entrance

I increment

· sampling location

The dotted lines indicate the volume represented by each individual sample.

Figure 4 — Example of the definition of a systematic sampling pattern on a soil stockpile

6.5.4 Systematic sampling

With systematic sampling the samples are evenly spaced across the population. Preferably, the systematic pattern is starting from a randomly chosen point.

Systematic sampling 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 which “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 approach.

NOTE Distinct spatial patterns that might interfere with the representativity of samples taken from a systematic sampling pattern are difficult to define in a stockpile. Therefore, in most cases, systematic sampling will result in samples that comply with probabilistic sampling.

6.5.5 Judgemental sampling

Judgemental sampling can embrace a wide variety of sampling patterns, but these can be broadly categorized as informative and non-informative.

Figure 3 d) shows an informative judgemental programme. The subpopulation is the narrow strip around the central region. Within this, however, there is a systematic sampling pattern (chosen such that there is no risk of the samples running in step with any systematic pattern that may be present within the subpopulation). As this is a form of probabilistic sampling, the statistical benefits associated with this approach may be exploited. That is, the methodology of Annex D can be used both to estimate the parameter of interest and also to calculate a confidence interval to quantify the uncertainty surrounding that estimate.

In contrast, the pattern in Figure 3 e) is typical of non-informative judgemental sampling. From this, nothing can reliably be inferred about mean quality except in the immediate vicinity of the sampling. There is, however, one situation in which this type of sampling can be preferable: that is when the purpose of sampling is simply to estimate the characteristics of an atypical material that is unexpectedly present in the population.

6.6 Determining the size and number of samples and increments

6.6.1 General

The sampling plan shall contain specific instructions on the number of increments and/or samples to be taken, the size of the increments and/or samples and, when relevant, the number of increments that should be put together in a composite sample.

NOTE For a good understanding of this clause, the definitions of increment (3.5), sample (3.16) and composite sample (3.4) are essential. Furthermore, the background principle for the estimation of minimum increment size (3.9), the minimum sample size (3.10) and the actual increment size (3.2) and actual sample size (3.3), as given in Annex B, are important.

In order to define the specific instructions needed for the sampling plan, the following activities should be performed in subsequent manner:

a) definition of the type of samples to be taken (see 6.6.2);

b) estimation of the minimum increment and/or sample size (see 6.6.3);

c) definition of the number of increments and/or samples to be taken (see 6.6.4);

d) calculation of the actual increment and/or sample sizes (see 6.6.5).

6.6.2 Definition of the type of samples

Depending on the purpose of sampling (see 6.3), different types of samples shall be taken from the stockpile. A distinction is made between two types of “samples”: increments (3.5) and samples (3.16). For each sampling situation, the type of samples which provide the desired information shall be chosen.

In general:

- One (or only a few) sample(s) will be taken when an indication of the quality of the stockpile is sufficient. Costs of sampling and analysis will be low (spot sampling).
- A (large) number of increments will be taken when a good estimate of the mean quality of the stockpile is desired. The resulting composite sample(s) in which the increments are put together prior to analysis will

result in a reliable estimate of the mean quality. Costs of sampling will be relatively high, but costs of analysis will be low (composite sampling).

- A (large) number of samples will be taken when a good estimate of the quality of the stockpile is desired. In addition to a reliable estimation of the mean quality information on the heterogeneity within the stockpile will also be obtained. Costs of sampling and analyses will be high (spot sampling).

When the purpose of sampling does not directly specify the type of samples, the project manager shall consult the involved parties prior to the definition of the sampling plan (and sampling).

6.6.3 Estimation of increment and sample size

6.6.3.1 General

As mentioned in 6.4.1, one of the requirements of probabilistic sampling is that all particles in the soil stockpile could be part of the sample. This has effect on the scale (volume) of both increments and samples. In this clause and subsequent subclauses, the increment and sample size will be determined. Increment and sample size are estimated in accordance with the following subsequent steps:

Determination of the minimum increment size	see 6.6.3.2;
Determination of the minimum sample size	see 6.6.3.3.

Subsequent steps are the definition of the number of increments and/or samples and the calculation of the actual increment and sample size, see 6.6.

If the sampling is carried out on the basis of a number of increments or samples, the individual increments or samples shall be equal size (± 25 %, mass fraction).

NOTE By using sampling equipment tailored to the material, the increment size is generally laid down much more precisely than the value of ± 25 % indicated for guidance purposes.

6.6.3.2 Estimation of the minimum increment size

The minimum increment size when sampling from a soil stockpile shall, under the different conditions under which the sampling shall be carried out, meet the following requirements:

- The actual width, height and length of the sampling equipment shall be at least equal to three times the maximum particle size (D_{95}) of the material to be sampled in the case of materials with a maximum particle size (D_{95}) of at least 3 mm.

- The actual width, height and length of the sampling equipment shall be at least equal to 10 mm in the case of materials with a maximum particle size (D_{95}) of less than 3 mm.

Annex B provides more detailed information on the estimation of the minimum increment size.

6.6.3.3 Estimation of the minimum sample size

Irrespective of whether composite sampling is to be used, it is important that each sample is sufficiently large for the effect of fundamental variability (see Annex B) to be negligible and meets the quantity requirements for testing and analyses. This is particularly important when the contaminant or characteristic of interest constitutes only a small proportion of the material. Annex B provides an equation for estimating the minimum sample size (by mass).

At the sampling stage, samples shall be taken of at least the size determined on the basis of the formula for the minimum sample size, see Annex B. However, when the actual sample's size is larger than the minimum sample size (see 6.6.5), samples should (at least) comply with the actual sample size.

6.6.4 Definition of the number of increments and/or samples

Irrespective of whether composite sampling is to be used, the next step is to determine the required number of increments and/or samples. E.2.7.1 provides a methodology for determining the required number of increments (m) and samples (n).

The number of increments and/or samples is directly related to the purpose of sampling (see 6.3) and the desired precision and confidence (see D.5.2).

The variability of the soil to be sampled also influences the precision and confidence realized in a sampling programme. It is therefore impossible to fulfil exact requirements for precision and confidence with a one-stage sampling programme.

When sampling consists of taking increments and putting these increments together into one or more composite samples, the number of increments in each composite sample shall be quantified in the sampling plan. In most cases, the number of increments will be equal for all composite samples within the sampling programme, which simplifies statistical analysis.

6.6.5 Calculation of the actual increment and/or sample size

Where composite sampling is not being considered, the question of increment size is irrelevant and the actual sample size is simply set to the minimum sample size as calculated in 6.6.3.3. Additionally, the resulting sample size shall be compared to the amount of material necessary for the desired testing and analysis. If insufficient material is sampled, the actual sample size is enlarged to accommodate all testing and analysis.

Where composite sampling is to be undertaken, there is a possible conflict between the previously calculated minimum values for increment size (see 6.6.3.2) and sample size (see 6.6.3.3), and the planned number of increments (see 6.6.4). Such conflict can be resolved by increasing either the number of increments, the increment size or the sample size, resulting in the actual increment size and/or sample size, as described in B.8. Additionally, the resulting sample size shall be compared to the amount of material necessary for the desired testing and analysis. If insufficient material is sampled, either the number of increments or the increment size is enlarged; resulting in a composite sample sufficiently large to accommodate all testing and analysis.

6.7 Incorporation in the sampling plan

The resulting sampling strategy derived on the basis of the earlier subclauses in Clause 6 shall be incorporated in the sampling plan (see Clause 5). Prior to the actual sampling, the right type of sampling equipment and appropriate sampling technique shall be selected (see Clause 7).

When the circumstances in the field deviate too much from the assumed situation in the sampling plan, the sampling plan should be altered. Depending on the type of alterations, the project manager who made the sampling plan should be consulted prior to actual sampling. Subclause 5.5 gives instructions on the type of alterations that can be made by the sampler, and the alterations for which the project manager should be consulted.

7 Sampling equipment and techniques

7.1 General

This clause describes suitable techniques for the sampling of soil stockpiles found in a variety of locations and consisting of a variety of soil types. This clause of ISO 10381 also gives guidance on the selection and appliance of equipment used in the sampling programme.

Prior to the selection of the sampling equipment and technique, the method of sampling has to be chosen. This involves combining the desired sampling strategy as chosen based on 6.5 and the local situation encountered.

The sampling equipment and sampling technique are closely related. Specific techniques can only be used adequately with specific types of sampling equipment. In 7.2, different sampling techniques suitable for sampling soil stockpiles are described. The sampling equipment to be used for these techniques is mentioned in 7.3, but a more detailed description is given in Annex I.

Finally, having chosen the correct sampling technique and equipment, the results should be incorporated in the sampling plan (see 7.4 and Clause 5). When the sampling plan is defined, sampling can be carried out according to the directions given in Clause 6 and this clause.

For simplicity reasons, in this clause, the material sampled will be referred to as “sample”, irrespective of the fact that it can be either an increment (3.5) or a sample (3.16).

7.2 Sampling techniques

A number of different sampling techniques are defined in this subclause and discussed in more detail in the subsequent subclauses. A distinction is made between sampling techniques that are potentially suitable for probabilistic sampling (see 6.4.1) and sampling techniques that will basically result in non-probabilistic samples and are therefore (in principle) only suitable for judgemental sampling (see 6.4.2). Under specific conditions, the “non-probabilistic” sampling techniques can also result in probabilistic samples, as well as vice versa, and therefore the distinction made in this subclause should only be considered as a rough outline.

7.2.1 Determination of the sampling method

As part of the sampling plan, determine the way in which the sampling shall be carried out.

NOTE 1 The prevailed sampling strategy is based on a random, stratified random, or, under specific conditions, systematic selection of the sampling location(s) by means of which probabilistic samples can be obtained.

The method of sampling is determined by the conditions under which the sampling shall be carried out. These conditions are determined by both the (type of) soil to be sampled, the size of the stockpile and the accessibility of the stockpile, as well as the time and financial possibilities for sampling.

NOTE 2 Sampling during transportation of the soil, by means of sampling from a conveyor belt or sampling from a mechanical shovel used to transfer the soil, is often to be preferred above sampling from a static stockpile. During transport probabilistic sampling is often much easier to accomplish than from the stockpile itself. However, in daily practice, it is most often not possible to sample the soil during transportation and thus sampling will be carried out from the static stockpile.

The sampling shall be carried out in accordance with probabilistic sampling as defined in 7.2.2 and H.1. Only if probabilistic sampling is not feasible shall the sampling be carried out in accordance with judgemental sampling, as defined in 7.2.3 and H.2. In view of the difference in quality between the samples obtained by probabilistic sampling and judgemental sampling, priority is given to probabilistic sampling.

7.2.2 Sampling techniques for probabilistic sampling

The following sampling techniques will, when applied correctly, result in probabilistic samples:

- simple random sampling;
- stratified random sampling;
- systematic sampling.

See also 6.5.

In the case of a stockpile which, due to the way it is available for sampling, cannot be satisfactorily differentiated from adjoining stockpiles, a “safety margin” shall be applied in connection with the spatial definition of the stockpile such that adjoining stockpiles do not partially overlap.

NOTE The size of the sampled stockpile is actually reduced by this safety margin. The material in the safety margin (the area where the adjoining stockpiles possibly overlap each other partially) can therefore not be assessed.

See Annex H for more details on the different sampling techniques.

7.2.3 Sampling techniques for judgemental sampling

When applying judgemental sampling, it can be preferred to deviate as little as possible from the probabilistic sampling. In those situations, the sampling techniques as mentioned in 7.2.2 and H.1 shall be used. In other situations, these sampling techniques are not applicable, or other types of sampling are preferential given the purpose of sampling. The following sampling techniques will normally not result in probabilistic samples, but are applicable for judgemental sampling:

- spot sampling;
- directional sampling.

See Annex H for more details on the different sampling techniques.

7.3 Sampling equipment

Selecting suitable sampling equipment is important for obtaining good quality samples. Advice should be sought on:

a) the risk assessment of the sampling activities and the safety procedures to be implemented during sampling and transport;

b) the moisture content of the soil;

c) the maximum size and size distribution of the soil particles;

d) the accessibility of sampling points;

e) the quantity of soil necessary for the tests and analyses.

In the case of soil stockpiles, augers, drill sampling tubes, scoops and shovels are used. Sampling equipment, ancillary apparatus (and sampling containers) should be made of materials that do not interact physically or chemically with the sample.

Basic requirements common to all sampling equipment and ancillary apparatus are

- suitability for purpose,
- safety in operation,
- ability to take a representative sample from the required sampling point,
- capability of preserving the integrity of the sample until it can be transferred to a sample container,
- ability to be cleaned,
- simplicity in use,
- practicality of use, and
- ability to withstand rough usage.

Suggested applications for generic types of equipment are detailed in Table 2.

Table 2 — Suggested applications for generic types of sampling equipment suitable for sampling soil stockpiles

Generic sampling apparatus	Dry fine grained soil	Moist fine grained soil	Dry coarse grained soil	Moist coarse grained soil	Very coarse soils [a]
Soil auger	+/–	+	+	+	–
Drill auger	–	+	+	+	–
Mechanical drill	–	–	–	–	+ [b]
Open sampling tube	–	+	–	–	–
Half cut sampling tube	+	+ [c]	–	–	–
Plunger sampling tube	+/–	+	–	–	–
Scoop	+/– [d]	+	+	+	+
Mechanical shovel	–	–	–	+	+

[a] Soils consisting of particles larger than 50 mm diameter.

[b] Suitable for taking part of the individual particle.

[c] Only suitable for a sludge.

[d] Suitability depending on wind velocity.

Detailed descriptions of the equipment identified in Table 2 are given in Annex I.

The size of the sampling equipment depends on the (maximum) particle size of the soil and the quantity of sample required. Depending on whether increments (3.5) or samples (3.16) are taken, the size of the sampling equipment should at least be equal to the actual increment or sample size respectively (see 6.6.5). In general, the opening used for sampling should at least be three times the diameter of the maximum particle size (D_{95}) in all directions that are relevant for the sampling process.

7.4 Incorporation in the sampling plan

Before actual sampling, the selected sampling equipment shall be prescribed in the sampling plan, see Clause 5.

When the circumstances in the field deviate too much from the assumed situation in the sampling plan, the sampling plan should be altered. Depending on the type of alterations, the project manager who made the sampling plan should be consulted prior to actual sampling. Subclause 5.5 gives instructions on the type of alterations that can be made by the sampler, and the alterations for which the project manager should be consulted.

7.5 Sampling

Prior to sampling, all elements of the sampling plan should be checked (see also 7.4). As a second step, the identity of the soil stockpile should be checked and recorded in such manner that it can be checked again on a later date. The most appropriate method is to photograph the stockpile.

NOTE Verification of the identity of the stockpile is often essential, specifically when the stockpile is publicly accessible or on a production site. Changes to the identity of the soil stockpile, either due to adding or removing (part of) the soil, will result in loss of validity of the sampling results.

For further identification of the stockpile, a number of visual characteristics can also be useful (e.g. colour, particle size, soil type). These characteristics should be noted by the sampler and be checked against the sampling plan when these kinds of characteristics are already mentioned there.

When the sampling plan is fully checked, the sampling locations shall be defined on the actual stockpile. This can be done either for all sampling locations at once or on individual bases prior to the sampling at that specific point.

After defining the sampling location, the sample is taken using the defined type of sampling equipment and applying the correct sampling technique.

Having obtained the sample, it is either directly stored in a suitable sample container (see Clause 9), or stored after appropriate sample pretreatment in the field, in accordance with Clause 8.

8 Sample pretreatment

8.1 General

Sample pretreatment is the process of subsampling, necessary to obtain a representative subsample for packaging and transport to the laboratory.

In sample pretreatment, two types of sample manipulation can be recognized:

- sample division: obtaining subsamples of smaller size than the original sample without reducing the particle size of the individual particles;
- particle size reduction: grinding the sample in order to reduce the particle size of the whole (sub)sample without reducing the sample size (mass).

Sample pretreatment is almost always necessary, as the amount of material sampled is larger than the amount of material necessary for the test or analysis.

When possible, the sample pretreatment shall take place in the laboratory, as sample integrity can be best guaranteed under laboratory conditions. However, the sampling of coarse material and the necessity to use large sampling equipment (e.g. a shovel) can result in (very) large samples. Sample pretreatment “in the field” can then be advisable in order to prohibit that these large samples have to be transported to the laboratory. In these situations, sample pretreatment “in the field”, directly after sampling, is advisable.

The requirements for sample pretreatment in the field are the same as for sample pretreatment in the laboratory. As the circumstances are in most situations not at all comparable to laboratory conditions, the type of sample pretreatment that is allowed in the field is limited to sample division. Only when laboratory conditions are available on site (there is a sample pretreatment laboratory/facility present), can the full range of sample pretreatment activities – thus also including particle size reduction – be carried out directly after sampling. As these conditions are only met in exceptional cases, sample pretreatment in this part of ISO 10381 only provides directions for the sample division that is possible in the field.

Whenever volatile components are to be determined, the process of sample pretreatment can result in a substantial loss of these components. Sample pretreatment shall be omitted in these cases by taking specific samples for the determination of volatile components. These samples shall be sealed directly after sampling, cooled and analysed as soon as possible after sampling.

In the case of very coarse soils, it can be necessary to reduce the particle size of the larger particles in the field in order to be able to send a representative sample of an acceptable size to the laboratory. When grinding or crushing “in the field” is truly necessary, measures have to be taken in order to prevent contamination and/or loss of both components and soil material. This part of ISO 10381 does not give instructions for these situations. In general, the basic International Standards for sample pretreatment are ISO 11464 and ISO 14507.

One should realise that the quality of subsampling in the field is less than the quality of subsampling in the laboratory, due both to the (environmental) circumstances for subsampling and to the inability to use the best possible subsampling method. When transfer of the individual sample(s) or composite sample(s) to the laboratory is possible, this should be considered as a preferable option.

8.2 Requirements

8.2.1 General

In most methods of sample pretreatment, there is a risk that the final composition of the subsample(s) will differ from the composition of the original individual sample or composite sample. This can be due to the nature of the material or the method selected for sample division. Especially the particle size reduction is a potential source of large changes in the composition of samples, and is therefore (in principle) only allowed in a fully-equipped pretreatment laboratory. Nevertheless, sample division can also result in significant changes in the composition of the material when no or inadequate precautions are taken. Examples include loss of moisture or volatile components due to evaporation and loss of fine particles due to air entrainment. When particle size reduction is applied contamination of the sample by abrasion or pick-up from the crushing surfaces and oxidation of newly exposed surfaces also influence the sample integrity.

It is therefore preferable to choose a method of sample pretreatment that causes the minimum possible change in composition particularly with respect to subsequent requirements of the material.

8.2.2 Minimum size of the subsample

The minimum size of the subsample is determined by the maximum size of the particles that are present in the sample. When the sample contains macro-aggregates, the maximum size of the macro-aggregates determines the minimum size of the subsamples whenever the macro-aggregates behave like individual particles during sample pretreatment, i.e. when macro-aggregates are not cut into pieces by the (sub)sampling equipment used. (See also 8.4.2 for macro-aggregate size reduction.)

The relation between the minimum size of the subsamples and the maximum size of the particles (D_{95}) in the original sample is given in Table 3.

- The relation is based on the formula for the minimum sample size as given in Annex B.
- For the variables, it was assumed that:
  - the density of the particles (ρ_{particle}) is 2,6 g/cm^3;
  - the coefficient of variation due to the fundamental error ($\text{CV}_{\text{fund. error}}$) is 0,1; and
  - the fraction of the particles that contains the constituent of interest (w_{particle}) is 0,02.

Table 3 — Minimum size of subsamples as a function of the maximum size of macro-aggregates or particles present in the sample

Maximum size of macro-aggregates or particles in the sample mm	Minimum size of subsample(s) g
0,2	0,01
0,4	0,1
0,6	0,4
0,8	0,8
1	2
2	15
4	110
6	360
8	850
10	1 600
12	2 900
14	4 600
16	6 800
18	9 700
20	13 000
22	18 000
24	23 000
26	29 000

8.2.3 Notes to Table 3 and practical considerations

a) Particle sizes: Although Table 3 gives the minimum sample size for pretreatment for soil with a maximum particle size varying between 0,2 mm and 26 mm, this does not imply a limitation to the particle sizes of soils.

b) Linking minimum subsample size and maximum particle size assumptions: In the above assumptions, for linking the minimum subsample size with the maximum particle size, it is assumed that (approximately) one in every fifty particles contains the constituent of interest, and the concentration of the component to be determined per particle does not vary too widely. If these assumptions are not valid, Annex B should be consulted for more information on the minimum (sub)sample size.

c) Minimum sizes of subsamples: The minimum sizes of the subsamples, as provided in Table 3, are only based on the theoretical relation between the particle size and the minimum sample size.

    1) The minimum sizes of the subsamples were calculated with the following assumptions: density of the particles (ρ_{particle}) is 2,6 g/cm^3; coefficient of variation due to the fundamental error ($\text{CV}_{\text{fund. error}}$) is 0,1; fraction of the particles that contains the constituent of interest (w_{particle}) is 0,02.

    2) In addition, it is also assumed that the subsamples are taken by a single action, and therefore the requirements of the increment size are of no influence.

   3) In practice, the minimum size of subsamples should also be determined by the amount of material necessary for laboratory analyses. Under the above assumptions and a given maximum particle size, subsamples might never be smaller than specified in Table 3. When more material is needed for analysis, the size of the (final) subsample shall be enlarged. When less material is needed for analysis, the particle size shall be reduced before further subsampling is applied to obtain the required amount of sample material. The latter type of sample pretreatment (particle size reduction) shall be carried out under laboratory conditions.

   4) Although the maximum particle size of a soil is 0,6 mm, and thus the minimum (sub)sample size is 0,4 g, the minimum subsample for a specific analysis should not be smaller than 20 g, as this is the amount of material necessary for the analysis.

d) Volume versus mass: The minimum sample size potentially can be defined as a volume or as a mass. However, as the volume per mass ratio can vary significantly, a volume-based minimum sample size is more variable than a mass-based minimum sample size, and thus the latter should be used as a basis.

e) Boulders: Some soils (partly) contain (very) large boulders. If these boulders were considered as part of the sample, this would result in extremely large samples, both in the field and for the material to be transferred to the laboratory. However, often only the smaller soil fraction is of interest, and therefore, these boulders can be neglected both during sampling and sample pretreatment. Whenever such a situation is encountered, the sampling plan should clearly define the material that is to be sampled/subsampled.

f) Small particle sizes: For small particle sizes, the minimum size of the subsample can be very small, which is relevant for subsampling in the laboratory, in order to obtain the analytical sample. For subsampling in the field, a minimum amount of approximately 200 g should at least be transferred as sample to the laboratory. Further subsampling will then take place in the laboratory.

g) Actual size: It shall be noted that the minimum size of the subsample(s) as given in Table 3 does not necessarily mean that this is the actual size to be used. Larger sizes of subsamples might be needed for analysis, and therefore the size of the subsample(s) shall be checked with the laboratory (see also Item c) 3).

h) Laboratory samples: For practical reasons, the maximum size of the samples to be sent to the laboratory should not be larger than approximately 20 kg to 30 kg. When larger subsamples are needed because of the large particle size, the particle size shall be reduced adjacent to sampling. As particle size reduction is (in principle) only allowed under laboratory conditions, see 8.1, for these situations either a mobile laboratory or on site laboratory is needed.

i) Integrity of sampling: Sample division into a number of representative subsamples can only be carried out in the field when the integrity of the sample and subsamples can be assured. To assure this effectively a sheltered area is necessary in most situations. Without adequate shelter, weather conditions like wind and rain can pose a serious threat to the quality of the samples.

j) Compounds to be analyzed / test: Finally, the compounds to be analyzed in the (sub)sample(s), or the test to be carried out, will in some cases affect the possibilities or methods of subsampling.

8.3 Equipment for sample pretreatment

For the purposes of sample pretreatment, one or more of the following apparatus, as identified in the sampling plan, is required:

- large heavy-duty plastic sheeting;
- spade;
- sledge hammer;
- mechanical shovel;

- riffle box;
- Tyler divider;
- mechanized turntable/rotating dividers.

8.4 Pretreatment methods

8.4.1 Making composite samples

When composite samples are produced, the basic principle is that the composite samples should contain equivalent quantities (mass fraction) of the individual increments. The quantities of the increments to be mixed together should be determined on the basis of the dry matter content of the individual increments. As it is not possible to determine the dry matter content in the field, two options for the composition of composite samples are possible:

- putting the increments together in the field;
- putting the increments together in the laboratory.

When increments are put together in the field, it is essential that it may be assumed that the dry matter content for all increments is (approximately) the same. In most cases, where increments are taken from the same stockpile, this will be the case. Increments of the same size/mass can then be put together in the field. Putting increments together of the same volume/mass from two or more stockpiles, can result in over sampling one of the stockpiles when the dry matter content of that stockpile is significantly higher than the others. When a significant deviation in dry matter content is expected, the increments should be put together in the laboratory after determining the dry matter content of each of the individual increments.

NOTE 1 In practice, it is often assumed that increments can actually be mixed, for instance by stirring. Mixing of particulate materials is however very difficult, especially when the increments have a different particle size distribution, moisture content or soil types. Appropriate mixing can only be realized by specific sample pretreatment methods.

Mixing increments in the field is therefore hardly possible and shall be avoided.

When the composite sample is larger than 20 kg to 30 kg, the size of the composite sample may be divided in accordance with Annex J.

When the composite sample is smaller than 20 kg to 30 kg, subsamples may only be taken after particle size reduction for the full composite sample. Particle size reduction is only allowed under laboratory conditions.

NOTE 2 When the composite sample exceeds 20 kg to 30 kg, it is preferable to transfer the individual increments to the laboratory to produce the composite sample and carry out the subsequent subsampling.

8.4.2 Procedure for macro-aggregate reduction by hand

In some cases, the soil is strongly aggregated. Macro-aggregates should be seen as individual “particles” when the method of sampling and sample pretreatment is not able to sample part of a macro-aggregate. For sample pretreatment, this happens for instance when a riffle box is used for dividing a moist or clay-like soil. As the particle size determines the minimum size of the subsample(s), it will be preferable when the size of macro-aggregates can be reduced during or prior to subsampling.

As reduction of macro-aggregates by hand will result in a relatively long and intense contact of the sample with the air, this method may only be applied when sample integrity is not influenced during this period.

Identify the maximum size of the macro-aggregates, using the minimum size of the subsample as a starting point, as given in Table 3. When the desired size of the subsample is smaller than a given minimum size of the subsample, further reduction of the macro-aggregate size is necessary.

- Identify an area of hard surface sheltered from the effects of wind and rain, preferably flat and large enough to allow ease of access around the whole sample when spread evenly on the surface.
- Place a clean protective floor covering, preferably heavy-duty plastic sheeting, to protect the sample from contamination by the surface.
- Place the sample on the covering/plastic sheeting and spread evenly to identify all macro-aggregates within the sample.
- Using the base of a spade or the head on a sledge hammer gently reduce the size of the macro-aggregates until all oversized material is less than or equal to the required particle size.

8.4.3 Subsampling methods

A sample can be divided into subsamples or analytical samples either mechanically or manually. Potentially, it is preferable to use a mechanical system for subsampling, since this results in more representative subsamples. This is however only true when the material is dry and particles can move through a stream of particles on an individual basis. This situation can be realised in the laboratory, but is not possible for subsampling in the field directly after sampling. If the particles in the sample behave cohesively, mechanical division is often impossible due to cohesion of soil in the system and subsequent blockage of the divider. And even when the mechanical division is still possible, mechanical sub-sampling devices will probably function incorrectly, and therefore will result in biased subsamples. As a consequence, the manual subsampling methods are often to be preferred for subsampling in the field.

Annex J describes the following subsampling methods:

- long pile and alternate shovel method, (see J.1);
- coning and quartering, (see J.2);
- riffling, (see J.3);
- application of Tyler divider, (see J.4);
- application of mechanized turntable (rotating divider), (see J.5).

8.5 Incorporation in the sampling plan

Before actual pretreatment, the selected pretreatment method and necessary equipment shall be prescribed in the sampling plan (see Clause 5).

When the circumstances in the field deviate too much from the assumed situation in the sampling plan, the sampling plan should be altered. Depending on the type of alterations, the project manager who made the sampling plan should be consulted prior to actual pretreatment. Subclause 5.5 gives instructions on the type of alterations that can be made by the sampler, and the alterations for which the project manager should be consulted.

8.6 Pretreatment

Prior to pretreatment, the apparatus and tools used for the pretreatment shall be cleaned in order to prohibit cross-contamination.

The characteristics of the soil to be pretreated shall be checked against the method described in the sampling plan. Basically, the maximum size of the particles is important, as is the moisture content. The latter is related to the inclination to macro-aggregate formation and cohesive behaviour.

When the assigned pretreatment method in the sampling plan is checked, the location for sample pretreatment shall be chosen and the location shall be made fit for use by cleaning it of all materials that can influence the integrity of the (sub)sample(s). When all preparations are ready, the sample pretreatment shall be carried out using the defined type of equipment and applying the correct pretreatment technique.

Having obtained the subsample(s), it/these shall be stored directly in a suitable sample container (see Clause 9).

9 Packing, preservation, storing, transport and delivery

9.1 General

Soil samples are liable to change as a result of various causes, including:

a) microbiological activity in the soil sample;

b) oxidation of compounds by atmospheric oxygen;

c) loss of volatile components;

d) changes in the basic characteristics of the soil, like the redox-potential;

e) irreversible adsorption on the surface of containers or transport through the packaging material.

To ensure the integrity and identity of the sample, this part of ISO 10381 therefore describes methods, materials and requirements for:

- packing the sample(s);
- preserving the sample(s);
- storing the sample(s) prior to transport;
- transporting the sample(s), and
- delivering the sample(s) to the laboratory.

In order to be able to give the correct instructions to the sampler, the project manager should consult the laboratory in order to determine the type and size of sample(s), type of containers, appropriate preservation method if applicable, maximum storage time prior to analysis and the labelling system. The maximum storage time prior to analysis will indicate the period of time available before the sample shall arrive at the laboratory. In general, this period shall be as short as possible.

Packing the sample is described in 9.2. In general terms, this subclause gives guidance for selecting a suitable package material, based on the different types of characteristics to be determined in the sample. In addition to packing the sample, the sample container shall be labelled, giving the sample a unique code. Subclause 9.2 also provides guidance for the labelling of the sample.

Preservation of the sample is described in 9.3. In most cases, the suitable method of preservation will only be storing the sample in a dark and cool environment. In very specific cases, preservation of the sample in a nitrogen gas atmosphere can be necessary; for these cases, a method is also described.

Storage of the sample, as described in this part of ISO 10381, deals only with the short-term storage of the sample between sampling and, when relevant, sample pretreatment in the field and transport of the sample. This short-term storage is described in 9.4. As the preservation of the sample will in most situations consist of storing the sample in a dark and cool environment, storage and preservation are in practice often the same.

After sampling and, when relevant, sample pretreatment in the field, the sample shall be transported to a facility where it shall be tested or analysed. In most situations, this facility will not be located on the sampling location, and as a consequence the sample has to be transported. Subclause 9.5 gives guidance for the handling and storage of the samples during transport.

Finally, the sample shall be delivered at the facility where it will be processed further. To ensure that the sample can be tracked, certain procedures should be followed. General instructions for the delivery of the sample are given in 9.6.

Having determined the methods and materials to apply, the results have to be incorporated in the sampling plan according to 9.7. Finally, the sample has to be packed, preserved, stored, transported and delivered at the laboratory. Guidance for these activities is given in 9.8.

9.2 Packing the sample

9.2.1 Selecting an appropriate sample container

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 soil sample and the components to be analysed. Contamination of the sample from contact with inappropriate sample containers or cleaning reagents should be avoided. The container size should be appropriate to the volume of the required sample. Sample containers should be properly sealed. The type and size of the container should prevent changes in the sampled.

The following general precautions are recommended to minimize chemical/physical and biological changes while the sample is stored within a container from the point of sampling to the time of analysis:

- The type and size of container should be such that the volume of headspace is appropriate to the sample and the required components, to prevent changes in the sample material. In the majority of cases, the containers should be completely filled. Air space should be minimal to prevent significant oxidation reactions, both at the top of the container, as well as in between the soil particles. When freezing is a method for prolonged preservation, some additional space is needed within the container to allow expansion.

NOTE Freezing (and drying) is obviously a preservation technique that falls outside the scope of this part of ISO 10381. When freezing is used for preservation, the sampler shall leave the necessary additional space in the sampling containers after consultation with the laboratory.

- Sample containers should be clean and dry. The choice of cleaning method will depend on the components to be analysed. Containers may be cleaned with mixtures of acids followed by rinsing with deionised water. Cleaning procedures may differ depending on the type of components to be analysed. Advice should be sought from the laboratory or other experts in order to establish the most suitable procedure in each case. In general, the re-use of sample containers is not advisable.

- Where possible, use wide neck rigid containers with screw caps and inert seals.

- When the sample is required for inorganic analysis, a suitable plastic bottle should be used. Where organic constituents are required, glass bottles should be employed. Tamperproof seals are essential for samples collected for regulatory purposes.

- All samples should be protected from light and heat and preferably be stored in a cool environment. Cooling of the sample is essential when volatile components are to be determined. When storage in a dark environment (e.g. cool box) is not possible, a bottle of dark glass should be used.

- Fitness for purpose is an important criterion for container selection, the following points should be considered when selecting and preparing sample containers:

  - adsorption into the walls of the container;

  - contamination of the container prior to sampling by improper cleaning;

- contamination of the sample by the material of which the container is made;
- reaction between components in the sample and the container;
- resistance to temperature extremes;
- resistance to breakage;
- water and gas tightness;
- ease of reopening;
- size, shape and mass volume;
- availability;
- cost.

Table 4 represents a summary of the advantages and disadvantages of collection containers in common usage.

Laboratory samples for dispatch or transport by third parties and reserved laboratory samples should be sealed in such manner that the integrity of the sample can be guaranteed. Appropriate sealing of the samples shall be required when samples are taken for (potential) regulatory investigations.

9.2.2 Labelling

All samples shall bear a clearly legible, unambiguous code that shall ensure the identity of the sample. The coding should comply with the sampler's quality system.

Care should be taken that the sample code remains clearly legible whatever the storage period and prevailing conditions (e.g. condense on cooling).

The labels should be secured on the sample containers, adequately preventing the losing or interchanging of labels between samples. Whenever the labels are attached to the lid, top or cap of the container, an identical label shall also be attached to the container itself.

The label should carry, in indelible ink, all the information necessary for unequivocal identification of the sample, but should be kept short and simple to avoid mistakes when transcribing numbers. More detailed paper-based or electronic record sheets should accompany the sample in order to provide an audit trail or relevant information.

Labels and inscriptions should be prepared using indelible ink. Pre-printed labels and bar-code labels can be a practical alternative.

Table 4 — Selection of sampling containers

Suitability for use									Inorganic analyses	Organic analyses
Type of container	Resistance to extreme temperature	Cost of purchase	Resistance to breakage	Degree of water and gas tightness	Ease of reopening	Suitability for volatiles	Size availability			
							Small	Large		
Rigid plastic with screw caps or snap on lids [a]	+	++	+	–	+	–	+	+	+	–
Glass bottles or jars with plastic caps and PTFE [b] seals [a]	+	+	–	+	+	+	+	–	–	+
Coloured plastic bottles with plastic caps and PTFE [b] seals	+	+	+	+	+	+	+	+	+	–
Coloured glass bottles with plastic caps and PTFE [b] seals	+	+	–	+	+	+	+	–	–	+
Heavy duty re-sealable plastic polythene bags	–	++	+	–	+	–	+	+	+	–

a) The use of wide neck plastic and glass bottles is preferable for soil sampling.

b) PTFE = Polytetrafluorethylene

9.3 Preserving the sample

9.3.1 General

Ideally, soil samples should be analysed immediately after collection. However, this is seldom possible. The components to be determined and the length of time for which the sample shall be kept prior to analysis influence the choice of preservation method. Although this part of ISO 10381 only considers the preservation between sampling and, when relevant, sample pretreatment in the field, the chosen method of preservation will often be the method for the whole period before analysis. A change of preservation method on arrival at the laboratory would often result in repackaging the sample, which could do more harm than good to the sample.

A number of preservation methods are available for soil samples:

- airtight storage;
- dark storage;
- cooled storage (< 6 °C);
- nitrogen atmosphere.

Other preservation methods are also available, like drying or freezing, but can not be applied in the field and are therefore not incorporated in this part of ISO 10381.

The method of preservation will influence the acceptable time between sampling and analysis. However, in general, the time between sampling and analysis should be kept to a minimum to avoid sample alteration. This is particularly important in samples in which biological degradation is likely to occur, or in which (semi-)volatile organic components are to be determined. For these types of samples, a period of more than 4 days between sampling and analysis can generally be considered as the maximum storage period, even though the samples were preserved as well as possible. Longer storage will result in significant loss of biodegradable, volatile and (semi-)volatile components, and will therefore make the sample worthless. The shorter the period between sampling and analysis, the better the representativity of these types of samples.

Specific types of soils are known to have a reducing character. If the materials' reducing character is to be preserved, special steps shall be taken when storing the sample. This implies that the presence of oxygen shall be eliminated.

9.3.2 Necessary preservation

In Table 5, necessary preservation measures are given for different types of components to be determined and for reducing soils.

Table 5 — Necessary (+) preservation circumstances and measures for different types of components and soils

	Volatile components	Semi-volatile components	Non-volatile inorganic components	Reducing soils [a]
Airtight storage	+	+	—	+
Dark storage	+	+	—	—
Cooled storage (< 6 °C) [e]	+	+	—	+
Nitrogen atmosphere	—	—	—	+
Maximum period of storage [b]	< 4	4	[c]	< 4 [d]

[a] When reducing characteristics are to be maintained.

[b] When stored with the appropriate preservation method.

[c] No maximum period when dried prior to storage.

[d] Maximum storage period depending on the airtightness of the sample container, but maximum of 4 days.

[e] The temperature should be (4 ± 2) °C.

The preservation measures in Table 5 are to be considered as minimum measures. Additional measures may be applied (like storage for the analysis of non-volatile inorganic compounds in a cool environment).

NOTE 1 The maximum period of 4 days is based on ISO 15009.

NOTE 2 Volatile components are organic and inorganic components with a boiling point under 300 °C. All organic components with a boiling point above 300 °C should be considered as semi-volatile according to ISO 14507.

The project manager should contact the laboratory that will carry out the analyses, or alternatively other experts, to get adequate advice for defining the method of preservation in the sampling plan.

9.3.3 Preservation methods

9.3.3.1 Airtight storage

Soil samples can only be stored airtight when:

- the closing mechanism of the sample container is completely clean of soil particles;
- the material of the sample container is airtight.

Most plastics are not to be considered as airtight.

NOTE Airtight storage will prevent volatilization of components and will reduce biological degradation. However, the air volume within the soil sample is always large; independent of tight filling of the sample container. For composite samples, the sample container will often be larger than the total volume of the number of increments within that composite sample. Nevertheless, care should be taken that the “head space” in the sample container is as small as practically possible. As a consequence, airtight storage is never a guarantee for keeping the characteristics of the sample constant during the storage period.

Best airtight storage of soil samples is obtained when storing the samples in glass bottles sealed with PTFE cap liners. In general, this is only possible for fine grained soils.

9.3.3.2 Dark storage

Dark storage is possible by using dark coloured sample containers or by storing the containers in a dark place. A cool box can provide both a cool and dark environment.

9.3.3.3 Cooled storage

Cool boxes and freezer packs are in general not capable of actual cooling of materials, they just keep cold material cold. When cooling is essential, a mobile (car) refrigerator should be used. Samples should be cooled to a temperature of 4 °C ± 2 °C. Samples should reach this temperature within 12 h, but preferably as soon as possible after sampling.

9.3.3.4 Nitrogen atmosphere

If the reducing character of the soil sample is to be preserved, exposure to oxygen shall be limited as much as possible after sampling. When possible, the sample container has to be flushed with nitrogen in the field. A sample that has not been flushed with nitrogen in the field and whose reducing character is to be preserved shall then be flushed with nitrogen in the laboratory as soon as possible, but at least within 24 h after sampling.

NOTE 1 Nitrogen gas of technical quality suffices.

With or without flushing, the sample should be packed in a gas-tight container.

The volume of nitrogen passed through the container should be at least ten times the container volume. It is recommended that the soil sample is flushed again with nitrogen after an interval of e.g. 24 h to remove any oxygen that may still be diffusing from the sample.

NOTE 2 Nitrogen flushing is easier if there is a gas connector (inlet/outlet) on the container.

Long-term storage under nitrogen is possible only in containers with a hermetic gas-tight seal such as a glass sample container welded shut.

9.4 Storing the sample prior to transport

Sample storing prior to transport is the period between the moment the sample is taken and packed until the moment the sample is transported towards the facility were it is delivered for further treatment (analysis).

Samples shall be stored prior to transport under the preservation conditions, as defined in 9.3.

In order to ensure the quality and representativity of the samples, it is essential to prohibit cross-contamination between samples as well as contamination of the samples from materials stored nearby the samples. When samples with high concentrations of volatile components are obtained, these should be separated from other samples.

9.5 Transporting the sample

Samples shall be stored during transport under the preservation conditions, as defined in 9.3.

To ensure the quality and representativity of the samples, it is essential to prohibit contamination of the samples from materials stored nearby the samples. Especially the storage of gasoline and or gasoline products in the same enclosed space as the samples can lead to serious contamination of the samples with volatile organic components and is therefore not allowed.

To ensure that a sample reaches its correct destination, it shall be correctly packaged, clearly labelled and sent with a clear chain of custody form. At each moment that the samples are transferred, the chain of custody form shall be checked and signed.

Details of the agreed transport and labelling arrangements should be written into the sampling plan as well as on the chain of custody form.

Laboratory samples for dispatch or transport by third parties and reserved laboratory samples should be sealed in such manner that the integrity of the sample can be guaranteed. Appropriate sealing of the samples shall be required when samples are taken for (potential) regulatory investigations.

The packaging should meet the requirements of the authorities or other organization(s) concerned with the transport of the sample.

Glass containers should be protected from potential breakage during transport by appropriate packaging.

9.6 Delivering the sample

Directly after sampling a chain of custody form shall be prepared for each (group of) sample(s), indicating the type of sample (e.g. date, size, specific conditions, necessary preservation and storing conditions) and the analyses to be conducted on the sample. An example of a chain of custody form is given in Annex A.

The chain of custody form shall be checked and signed at each transfer of the samples.

NOTE Samples will under normal circumstances at least be transferred between the sampler and the laboratory. When a third party transports the samples, there will be at least two moments when the samples are transferred.

9.7 Incorporation in the sampling plan

Preservation techniques, storage time, sample containers and collection of duplicate samples and blanks for quality control and quality assurance should be established at the design phase of the sample programme, in consultation with the laboratory analyst.

Before actually packing, preserving, storing, transporting and delivering the sample, the selected methods and necessary materials shall be prescribed in the sampling plan (see Clause 5).

When the circumstances of one or more of these steps deviate too much from the assumed situation in the sampling plan, the sampling plan should be altered. Depending on the type of alterations, the project manager who made the sampling plan should be consulted prior to any further activities. Subclause 5.5 gives instructions on the type of alterations that can be made by the sampler, and the alterations for which the project manager should be consulted.

9.8 Actual packing, preservation, storing, transport and delivery

The procedure consists of the following steps.

- Put the sample into the sample container selected in accordance with 9.2.1.
- Fill the sample container completely.

- When necessary, clean the outside of the sample container enabling a good closure of the container.
- Close the sample container securely.
- Wipe the outside of the container.
- Attach a label in accordance with 9.2.2.
- Preserve the sample in accordance with 9.3.
- Store the sample in accordance with 9.4.
- Fill in the chain of custody form for the sample in accordance with 9.6.
- Transport the sample to the laboratory/facility where the sample will be treated further in accordance with 9.5.
- Check and sign the chain of custody form at each point where (i.e. moment when) the sample is transferred.
- Deliver the sample at the designated laboratory in accordance with 9.6.

10 Report

Usually, a report of the sampling has to be provided for the client. The report shall mention that the sampling has been carried out in accordance with this part of ISO 10381, and shall specify the exact method of sampling performed. Any deviation from the sampling plan shall be mentioned and motivated in the report.

The sampling plan and in addition to that the observations of the sampler form the basis of the sampling report.

In practice, the sampling report will often be part of a total report that also presents the analytical and testing results obtained with the samples and a discussion of these results.

The report shall at least provide the following information:

a) a description of the available background information on the soil stockpile;

b) a list of the involved parties together with a specification of their interest to the sampling programme;

c) the purpose of sampling;

d) the primary sampling goal that had to be achieved through sampling;

e) a list of components that had to be analysed in the samples, together with the consequences towards sampling, handling, preservation, packaging and storage;

f) a description of the applied sampling exercise, including:

    1) the number of samples or increments to be taken;

    2) the number of composite samples to make (if relevant);

    3) the sampling locations;

    4) the used sampling equipment and technique;

   5) any remarks on the status of the soil stockpile as made by the sampler during sampling and as described in the sampling record;

   6) the sample division in the field (if relevant);

   7) packing, preservation, storage, transport and delivery;

   8) any health and safety measures taken during sampling;

g) the full sampling plan;

h) all alterations to the sampling plan as made by the sampler and recorded in the sampling record together with a motivation of these alterations;

i) a description of the sampled soil stockpile that provides insight in the material sampled as well as providing information for the identification of the soil stockpile.

Photographs of the soil stockpile are useful for identification and when available should be part of the report.

Annex A
(informative)

Forms

A.1 Example of a sampling plan

SAMPLING PLAN	
GENERAL INFORMATION	
CLIENT (company): CONTACT:	CARRIED OUT BY (company): CONSULTANT: SAMPLER:
OWNER OF THE STOCKPILE:	
LANDOWNER:	
PURPOSE OF SAMPLING:	
SAMPLING LOCATION:	SAMPLING DATE:
MATERIAL	
NATURE OF MATERIAL:	
DETAILED SPECIFICATIONS:	BATCH SIZE:
WAY IN WHICH THE MATERIAL IS MADE AVAILABLE FOR SAMPLING:	
SAMPLING	
SAMPLING METHOD:	
EQUIPMENT TO BE USED:	
NUMBER OF INCREMENTS/SAMPLES TO BE TAKEN*):	
INCREMENT SIZE/SAMPLE SIZE*):	
SAMPLE CODING:	
NECESSARY SAFETY MEASURES:	
DATE OF SAMPLING:	
SAMPLE PRETREATMENT TO BE PERFORMED ON-LINE/OFF-LINE*)	
INSTRUCTIONS:	
PACKAGING, STORAGE AND TRANSPORT DETAILS	
PACKAGING:	
STORAGE:	
TRANSPORT:	
DELIVERY	
COMPANY: DELIVERY DATE:	
SIGNATURE FOR APPROVAL OF CONSULTANT:	DATE:
*) delete whichever is not applicable	

A.2 Example of a chain of custody form

Protocol ref. No.

Title: Sample Custody Form

Issued by:

External laboratories:
(please attach a copy of this form with reported results)

Contact name and number:	

Site visited: Site owner:	Site address: Tel No. Contact name:
Analysis subcontracted to: Laboratory name: Quotation ref. No.	Address: Tel: Contact name:
Carrier:	Address: Tel. No.
Sample collected by: Name: Signature: Sampling protocol used:	Date: Location:
Sampling delivered by: Name: Signature: Date: Time:	Samples accepted at laboratory by: Name: Signature: Date: Time:
Sample description:	Necessary preservation/storage: Dark: Cooled: Airtight sealing: Nitrogen gas atmosphere: Other:

Additional comments/instructions:

Analyses (when possible use laboratory codes):
Pretreatment method:
Inorganic components:
Organic components:

External laboratory sample job No.

Date sample received:

Sample storage time:

Date analysis undertaken:

Annex B
(informative)

Estimation of minimum increment and sample size

B.1 General

This annex provides methods for the estimation of the minimum and actual increment and sample size. These estimations need certain specific knowledge about the material to be sampled. In the clauses of this annex, the estimation methods are explained, and information and methods are provided for obtaining information necessary for making the estimates.

NOTE For a good understanding of this annex, the distinction between the terms “increment”, “sample” and “composite sample” is essential. See the definitions 3.5, 3.16 and 3.4. An increment is obtained by a single operation of a sampling device and is – per definition – put together with other increments in a composite sample. The term “sample” can either be used for an increment or a composite sample, depending on the method through which it is obtained.

B.2 Background of the estimation of the minimum increment size

An increment is the amount of material that is obtained in one single sampling activity. For instance, the filling of a scoop or auger. In order to ascertain non-biased sampling, it is essential that every particle in the stockpile can potentially be sampled.

This is obviously not the case when the size of the scoop or auger is smaller than the maximum particle size - for instance, sampling a material with a maximum particle size of 50 mm when the diameter of the auger is only 3 cm. Also, when the diameter of the auger is little over 50 mm, there is still a possibility that the larger particles cannot be sampled when two or more of these larger particles are in front of the auger at the same time. The particles would then obstruct each other from “entering” the auger.

In general, the effective opening of the auger will have to be (at least) three times the size of the maximum particles. The consequence of this on the size of the sampling equipment depends on the dimensions of the used sample equipment. This can either be one, two or three dimensions. When a sampling tube is used, the material enters the tube at the front end of the tube and therefore only the diameter has to be larger than three times the diameter of the maximum particles (assuming that the length of the tube is well over the dimensions of the individual particles). On the other hand, using a scoop would result in a three-dimensional definition of the minimum increment size, as both the front size, the height and the depth of the scoop would have to be over three times the maximum particle size.

Two major items are relevant for determining the minimum increment size:

- there is in general a wide particle size distribution;
- the presence of macro-aggregates.

Due to the fact that there is in general a wide particle size distribution, we can assume that the probability of two large particles being at the front end of a sampling device is relatively small. This implies that to some extent, the opening of the sampling device can be a bit smaller than three times the diameter of the largest particles. In practical applications, it is therefore assumed that the opening is still large enough when instead of the real largest particle size, the 95-percentile of the distribution of particle sizes is used for estimating the dimensions of the sampling equipment. This 95-percentile is known as D_{95}. Care should be taken that with the dimensions of three times D_{95} (for a one-dimensional definition), the opening still is large enough for the largest particles to enter. In a situation where the largest particles are much larger than the bulk of the soil, the assumption of using D_{95} can still result in biased samples.

When macro-aggregates are present, they can have important consequences on the size of the sampling equipment. Two situations should be discussed:

- the sampling equipment cuts through macro-aggregates;
- the sampling equipment encounters the macro-aggregates as if these are individual particles.

In the first situation, the presence of macro-aggregates has no consequences on the dimensions of the sampling equipment. This is for instance to be expected when using a sampling tube or auger.

In the second situation, however, the sampling equipment is apparently not able to sample part of a macro-aggregate and the macro-aggregates are therefore to be seen as individual particles. This is for instance the case when sampling soil in a falling stream. Then the presence of macro-aggregates will have severe consequences on the necessary dimensions of the sampling equipment.

Now having discussed the physical background of the minimum increment size, the dimensions can also be defined in terms of volume or weight of the increment. The volume or weight is a three-dimensional definition, independent of the question of how the dimensions of the sampling equipment are to be defined (one-, two- or three-dimensional). The volume of the minimum increment size can now be easily defined as $(3D_{95})^3 = 27(D_{95})^3$. As D_{95} is usually defined in millimetres and volumes are defined in litres, the minimum volume of the increment can be defined according to Equation (B.1):

$$V_{\text{min. increment}} = 2{,}7 \times 10^{-8}(D_{95})^3 \tag{B.1}$$

where

$V_{\text{min. increment}}$ is the minimal volume of the increment, in litres (l);

D_{95} is the maximum particle size (95 % of the particles smaller than D_{95}), in millimetres (mm).

To calculate the mass of this volume, the bulk density of the material has to be implemented in the formula. It is the bulk density because, apart from the sampled soil material, the air between the soil particles is sampled. Assuming that the width, height and length of the increment are chosen to be equal to three times the maximum particle size (D_{95}) and the maximum particle size is at least 3 mm, the minimum increment size is defined according to Equation (B.2):

$$M_{\text{min. increment}} = 2{,}7 \times 10^{-8}(D_{95})^3 \rho_{\text{bulk}} \tag{B.2}$$

where

$M_{\text{min. increment}}$ is the minimal mass of the increment, in kilograms (kg);

D_{95} is the maximum particle size (95 % of the particles smaller than D_{95}), in millimetres (mm);

ρ_{bulk} is the bulk density of the material, in kilograms per cubic metre (kg/m^3).

In the case of materials with a maximum particle size (D_{95}) of less than 3 mm, Equation (B.3) applies to the mass of the minimum increment size:

$$M_{\text{min. increment}} = 1 \times 10^{-6} \rho_{\text{bulk}} \tag{B.3}$$

where

$M_{\text{min. increment}}$ is the mass of the minimum increment size, in kilograms (kg);

ρ_{bulk} is the bulk density of the material, in kilograms per cubic metre (kg/m^3).

B.3 Background of the estimation of the minimum sample size

For a granular material, in which each particle possesses a certain characteristic to the same degree, no minimum sample size is required in order to determine this characteristic; every particle from a batch of this kind will be representative of this characteristic. The size of the sample (or increment) should only be as large as one particle, assuming that this would be enough material to determine that specific characteristic.

However, when only a fraction $w_{\text{particle}} < 1$ of the particles from the material possesses that characteristic, the sample shall have a minimum size to be regarded as sufficiently representative of the batch. Assume the presence of copper particles in a silica particle matrix. When sampling this mixture with a sample size of only one particle, one would either find 0 % copper or 100 % copper. It is clear that both of these results are inaccurate estimates of the mean copper concentration to be found in the material.

The fact that in a particulate material the particles possess different characteristics, and therefore vary from each other in a way that is characteristic for that material, results in the conclusion that there is always a certain degree of variability within the material. This fundamental variability results in what is known as the "fundamental error" in sampling. In order to reduce this undesired degree of variability, the samples should at least contain such an amount of particles that the variability between the particles has no significant influence on the mean concentration of the sample. The formula for estimating the minimum sample size is defined as an approximation of the size (expressed in mass), necessary to reduce the effect of the fundamental error.

The calculation of the minimum sample size is based on the binomial distribution, characterized by two parameters, i.e. the number of particles n and the fraction w_{particle} of these with a certain characteristic. The following applies to a number of particles y in a random sample which have a certain characteristic as in Equations (B.4) and (B.5):

$$E(y) = \mu = n w_{\text{particle}} \tag{B.4}$$

and

$$\sigma(y) = \sqrt{n w_{\text{particle}} (1 - p)} \tag{B.5}$$

where

$E(y)$ is the expected value (mean) of y;

μ is the mean (for example, the mean concentration);

n is the number of particles;

w_{particle} is the fraction of the particles with a certain characteristic (mass fraction);

p is probability for the occurrence of a particle with the characteristic to be determined ($0 < p < 1$);

$\sigma(y)$ is the standard deviation of the mean.

The coefficient of variation due to the fundamental error ($\text{CV}_{\text{fund. error}}$) is then according to Equations (B.6) and (B.7):

$$\text{CV}_{\text{fund. error}} = \frac{\sigma(y)}{E(y)} = \sqrt{\frac{(1 - p)}{n w_{\text{particle}}}} \tag{B.6}$$

or:

$$n = \frac{(1 - p)}{(\text{CV}_{\text{fund. error}})^2 \, w_{\text{particle}}} \tag{B.7}$$

The mass of a sample consisting of n particles is then equivalent to the product of the number of particles, the volume of the particles and the density ρ_{particle} of these particles. Assuming that the particles are spherical and of diameter d, see Equation (B.8):

$$m_{\text{min. sample}} = \frac{1}{6} \pi d^3 \rho_{\text{particle}} \frac{(1-p)}{(\text{CV}_{\text{fund. error}})^2 w_{\text{particle}}} \tag{B.8}$$

In order to take into account the fact that not all particles are of the same size and that the largest particles could be present in the sample, the diameter of the particles (d) that should be used in the equation is the diameter of the largest particles. For practical reasons, the particle size for which 95 % of the particles is smaller (D_{95}) is used. Additionally, to take into account the differences in particle size, a correction factor c is added [see Equation (B.9)]:

$$m_{\text{min. sample}} = \frac{1}{6} \pi (D_{95})^3 \rho_{\text{particle}} \times c \frac{(1-p)}{(\text{CV}_{\text{fund. error}})^2 w_{\text{particle}}} \tag{B.9}$$

where

$m_{\text{min. sample}}$ is the mass of the sample, rounded to two significant figures, in grams (g);

D_{95} is the maximum particle size (95 % of the particles smaller than D_{95}), in centimetres (cm);

ρ_{particle} is the density of the particles in the material, in grams per cubic centimetre (g/cm^3);

c is the correction factor for the particle size distribution of the material to be sampled (see B.4.2);

$\text{CV}_{\text{fund. error}}$ is the coefficient of variation caused by the fundamental error;

w_{particle} is the fraction of the particles with a certain characteristic (mass fraction).

The correction factor (c) for the particle size distribution is as follows:

- Broad distribution: $D_{95}/D_{05} > 4$ $c = 0{,}25$
- Medium distribution: $2 < D_{95}/D_{05} \leqslant 4$ $c = 0{,}50$
- Narrow distribution: $1 < D_{95}/D_{05} \leqslant 2$ $c = 0{,}75$
- Uniform particles: $D_{95}/D_{05} = 1$ $c = 1{,}00$

where

D_{95} is the maximum particle size (approximately 95 % of particles smaller than D_{95}), in centimetres (cm);

D_{05} is the minimum particle size (approximately 5 % of particles smaller than D_{05}), in centimetres (cm).

For practical reasons, the units that are used in the equation are to some extent deviating from the units that are usually used (mass in grams and particle size in centimetres).

It is essential to understand that Equation (B.9) is only an estimation of the size of a sample for which the fundamental variability will no longer have a significant effect on the variability of the samples. It should therefore only be used as an estimation and correspondingly the estimated minimum sample size should be given with only two significant figures (e.g. 1 523 = 1 500 and 2,365 = 2,4).

NOTE 1 The variables in the equation for the estimation of the minimum sample size are expressed in CGS units for practical reasons.

NOTE 2 The minimum sample size is directly related to the selected coefficient of variation of the fundamental error ($\mathrm{CV}_{\text{fund. error}}$) and to the size of fraction of the particles with the characteristic to be determined (w_{particle}).

NOTE 3 For the sampling of soil, in most cases, the following estimates of the factors in the equation are used:

— D_{95} = 1,6 cm (see also Note 4);

— ρ_{particle} = 2,6 g/cm^3;

— c = 0,25;

— w_{particle} = 0,02 (see also Note 5);

— $\mathrm{CV}_{\text{fund. error}}$ = 0,1.

NOTE 4 The maximum particle size (D_{95}) is for most soil assumed to be less than 1,6 cm. Using a standard value of 1,6 cm will, for most soils, result in a safe estimate of the minimum sample size. The value of 1,6 cm assumes that the sampling equipment/sampling technique is capable of taking a part of a macro-aggregate, and macro-aggregates are therefore not to be considered equal to individual particles. The actual maximum particle size (D_{95}) can be estimated, using the method given in B.5.

NOTE 5 The actual value of w_{particle}, the fraction of particles with a certain characteristic, depends on the soil to be sampled and the substances to be determined in that soil. Knowledge of the soil consistency is required in order to determine this value. Instructions for selecting w_{particle} are given in B.4.5, but as the information for a good estimation of w_{particle} will often not be available, the value of 0,1 is most commonly used. For soil sampling, a “safer” estimate of 0,02 is suggested in Note 3. It should be noted that w_{particle} is not equivalent to the concentration of the component to be determined!

NOTE 6 The equation for estimating the minimum sample size is derived for spherical particles of diameter d. For non-spherical particles, the formula gives an approximation of the sample size for which the coefficient of variation of the fundamental error ($\mathrm{CV}_{\text{fund. error}}$) will comply with the pre-defined value.

B.4 Use of the equation for the minimum sample size

B.4.1 General

For the application of the equation for the minimum sample size derived under B.3, a number of requirements are important. These requirements are discussed in more detail hereafter. However, the most important point is that the equation is only an estimation tool. The minimum sample size calculated with the aid of the equation should never be seen as “the physical reality”. It is only a method to obtain an estimation of the size of the samples that should be used in order to prohibit biased sampling results due to the fundamental heterogeneity of the material. As biased sampling results can have important consequences for decisions based on these results, the equation is important for obtaining good quality results.

Some of the hereafter discussed requirements do not, or only partially, apply for soil sampling. Nevertheless, even in situations where the equation strictly speaking does not apply, the equation should be used. As the result of the equation should always be used as “the order of magnitude” of the minimum sample size, the usage of the equation in less well fitting situations is acceptable.

In order to express the fact that the equation only provides us with “the order of magnitude” of the minimum sample size, the results of the equation are always to be rounded at two significant figures.

B.4.2 Spherical particles

The calculation of mass for the minimum sample size is carried out assuming that the particles are spherical $[\frac{1}{6}\pi(D_{95})^3]$. If the particles are not spherical, this leads to a miscalculation of the volume of the particles and thus of the mass of the minimum sample size. This error will increase as the average particle shape increasingly deviates from a spherical form. It is clear that soil particles only in average can be assumed to be spherical, and thus that the equation will result in just an approximation of the true mass of the largest particles.

B.4.3 Particle size distribution, factors D_{95} and c

Two characteristics of the particle size distribution of the material are relevant, i.e. the particle size above which (approximately) 5 % of larger particles occur (D_{95}) and the particle size under which (approximately) 5 % of smaller particles occur (D_{05}).

The estimation or determination of D_{95} is more important than that of D_{05}, in view of the fact that D_{95} is expressed directly in the equation for the minimum sample size. In B.5, a method is described that can be used to obtain a suitable estimation of D_{95}.

For materials with a broad distribution of particle size, it is not necessary to quantify D_{05}, an estimate may suffice to determine whether $D_{95} / D_{05} > 4$.

For materials with a narrower particle size distribution, it is relevant to have a good estimate of D_{05} or to determine it as well as D_{95}. For soil, in general, it can be assumed that $D_{95} / D_{05} > 4$ and therefore $c = 0{,}25$.

NOTE As mentioned for soil in general, it can be assumed that $c = 0{,}25$. In order to check this assumption, an estimate of the D_{95} and D_{05} by an experienced sampler will, in most cases, be sufficient.

B.4.4 Density of the particle

For the density of the particle ρ_{particle}, the density of the individual particles should be used, not the density of the sample or the (loosely) packed material. If the material in question contains particles of varying density, the mean value shall be used.

B.4.5 Fraction of the particles with the characteristic to be determined, factor w_{particle}

The equation applied to determine the minimum sample size is based on the equation for the binomial distribution. This means that w_{particle} is the fraction of the particles possessing a certain characteristic. If all the particles contain the material to some extent, the binomial distribution does not apply. Values higher than $w_{\text{particle}} = 0{,}1$ (more than 10 % of the particles contains the material to be determined) therefore cannot be used in the equation. However, for such fairly homogeneous materials, the minimum sample size can be determined on the basis of the assumption that $w_{\text{particle}} = 0{,}1$. The resulting minimum sample size should then be an overestimate of the true minimum sample size (given a certain admissible amount of variation due to the fundamental error); the samples used are larger than strictly necessary and the variation realized due to the fundamental error is less than 0,1.

One should realize that the concentration of a certain component is a significantly different parameter than the fraction w_{particle} of the particles that contain that component. Nevertheless, there will be a certain relationship between the concentration and w_{particle}. In other words, generally speaking, for very high concentrations w_{particle} will not be extremely small, while for very low concentrations w_{particle} will normally also be (very) small.

B.4.6 Coefficient of variation from the fundamental error, factor $\mathrm{CV}_{\text{fund. error}}$

A choice has to be made for the amount of fundamental variation that is thought to be acceptable in relation to the quality of the sampling. Principally, this choice can not be made without knowledge about the size of other sources of variation. When, for example, the variation of the analysis would be very large, there is no need to have a very small fundamental error. The analytical variance would then be dominant over the other sources of variability, and a (much) larger sample would only result in a small enhancement of the total quality. As the quantity of variance added from all sources is often unknown or only partly known, in most situations an assumption of the maximum size of the fundamental error has to be made. Often the value 0,1 is therefore applied for the coefficient of variation of the fundamental error.

Looking only at the variability that results from sampling, the degree of large-scale variability (the heterogeneity of the material) will result in the degree of variability between the samples. This will be true as long as these samples are large enough to make the fundamental error negligible. This again means that in a highly variable soil stockpile, the effect of the fundamental error may be larger than for a more homogeneous stockpile. For more homogeneous materials, a coefficient of variation of 0,1 can still mean a relatively large contribution to the total variation among the samples. In these cases, the differences between the individual particles thus do make a significant contribution to the total variation. In view of the relatively small value of CV, however, the total variation shall also be small in such a situation.

B.5 Determination of the maximum particle size

B.5.1 Step 1: Sampling

Determine the mass of the necessary sample by applying Equation (B.10):

$$m = 150\rho_{\text{bulk}}(D_{\text{est. 95}})^3 \geqslant 1\ \text{kg} \tag{B.10}$$

where

m is the mass of the sample used for determining D_{95}, in grams (g);

ρ_{bulk} is the bulk density of the material, in grams per cubic centimetre (g/cm^3);

$D_{\text{est. 95}}$ is the estimated maximum particle size, in centimetres (cm).

Take a sample of this mass from the stockpile.

The purpose of a specific sampling programme should be kept in mind. As the purpose is only to obtain an estimation of D_{95}, the sample can be taken based on the knowledge of the sampler. This means that the sampler can take a sample for which he can assume that all particle sizes are present.

B.5.2 Step 2: Weighing the sample

Weigh the sample (m_0).

For the accuracy of the weighing, Table B.1 is used:

Table B.1 — Accuracy of weighing

Size of the sample kg	**Accuracy of the weighing** g
1 to 5	0,1
5 to 10	1
10 to 50	10
> 50	100

B.5.3 Step 3: Sieving the sample

Transfer the entire sample on a sieve with a sieve aperture equal to the estimated D_{95}. If there is no sieve aperture equal to the estimated D_{95}, two sieves shall be used. The sieve with the largest aperture has an aperture adjacent to but larger than D_{95}, while the sieve with the smallest aperture has an aperture adjacent to but smaller than D_{95}.

Sieve the sample either manually or mechanically. Check if the material that remains on the sieve(s) consists of individual particles. If so, proceed to Step 4.

When macro-aggregates are present, the macro-aggregates are pushed through the sieve. However, this may only be done when the method of sampling is capable of taking a part of a sample. When macro-aggregates are to be seen as individual particles during sampling, the macro-aggregates are left on the sieve.

B.5.4 Step 4: Weighing the sample part(s)

Weigh the part of the sample that remains on the sieve (m_1) or weigh the parts that remain on both sieves (m_1 and m_2). The accuracy of the weighing should fulfil the conditions given in the table in Step 2.

B.5.5 Step 5: Determination of the maximum particle size

B.5.5.1 Step 5.1: Calculation for one sieve

If only one sieve is used, the maximum particle size is calculated according to Equation (B.11):

$$w_{\text{sample}} = 100 \frac{m_1}{m_0} \% \tag{B.11}$$

where

w_{sample} is the mass fraction of the sample that remains on the sieve, in percent (%);

m_1 is the mass of the part of the sample that remains on the sieve, in grams (g);

m_0 is the total mass of the sample, in grams (g).

If

- $w_{\text{sample}} < 5\ \%$ the used sieve aperture was too big. The aperture size is used as an assumption for D_{95}, or the sieved part of the sample is sieved again with a sieve with smaller aperture. In the latter situation, go back to Step 3 and the calculation of the D_{95} is then carried out in accordance with Step 5.2.
- $w_{\text{sample}} = 5\ \%$ the used aperture was exactly right and is used as an estimation of D_{95}.
- $w_{\text{sample}} > 5\ \%$ the used aperture is too small. Go back to Step 3 and use the fraction m_1 for a second sieving. D_{95} is calculated according to Step 5.2.

B.5.5.2 Step 5.2: Calculation when applying two sieves

If two sieves were applied, D_{95} is calculated according to Equation (B.12):

$$\begin{aligned} w_{\text{sample},1} &= 100\,\frac{m_1}{m_0}\ \% \\ w_{\text{sample},2} &= 100\,\frac{m_2}{m_0}\ \% \end{aligned} \tag{B.12}$$

where

$w_{\text{sample},1}$	is the mass fraction of the sample that remains on the sieve with the largest aperture, in percent (%);
$w_{\text{sample},2}$	is the mass fraction of the sample that remains on the sieve with the smallest aperture, in percent (%);
m_1	is the mass of the part of the sample that remains on the sieve with the largest aperture, in grams (g);
m_2	is the mass of the part of the sample that remains on the sieve with the smallest aperture, in grams (g);
m_0	is the total mass of the sample, in grams (g).

If

- $w_{\text{sample},1} = 5\ \%$ the aperture of sieve 1 is exactly right. This aperture size is used as an estimate for the D_{95}.
- $w_{\text{sample},1} > 5\ \%$ the used aperture of the sieves is too small. Go back to Step 3 and use fraction m_1 for a second sieve step. The D_{95} is determined according to Step 5.2 using the newly produced sieve fractions.
- $w_{\text{sample},1} + 2 < 5\ \%$ both applied sieves are too big. Assume that D_{95} is equal to the aperture of sieve 2 or go back to Step 3 and sieve the total sample again on a set of smaller sieves. D_{95} is then estimated according to Step 5.2 using the newly produced sieve fractions.
- All other situations: estimate D_{95} by interpolation between the two sieve aperture sizes. Linear regression can be used for this interpolation.

B.6 Commonly used assumptions

For sampling soil stockpiles, a number of assumptions can be made for using the equation for the minimum sample size, as given in B.3. These assumptions can be used in common situations. However, the assumptions may only be used after careful study of the backgrounds of the equation for the minimum sample size.

NOTE The assumptions might be considered as valid for fine grained soils.

The commonly used estimates are:

ρ_{particle} = 2,6 g/cm^3, the particle density for silicium oxide;

c = 0,25;

w_{particle} = 0,02;

$\text{CV}_{\text{fund. error}}$ = 0,1;

D_{95} = 1,6 cm.

The maximum particle size (D_{95}) is for most soil assumed to be less than 1,6 cm. Using a standard value of 1,6 cm will for most soils result in a safe estimate of the minimum sample size.

B.7 Tables for the minimum sample size

To determine the minimum sample size in probabilistic sampling, the criterion is that variations in the composition of the material to be analysed on the scale of the individual particles is regarded as irrelevant, in other words: the variation that is caused by the fundamental error is small.

Applying part of the assumptions made in B.6, the minimum sample size can be calculated for different mass fractions (w_{sample}) of the material that are thought to obtain the characteristic of interest.

Minimum sample sizes are given in the following tables.

Table B.2 — Minimum sample size in grams for soil with D_{95} = 0,4 cm

D_{95}	0,4 cm
ρ_{particle}	in table (g/cm^3)
c	0,25
w_{sample}	in table
$\text{CV}_{\text{fund. error}}$	0,1

ρ_{particle}	**Mass fraction**, w_{sample}			
	0,1	0,05	0,01	0,005
2,2	17	35	180	370
2,4	18	38	200	400
2,6	20	41	220	430
2,8	21	45	230	470
3	23	48	250	500

Table B.3 — Minimum sample size in grams for soil with D_{95} = 0,6 cm

D_{95}	0,6 cm
ρ_{particle}	in table (g/cm^3)
c	0,25
w_{sample}	in table
$\text{CV}_{\text{fund. error}}$	0,1

ρ_{particle}	**Mass fraction**, w_{sample}			
	0,1	0,05	0,01	0,005
2,2	56	120	620	1 200
2,4	61	130	670	1 400
2,6	66	140	730	1 500
2,8	71	150	780	1 600
3	76	160	840	1 700

Table B.4 — Minimum sample size in grams for soil with D_{95} = 0,8 cm

D_{95}	0,8 cm
ρ_{particle}	in table (g/cm^3)
c	0,25
w_{sample}	in table
$\text{CV}_{\text{fund. error}}$	0,1

ρ_{particle}	**Mass fraction**, w_{sample}			
	0,1	0,05	0,01	0,005
2,2	130	280	1 500	2 900
2,4	140	310	1 600	3 200
2,6	160	330	1 700	3 500
2,8	170	360	1 900	3 700
3	180	380	2 000	4 000

Table B.5 — Minimum sample size in grams for soil with D_{95} = 1,0 cm

D_{95}	1,0 cm
ρ_{particle}	in table (g/cm^3)
c	0,25
w_{sample}	in table
$\text{CV}_{\text{fund. error}}$	0,1

ρ_{particle}	**Mass fraction**, w_{sample}			
	0,1	0,05	0,01	0,005
2,2	260	550	2 800	5 700
2,4	280	600	3 100	6 200
2,6	310	650	3 400	6 800
2,8	330	700	3 600	7 300
3	350	750	3 900	7 800

Table B.6 — Minimum sample size in grams for soil with D_{95} = 1,2 cm

D_{95}	1,2 cm
ρ_{particle}	in table (g/cm^3)
c	0,25
w_{sample}	in table
$\text{CV}_{\text{fund. error}}$	0,1

ρ_{particle}	**Mass fraction**, w_{sample}			
	0,1	0,05	0,01	0,005
2,2	450	940	4 900	9 900
2,4	490	1 000	5 400	11 000
2,6	530	1 100	5 800	12 000
2,8	570	1 200	6 300	13 000
3	610	1 300	6 700	14 000

Table B.7 — Minimum sample size in grams for soil with D_{95} = 1,4 cm

D_{95}	1,4 cm
ρ_{particle}	in table (g/cm^3)
c	0,25
w_{sample}	in table
$\text{CV}_{\text{fund. error}}$	0,1

ρ_{particle}	**Mass fraction**, w_{sample}			
	0,1	0,05	0,01	0,005
2,2	710	1 500	7 800	16 000
2,4	780	1 600	8 500	17 000
2,6	840	1 800	9 200	19 000
2,8	900	1 900	10 000	20 000
3	970	2 000	11 000	21 000

Table B.8 — Minimum sample size in kilograms for soil with D_{95} = 1,6 cm

D_{95}	1,6 cm
ρ_{particle}	in table (g/cm^3)
c	0,25
w_{sample}	in table
$\text{CV}_{\text{fund. error}}$	0,1

ρ_{particle}	**Mass fraction**, w_{sample}			
	0,1	0,05	0,01	0,005
2,2	1,1	2,2	12	23
2,4	1,1	2,4	13	26
2,6	1,2	2,6	14	28
2,8	1,4	2,8	15	30
3	1,4	3,0	16	32

Table B.9 — Minimum sample size in kilograms for soil with D_{95} = 1,8 cm

D_{95}	1,8 cm
ρ_{particle}	in table (g/cm^3)
c	0,25
w_{sample}	in table
$\text{CV}_{\text{fund. error}}$	0,1

ρ_{particle}	**Mass fraction**, w_{sample}			
	0,1	0,05	0,01	0,005
2,2	1,5	3,2	17	33
2,4	1,6	3,5	18	36
2,6	1,8	3,8	20	39
2,8	1,9	4,1	21	42
3	2,1	4,4	23	45

Table B.10 — Minimum sample size in kilograms for soil with D_{95} = 2,0 cm

D_{95}	2,0 cm
ρ_{particle}	in table (g/cm^3)
c	0,25
w_{sample}	in table
$\text{CV}_{\text{fund. error}}$	0,1

ρ_{particle}	**Mass fraction**, w_{sample}			
	0,1	0,05	0,01	0,005
2,2	2,1	4,4	23	46
2,4	2,3	4,8	25	50
2,6	2,4	5,2	27	54
2,8	2,6	5,6	29	58
3	2,8	6,0	31	62

B.8 Calculation of the actual increment and sample size

On the basis of the relationship between the minimum increment size, the minimum sample size and the number of increments to be included in a composite sample, the actual increment size and the actual sample size shall be determined according to the following rules:

If: $a \times M_{\text{min. increment}} > M_{\text{min. sample}}$

then: $M_{\text{act. sample}} = a \times M_{\text{min. increment}}$ and $M_{\text{act. increment}} = M_{\text{min. increment}}$

If: $a \times M_{\text{min. increment}} = M_{\text{min. sample}}$

then: $M_{\text{act. sample}} = M_{\text{min. sample}}$ and $M_{\text{act. increment}} = M_{\text{min. increment}}$

If: $a \times M_{\text{min. increment}} < M_{\text{min. sample}}$

then: $M_{\text{act. sample}} = M_{\text{min. sample}}$ and $M_{\text{act. increment}} = M_{\text{min. sample}}/a$

where

a	is the number of increments which are put together in a composite sample;
$M_{\text{min. sample}}$	is the minimum sample size in kilograms (kg);
$M_{\text{act. sample}}$	is the actual sample size of the composite sample in kilograms (kg);
$M_{\text{min. increment}}$	is the minimum increment size in kilograms (kg);
$M_{\text{act. increment}}$	is actual increment size in kilograms (kg).

NOTE Depending upon the situation:

— the actual increment size is larger than the minimum increment size (the minimum sample size is a determinant factor for the increment size given the number of increments);

or

— the actual composite sample size is larger than the minimum sample size (the minimum increment size is a determinant factor for the composite sample size given the number of increments).

The examples given below are for a soil stockpile with the following estimated minimum increment and sample size:

— $M_{\text{min. increment}}$ = 0,34 kg;

— $M_{\text{min. sample}}$ = 4,2 kg.

EXAMPLE 1

Situation: 12 increments for 3 composite samples, i.e. 4 increments (a) per composite sample:

$a \times M_{\text{min. increment}} = 4 \times 0{,}34\ \text{kg} = 1{,}3\ \text{kg}$, but $M_{\text{min. sample}} = 4{,}2\ \text{kg}$,

therefore

$M_{\text{act. sample}} = M_{\text{min. sample}}$ and $M_{\text{act. increment}} = 4{,}2 / 4 = 1\ \text{kg}$.

Although it is also possible to take more increments of the predetermined size of 0,34 kg (with 13 increments, the actual sample size is a bit larger than the minimum sample size), this is often not preferred.

The number of increments per composite sample would be changed (and, as in this example, not even the same number of increments would have to be mixed), but at the same time, the resulting confidence would be better than asked for. Although this would be no problem in most situations, a larger number of increments would also result in higher sampling costs.

EXAMPLE 2

Situation: 54 increments for 3 composite samples, i.e. 18 increments (a) per composite sample:

$a \times M_{\text{min. increment}} = 18 \times 0{,}34\text{ kg} = 6{,}1\text{ kg}$, but $M_{\text{min. sample}} = 4{,}2\text{ kg}$,

therefore:

$M_{\text{act. increment}} = M_{\text{min. increment}} = 0{,}34\text{ kg}$, and $M_{\text{act. sample}} = 6{,}1\text{ kg}$.

Annex C
(informative)

Scale of sampling

C.1 Spatial variability and scale

C.1.1 General

Scale is one of the essential issues of sampling. The scale defines the volume or mass that a sample directly represents. This implies that when the assessment of the soil stockpile is needed, for example, on a scale of 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 soil. At the same time spatial variability within the stockpile is determined between "units" of one cubic metre.

Depending on the objective of sampling, theoretically, the scale of sampling may be equal to the size of individual particles of the soil, the size of a specified volume that is part of the stockpile (like the previous one cubic metre example), or the whole stockpile.

Defining the scale is important, as heterogeneity is a scale-dependent characteristic.

EXAMPLE Taking the example of a stockpile that consists of small particles that only vary in colour, and with the particles 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. For a larger scale (larger volume), the observed heterogeneity can be seen as a worst case assumption. 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.

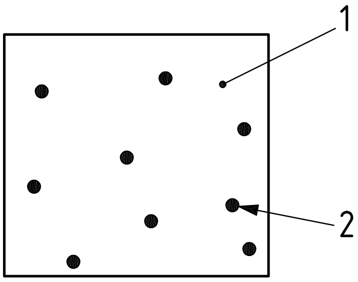
C.1.2 Three specific situations for which the scale is defined

C.1.2.1 Situation 1

Situation 1 describes a soil stockpile of 2 000 t from which randomly 50 increments are taken. 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 stockpile, the scale for the composite sample in this example is 2 000 t.

Note that although the variability within the stockpile (on the scale of the increments) is fully incorporated in the composite sample, the sampling method will not provide any information on the variability.



Key

1 population 2 000 t

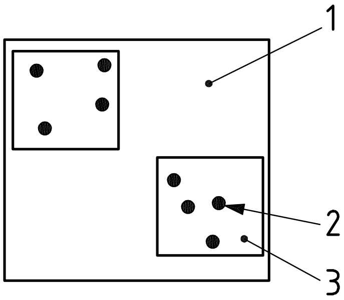
2 increment 200 g (50 increments in a composite sample of 10 kg)

Figure C.1 — Situation 1 — Soil stockpile of 2 000 t with 50 random increments

C.1.2.2 Situation 2

Situation 2 describes a stockpile of 2 000 t. Within this stockpile – perhaps only for the purpose of sampling – subpopulations are defined of 50 t each. From each subpopulation 50 increments are taken. The resulting composite samples are 10 kg, each representing a subpopulation.

The mass represented by each composite sample is now the mass of the individual subpopulations; thus 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 stockpile of 2 000 t and the variability within the whole stockpile is estimated on a scale of 50 t.



Key

1 population 2 000 t

2 increment 200 g (50 increments in a composite sample of 10 kg)

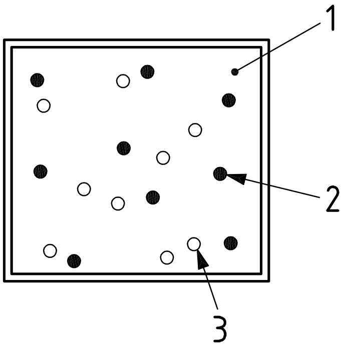
3 subpopulation 50 t

Figure C.2 — Situation 2 — Soil stockpile of 2 000 t with 50 increments from each subpopulation

C.1.2.3 Situation 3

Situation 3 describes a stockpile 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 stockpile. The mass represented by each composite sample is now equal to the mass of the whole stockpile; thus 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 stockpile of 2 000 t is estimated on a scale of 200 g (the mass of the increments).



Key

1 population 2 000 t
2 increment 200 g (50 increments in a composite sample of 10 kg)
3 increment 200 g (50 increments in a composite sample of 10 kg)

Figure C.3 — Situation 3 — Soil stockpile of 2 000 t with more than one composite sample taken

C.1.3 Effects of different definitions of the scale on sampling

The following example illustrates the effects of different definitions of the scale of sampling. Depending on the objective of sampling, the involved parties shall make a choice on the scale on which information about the soil stockpile is desired.

Consider within a stockpile the three subpopulations as shown in Table C.1 (comparable to situation 2 as described above). Each subpopulation consists of thirteen individual samples that have a “quality” that is symbolized 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 subpopulation, the sampling result will only be an estimate of the mean concentration for each subpopulation (26,2 / 26,2 and 32,5). Comparing the subpopulations in Table C.1, subpopulations 1 and 2 are comparable while subpopulation 3 has a higher mean.

When the scale of sampling is equal to the size of the individual samples within each subpopulation, we obtain not only an estimate for the mean concentration of the subpopulation (26,2 / 26,2 and 32,5), but also an estimate for the heterogeneity within that subpopulation (33,3 / 84,2 and 33,2). Comparing the subpopulations in Table C.1 now still gives the same result for the mean of the whole subpopulation, but additionally we discover that subpopulation 2 has a higher degree of variability than subpopulations 1 and 3.

Table C.1 — Example of three different subpopulations, characterized on the individual samples, the mean and coefficient of variation (CV). A high CV indicates a heterogeneous sample.

Statistical parameter	Sub-population 1	Sub-population 2	Sub-population 3	Population
	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	
Mean	26,2	26,2	32,5	28,3
Coefficient of variation CV	33,3 %	84,2 %	33,2 %	

Finally, when the scale of sampling is equal to the whole stockpile (population), we obtain only an estimate of the mean for the whole stockpile (28,3).

C.1.4 Choices on the scale of sampling

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

a) The scale of sampling is equal to the volume or mass of the individual samples.

   1) For each defined (sub)population, a number of samples are taken. The result of this definition of the scale is that information on the heterogeneity within the subpopulations can be obtained by calculating, for example, the coefficient of variation. Additionally, the heterogeneity between the subpopulations, and thus the heterogeneity within the stockpile, can be calculated. In this approach, the presumptions that led to identification of the subpopulation as a relatively homogeneous part within the stockpile can be verified.

   2) For example, it may be argued that subpopulation 2 in Table C.1 is so heterogeneous that at least a part of subpopulation 2 will not comply with certain quality standards, although the mean value is within the quality range.

   3) Many subpopulations of high heterogeneity may lead to a re-evaluation of the sampling plan. An important disadvantage is the cost for measuring the individual samples, in this example thirteen per subpopulation1).

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1) Note that it is not necessary (nor practical) to measure each individual volume on the scale of the sample within a subpopulation. A sample survey within each subpopulation might be sufficient.

b) The scale of sampling is equal to the scale of the subpopulations.

   1) Therefore no information on individual samples within a subpopulation is gathered.

   2) Characterization of the subpopulation is done by means of a composite sample per subpopulation in which a number of increments (13 increments in the example of Table C.1) is put together prior to analysis. If this composite sample consists of sufficient increments and the analytical sample is taken and analysed correctly, the result of the composite sample will be a good estimate of the true mean of the subpopulation.

   3) 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.

   4) 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 pretreatment 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 subpopulation.

c) The scale of sampling is equal to the scale of the whole stockpile.

   1) In the example given in Table C.1, the whole stockpile is defined as the combination of the three subpopulations. Individual increments are gathered from the whole stockpile. The increments are put together in one composite sample for the whole stockpile. Now, there will be no information available on a smaller scale than the scale of the whole stockpile.

   2) An important advantage is the (very) low costs for measuring, while, as long as it is technically possible to obtain a representative analytical sample from a composite sample containing a large number of increments, the analytical result will still be representative for the true mean of the whole stockpile. But the stockpile has to be treated as one entity.

   3) In the case of a heterogeneous stockpile (e.g. subpopulation 2 in Table C.1), sampling on the scale of subpopulations or individual samples would have given the involved parties information that may have led to different choices for the destination of subpopulations of different quality.

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

The type of information that is desired, the possible destination, the financial means available and the technical possibilities of working with composite samples determine the choice on the scale of sampling.

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 that is considered relevant from a policy perspective.

EXAMPLE An example of a policy-defined scale of sampling might be as follows:

Based on the radius of action of small animals living in soil, the mean concentration of a soil volume of 25 m^3 is considered as relevant for assessing the seriousness of soil contamination. It is assumed that these animals throughout their whole life span are exposed to the mean concentration of the pollutants in this soil volume. Thus, when assessing the seriousness of polluted soil, we are interested in the mean concentration within this volume of 25 m^3. When acute exposure to (very) high concentrations is considered not to be relevant, there is no need to gather information on a smaller scale than 25 m^3. The scale of sampling is therefore 25 m^3, and is achieved by taking a number of increments within this volume. An estimate of the true mean concentration on the scale of 25 m^3 can thus be obtained.

C.2 Fundamental variability

Granular material like soil will generally consist of different types and shapes of particles. As a consequence, there is a degree of variability on the scale of the individual particles. This variability cannot be reduced without particle size reduction. This is called the “fundamental variability”. It will be the cause of variability between samples whenever the characteristic of interest (e.g. the concentration of metals, or organic matter) is directly related to a specific portion or subset of the particles. Also, when the concentration of the constituent of interest varies over the different particles, there is fundamental variability.

As the average number of particles per sample increases, so the effect of the fundamental variability becomes less dominant. Nevertheless, the effect can remain large even with a large number of particles in the sample if the constituent of interest (e.g. copper occurring incidentally within a soil stockpile due to small pieces of copper wire) arises in only a small proportion of particles, but at very high concentrations. Annex B provides the details of a method that can be used to estimate the minimum size of samples to ensure that the error due to fundamental variability is as small as required.

Annex D
(informative)

Statistical principles

D.1 General

This annex briefly describes the basic statistical concepts of sampling. The text is not intended to be a statistical textbook, and gives only a basic outline of the statistical elements relevant to this part of ISO 10381. These are grouped under five headings:

- Population (D.2). This is a statistical term for defining the entirety of material – in general the stockpile – about which information is required. Specification of the population should be the starting point of any sampling exercise.
- Types of variability (D.3). A good awareness of the variability in the material being sampled is a vital element in arriving at an effective sampling programme. Linked with this (for sampling granular material) is the need to decide on the “scale” of the sampling, i.e. the maximum volume of material within which variations in quality are of no concern.
- Error (D.4). Apart from the variability characteristic of the material itself, sampling introduces additional uncertainty, known as “sampling error”. Analysis similarly introduces a further degree of uncertainty, termed “analytical error”.
- Statistical parameters (D.5). A variety of summary measures, or “parameters”, may be used to characterize a population (e.g. the mean, the median or the 90-percentile). It is important to consider the choice of parameter because this can have a big influence on the size of the sampling error.
- Reliability (D.6). The greater the amount of sampling, the more reliable the results are likely to be. The major benefit of a statistical approach is that it enables this link between sampling effort and reliability to be quantified.

D.2 Population and subpopulation

D.2.1 Population

The “population” is the entire stockpile of soil about which information is to be sought via sampling. It is important for this to be defined explicitly over space and/or time (when relevant, see D.3.3); if this is not done, it is impossible to say whether a particular sampling programme will result in representative samples.

NOTE For some sampling purposes, spatial variation may not be relevant (e.g. when sampling a specific pre-defined portion of a stockpile), whilst for other purposes, temporal variation may not be relevant (e.g. when sampling from a stockpile and there is no (expected) relation between the build-up and quality of the soil within the stockpile).

Where the population relates to a granular material like soil, an important related consideration is the “scale” at which the individual elements of the population are defined, as this can greatly influence the type and amount of sampling that is required. See in this context D.3.3 for the definition of this scale.

D.2.2 Subpopulation

Commonly, it is difficult or even impossible to sample certain parts of the population. For example, to sample the material at the core of a large stockpile could require expensive equipment or be very time-consuming. In such cases, it can be useful to define a subset of the population – termed the “subpopulation” – and restrict the sampling to that more convenient region.

It is important to appreciate, however, that the resulting samples can be representative only for that subpopulation. The relevance or otherwise of those results to the total population rests entirely on the project manager's assumptions (which are usually unverifiable).

Given the risk of multiple interpretations, it is important always to check that all involved parties are talking about “the same amount of soil”.

EXAMPLE 1:

Population:	The contents of the total stockpile.
Subpopulation:	All material within 2 m of the perimeter of the stockpile.

EXAMPLE 2:

Population:	All soil from a soil treatment plant over a particular month.
Subpopulation:	All soil produced through the month during the working day (e.g. 08:00 to 16:00).

D.3 Types of variability

D.3.1 General

A key element of sampling programme design is an understanding of the main components of variability in the soil stockpile being sampled.

Two distinctive types of variability can be distinguished:

- spatial variability;
- temporal variability.

Temporal variability is only relevant in stockpile sampling when temporal processes determine the spatial variability within a stockpile or between stockpiles. When temporal effects are expected based on the process wherein the stockpile is formed, these effects should be accounted for in the assumptions on the spatial variability.

The spatial variability is discussed under the next three headings.

D.3.2 Fundamental variability

Soil consists of different types and shapes of (soil) particles. On the scale of the individual particles, there is a degree of variability that cannot be reduced without particle size reduction. This is called the “fundamental variability”. It will be the cause of variability between samples whenever the characteristic of interest (e.g. the concentration of metals related to the occurrence of organic matter) is directly related to a specific portion or subset of the particles.

As the average number of particles per sample increases, so the effect of the fundamental variability becomes less dominant. Nevertheless, the effect can remain large even with a large number of particles in the sample if the constituent of interest (e.g. small pieces of copper wire occurring incidentally as a waste material within soil) arises in only a small proportion of particles, but at very high concentrations. Annex B provides the details of a method that can be used to estimate the minimum size of samples to ensure that the error due to fundamental variability is as small as required.

In general it is assumed that the heterogeneity between individual particles is of no importance when assessing the quality of the soil. Therefore, the sample size (volume) should be of such size that the effect of individual particles on the results from the sample is small.

D.3.3 Variability within stockpile

The variability that is encountered when sampling soil stockpiles will generally be the variation that occurs within an individual stockpile. This *spatial* variability is essential in quality assessment of soil stockpiles. Whether one is interested in the variability within the stockpile or not, the potential occurrence of spatial variability has a direct effect on the sampling strategy to be chosen.

Two different situations are distinguished.

- The spatial variability within the stockpile should be known.
- There is no specific interest in the spatial variability.

The spatial variability is an inherent characteristic of the population. Without manipulation of the material (e.g. mixing the stockpile), it will not change.

The amount of spatial variability in the population cannot be quantified without defining the “scale” on which that variability occurs. For example, the variability from gram to gram of material in a stockpile is likely to be greater than that from kilogram to kilogram. If concentration variations on so fine a scale as this are believed to be important, then that is the scale on which the sampling shall operate. If, conversely, concentration variations within any one kilogram of material are irrelevant, the primary aim of the sampling should be to quantify variability solely on the kilogram-to-kilogram scale. It is therefore of vital importance that the scale is stated explicitly.

The scale of sampling is discussed in Annex C.

D.3.4 Variability between stockpiles

For quantification of the variability between stockpiles, the variability within the individual stockpiles should either be quantified or should be incorporated in the composite samples that are used to quantify the quality of each individual stockpile.

Again the scale on which the variability is quantified – now both within and between stockpiles – is of vital importance. See also Annex C.

D.4 Error

D.4.1 Sampling error

D.4.1.1 General

Based on the principle that causes it, the sampling error can be divided into two separate terms:

- the statistical sampling error;
- the physical sampling error.

D.4.1.2 Statistical sampling error

It is seldom possible (or desirable) to sample the whole population. Consequently, any (statistical) parameter (e.g. an average concentration) that is calculated from the results of a sampling programme will differ from the “true” value – that is, the value that would have been obtained if the whole population could have been sampled – except by a lucky chance. This difference is known as “statistical sampling error”.

D.4.1.3 Physical sampling error

In addition to the statistical sampling error that arises as an inevitable consequence of the random selection process, the sampling activity may itself introduce an additional error. This can be termed as the "physical sampling error", and may take the form of either bias or random error.

EXAMPLE Suppose a 3 cm diameter auger is used when sampling a soil stockpile in which particle size may be as large as 5 cm. The larger particles cannot be sampled and will therefore have no contribution to the measurements. As a consequence, the measurements will be biased.

D.4.2 Sampling error due to pretreatment

After sampling sample pretreatment will be necessary, either in field directly after sampling and/or in the laboratory in order to get a representative analytical sample. These activities themselves will add some variability; both in the sense of the statistical and the physical sampling error.

As pretreatment still is a form of (sub)sampling, the additional variation caused by sample pretreatment is part of the sampling error.

NOTE Due to the fact that the degree of variation reduction in sample pretreatment is usually (much) larger than the additional variation caused by it, the overall effect of pretreatment will be a reduction of variation.

D.4.3 Analytical error

The analytical error is the error that occurs during the analytical activities necessary to obtain the desired results. Starting with the analytical sample, this means the extraction or destruction of the sample or the test carried out on the sample and the subsequent analysis of the extract, destruate or eluate.

NOTE A reliable estimate of the random error attributable to analytical error will generally be available from the laboratory through its analytical quality control procedures.

D.4.4 Total error

The total error is the amount of variation that is totally added due to all the activities necessary to obtain the desired results. Based on the definitions of the sampling error in D.4.1 and D.4.2 and the analytical error in D.4.3, the total error is the sum of these two errors, or written as Equation (D.1):

$$\mathrm{CV}_{\mathrm{total}} = \sqrt{\mathrm{CV}_{\mathrm{sampling}}{}^2 + \mathrm{CV}_{\mathrm{analysis}}{}^2} \tag{D.1}$$

where

CV is the coefficient of variation.

D.5 Population parameters

A "population parameter" is any numerical characteristic of a population, such as its mean or its standard deviation. An alternative term sometimes used is "statistical parameter" – in order to emphasise the distinction between this and other usages of the word "parameter".

A key step in planning a sampling programme is to specify the population parameter that should be estimated. It is important to do this because the choice generally has a critical bearing on both the type of sampling and the number of samples needed. For a number of commonly used parameters, Annex E provides statistical expressions both for estimating the parameter itself, and for calculating the uncertainty associated with that estimate. The second of these is a critical piece of information, because it provides the quantitative link between the number of samples and the achievable reliability, i.e. precision and confidence (see D.6 and 6.6.3 and E.2.7).

For estimating percentiles, as indicated in Annex E, the choice of method depends on what can be assumed about the underlying “probability distribution” – a statistical term used to describe the relative frequencies with which different values arise in a given population. Two probability distributions are particularly useful – the Normal and logNormal distributions. Annex E accordingly provides a brief description of these distributions. It also introduces the binomial distribution because of its importance to the handling and interpretation of “presence/absence” data.

D.6 Reliability

The reliability of a sampling programme is a general term embracing three statistical concepts: bias, precision and confidence.

D.6.1 Bias

A biased sampling programme is one that has a persistent tendency either to underestimate or to overestimate the parameter of interest. Bias is a particular risk when sampling takes place from a subpopulation.

The total bias (B_{total}) of the sampling programme equals:

$$B_{\text{total}} = B_{\text{sampling}} + B_{\text{analysis}} \tag{D.2}$$

D.6.2 Precision and confidence

A unique property of probabilistic sampling is that it allows an error band – known as a “confidence interval” – to be placed around any parameter estimate. The semi-width of the confidence interval is usually known as the “precision”. This depends on:

- the desired confidence level;
- the variability in the population or subpopulation;
- the pattern of sampling (see 6.5), and
- the chosen number of samples.

The key benefit of being able to estimate the achievable precision and confidence associated with any proposed sampling programme is that it provides a quantitative link between the sampling resources used and the reliability of the resulting answers.

EXAMPLE

Suppose the summary statistics for the cadmium concentrations found in 30 random samples of contaminated soil are

$\bar{x}$ = 45 mg/kg and s = 35 mg/kg.

A 90 % confidence interval for the mean cadmium would be

45 mg/kg ± 10,9 mg/kg, namely 34,1 mg/kg to 55,9 mg/kg.

Annex E
(informative)

Statistical methods for characterizing a population

E.1 Probability distributions

E.1.1 General

The “probability distribution” is a statistical term used to describe the relative frequencies with which different values arise in a given population. The probability distribution plays an important role in sampling programme design, because the reliability of a programme can be improved if the form of the underlying distribution is known (or can reasonably be assumed).

Three distributions of particular relevance to the design of sampling programmes are described in the following subclauses.

E.1.2 Normal distribution

The probability distribution used most widely in statistics is the Normal distribution. This has a characteristic “bell” shape, and is defined by two quantities or “parameters”: the mean (which fixes the centre of the distribution), and the standard deviation (which determines the degree of spread). These and other statistical parameters are discussed further in E.2.

Figure E.1 shows an example of a Normal distribution with mean 12 mg/kg and standard deviation 2. A characteristic property of a Normally distributed population is that about 68 % of its observations fall within a range of ± 1 standard deviation from the mean, and about 95 % fall within ± 2 standard deviations. Here, therefore, most of the area under the curve lies in the range $12 \pm 2 \times 2$, namely 8 mg/kg to 16 mg/kg.

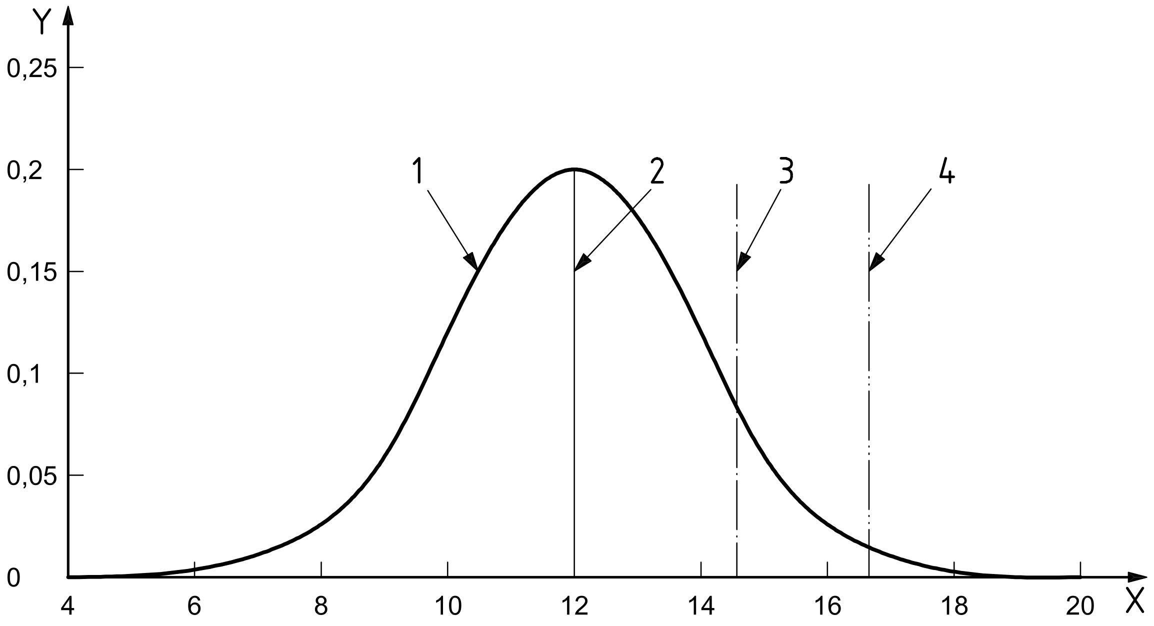
The Normal distribution is important for two main reasons. One is that many standard statistical test procedures (e.g. t-tests, F-tests) rest on the assumption that the sample values have been drawn from a Normal population.

Normality is nearly always a reliable assumption for analytical error, but is less generally applicable for variables describing the composition of a heterogeneous material like soil. It is more common to find a positively skewed distribution, whereby the majority of values are grouped relatively close to zero, but a minority of values form a tail of increasingly larger concentrations. It is easy to see how this can arise: concentrations (or other relevant characteristics of the material) can never be less than zero, but occasional high concentrations can occur.

Such populations are often better described by the logNormal distribution (see E.1.3).

E.1.3 LogNormal distribution

Figure E.2 shows an example of a logNormal distribution with mean 3,0 and standard deviation 2,4 (giving a relative standard deviation, or coefficient of variation, of 2,4 / 3,0 = 0,8). The right-hand skewness can clearly be seen: more than 90 % of the population falls below 6 mg/kg, whilst the greatest 1 % of the population lies beyond 12 mg/kg.

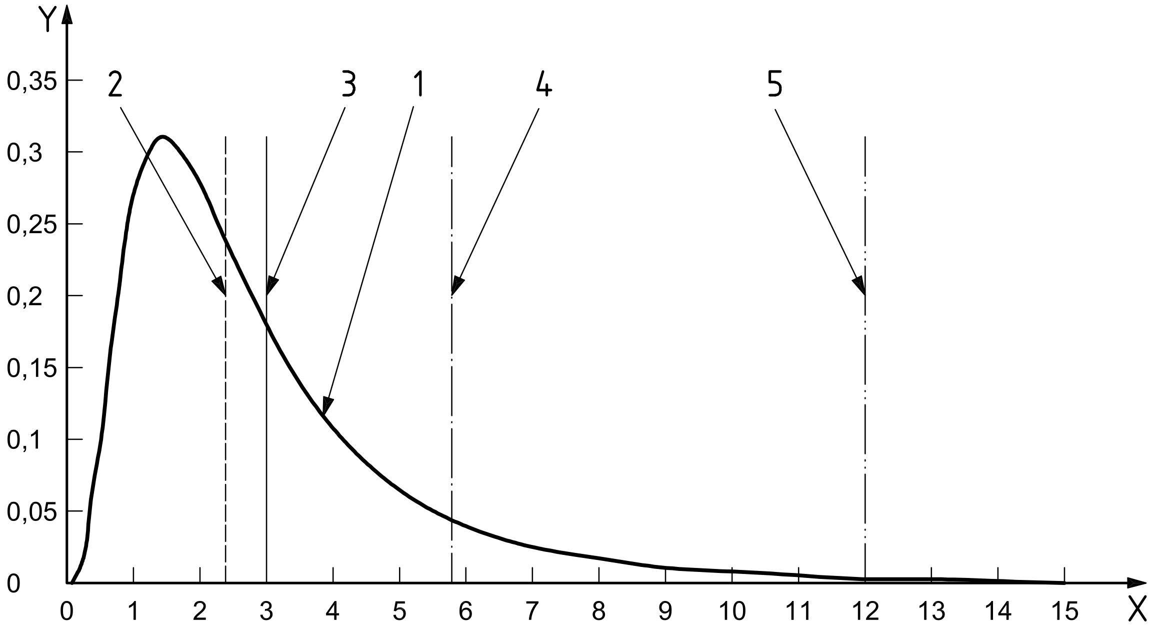


Key

X	concentration	1	normal curve	3	90-percentile
Y	probability density	2	mean, median	4	99-percentile

NOTE Standard deviation = 2,0

Figure E.1 — Example of a Normal distribution



Key

X	concentration	1	logNormal curve	4	90-percentile
Y	probability density	2	median	5	99-percentile
		3	mean		

NOTE Relative standard deviation = 0,8

Figure E.2 — Example of a logNormal distribution

The logNormal distribution is only a convenient approximation, and cannot always reflect the extreme skewness seen in some types of data. Nevertheless, it often provides an acceptable assumption, especially where the purpose of the sampling is to estimate mean concentrations (see E.2.3). There is also a practical advantage in assuming a population to be logNormally distributed, in that after logarithmic transformation the values become Normally distributed. This makes statistical analysis more straightforward, and in particular allows methods based on standard Normal theory to be used.

E.1.4 Binomial distribution

Some cases will arise where the measurement of interest is not a continuous variable, but is instead an attribute or characteristic of the population that can be either “present” or “absent”. In such cases, a widely applicable distribution is the “binomial distribution”. This is defined by two parameters: the number of samples to be taken (n), and the proportion (p) of the population that has the attribute in question. The probability of observing a specific number of samples, r, exhibiting the attribute of interest is given by:

$$B(r; n,p) = \frac{n!}{(n-r)!r!} p^{r} (1-p)^{n-r} \tag{E.1}$$

For small values of n, individual binomial probabilities can be evaluated by the straightforward application of this equation. However, it soon becomes a problem for larger values of n. Where binomial or cumulative binomial probabilities are needed, therefore, it is advisable that these are calculated using the statistical functions available in most popular spreadsheet packages.

EXAMPLE 1

Theoretical example:

If a fair coin is tossed 10 times, “tails” will on average appear 5 times, but because of sampling error the actual number may well be less than or greater than 5. The binomial distribution $B(r;\ 10,\ 0{,}5)$ determines the precise probabilities with which 0, 1, 2, …, 9, or 10 tails will be seen. For example, the probability of getting exactly 5 tails is 24,6 %.

EXAMPLE 2

Practical application:

Suppose it has been agreed that the mean concentration in no more than 50 % of daily produced soil stockpiles by a soil treatment plant may exceed a given concentration level. From daily analysis over 30 days, 19 stockpiles are identified as exceeding the given concentration level. The binomial distribution can quantify just how unusual it would be to get a proportion as high as 19/30 through sampling error alone, assuming that the process had truly been complying with the allowed rate of 50 %. (In this example, the probability of getting a result at least as extreme as this is 8 %, which is small but not unbelievably so.)

E.2 Statistical parameter

E.2.1 General

A key step in planning a sampling programme is to specify the statistical parameter that is to be estimated. This is important because the choice generally has a critical bearing on both the type of sampling and the number of samples needed.

NOTE For example, composite sampling is an effective method for estimating the mean concentration, but is inappropriate for a percentile- or maximum-related purpose.

Except for the expression for the estimation of the statistical parameter itself, a second expression is needed for calculating the statistical uncertainty associated with the estimate. The second of these is a critical piece of information, because it provides the quantitative link between the number of samples and the achievable reliability (i.e. precision and confidence). This is addressed in detail in E.2.8.2.

The following subclauses provide both expressions for each of a number of commonly used parameters.

E.2.2 Symbols and abbreviated terms

The symbols and abbreviated terms used in this annex are as follows:

n	is the total number of samples or observations
x_i	is the ith sample value (with i running from 1 to n)
$x(i)$	is the ith ranked value, i.e. the ith value after sorting the n values into increasing order
μ	is the population mean
$\overline{x}$	is the sample mean
σ	is the population standard deviation
s	is the estimated standard deviation
u_p	is the standard normal deviation corresponding to cumulative probability p
$\chi_p{}^2$	is the chi-squared deviate corresponding to cumulative probability p
X_P	is the population P-percentile
$\hat{X}_\mathrm{P}$	is the estimated P-percentile
$e_\mathrm{s}(z)$	is the standard error of the statistic z
$B(r;n,p)$	is the binomial probability that exactly r out of n random samples have a particular characteristic of interest, when the proportion of the entire population having this characteristic is p.
Cum$B(r;n,p)$	is the cumulative binomial probability that up to r out of n random samples have a particular characteristic of interest, when the proportion of the entire population having this characteristic is p.

E.2.3 Mean

The arithmetic mean – usually abbreviated to “mean” – is the most commonly encountered parameter. It is a very useful measure of the “central tendency” of a population. An unbiased estimate of the population mean is provided by the sample mean, given by:

$$\overline{x} = \frac{\sum x_i}{n} \tag{E.2}$$

The uncertainty is given by:

$$e_\mathrm{s}(\overline{x}) = \frac{s}{\sqrt{n}} \tag{E.3}$$

E.2.4 Standard deviation

The standard deviation is a widely used measure of the variability of the population. It can be thought of as the root-mean-square of all the units in the population. A (nearly) unbiased estimate of the population standard deviation is calculated as:

$$s = \sqrt{\frac{\sum (x_i - \overline{x})^2}{(n-1)}} \tag{E.4}$$

For Normal populations, the uncertainty in s can be assessed using the chi-squared distribution. A C % confidence interval for σ given s can be calculated as:

$$s\sqrt{\frac{(n-1)}{\chi^2_{1-p}}} \text{ to } s\sqrt{\frac{(n-1)}{\chi^2_{p}}} \tag{E.5}$$

where

$$p=\frac{\left(1-\frac{C}{100}\right)}{2} \tag{E.6}$$

The square of the standard deviation, s^2, is known as the “variance”. The variance is of great importance in statistical theory, but is not a practically useful measure for reporting variability as it is not defined in the same dimensions as the observed data.

EXAMPLE

Suppose a set of concentrations had a mean of 1,1 mg/kg and a standard deviation of 0,3 mg/kg.

The variance would be 0,09 mg^2/kg^2.

E.2.5 Coefficient of variation

The variability of a population can also be defined in a non-dimensional manner by the coefficient of variation, CV. An approximately unbiased estimate of the coefficient of variation is given by:

$$\text{CV}=\frac{s}{\bar{x}} \tag{E.7}$$

The uncertainty in CV can be quantified for Normal populations, but this information is not required for the present applications.

The coefficient of variation is particularly useful when the variabilities of different populations are to be compared. For many types of material, it is found that the standard deviation of a determinant tends to increase in proportion with its mean. Thus, the relative standard deviation, i.e. the CV, is approximately constant, and so this forms a good basis for comparison.

E.2.6 Percentiles

E.2.6.1 General

The P-percentile of a population is that value below which P % of the population lies.

EXAMPLE In Figure E.1, the 90-percentile has a value of about 14,6 mg/kg. This means that 90 % of the population is less than or equal to 14,6 mg/kg. Equivalently, 10 % of the population lies above 14,6 mg/kg.

Depending on what information is available about the underlying probability distribution, percentiles can be estimated in a variety of different ways, which will result in different estimates for the same percentile. Three methods to estimate a percentile are described below. Given the variety of methods to estimate the percentiles and the differences between these estimates, it is important to specify how percentiles are calculated.

E.2.6.2 Percentiles assuming Normality

The P-percentile is defined as $\mu + u_p\sigma$,

where

p = P/100

Standard Normal deviates u_p for various values of p are given in Table E.1:

Table E.1 — Standard Normal deviates u_p

P	1	5	10	50	75	90	95	97,5
p	0,01	0,050	0,1	0,5	0,75	0,9	0,95	0,975
u_p	−2,326	−1,645	−1,282	0,000	0,675	1,282	1,645	1,960

EXAMPLE For example, the 95-percentile is $\mu + 1{,}645\sigma$, and the 1-percentile is $\mu - 2{,}326\sigma$.

An (almost) unbiased estimate of the p-percentile is given by:

$$X_p = \overline{x} + u_p s \tag{E.8}$$

where

p = P/100

An approximate equation for the uncertainty in $\hat{X}p$ is:

$$e_S(X_p) = s\sqrt{\frac{1}{n} + \frac{{u_p}^2}{2(n-1)}} \tag{E.9}$$

E.2.6.3 Percentiles assuming logNormality

E.2.5.1 applies equally to the case of logNormally distributed data, with the following adjustments.

- The standard deviation s refers to the log-transformed data (it being immaterial whether base-10 or base-e is used);
- At the end of the calculation $\hat{X}p$, the estimate of the P-percentile, should finally be antilogged to return to the unlogged domain.

E.2.6.4 Percentiles – non-parametric approach

If nothing can reliably be assumed about the probability distribution, a “non-parametric” method is recommended. This is somewhat less precise than a parametric method – such as those in the preceding clauses – but is clearly a safer option when the parametric approach cannot be relied upon.

There are numerous slight variants of the non-parametric approach. The one proposed here is the so-called “Weibull” convention, whereby the P-percentile is estimated as follows:

$$X_p = X(r) \tag{E.10}$$

where

$$r = \left(\frac{p}{100}\right)(n+1) \tag{E.11}$$

If r is not an exact integer, linear interpolation should be used as follows:

$$X_p = (1-d)X(s) + dX(s+1) \tag{E.12}$$

where

s = integer part of r; and

d = $r - s$.

The concept of standard error is less appropriate for non-parametric methods.

Instead, the uncertainty in $\hat{X}_p$ can be quantified by a conservative confidence interval

$\{X(r_1)$ to $X(r_2)\}$,

where

r_1 and r_2 are defined by the following cumulative binomial expressions:

r_1 is the largest integer satisfying the condition Cum$B(r_1 - 1; n,p) \leqslant (1 - C/100)/2$, and

r_2 is the smallest integer satisfying the condition Cum$B(r_2 - 1; n,p) \geqslant 1 - (1 - C/100)/2$.

NOTE The resulting interval will in general have a confidence coefficient rather larger than C %, because of the discrete nature of binomial probabilities.

EXAMPLE

Suppose it is required to estimate the 80-percentile cadmium concentration from 39 random samples taken from a soil stockpile, together with a 90 % confidence interval.

Estimating the 80-percentile cadmium concentration by the Weibull method:

$\hat{X}_{80} = \hat{X}(r)$, where r = (80/100)(39 + 1) = 32.

Thus $\hat{X}_{80}$ is estimated by $\hat{X}$ (32), the ordered sample value with rank 32 (or, equivalently, the 8th largest value).

With a 90 percent confidence level:

C = 90 %, and so the conditions for r_1 and r_2 are:

Cum$B(r_1 - 1; 39, 0{,}8) \leqslant 0{,}05$ and Cum$B(r_2 - 1; 39, 0{,}8) \geqslant 0{,}95$.

Using appropriate software, we find by experimentation that:

Cum$B$(26; 39, 0,8) = 0,035 5 and Cum$B$(35; 39, 0,8) = 0,966 8.

Thus the interval $\hat{X}$(27) to $\hat{X}$(36), i.e. the interval from the 13th biggest to the 4th biggest sample value, provides a conservative 90 % confidence interval for the true 80-percentile cadmium concentration. (The actual confidence coefficient is 0,966 8 − 0,035 5 = 0,931, or 93,1 %).

E.2.7 Maximum

The population maximum should not be used as the desired statistical parameter (except in the unlikely event of the sampling being of very high frequency). This is because no reliable estimate of the maximum can ever be obtained from a set of sample values. The sample maximum will always be an underestimate of the population maximum, and furthermore, there is no straightforward method available for quantifying the extent of that bias.

Where the primary goal is concerned with "worst case" values, the recommended approach is to recast the goal in terms of a suitably high percentile – say the 99-percentile. The methods described in E.2.5 can then be applied.

E.2.8 Percentage compliance with a given limit

E.2.8.1 General

The primary sampling goal often relates to the percentage of a population that complies with a specific limit (e.g. a target or intervention value).

As with percentile-type goals, both parametric and non-parametric approaches can be taken. To contrast the two approaches, imagine that the limit L shall be complied with for P % of the time or better.

E.2.8.2 Percentage compliance – parametric approach

Using the parametric approach, the P-percentile would be estimated assuming a particular distribution (e.g. Normal), and the resulting estimate $\hat{X}_p$ would be compared with L. The statistical uncertainty in the compliance result would then be assessed using the quantity $e_s(\hat{X}_p)$.

The parametric approach is not, however, generally suggested unless there is reliable information about the nature of the underlying distribution, because of the confusion that can be caused whenever the parametric estimate differs markedly from the non-parametric compliance figure — that is, the simple pass rate calculated directly from the data. Moreover, the details of the statistical method go beyond the scope of this document (even in the case where Normality can be assumed), and so specialist statistical advice should be sought for its application.

E.2.8.3 Percentage compliance – non-parametric approach

By the non-parametric approach, the quantity r (the number of sample values $\leqslant L$) is first calculated.

The sample compliance $100(r/n)$ % can then be determined.

The advantage now is that $100(r/n)$ is binomially distributed (irrespective of the distribution followed by the original samples), and so the statistical uncertainty in the compliance result can be assessed without the need for any distributional assumptions about the population.

Specifically, a C % confidence interval for the true population compliance is given by [$100p_{\mathrm{LO}}$ to $100p_{\mathrm{UP}}$], where:

p_{LO} is chosen so that $1 - \mathrm{Cum}B(r - 1; p_{\mathrm{LO}}, n) = (1 - C/100)/2$,

and

p_{UP} is chosen so that $\mathrm{Cum}B(r; p_{\mathrm{UP}}, n) = (100 - C)/2$.

$\mathrm{Cum}B(r;n,p)$ is the cumulative binomial probability that up to r out of n random samples have a particular characteristic of interest, when the proportion of the entire population having this characteristic is p.

Although the definition of the limit with which the observations are to be compared falls outside the scope of this part of ISO 10381, it is important to realize that the (often implicit) statement that “no observation may exceed the limit” is statistically unusable. It implies that not even one single unit of the population (at the investigated scale, see also D.2.2) might have a concentration above that limit. In order to test this hypothesis, it would be necessary to test the entire population at the predefined scale!

However, an almost equivalent but statistically “coherent” level of protection can be obtained by requiring that 99 % (or even 99,9 %) of the population at the defined scale, rather than 100 %, should comply with the limit.

Annex F
(informative)

Calculating the required number of samples

F.1 Symbols and abbreviated terms

The symbols and abbreviated terms used in this annex are as follows:

n	is the total number of samples or observations
m	is the number of increments per composite sample
μ	is the population mean
u_p	is the standard normal deviation corresponding to cumulative probability p
$\chi_p{}^2$	is the chi-squared deviation corresponding to cumulative probability p
X_p	is the population P-percentile
$\hat{X}p$	is the estimated P-percentile
$e_\mathrm{s}(z)$	is the standard error of the statistic z
σ_w	is the standard deviation of local (i.e. within-composite) spatial variation
σ_b	is the standard deviation of between-composites spatial and/or temporal variation
σ_s	is the standard deviation of total spatial and/or temporal variation (= $\sqrt{[\sigma_\mathrm{w}{}^2 + \sigma_\mathrm{b}{}^2]}$)
σ_e	is the standard deviation of analytical error
C	is the desired confidence level (%)
a	is the cumulative probability related to the desired confidence level
d_prec	is the desired precision

F.2 Estimating a mean concentration

F.2.1 Using composite sampling

The standard error of the mean is given by:

$$e_\mathrm{s}(\mathrm{mean}) = \sqrt{\left[\frac{\left(\dfrac{\sigma_\mathrm{w}^2}{m} + \sigma_\mathrm{b}^2 + \sigma_\mathrm{e}^2\right)}{n}\right]} \qquad (\mathrm{F.1})$$

Thus for a given value of m, and assuming Normality, the number of composites required to achieve the desired precision and confidence is given approximately by:

$$n = \left(\frac{u_a}{d_{\text{prec}}} \right)^2 \times \left(\frac{\sigma_{\text{w}}^2}{m} + \sigma_{\text{b}}^2 + \sigma_{\text{e}}^2 \right) \tag{F.2}$$

where

a is 1 – (1 – C/100)/2.

Alternatively, Equation (F.1) can be rewritten to determine the number of increments (m) needed per composite sample if n, the total number of composite samples, has been set in advance. Thus:

$$m = \frac{\sigma_{\text{w}}^2}{\left[n \left(\frac{d_{\text{prec}}}{u_{\text{a}}} \right)^2 - \sigma_{\text{b}}^2 - \sigma_{\text{e}}^2 \right]} \tag{F.3}$$

NOTE 1 It may be desirable to plan to take only a single composite sample. Provided $\sigma_{\text{b}}^2 + \sigma_{\text{e}}^2$ is sufficiently small, this can be achieved by setting n equal to 1 in Equation (F.2).

In practice, the true standard deviations are unknown and so estimates must be used. In some cases, it may be appropriate to use the values obtained from the past analysis of sample data from similar investigations. Otherwise the estimates should where possible be obtained from a preliminary pilot study.

EXAMPLE 1

Suppose that:

- estimates of σ_{w}, σ_{b} and σ_a are 4 mg/kg, 2 mg/kg and 0,5 mg/kg;
- 10 increments are to be taken per composite (i.e. m = 10), and
- the mean is required to be estimated to a precision of d_{prec} = 1 mg/kg with 90 % confidence.

Calculating a for a 90-percent confidence level:

For C = 90, a = 1 – (1 – 90/100)/2 = 0,95, and so u_a = 1,65.

Calculating n from Equation (F.1),

$n = (1{,}65)^2(16/10 + 4 + 0{,}25) = 15{,}9$.

Thus, about 16 composite samples would be needed to produce a mean to the required reliability.

To decide on the most appropriate value of m, it is necessary to consider the relative costs of sampling and analysis.

EXAMPLE 2

Suppose that

A is the sampling cost per increment, and

B is the analysis cost per sample (considerably greater than A).

The total cost T_{c} is accordingly given by:

$$T_{\text{c}} = (A_m + B)n \tag{F.4}$$

Thus, using Equation (F.1) with various trial values of m, it is possible to find the combination of m and n which minimizes T_{c}.

EXAMPLE 3

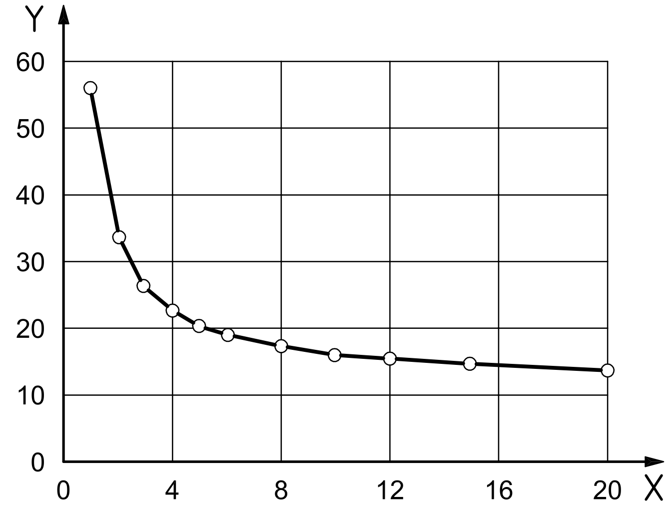
Continuing with the earlier example, suppose that:

- values of m ranging from 1 to 20 are considered, and
- $B/A = 30$ – that is, a sample analysis is 30 times more expensive than the cost of a sample increment.

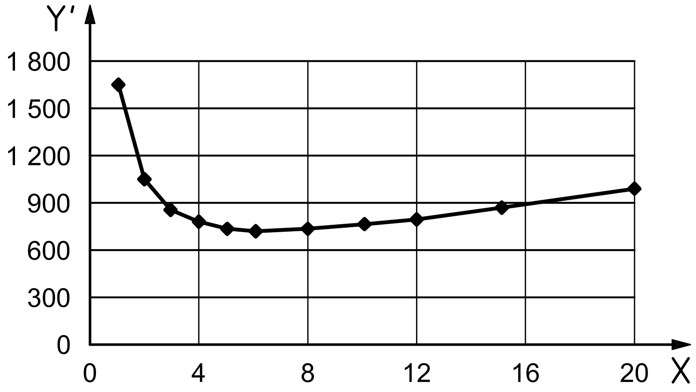
Getting the n and T_c values:

> The upper panel of Figure F.1 shows the n value given by Equation (F.1) for each trial value of m.
>
> The lower panel then shows the corresponding values of the total sampling cost T_c (in arbitrary units).

Thus, it is apparent that the optimum number of increments per composite sample is about 6.



a) Samples needed to achieve specified precision and confidence



b) Cost of sampling in relation to number of increments per composite sample

Key

X number of increments, m
Y required number of composites, n
Y′ total cost of sampling, T_c

Figure F.1 — Illustration of the relationships between m, n and T_c (see text for details)

F.2.2 Using individual samples

The standard error of the mean is given by:

$$e_\mathrm{s}(\text{mean}) = \sqrt{\left[\frac{\left(\sigma_\mathrm{s}^2 + \sigma_\mathrm{e}^2\right)}{n}\right]} \tag{F.5}$$

Thus, the number of samples required to achieve the desired precision and confidence is given approximately by:

$$n = \left(\frac{u_a}{d_\mathrm{prec}}\right)^2 \times \left(\sigma_\mathrm{s}^2 + \sigma_\mathrm{e}^2\right) \tag{F.6}$$

where

a = 1 – (1 – C/100)/2.

NOTE Spot sampling can be thought of as composite sampling with just one increment per composite. Thus, the results of the previous section apply to the case of spot sampling by substituting m = 1 and replacing $\sigma_\mathrm{w}^2 + \sigma_\mathrm{b}^2$ by σ_s^2.

In practice, the true standard deviations are unknown and so estimates must be used. In some cases, it may be appropriate to use the values obtained from the past analysis of sample data from similar investigations. Otherwise the estimates should where possible be obtained from a preliminary pilot study.

EXAMPLE

Suppose that:

- estimates of σ_s and σ_e are 4,5 mg/kg and 0,5 mg/kg, and
- the mean is required to be estimated to a precision of d = 2 mg/kg with 90 % confidence.

Getting u_a:

For C = 90, a = 1 – (1 – 90/100)/2 = 0,95, and so u_a = 1,65.

Getting the n value:

From Equation (F.5), $n = (0{,}825)^2(20{,}25 + 0{,}25) = 13{,}9$.

Thus:

About 14 spot samples would be needed to produce a mean to the required reliability.

F.3 Estimating a standard deviation

The following approach is applicable when the population can be assumed to be normally distributed. Even for non-Normal populations, however, the method is useful as a rough approximation.

Confidence intervals for σ can be calculated using the equation given in E.2.4. For a given choice of confidence C, this can be evaluated for a range of trial n values, and this will identify the number of samples that provides the required precision.

EXAMPLE

Suppose it is required to estimate the standard deviation to a precision of 20 % with 90 % confidence.

For 90 % confidence, the lower and upper p values are = 0,5 ± C/200 = 0,05 and 0,95.

With the help of statistical tables of the χ^2 distribution at the P 0,05 and 0,95 points, the following Table F.1 can be developed.

Table F.1 — 90 % confidence limits for σ/s for various numbers of samples

No. of samples	Lower 90 % confidence limit for σ/s	Upper 90 % confidence limit for σ/s
n	$\sqrt{[(n-1)/\chi^2]}$ (p = 0,05)	$\sqrt{[(n-1)/\chi^2]}$ (p = 0,95)
20	0,79	1,37
30	0,83	1,28
40	0,85	1,23
50	0,86	1,20
60	0,87	1,18
70	0,88	1,16
80	0,89	1,15
90	0,89	1,14
100	0,90	1,13
120	0,90	1,12
150	0,91	1,11
200	0,92	1,09

It can be seen that with 50 samples, the lower and upper confidence limits are 0,86 and 1,20. That is, the population standard deviation σ may be 14 % below or 20 % above s, the observed standard deviation (note that the interval is asymmetrical). Thus, a precision of 20 % or better will be achieved by a standard deviation calculated from 50 random samples.

F.4 Estimating a percentile

F.4.1 Assuming normality

The standard error of the P-percentile X_P is given by:

$$e_\mathrm{s}(X_\mathrm{P}) = \sigma\sqrt{\frac{1}{n} + \frac{{u_p}^2}{2(n-1)}} \qquad \text{(F.7)}$$

where

p = P/100

σ = $\sqrt{(\sigma_\mathrm{s}^2 + \sigma_\mathrm{e}^2)}$

Thus, the number of samples required to achieve the desired precision and confidence is given approximately by:

$$n = \left(\frac{u_a s}{d_{\text{prec}}}\right)^2 \times \left(\frac{1+u_p^2}{2}\right) \qquad \text{(F.8)}$$

where

a is 1 − (1 − C/100)/2

s is an estimate of σ

EXAMPLE

Suppose that:

- σ is estimated by s = 3,5 mg/kg, and
- the 95-percentile is required to be estimated to a precision of d_{prec} = 1,46 mg/kg with 90 % confidence.

Finding u_p and u_a:

- For the 95-percentile, p = 0,95 and so u_p = 1,65.
- For C = 90, a = 1 − (1 − 90/100)/2 = 0,95, and so u_a = 1,65.

Finding the n value:

- Thus from Equation (F.7), $n = (1{,}65 \text{ x } 3{,}5/1{,}46)^2 \times (1 + 1{,}65^2/2) = 36{,}9$.

Result:

- Thus, about 37 samples would be needed for the 95-percentile to be estimated to the required reliability.

F.4.2 Non-parametric approach

For determining the precision achievable by a non-parametric approach, there is no direct expression available corresponding to the one given in F.4.1 for the Normal case. As a rough approximation, however, the expression given in F.4.1 can still be used, but with an additional multiplicative factor of 1,3 applied to represent the poorer precision typically attained by the non-parametric rather than the Normal-based approach.

Alternatively, exact results can be obtained using the following more time-consuming approach. The first step is to select a trial number of samples and desired confidence level, C. The methodology described in E.2.6.4 for calculating C % confidence intervals around non-parametric percentile estimates is then applied. This should be repeated for different trial sample numbers. The various confidence intervals will be expressed as ranked values, but these can be converted into equivalent actual measurements as long as a suitable historical data set is available. These trial calculations will give an indication of the precision that can typically be achieved at C % confidence for various numbers of samples; and from this an appropriate choice can be made.

EXAMPLE

Suppose that the 80-percentile cadmium concentration from a particular soil stockpile is required to be estimated to a precision of d = 15 mg/kg with 90 % confidence.

Select n = 39 as the trial number of samples.

From E.2.6.4, a conservative 90 % confidence interval is provided by the interval $\hat{X}(27)$ to $\hat{X}(36)$.

- From a set of 39 cadmium concentrations taken at random from prior comparable soil stockpiles, the 15 highest values are: (12, 12, 13, 15, 17, 20, 20, 25, 26, 31, 31, 35, 36, 40 and 55) mg/kg.
- Thus, the 27th and 36th ranked values are 13 mg/kg and 35 mg/kg, and so the expected precision is

  (35 − 13)/2 = 11 mg/kg.
- This is better than required, and so a lower trial value of n is selected.

Select n = 29 as the new trial number of samples.

- From E.2.6.4, a conservative 90 % confidence interval is provided by the interval $\hat{X}(20)$ to $\hat{X}(28)$.
- From a set of 29 values taken at random from the historical data, the 12 highest values are:

  (10, 12, 12, 15, 20, 20, 25, 26, 31, 35, 40 and 55) mg/kg.
- Thus, the 20th and 28th ranked values are 12 mg/kg and 40 mg/kg and so the expected precision is (40 − 12)/2 = 14 mg/kg.
- This is adequately close to the required precision.

Result:

- About 29 samples would therefore be needed for the 80-percentile to be estimated to the required reliability.

F.5 Estimating a percentage compliance with a given limit

The approach here is similar to that described in F.4.2. First, the desired confidence level, C, is chosen. Then, for each of a range of trial sample numbers, the C % confidence interval for the true percent compliance is calculated using the methodology described in E.2.8. The resulting set of confidence intervals shows the quantitative link between achievable precision and samples taken, and hence provides a rational basis for arriving at an acceptable compromise.

EXAMPLE

Suppose that:

- the percentage of a soil stockpile meeting a particular cadmium concentration limit is thought to be about 80 %;
- this percentage must be estimated to a precision of 10 % with 90 % confidence, and
- nothing is known about the statistical nature of the cadmium distribution.

Select a trial number of samples of n = 20, and suppose that 16 samples meet the required cadmium limit (that is, the observed compliance rate is 80 %).

- Using the non-parametric binomial method described in E.2.8.3, calculate a 90 % confidence interval for the true compliance percentage.
- This is 71,7 % to 98,2 %, giving a precision of about 13 %.
- Thus, a greater number of samples is needed.

Select a trial number of samples of n = 40, and suppose that 32 samples meet the required cadmium limit (to keep the observed compliance rate at 80 %).

- Using E.2.8.3, calculate a 90 % confidence interval for the true compliance percentage.
- This is 78,6 % to 96,5 %, giving a precision of about 9 %.
- This is adequately close to the required precision.

Result:

- About 40 samples would therefore be needed for the compliance percentage to be estimated to the required reliability.

Annex G
(informative)

Examples of types of sampling suitable for the goal

G.1 General

The definition of the type of sampling is determined on the basis of the purpose of sampling, the primary sampling goal and the secondary sampling goals. By definining the secondary sampling goals, essential elements of the sampling plan are also defined, and these goals are therefore the guidance for the definition of the sampling programme. However, the secondary goals cannot be defined without clear definition of the purpose of sampling and of the primary sampling goals.

In this annex, examples are given on how to derive the secondary sampling goals (the detailed definition of the technical aspects necessary for defining the sampling) in subsequent steps from:

- the purpose of sampling (the reason why the stockpile is sampled), to
- the primary sampling goal (the definition of the sampling in short, general statements, giving direction towards the type of sampling, but still lacking the necessary detail to define a sampling plan).

Secondary sampling goals are given below:

a) definition of the stockpile to be sampled (the population);

b) definition of the components to be determined and/or tests to be carried out on the samples;

c) definition of the statistical parameter to be determined (e.g. mean concentration, degree of heterogeneity, percentile);

d) definition of the type of sampling (probabilistic or judgemental);

e) definition of the scale of sampling (the use of increments and composite samples or individual samples and the scale on which the soil should be tested);

f) definition of the desired precision and confidence.

G.2 Example of the basic characterization of a soil stockpile

G.2.1 Purpose of sampling

This example deals with the characterization of the quality of a stockpile of soil to be used for laboratory experiments. The general quality of soil material to be used in the laboratory experiments is already known, for instance due to prior compliance testing, but in order to determine whether it is truly fit for the planned experiments, the quality of the soil has to be determined in much more detail.

G.2.2 Primary sampling goal

Level 1 testing is a basic (comprehensive) characterization, consisting of a thorough determination of the behaviour and properties of interest of the material.

G.2.3 Definition of secondary sampling goals

The following secondary sampling goals are defined:

a) definition of the stockpile

   The stockpile is a predefined heap of soil material already available at the laboratory that will conduct the planned experiments.

b) definition of the components and tests

   As the experiments involve the plant uptake of heavy metals, the concentrations of the heavy metals is to be determined in order to know the original concentrations in the soil.

c) definition of the statistical parameter

   1) The concentrations of heavy metals in the soil stockpile are assumed to be homogeneous on the scale on which the laboratory experiments will take place (1 m^3).

   2) In order to determine the variability on this scale, the mean concentration on a 1 m^3 has to be determined.

d) definition of the type of sampling

   It is obvious that, for a thorough quality check of the soil, a probabilistic sampling approach is essential.

e) definition of the scale of sampling

   1) As mentioned, the laboratory experiments will be conducted at a 1 m^3 scale.

   2) The individual experiments need the same soil quality (for heavy metals) on this 1 m^3 scale.

   3) Therefore, the variability of the soil has to be determined on a 1 m^3 scale.

   4) This implies that for each potential part of 1 m^3, the mean concentration must be known to lie within a specified bandwidth.

f) definition of the desired precision and confidence

   1) A reliable estimate has to be obtained for the mean concentration within a soil volume of 1 m^3.

   2) In order to obtain this reliability, sufficient “units” of 1 m^3 have to be investigated in order to know the degree of variability within the stockpile at this scale.

As there is no prior indication of the variability between the “units”, a two-phased sampling programme is defined. However, as it is not efficient to sample the stockpile twice, all samples are taken at the same time. It is decided that the investigation of a maximum of 30 “units” should be sufficient, so in the sampling stage, 30 “units” are chosen at random and sampled.

In order to get a good estimate of the mean concentration within a soil volume of 1 m^3, each unit is sampled by means of three directional samples of 1 m each. From each of these three 1 m samples, a composite sample is taken in the field. This results in 30 × 3 borings: a total of 90 borings and 90 composite samples.

In order to obtain an indication of the variability within the stockpile on the scale of 1 m^3, 5 of the 30 “units” are chosen randomly. For each of these 5 “units”, the three composite samples are selected and analysed individually. The mean concentration for each “unit” is then used to estimate the variability between the “units”. Based on this estimation and the desired precision, the total number of “units” that are to be analysed is calculated. When 5 appears to be sufficient the investigation is finished after the first stage, if not an additional number of “units” is analysed up to a maximum of 25.

G.3 Example of the compliance of a soil stockpile with national limit values for re-usability

G.3.1 Purpose of sampling

In this example, national legislation determines that for the re-use of soil, the mean concentrations for a defined number of components within a soil stockpile should be in compliance with the limit values stated in the legislation. If they are in compliance, the soil can be re-used. If not, it has to be treated in a soil treatment plant, or be dumped on a waste disposal site.

G.3.2 Primary sampling goal

Level 2 testing is testing to determine compliance with specific reference conditions (e.g. legislation).

G.3.3 Definition of secondary sampling goals

The following secondary sampling goals are defined:

a) definition of the stockpile

   The stockpile is known, as this is the material that has to be tested prior to re-use.

b) definition of the components and tests

   1) The components to be tested are given in legislation: 8 (heavy) metals, sum of 10 specified PAHs[2)], mineral oil and EOX[3)].

   2) The two composite samples are to be analysed after thorough sample pretreatment in the laboratory.

c) definition of the statistical parameter

   The mean concentration of the stockpile has to comply with the reference levels given in legislation.

d) definition of the type of sampling: Probabilistic sampling

   1) The number of increments is defined in legislation, i.e. 2 × 50 increments, to be taken at random locations throughout the soil stockpile.

   2) The total of 100 increments are divided into two composite samples of 50 increments each.

e) definition of the scale of sampling

   The scale of interest is the size of the increments, for example 200 g.

f) definition of the desired precision and confidence

   The true mean concentration should be within the 90 % confidence interval of the determined mean concentration.

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2) PAH Polycyclic aromatic hydrocarbons.

3) EOX Extractable organic halides.

The number of increments to be taken can only be calculated when the variability of the increments within the stockpile is known. This is obviously not the case without sampling the stockpile and analyses of all individual increments. However, based on a large number of investigated stockpiles, it is known that the true mean for 85 % to 98 % of the stockpiles (depending on the components to be determined) will be within the 90 % confidence interval of the estimated mean when 100 increments are taken.

NOTE The described sampling method is used in Dutch legislation, and is validated by statistical analysis of 2 570 soil stockpiles which were sampled by means of this strategy. Depending on the assumed size of the sampling error, it has been proven that the strategy is sufficiently reliable for the percentages of soil stockpiles, in the Netherlands, as given below:

Table G.1 — Percentage of soil stockpiles for which the sampling strategy is sufficiently reliable

Assumed sampling error	**Inorganic components**		**Organic components**	
	5 %	10 %	5 %	20 %
Strategy 2 × 50	97 %	98 %	75 %	82 %

G.4 Example of on-site verification

G.4.1 Purpose of sampling

In this example, the soil delivered to a farmer appears to consist of a different soil type than that which was ordered (e.g. peat instead of clay).

G.4.2 Primary sampling goal

Level 3 testing involves a “quick check” to establish consistency with soil characteristics for clay (e.g. percentage fines, dry matter and organic matter content).

G.4.3 Definition of secondary sampling goals

The following secondary sampling goals are defined:

a) definition of the stockpile

    1) The material as delivered at the farm.

    2) As the stockpile is lying on top of the land of the farmer, a clear distinction between the soil stockpile and the underlying land is difficult or impossible.

    3) Therefore, the stockpile should not be sampled in this “zone”.

b) definition of the components and tests

    In order to determine whether the soil is of the soil type specified in the contract (clay), the percentage of fines (< 2 µm and < 63 µm) has to be determined, as well as the dry matter and organic matter content.

c) definition of the statistical parameter

    The concentration in a composite sample obtained from different locations throughout the soil stockpile.

d) definition of the type of sampling

    1) Simple random sampling on a limited number of locations (e.g. 10) throughout the soil stockpile will be sufficient.

    2) Full probabilistic sampling will not be necessary (for example in the centre of the stockpile) as long as there is no tendency to select sampling.

e) definition of the scale of sampling

There is now no real need for a pre-defined scale of interest; the normal size of an increment (e.g. 200 g) will be sufficient, resulting in a composite sample of limited size (e.g. 2 kg).

f) definition of the desired precision and confidence

Limited precision and confidence, these are not to be quantified.

Annex H
(informative)

Sampling techniques

H.1 Sampling techniques for probabilistic sampling

H.1.1 General

The following sampling techniques will, when applied correctly, result in probabilistic samples:

- simple random sampling;
- stratified random sampling;
- systematic sampling.

See also 6.5.

In the case of a stockpile which, due to the way it is available for sampling, cannot be satisfactorily differentiated from adjoining stockpiles, a “safety margin” shall be applied in connection with the spatial definition of the stockpile such that adjoining stockpiles do not partially overlap.

NOTE The size of the sampled stockpile is actually reduced by this safety margin. The material in the safety margin (the area where the adjoining stockpiles possibly overlap each other partially) can therefore not be assessed.

H.1.2 Simple random sampling

H.1.2.1 Principle

In simple random sampling, the sampling locations are selected by using random numbers. In order to apply these random numbers, an imaginary spatial co-ordinate system is defined around the stockpile, giving each possible location within the stockpile a spatial co-ordinate. Random numbers are generated by a random numbers table, calculator or computer (programme) or likewise.

H.1.2.2 Sampling equipment

All sampling equipment that fulfils the requirements for probabilistic sampling, as defined in 7.3, 6.4, 6.5 and 6.6.

H.1.2.3 Circumstances

All circumstances where application of the chosen sampling equipment in conjunction with the appearance of the stockpile (e.g. maximum particle size, degree of consolidation, size/height) enables the sampler to take the samples in a safe way whilst the identity of the sampled material is guaranteed.

The sampling process shall result in sampling the soil material at the pre-defined locations within the stockpile. The process of inserting and retrieving the sampling apparatus should not result in the pick-up of soil material from other locations than the selected sampling location, or in loss of material from the selected sampling location.

H.1.2.4 Procedure

In order to facilitate the random selection of sampling locations, an imaginary spatial co-ordinate system is defined for the spatial definition of the sampling locations.

Measure the stockpile precisely. Apply an imaginary three-dimensional co-ordinate system by which every point within the stockpile is defined. Samples will be taken on coordinates that are randomly chosen within the volume of the stockpile.

Using random numbers, determine the sampling points within the stockpile. Begin by determining the X coordinate in accordance with Equation (H.1):

$$X = r\left(X_{\text{max}} - X_0\right) \tag{H.1}$$

where:

X	is the X coordinate of the sampling point, in metres (m);
r	is a random number;
X_{max}	is the maximum value of X; the stockpile ends here (in the X-direction), in metres (m);
X_0	is the minimum value of X; the stockpile begins here (in the X-direction), in metres (m).

Then, determine the value of the Y coordinate in accordance with Equation (H.1):

$$Y = r\left(Y_{\text{max}} - Y_0\right) \tag{H.2}$$

where:

Y	is the Y coordinate of the sampling point, in metres (m);
r	is a random number;
Y_{max}	is the maximum value of Y; the stockpile ends here (in the Y-direction), in metres (m);
Y_0	is the minimum value of Y; the stockpile begins here (in the Y-direction), in metres (m).

Determine whether the point X,Y occurs in the stockpile. If not, a new value for Y is determined.

Then, determine the value of the Z coordinate in accordance with Equation H.3:

$$Z = r\left(Z_{\text{max}} - Z_0\right) \tag{H.3}$$

where:

Z	is the Z coordinate of the sampling point, in metres (m);
r	is a random number;
Z_{max}	is the maximum value of Z; the stockpile ends here (in the Z-direction), in metres (m);
Z_0	is the minimum value of Z; the stockpile begins here (in the Z-direction), in metres (m).

Determine whether the point X,Y,Z occurs in the stockpile. If not, a new value for Z is determined.

Use the selected sampling equipment in order to obtain a sample from the designated location. Withdraw the sampling equipment and make sure that the sample volume of the equipment is correctly filled.

If the stockpile is non-consolidated, it may be difficult or even impossible to reach the randomly selected sample locations for sampling. If so, the type of sampling equipment should be adapted to this type of sampling situation or the sampling technique should be altered (see Notes 2 and 3). A final option would be not to sample the inside of the stockpile, but this would mean that a non-probabilistic sampling method is used.

NOTE 1 Simple random sampling of soil stockpiles is not (readily) practicable if the soil has a maximum particle size (D_{95}) of more than approximately 20 mm. The minimum increment size is then so large that sampling cannot be carried out with the usual (manual) sampling equipment. In addition, this method of sampling is also not (readily) practicable if the depth at which a sample is taken is more than 6 m to 8 m.

NOTE 2 As the sampling depth increases, so the costs of sampling will rise (substantially). When sampling a non-consolidated soil stockpile, special precautions need to be taken for depths of more than about 0,5 m (e.g. tube-enclosed drilling) in order to ensure that the sample material does indeed originate from the desired depth. This results in a considerable rise of sampling costs.

NOTE 3 To take samples at greater depths, it is also possible to partially excavate the stockpile, if, in addition to sufficient space for the storage of the part dug up, a suitable excavating machine is available. The integrity of the sample at the locations defined in the spatial coordinate system needs to be preserved. Less than complete excavation prevents undesirable separation of particles at the sampling location. The distance to be maintained from the sampling location depends on the size of the particles of the soil and the slope of the soil above the excavation.

Apply suitable sample pretreatment when appropriate and defined in the sampling plan (see 5.2.8 and Clause 8).

Transfer the (sub)sample into an appropriately sized sample container. Wipe the outside of the sample container and apply a label recording sample details and any observations. Apply suitable sample preservation and handling procedures as identified in the sampling plan (see 5.2.9 and Clause 9).

Record the operations carried out including the specific coordinates and any special conditions in the sampling form.

H.1.3 Stratified random sampling

H.1.3.1 Principle

In stratified random sampling a number of strata is defined prior to sampling. Each stratum has the same size/volume (also see 6.5.3) and therefore covers an equal amount of material as present in the stockpile. The total number of strata covers the complete stockpile.

From each stratum an equal number of samples is taken. The positions of the samples within the stratum are defined by random selection of sampling locations.

H.1.3.2 Sampling equipment

All sampling equipment that fulfils the requirements for probabilistic sampling as defined in 7.3, 6.4, 6.5 and 6.6.

H.1.3.3 Circumstances

All circumstances where application of the chosen sampling equipment in conjunction with the appearance of the stockpile (e.g. maximum particle size, degree of consolidation, size/height) enables the sampler to take the samples in a safe way whilst the identity of the sampled material is guaranteed.

The sampling process shall result in sampling the soil material at the pre-defined locations within the stockpile. The process of inserting and retrieving the sampling apparatus should not result in the pick-up of soil material from other locations than the selected sampling location, or in loss of material from the selected sampling location.

H.1.3.4 Procedure

Define the strata based on the spatial co-ordinates system as mentioned in H.1.2. Use the procedure of H.1.1 for defining the sampling location(s) within each stratum, where “stockpile” should be read as “stratum”.

NOTE See the comments in H.1.2.

Use the selected sampling equipment in order to obtain a sample from the designated location. Withdraw the sampling equipment and make sure that the sample volume of the equipment is correctly filled.

Apply suitable sample pretreatment when appropriate and defined in the sampling plan (see 5.2.8 and Clause 8).

Transfer the (sub)sample into an appropriately sized sample container. Wipe the outside of the sample container and apply a label recording sample details and any observations. Apply suitable sample preservation and handling procedures as identified in the sampling plan (see 5.2.9 and Clause 9).

Record the operations carried out including the specific coordinates and any special conditions in the sampling form.

H.1.4 Systematic sampling

H.1.4.1 Principle

In systematic sampling a systematic pattern of sampling points covers the stockpile. The size of the pattern will be determined by the number of samples to be taken. The systematic pattern is applied both in horizontal and vertical direction.

H.1.4.2 Sampling equipment

All sampling equipment that fulfils the requirements for probabilistic sampling as defined in 7.3, 6.4, 6.5 and 6.6.

H.1.4.3 Circumstances

All circumstances where application of the chosen sampling equipment in conjunction with the appearance of the stockpile (e.g. maximum particle size, degree of consolidation, size/height) enables the sampler to take the samples in a safe way whilst the identity of the sampled material is guaranteed.

The sampling process shall result in sampling the soil material at the pre-defined locations within the stockpile. The process of inserting and retrieving the sampling apparatus should not result in the pick-up of soil material from other locations than the selected sampling location, or in loss of material from the selected sampling location.

H.1.4.4 Procedure

The principle of the systematic sampling pattern is that the distance between sampling locations both in horizontal and vertical direction is constant, however there is no need that the horizontal distance is equal to the vertical distance.

In order to define the distance(s) between sampling locations:

- the volume of the stockpile needs to be estimated;
- the number of samples or increments that shall be gathered must be known; and
- the vertical distance between samples shall be chosen.

The distance (in a horizontal plane) between the sampling locations can then be calculated, using Equation (H.4):

$$\Delta_{XY} = \sqrt{\frac{V_s}{\Delta_Z}} \quad \text{(H.4)}$$

with Equation (H.5):

$$V_s = \frac{V}{n} \quad \text{(H.5)}$$

where:

Δ_{XY} is the distance between sample locations in a horizontal plane, in metres (m);

V_s is the volume per sample, in cubic metres (m^3);

Δ_Z is the distance between sample locations in vertical direction, in metres (m);

V is the total volume of the stockpile, in cubic metres (m^3);

n is the number of samples or increments to be taken.

Example 1:

From a stockpile of approximately 1 250 m^3, 100 increments have to be taken at a vertical distance of 0,5 m.

V_s = 1 250/100 = 12,5 m^3 per sample

Δ_{XY} = $\sqrt{}$(12,5/0,5) = 5 m

Figure H.1 gives the sampling locations for this example.

Example 2:

From a stockpile of approximately 1 000 m^3, 30 samples have to be taken at a vertical distance of 2 m.

V_s = 1 000/30 = 33 m^3 per sample

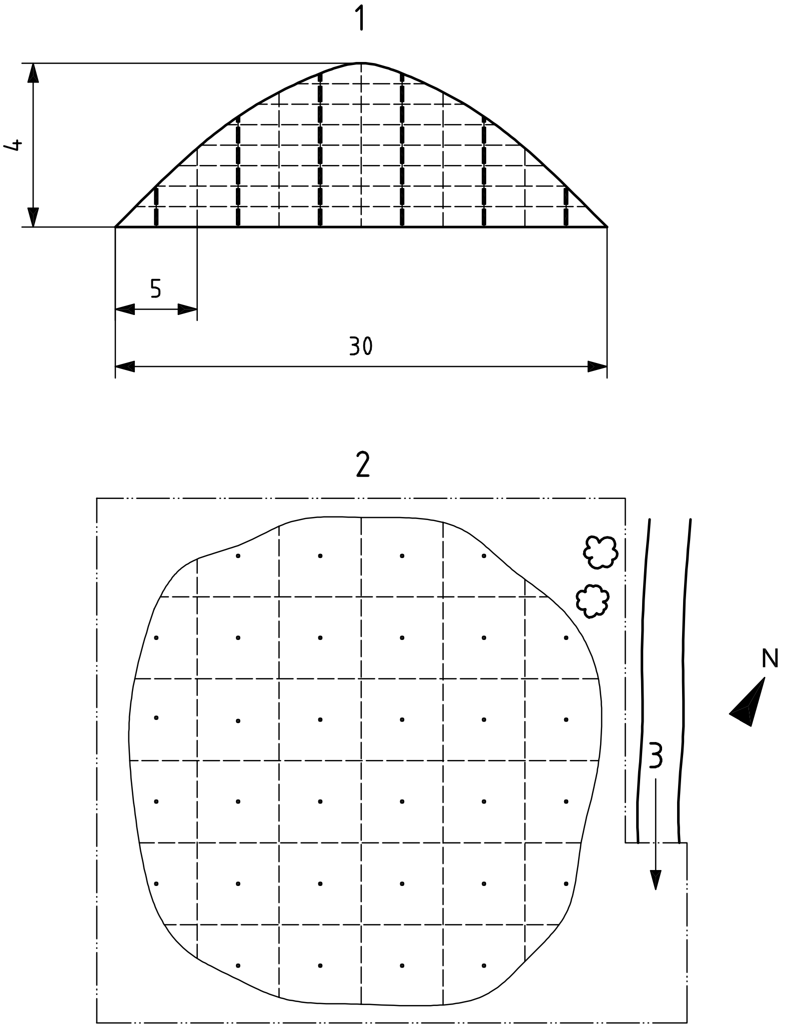
Δ_{XY} = $\sqrt{}$(33/2) = 4 m

It is not necessary to sample the stockpile in the vertical direction, horizontal sampling may, in some cases, be more practical and might therefore be preferred. The principle of (systematic) sampling does not change due to a change in direction of sampling. It should be noted that the direction of sampling should be constant whenever systematic sampling is applied.

NOTE 1 The stockpile in Figure H.1 shows to some extent a hypothetical situation, as in daily practice the shapes of stockpiles might be more irregular. For the purpose of sampling, relatively small irregularities in the surface/shape of the stockpile might be neglected as long as these irregularities represent only a small portion of the total amount of material (guidance: less then 5 %).

NOTE 2 Soil stockpiles can often be modelled by either a conical form (with or without a flat upper surface) or a trapezium shape. In both cases, mathematical calculations can be used to estimate the amount of material on a specific height/depth. Based on these estimates, a (systematic) sampling pattern can be calculated, where the number of samples on a specific depth equals the estimated amount of soil material on that depth.

Dimensions in metres



Key

1 cross section
2 top view
3 entrance

I increment
· sampling location

The dotted lines indicate the volume represented by each individual sample.

Figure H.1 — Example of the definition of a systematic sampling pattern on a soil stockpile

Use the selected sampling equipment in order to obtain a sample from the designated location. Withdraw the sampling equipment and make sure that the sample volume of the equipment is correctly filled.

Apply suitable sample pretreatment when appropriate and defined in the sampling plan (see 5.2.8 and Clause 8).

Transfer the (sub)sample into an appropriately sized sample container. Wipe the outside of the sample container and apply a label recording sample details and any observations. Apply suitable sample preservation and handling procedures as identified in the sampling plan (see 5.2.9 and Clause 9).

Record the operations carried out including the specific coordinates and any special conditions in the sampling form.

H.2 Sampling techniques for judgemental sampling

H.2.1 General

When applying judgemental sampling, it can be preferred to deviate as little as possible from the probabilistic sampling. In those situations, the sampling techniques as mentioned in H.1 shall be used. In other situations, these sampling techniques are not applicable or other types of sampling are preferential given the purpose of sampling.

The following sampling techniques will usually not result in probabilistic samples, but are applicable for judgemental sampling:

- spot sampling;
- directional sampling.

H.2.2 Spot sampling

H.2.2.1 Principle

A sample of the appropriate size is taken on a specific spot or location that has either been:

- randomly chosen in advance of sampling;
- chosen based on accessibility for sampling or a similar type of motivation;
- chosen based on the appearance of specific types of particles/material as encountered during sampling.

In the first situation, spot sampling is equal to simple random sampling (see H.1) and will therefore result in probabilistic samples, when applying sampling equipment of appropriate size (see 7.3, 6.4, 6.5 and 6.6).

The method of choosing the sampling locations as specified in the latter two options will not result in a probabilistic sample.

H.2.2.2 Sampling equipment

All sampling equipment that (in principle) fulfils the requirements for probabilistic sampling, as defined in 7.3, 6.4, 6.5 and 6.7.

NOTE Although the use of sampling equipment that enables probabilistic sampling is not per definition necessary when applying judgemental sampling, it is still to be preferred. When combining a non-probabilistic sampling strategy with a non-probabilistic sampling device, the representativity of the resulting samples is highly questionable.

H.2.2.3 Circumstances

Spot sampling will for instance be practical, when:

- a quick characterisation of the material is necessary and representativity is not directly relevant;
- specific parts of the soil stockpile seem to deviate from the bulk and characterization of these parts is desired.

H.2.2.4 Procedure

Push the sampling equipment of specified size into the soil at the point identified in the sampling plan. Withdraw the sampling equipment and make sure that the sample volume is correctly filled. Apply suitable sample pretreatment when appropriate and defined in the sampling plan (see 5.2.8 and Clause 8).

Transfer the (sub)sample into an appropriately sized sample container. Wipe the outside of the sample container and apply a label recording sample details and any observations. Apply suitable sample preservation and handling procedures as identified in the sampling plan (see 5.2.9 and Clause 9).

Record the operations carried out including the specific coordinates and any special conditions in the sampling form.

H.2.3 Directional sampling

H.2.3.1 Principle

A number of samples or a full-length sample will be taken in a specific direction through the soil stockpile, resulting in a composite sample throughout the full direction of sampling.

H.2.3.2 Sampling equipment

All sampling equipment that, in principle, fulfils the requirements for probabilistic sampling as defined in 7.3 and 6.4, 6.5 and 6.6.

NOTE Although the use of sampling equipment that enables probabilistic sampling is not per definition necessary when applying judgemental sampling, it is still to be preferred. When combining a non-probabilistic sampling strategy with a non-probabilistic sampling device, the representativity of the resulting samples is highly questionable.

H.2.3.3 Circumstances

A directional sample will be taken when it is expected that the quality of the soil will vary in the direction of sampling. This can occur when the soil stockpile is expected or known to consist of different layers of soil with (potential) varying quality.

H.2.3.4 Procedure

Push the sampling equipment of the appropriate size through the material in the identified direction, taking a series of spot samples or a full directional sample until the traverse is complete as specified in the sampling plan. Make sure that the sampling equipment is correctly filled at the sampling locations. In the case of spot samples, these shall be combined in order to give a directional sample.

Apply suitable sample pretreatment when appropriate and defined in the sampling plan (see 5.2.8 and Clause 8).

Transfer the (sub)sample into an appropriately sized sample container. Wipe the outside of the sample container and apply a label recording sample details and any observations. Apply suitable sample preservation and handling procedures as identified in the sampling plan (see 5.2.9 and Clause 9).

Record the operations carried out including the specific coordinates and any special conditions in the sampling form.

Annex I
(informative)

Description of sampling equipment

I.1 Augers

I.1.1 Soil auger

The soil auger is the most commonly used sampling equipment. It consists of a central shaft on the end of which a two bladed drill is connected. The width of the blades varies for different types of soil augers, making them suitable for different soil types. The small bladed types (wide opening) are suitable for moist or well-consolidated soils. The wide blade types (small opening) are suitable for dry or non-consolidated soils.

The soil auger is pushed into the soil with a turning motion. Usually two to four full twists will fully fill the soil auger. When filled, the soil auger will be pulled up, where after the soil material can be discharged from the auger.

Using a soil auger will result in a more or less disturbed sample, although the continuous discharge of the auger will give a good description of present soil layering.

I.1.2 Drill auger

An auger consists of a hard metal central shaft with sharpened spiral blades around, that discharge cuttings upwards as the shaft is rotated down through the soil. A disturbed sample is obtained (i.e. it is not possible to distinguish layered material during one sampling movement).

A large variety of soils can be sampled with a drill auger. However, the equipment will not be useful for non-cohesive soils (e.g. dry sandy soil or highly moist sandy soils).

I.1.3 Mechanical drill

This type of auger is used for drilling hard and tough materials. Using the sampling drill, the material sampled will not consist of full individual particles, but will only result in powdered material from one or more individual particles.

I.2 Sampling tubes

There are different types of materials used for sampling tubes. In most cases, tubes made from stainless steel are applicable.

I.2.1 Open sampling tube

The open sampling tube is the simplest sampling tube as it consists only of a tube that is cut in half over the full length of the tube. The tube is pushed into the soil by a vertical or (partly) rotating motion until it is fully filled and then pulled back. This type of sampling tube is only suitable for fine-grained slightly moist soils.

I.2.2 Half cut sampling tube

One of the options is that the tube is cut in half, and consisting of two concentric tubes closely fitted into each other throughout their entire length, so that one tube can be rotated within the other. Longitudinal openings are cut in each tube. In one position, the tube is open and admits the sample. By turning the inner tube, it becomes a sealed container. This type of sampling tube is only suitable for fine grained dry soils or fine grained soil sludge. In both cases, the soil is free flowing and can enter the sampling tube without further handling of the soil.

I.2.3 Plunger sampling tube

Another option is that the sampling tube contains a plunger. The plunger is moved upwards while inserting the sampling tube in the soil and thereby letting the soil into the tube. Sampled soil can be discharged from the tube by pressing the plunger down. A more or less undisturbed sample can be obtained, although the soil will be compressed when not fully consolidated. This type of sampling tube is most suitable for sampling sludge, but also fine-grained soils with a relatively high moisture content can be sampled.

I.3 Scoops

Soils can be sampled with a sampling scoop. This equipment is however not suitable for sampling at significant depths within the soil stockpile without the aid of other equipment to reach the sampling location (like a mechanical shovel).

The sampling scoop is pushed into the soil at the sampling location and withdrawn. The excess material above the sides of the scope is pushed off, the rest of the material is the sample.

A usual handheld shovel shall not be used as a sampling scoop. As a shovel does not meet the special characteristics of a sampling scoop, the consistency of the sample can not be guaranteed when applying a shovel. Soil that is originally sampled will fall off the shovel after sampling and additionally the larger particles will have a larger tendency to roll off the heap that is formed on a shovel.

I.4 Mechanical shovel

A mechanical shovel, or comparable mechanized equipment for digging and excavation work, can be suitable for taking samples from a soil stockpile. The mechanical shovel can be used in two ways:

- for sampling;
- for excavating the soil stockpile, making the sampling location accessible for other sampling equipment.

Due to the large size of the shovel, in most situations a full load of the shovel will be much too large as a sample. Using the mechanical shovel as a sampling tool therefore necessitates sample pretreatment directly after sampling (see Clause 8). Only for soils that consist of (very) large particles will a mechanical shovel be the most appropriate sampling equipment.

When applying the mechanical shovel for excavating the soil stockpile in order to make it accessible for other sampling equipment, a safe boundary layer should be maintained between the excavation and the sampling location. This is necessary to ensure the consistency of the sample.

NOTE The dimensions of the boundary layer will depend on the particle size of the soil, the slope of the soil at the sampling location and the degree of consolidation and can therefore not be described specifically.

Annex J
(informative)

Subsampling methods

J.1 Long pile and alternate shovel method

This subsampling method is suitable for samples in excess of approximately 100 kg.

Identify the maximum particle size of the sample and determine the minimum size of the subsample(s) according to Table 3. When the minimum size of the subsamples is larger than desired and the maximum particle size is related to the size of macro-aggregates, the macro-aggregate size can be reduced according to 8.4.2. The subsampling process shall be stopped when the size of the subsample is equal to or larger than the minimum size of the subsample as derived from Table 3.

The method consists of the following steps.

- Identify an area of hard surface sheltered from the effects of wind and rain, preferably flat and large enough to allow ease of access around the whole sample when spread on the surface.
- Place a clean protective floor covering, preferably heavy duty plastic sheeting, to protect the sample from contamination by the surface.
- Shovel the soil sample into a conical pile on the protective floor covering, placing each shovelful on the top of the preceding one. For samples in excess of approximately 500 kg, the use of a mechanical shovel is to be preferred above the use of a (manually handled) spade.
- When the entire soil sample is on the floor, circumvent the cone systematically depositing shovelfuls from the base to the apex of the cone so that the centre of the cone is not displaced. Repeat the process twice.
- Form the cone into a long pile as follows.
  - Taking a shovelful from the base of the cone, spread the material into a ribbon having an initial width equal to that of a shovel and a length of 1,5 m to 3,0 m.
  - Take the next shovelful from a different point at the base of the cone, and spread it directly over the previous shovelful, but in the opposite direction.
  - Repeat the above step until one long pile is formed.
- Discard half the soil sample in the following manner.
  - Take a shovelful from the bottom of one end of the pile and set it aside.
  - Take the next shovelful immediately adjacent to the first by advancing along the side of a pile a distance equal to the width of the shovel and discard this shovelful.
  - Again, advancing in the same direction a distance of one shovel width, take the third shovelful and add it to the first.
  - Continue along the pile following the above procedure, discarding alternate shovelfuls so that the pile is decreased gradually and uniformly.

  - Repeat the above procedure (from forming the coning to halving the pile) until the retained amount of material is equal to the desired size of the subsample (but no less than the minimum size of the subsample in accordance with Table 3).

- Transfer the subsample to an appropriate sample container in accordance with Clause 9.

J.2 Coning and quartering

This procedure is suitable for all samples down to approximately 1 kg.

- Identify the maximum particle size of the sample and determine the minimum size of the subsample(s) according to Table 3. When the minimum size of the subsamples is larger than desired and the maximum particle size is related to the size of macro-aggregates, the macro-aggregate size can be reduced according to 8.4.2. The subsampling process shall be stopped when the size of the subsample is equal to or larger than the minimum size of the subsample as derived from Table 3.

- Identify an area of hard surface sheltered from the effects of wind and rain, preferably flat and large enough to allow ease of access around the whole sample when spread on the surface.

- Place a clean protective floor covering, preferably heavy-duty plastic sheeting, to protect the sample from contamination by the surface.

- Shovel the soil sample into a conical pile on the protective floor covering, placing each shovelful on the top of the preceding one. For samples in excess of approximately 500 kg, the use of a mechanical shovel is to be preferred above the use of a (manually handled) spade. Manual handling is preferred for samples smaller than 100 kg.

- When the entire soil sample is on the floor circumvent the cone systematically taking shovelfuls from the base and forming a second cone with all the material from the first cone transferred to the apex of the second cone. Repeat the process twice.

- Flatten the cone so that the height is less than or equal to the height of the shovel or spade used.

- Divide the pile into quarters along two lines intersecting at 90° to each other, using one of the following methods:

  - Method 1:

    - Place the centre of a sheet metal cross, made with four blades joined together at the centre at 90° to each other, at the centre of the flattened cone and press the lower edges of the metal cross through the soil sample. The height and length of the blades forming the cross should be greater than that of the flattened cone.

    - With the metal cross left in position, discard opposite diagonal quarters and brush clean the space they occupied.

    - Remove the metal cross and mix together the remaining two quarters.

    - Cone and quarter again, using the previous stages, until the volume of remaining soil is equal to the desired size of the subsample (but no less than the minimum size of the subsample in accordance with Table 3).

  - Method 2:

    - Quarter the flattened cone along two diagonals intersecting at right angles, using a shovel inserted vertically into the soil.

  - Discard one pair of opposite quarters and shovel the remainder into a stockpile.
  - Check if the mass of the discarded material is equal to half the mass of the (sub)sample before subdivision, allowing a variation of ± 10 % (mass fraction). When this condition is not met, the discarded material should be added and mixed again, whereafter the subdivision can continue.
  - Repeat the process of mixing and quartering until the volume of remaining soil is equal to the desired size of the subsample (but no less than the minimum size of the subsample in accordance with Table 3).
- Transfer the subsample to an appropriate sample container in accordance with Clause 9.

NOTE Coning and quartering are known to be subject to bias. This bias is partly caused by the tendency of larger particles to roll down the side of the cone and to collect at the base. This results in segregation of particles from the top to the bottom of the cone. The same problem arises when taking subsamples when the areas to be subsampled are not previously separated (for instance, by the metal cross as described in the first method of quartering).

J.3 Riffling

The use of a riffle box is possible when the soil is dry enough to allow free flow of the soil particles through the riffle box. Division of the sample with a riffle box is most often only practical for samples less than approximately 100 kg (but depending on the size of the riffle box).

Division of the sample with a riffle box will result in a reduction to one half or one quarter (depending on the riffle) at each operation.

- Identify the maximum particle size of the sample and determine the minimum size of the subsample(s) according to Table 3. When the minimum size of the subsamples is larger than desired and the maximum particle size is related to the size of macro-aggregates, the macro-aggregate size can be reduced according to 8.4.2. The subsampling process shall be stopped when the size of the subsample is equal to or larger than the minimum size of the subsample as derived from Table 3.
- Identify an area of hard surface sheltered from the effects of wind and rain, preferably flat and large enough to allow ease of access around the whole sample when spread on the surface.
- Place a clean protective floor covering, preferably heavy-duty plastic sheeting, to protect the sample from contamination by the surface.
- Shovel the soil sample into a conical pile on the protective floor covering, placing each shovelful on the top of the preceding one. Manual handling is preferred for samples smaller than 100 kg.
- When the entire soil sample is on the floor circumvent the cone systematically taking shovelfuls from the base and forming a second cone with all the material from the first cone transferred to the apex of the second cone. Repeat the process twice.
- Check that the slot widths of the riffle box are at least three times larger than the maximum particle size of the soil to be subsampled.
- Using a shovel or container, pour the material into the riffle box. It is essential that the soil is poured evenly over the whole riffle in order to prohibit biased subsampling.
- Remove one subsample as the reduced sample, discarding the remaining material.
- Check if the mass of the discarded material is equal to half (or three quarters of) the mass of the (sub)sample before subdivision, allowing a variation of ± 10 % (mass fraction). When this condition is not met, the discarded material should be added and mixed again, whereafter the subdivision can continue.

- Repeat the process of riffling until the volume of remaining soil is equal to the desired size of the subsample (but no less than the minimum size of the subsample in accordance with Table 3).
- Transfer the subsample to an appropriate sample container in accordance with Clause 9.

J.4 Application of Tyler divider

The sloping plate of the Tyler divider provides a reduction ratio of 16:1. Material flows over the plate and is reduced successively in steps at each station down the plate by means of slots or holes placed in the plate. Each reduction is to one half the amount passing the station and a means for re-mixing after each stage is incorporated in the plate. An essential requirement in applying a Tyler divider is that the soil is dry enough to allow free flow of the soil particles.

The mechanical feed should be set at a constant rate suitable for the material being sampled and as identified in the sampling plan. This implies the requirement for the hopper width to be equal to that of the sloping plate and a gate of variable height.

The application of the Tyler divider calls for the following steps.

- Identify the maximum particle size of the sample.
- Check that the slot width of the Tyler divider is at least three times larger than the maximum particle size.
- Determine the minimum size of the subsample(s) according to Table 3 and calculate if the reduction ratio of the divider will result in a subsample that is equal to or larger than the minimum size of the subsample. If not, this type of divider shall not be used.
- Start the division process by pouring the sample into the divider with a constant rate and catch the subsamples(s) in (an) appropriate sample container(s).
- When necessary repeat the process of subsampling using one or more of the resulting subsamples until a subsample of the required size is obtained (but is no less than the minimum size of the subsample in accordance with Table 3).
- Transfer the subsample to an appropriate sample container in accordance with Clause 9.

J.5 Application of mechanized turntable (rotating divider)

The mechanised turntable comprises a number of prismatic containers, of equal size, mounted round the periphery of a circle which pass under the falling stream of the sample fed from a hopper mounted above the turntable, and off-set from the centre.

The turntable should operate at a constant speed of rotation that should not change (significantly) while sample material is coming into the turntable.

The application of the mechanised turntable calls for the following steps.

- Check that the slot width of the turntable is at least three times larger than the maximum particle size.
- Transfer the soil with a constant speed into the turntable. The speed should be relatively low in order to allow all particles to fall freely into the slot of the turntable and it will take a large number of rotations of the turntable before the full amount of soil is transferred into the slot.
- After completion of the division process, one or more of the subsamples is(are) collected.

- Check the mass of one of the subsamples. If the mass is not equal to the product of the total mass and the inverse number of subsamples in the rotating divider, allowing a variation of ± 10 % (mass fraction), all subsamples shall be added and the subsampling step shall be repeated.

- The subsamples obtained are (if necessary) divided again, until a subsample of the required size is obtained, or until the minimum sample size is achieved, see Table 3.

- Transfer the subsample to an appropriate sample container in accordance with Clause 9.

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